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Fabrication of multi-walled carbon nanotube-reinforced carbon fiber/silicon carbide composites by polymer infiltration and pyrolysis process

Hai-zhe Wang *, Xiao-dong Li, Jun Ma, Gong-yi Li, Tian-jiao Hu

College of Science, National University of Defense Technology, Changsha 410073, China

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ABSTRACT

Multi-walled carbon nanotube (MWNT)-reinforced carbon fiber/silicon carbide (C_f/SiC) composites were prepared using a polymer infiltration and pyrolysis (PIP) process. The MWNTs used in this study were modified using a chemical treatment. The MWNTs were found to be well dispersed in the matrix after ultrasonic dispersion, and the mechanical properties of the C_f/SiC composite were significantly improved by the addition of MWNTs. The addition of 1.5 wt.% of MWNTs to the C_f/SiC composite led to a 29.7% increase in the flexural strength, and a 27.9% increase in the fracture toughness.

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1. Introduction

C_f/SiC composites—which exhibit excellent properties in terms of their low density, high strength, high fracture toughness, and oxidation resistance—are considered as desirable candidate materials for high-tech applications. The automotive, aerospace, and aviation industries are particularly concerned with the application of C_f/SiC composites [1,2].

It is well known that the mechanical properties of C_f/SiC composites are generally determined by three factors: (1) the properties of the fibers, (2) the interface between the fibers and the matrices, and (3) the microstructure of the matrices [3,4]. The addition of reinforcing agents with unique mechanical properties (and consistency with the matrices) is therefore expected to play a positive role in improving the mechanical properties of C_f/SiC composites. In recent work, nanoscale additives were introduced into C_f/SiC composites to make them "stronger"; carbon nanotubes (CNTs) were considered as one of the most outstanding candidates [5].

CNTs have attracted much attention because of their excellent mechanical [6–8] and electronic properties [9]. The incorporation of CNTs can improve the mechanical properties of materials, and CNTs have been extensively applied in fabricating polymer/CNT [10–12] and metal/CNT [13–15] composites. However, the number of studies and the achievements in the ceramic/CNT composite field [16–18] have been less significant than those in the polymer/CNT and metal/CNT fields. The matrices used in CNT-reinforced ceramic composites in previous studies were primarily Al_2O_3 [5,13,19,20], and SiC [21].

In the preparation of ceramic/CNT composites, several key issues must be considered [19]. Firstly, the CNTs should be dispersed uniformly in the matrices. Both single- and multi-walled CNTs can easily form twisted aggregate structures, because of the high aspect ratio of CNTs, and the strong Van der Waals forces among the tubes. As a result, it is difficult to disperse CNTs homogeneously in the matrices. The second problem is the interphase between the MWNTs and the matrices. Only an appropriate interface will lead to an enhanced stress transfer capability from the matrices to the CNTs [21].

In this study, MWNT-reinforced C_f/SiC composites were prepared using a PIP [22–24] method, with antimony-substituted polymethysilane (A-PMS) used as a precursor. The mechanical properties and microstructures of these composites were investigated.

2. Experimental procedures

The C_f/SiC/MWNT composites were prepared using a PIP process; one cycle of this process had four steps. Firstly, pretreated MWNTs were added to the A-PMS (the MWNT:A-PMS weight ratios were 0%, 0.5% and 1.5% for the different samples), with subsequent agitation and ultrasonic dispersion performed at 800 W for 60 min at room temperature. The chemical modification of the MWNTs was performed as follows: MWNTs with a diameter of 30–80 nm and a length of 20–100 μm were prepared in our lab by chemical vapor deposition (CVD). These MWNTs were treated

* Corresponding author. Fax: +86 84574786.
E-mail address: hezaijushi@163.com (H.-z. Wang).

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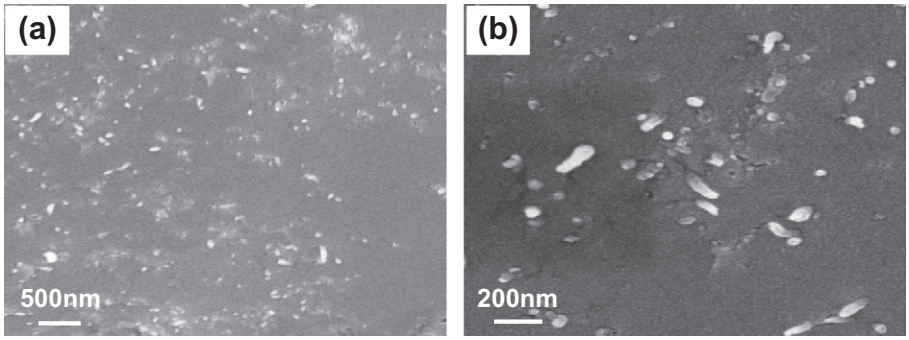

Fig. 1. Cross-sectional SEM images of cured A-PMS/1.5 wt.% MWNT composite.

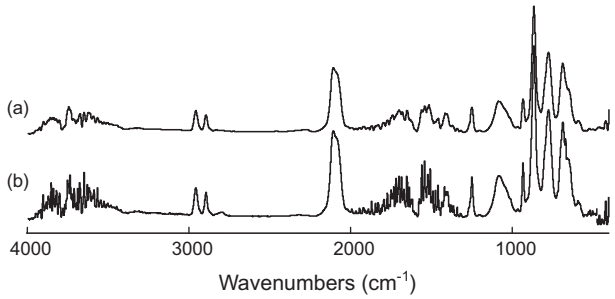

Fig. 2. FT-IR spectra for (a) A-PMS after ultrasonic treatment, and (b) A-PMS. The strong peaks at 2106-2166 cm^{-1} correspond to the Si–H bonds' stretching vibration.

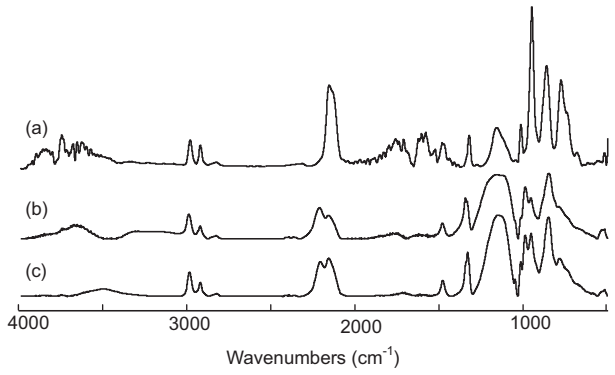

Fig. 3. FT-IR spectra for (a) A-PMS, (b) cured A-PMS, and (c) cured A-PMS/1.5 wt.% MWNT composite. The strong peaks at 2106–2166 cm^{-1} correspond to the Si-H bonds' stretching vibration.

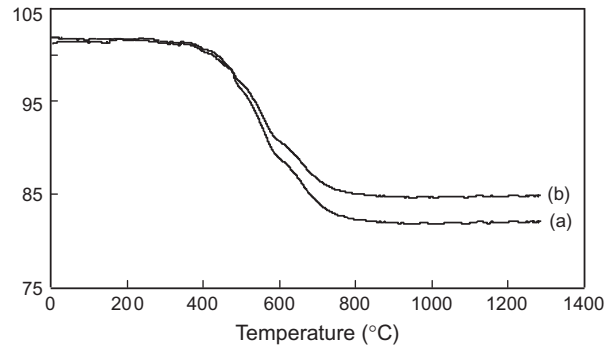

Fig. 4. TG curves for (a) cured A-PMS and (b) cured A-PMS/MWNT composite after ultrasonic treatment.

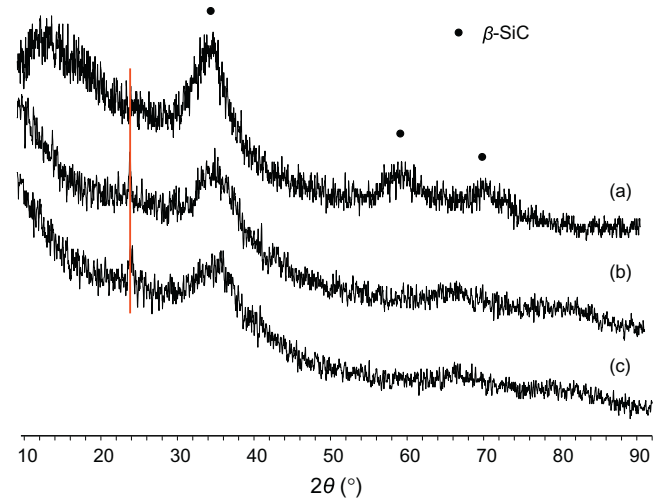

Fig. 5. XRD patterns for the ceramics derived from (a) A-PMS, (b) A-PMS/0.5 wt.% MWNTs, and (c) A-PMS/1.5 wt.% MWNTs.

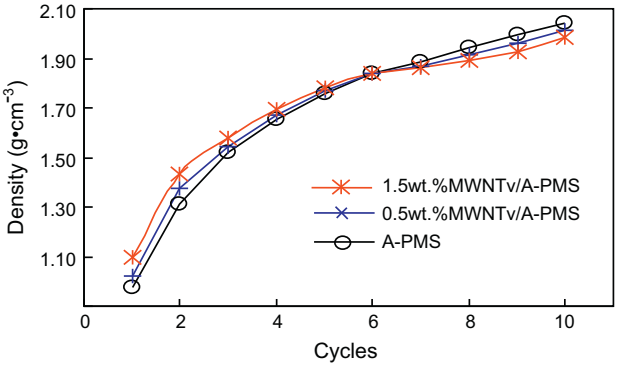

Fig. 6. Density–cycle curves for the composites prepared by PIP technology using A-PMS, A-PMS/0.5 wt.% MWNTs, and A-PMS/1.5 wt.% MWNTs.

in concentrated HNO_3 for 8 h at room temperature; they were then reacted with acryloyl chloride at 66 °C for 12 h. After this chemical modification process, it was found that the length of the MWNTs had decreased to about 5–10 μm, and the acryloyl chloride had polymerized on the surface of MWNTs and had encapsulated them (this work has been submitted for publication in separate manuscripts). Secondly, 3D carbon fiber preforms (Jilin Carbon, China, braided in Nanjing, China) were infiltrated with the A-PMS/MWNTs, under vacuum. Afterwards, the samples were cured at 470 °C for 1 h in flowing N_2. Thirdly, the cured samples were

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