Applied Surface Science 270 (2013) 611–616

Contents lists available at SciVerse ScienceDirect

Applied Surface Science

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

The improvement of wave-absorbing ability of silicon carbide fibers by depositing boron nitride coating

Fang Ye, Litong Zhang, Xiaowei Yin*, Yongsheng Liu, Laifei Cheng

Science and Technology on Thermostructural Composite Materials Laboratory, Northwestern Polytechnical University, Xi'an, Shaanxi 710072, PR China

ARTICLE INFO

Article history:
Received 20 November 2012
Received in revised form
28 December 2012
Accepted 15 January 2013
Available online 23 January 2013

Keywords:
SiC fibers
BN coating
Dielectric property
Impedance matching
Tensile strength

ABSTRACT

This work investigated electromagnetic wave (EMW) absorption and mechanical properties of silicon carbide (SiC) fibers with and without boron nitride (BN) coating by chemical vapor infiltration (CVI). The dielectric property and EM shielding effectiveness of SiC fiber bundles before and after being coated by BN were measured by wave guide method. The EM reflection coefficient of SiC fiber laminates with and without BN coating was determined by model calculation and NRL-arc method, respectively. Tensile properties of SiC fiber bundles with and without BN coating were tested at room temperature. Results show that SiC fibers with BN coating had a great improvement of EMW absorbing property because the composites achieved the impedance matching. BN with the low permittivity and dielectric loss contributed to the enhancive introduction and reduced reflection of EMW. The tensile strength and Weibull modulus of SiC fiber bundles coated by BN increased owing to the decrease of defects in SiC fibers and the protection of coating during loading.

1. Introduction

Nowadays, stealthiness is an indispensable specification for modern weapons, and parts made of specific materials are required to absorb the emitted electromagnetic wave (EMW) energy and to minimize the reflection of EMW in the direction of the enemy radar receiver. In some harsh environments, such as high velocity missiles, the materials also need to endure high thermomechanical stress [1–4]. For these applications, ceramic fibers reinforced ceramic matrix composites are considered as the most promising materials [1].

Silicon carbide (SiC) fibers are a crucial reinforcement candidate for high-temperature structural ceramic matrix composites due to the excellent strength and stability at high temperatures, low creep, high oxidation and corrosion resistance, high thermal conductivity and high thermal shock resistance [5–7]. SiC fibers are also an available EMW absorbing agent, they have a wide range of electrical resistance from 10^{-3} to 10^{4} Ωm, and represent the optimal EMW absorbing property in GHz as electrical resistance varies at a range of 10^{1}–10^{3} Ωcm [8,9].

The study on the core/shell structure of SiC indicated the positive effect of the interfacial polarization on the good EMW absorbing properties [10]. It may be an efficient way of improving the wave-absorbing performance of SiC fibers through reducing the surface reflection and enhancing the wave-transmitting capacity by the interface scattering loss [11]. For SiC fibers reinforced ceramic matrix composites, such as SiC_f/SiC composites, the property of fiber/matrix interface is one of the key elements which determine the materials performance, primarily because the damage tolerance results from the deviation of matrix cracks into the interface [12,13]. Pyrolytic carbon (PyC) and boron nitride (BN) with layered structure are the most frequently used and effective interface materials for SiC_f/SiC composites [14,15]. However, PyC interface is inappropriate for the EMW absorbing applications, because the continuous PyC coating with high electrical conductivity can lead to a strong reflection of EMW [16]. BN is considered as the primarily leading interface material not only because of its improved oxidation resistance than PyC [17,18] but also its low dielectric constant and extremely low dielectric loss, which makes BN a promising interface material for the EMW absorbing applications [19]. To develop SiC_f/SiC composites with excellent functional (EMW absorbing) and structural properties, the investigation on the effect of BN coating on the EM property of SiC fibers is very necessary. However, little has so far been known about the interaction between SiC fibers with BN coating and EM radiation.

The present work focused on the mechanical and EM properties of SiC fibers with and without BN coating. The tensile strength and dielectric property of SiC fiber bundles and SiC/BN mini-composites were measured. The EM reflection coefficient (RC) of single layer SiC fiber laminates with and without BN coating was determined. The effects of BN coating on the surface structure of SiC fibers and the interaction between SiC fibers and EMW were discussed.

* Corresponding author. Tel.: +86 029 88494620.
E-mail address: yinxw@nwpu.edu.cn (X. Yin).

0169-4332/$ – see front matter
http://dx.doi.org/10.1016/j.apsusc.2013.01.093

Table 1
Preparation processing and dielectric property of pyrolytic BN.

CVI processing parameter						Dielectric property [19]		
T (°C)	Q_{BCl_3} (ml min^{-1})	Q_{NH_3} (ml min^{-1})	Q_{H_2} (ml min^{-1})	Q_{Ar} (ml min^{-1})	t (h)	ε'	ε''	$tg\delta$
650	10	30	50	25	7	3.2	0.00055	0.00017

2. Experimental

2.1. SiC fiber

SiC fibers in this study were made in China. Each fiber bundle contained 250 filaments.

2.2. BN coating

The BN coating was prepared by chemical vapor infiltration (CVI). Boron trichloride (BCl_3 of 99.99% purity, Beijing Multi Technology Co., Ltd., China) and ammonia (NH_3 of 99.99% purity, Sichuan Messer Co., Ltd., China) were used as boron and nitrogen source for BN preparation respectively. Hydrogen (H_2 of 99.999% purity, Sichuan Messer Co., Ltd., China) was carrier gas and reactant. Argon (Ar of 99.999% purity, Sichuan Messer Co., Ltd., China) was used as the dilution gas. The preparation processing was listed in Table 1. Due to the moist sensitivity of BN coating, the samples after CVI should be kept in vacuum drying apparatus.

2.3. Morphology and composition

The cross-section and surface morphology of SiC fibers with and without BN coating was observed by SEM (S4700, Hitachi, Japan). The element composition was qualitatively analyzed by EDS (Genesis XM2, EDXA, USA) attached to SEM. XPS analysis was performed on a Thermo Scientific apparatus with Al Kα radiation to determine the bonding form of elements.

2.4. EM property test

The dielectric property was measured by a vector network analyzer (VNA, MS4644A, Anritsu, Japan) via waveguide method. 25 vol.% of SiC fiber bundles with and without BN coating were mixed with the epoxy resin and cast into the flange to be made into the measurement samples with the dimension of 3 mm × 10.16 mm × 22.86 mm. The permittivity, dielectric loss and shielding effectiveness (SE) of each sample were obtained at frequencies of 8.2–12.4 GHz (X-band). The EMW absorbing property was evaluated by calculating and measuring the EM RC of material. The RC of unidirectional SiC fiber laminates was directly determined by NRL-arc method at frequencies of 8–18 GHz. NRL-arc measurement technique is carried out in a non-destructive manner and without contact. An accurate measurement for the EM property of anisotropic and inhomogeneous media, such as composites, can be performed without the excitation of higher-order modes [20].

2.5. Tensile property test

The tensile strength of SiC fiber bundles with and without BN coating was measured by Instron 3345. The gauge length was 50 mm, and loading speed was 0.3 mm/min. The valid data were not less than 10 for each series.

3. Results and discussion

The filament diameter of SiC fibers was about 15 μm observed by SEM as shown in Fig. 1 (a). Fig. 1 (b) shows the cross-section morphology of SiC fibers coated by a thin layer (about 1000 nm). It can be found that after the deposition of coating the surface of SiC fibers was smooth and even (Fig. 1 (c)). Fig. 1 (d) is the EDS spectrum of coating with 5 μm in thickness, which demonstrates that both B and N elements were detected, and the as-prepared BN was composed of nearly stoichiometric B and N (their atomic ratio was estimated to be 47.38:52.45) hardly with any impurities. Au was sprayed on the surface of samples before SEM observation considering the poor conductivity of BN.

XPS was conducted from the surface of as-prepared BN coating. Photoelectron peaks from B 1s and N 1s were clearly recognized. Fig. 2 exhibits the narrow scanning spectrum of B 1s and N 1s of coating. The B 1s peak at 190.3 eV (Fig. 2 (a)) should be assigned to an atomic circle surrounding the boron atom only consisting of nitrogen atoms [21]. The N 1s peak at 398.2 eV corresponded to the N—B bond [22]. The presence of weak peaks of B—O and N—C bonds indicated that a small quantity of carbon and oxygen impurities was distributed in the surface of coating.

3.1. Dielectric and EMW absorbing property

When an EM plane wave strikes a monolithic conductive material with different intrinsic impedance from that of the medium in which EM plane wave is propagating, the reflected wave and the transmitted wave will be created at the external surface. During the transmitted wave propagates in the material and reaches the second surface of material, the EMW energy will be decreased due to the attenuation and absorption by material. The remaining is reflected back into material interior at the second surface, and then is absorbed by material or transmits out of material [23]. So the total EM SE of material (SE_T) consists of surface reflection (SE_R), the internal absorption (SE_A) and the multiple internal reflection (SE_{MR}) SE. It can be described as follows:

$$SE_T = SE_R + SE_A + SE_{MR} \tag{1}$$

Specially, if the shield is thicker than the skin depth of material, the SE_{MR} can be ignored as a result of the reflected wave from the internal surface being absorbed adequately by the conductive material [23]. Therefore, Eq. (1) can be deformed as follows:

$$SE_T = SE_R + SE_A \tag{2}$$

In the study, SE was calculated according to the S-parameters (S_{11}, S_{12}, S_{22} and S_{21}) given by VNA. Fig. 3 shows SE_T, SE_R and SE_A of SiC fibers with and without BN coating inlaid in the epoxy resin as a function of frequency. As can be seen, the SE_R at 10 GHz of fiber bundles decreased from 2.90 to 2.38 dB with the SE_A at 10 GHz increasing from 1.47 to 2.23 dB after the deposition of BN. Consequently the SE_T at 10 GHz increased from 4.36 to 4.61 dB. It demonstrates that SE_A and SE_T of fiber bundles coated by BN were measurably improved.

The complex permittivity and dielectric loss are two important microwave interaction performance parameters of a dielectric material [24,25]. The real part (ε') i.e. dielectric constant is related to the polarization, and the imaginary part (ε'') represents dielectric loss ability of material [26]. The dielectric loss ($tg\delta = \varepsilon''/\varepsilon'$) can forecast the EMW absorbing property of material, which can be a promising EMW absorber if its dielectric loss is appropriately high [27]. Fig. 4 shows the real part, imaginary part and dielectric loss