Fabrication and Properties of SiNO Continuous Fiber Reinforced BN Wave-Transparent Composites

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SiNO continuous fiber reinforced boron nitride (BN) wave-transparent composites (SiNO_f/BN) have been fabricated by a precursor infiltration pyrolysis (PIP) method using borazine as the precursor. The densification behavior, microstructures, mechanical properties, and dielectric properties of the composites have been investigated. After four PIP cycles, the density of the composites had increased from 1.1 g·cm⁻³ to 1.81 g·cm⁻³. A flexural strength of 128.9 MPa and an elastic modulus of 23.5 GPa were achieved. The obtained composites have relatively high density and the fracture faces show distinct fiber pull-out and interface de-bonding features. The dielectric properties of the SiNO_f/BN composites, including the dielectric constant of 3.61 and the dielectric loss angle tangent of 5.7×10⁻³, are excellent for application as wave-transparent materials.

Keywords: SiNO continuous fiber, boron nitride, wave-transparent composites, mechanical properties, dielectric properties

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1. Introduction

With the rapid development of hyper-velocity aircraft, a radome for protecting the antennas [1, 2], characterized by high-temperature and erosion resistance, becomes essential in the harsh environment [3, 4]. However, because the radome may affect the antenna operation, it must be made of a kind of material with enhanced electromagnetic transmission. Therefore, the search for wave-transparent materials with low dielectric constant, low dielectric loss and excellent mechanical properties has attracted a great deal of attention in recent years.

So far, continuous fiber-reinforced ceramic matrix composites (CMCs), due to their excellent mechanical and dielectric properties, seem to fulfill the abovementioned requirements. Due to their outstanding dielectric and mechanical properties as well as resistance to high-temperature, boron nitride (BN) ceramics represent the most promising prospective wave-transparent materials. Furthermore, BN ceramics, synthesized by pyrolyzing borazine at high temperature [5], maintain stable mechanical and dielectric properties over a wide temperature range [6]. Generally, SiO₂ fibers are used as reinforcements in BN ceramics. However, the performance of SiO₂ fibers cannot be maintained when the temperature exceeds 1100 °C because of crystallization and softening at such high temperature. SiNO fiber is another very promising high-temperature ceramic fiber which has been developed and attracted a great deal of attention worldwide. In many respects, the material is far superior compared with the traditional SiO₂ fiber. In the work described herein, SiNO continuous fiber reinforced BN wave-transparent composites (SiNO_f/BN) have

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been fabricated by a precursor infiltration pyrolysis (PIP) method using borazine as the precursor. The densification behavior, microstructure, and mechanical and dielectric properties of the composites have been investigated.

2. Experimental

2.1. Raw materials

Borazine, the precursor for the BN matrix, was synthesized from NaBH$_4$ and (NH$_4$)$_2$SO$_4$ and then purified according to literature reports [7–9]. The precursor was obtained as a transparent, homogeneous, volatile liquid with the density of 0.81 g·cm$^{-3}$, the purity of 99% (the ceramics yield was above 89%). The SiNO continuous fibers, produced at the National University of Defence Technology (NUDT), China, had a density of 2.02 g·cm$^{-3}$, a tensile strength of 1120 MPa, and an average diameter of 12 µm.

2.2. Preparation of the composites

In the composites, the volume fractions of SiNO fibers, BN matrix, and pores were 48%, 36%, and 16%, respectively. The fabrication of SiNO$_f$/BN composites by the PIP method involved the following steps. Firstly, the continuous SiNO fibers were arranged in the same orientation in the mold, and then the borazine precursor was allowed to infiltrate in vacuum. Secondly, the borazine precursor filled with fibers was cured at 70 °C in an inert atmosphere for 50 h to form a SiNO fiber reinforced polyborazine composite. Finally, the composite was subjected to pyrolysis in a nitrogen atmosphere at 900 °C for 3 h to obtain the SiNO$_f$/BN composite. In order to further densify the composites, the borazine infiltration, curing, and pyrolysis steps were repeated four times. To identify the ceramic matrix formed after the pyrolysis at 900 °C, borazine was cured at 70 °C in an inert atmosphere for 50 h, and the material obtained was pyrolyzed in a nitrogen atmosphere at 900 °C for 3 h.

2.3. Characterization of the composites

The apparent density of the composites was determined by the Archimedes displacement method. The powder was characterized by X-ray diffraction (XRD) with a Bruker Advance D8 diffractometer. Fracture surfaces of the specimens after three-point bending tests were observed with a scanning electron microscope (JSM-5600LV). The mechanical properties of a specimen with dimensions of 3×4×35 mm, including flexural strength and elastic modulus, were determined using a WDW-100 three-point testing machine with a span of 30 mm and a crosshead speed of 0.5 mm/min. The tensile strength was calculated as the failure force divided by the cross-section of the measured part, and the tensile modulus was determined from the initial part of the stress–strain curves. Interlaminar shear strength (ILSS) was measured by the three-point short-beam bending test method to estimate the interfacial adhesion strength of the fibers to the matrix in the composites, according to the ASTM D-2344 standard. The dielectric properties were examined by the short-cutting waveguide method with specimen dimensions of 15.8×7.9×(5–10) mm.

3. Results and discussion

3.1. Densification and microstructures of the composites

As pores would inevitably be generated during the infiltration and pyrolysis processes, repeated infiltration – pyrolysis cycles were carried out to densify the CMCs. Fig. 1 shows the effect of the number of PIP cycles on the density of an SiNO$_f$/BN composite. It is clear that after the first cycle the density of the SiNO$_f$/BN composite increased remarkably due to the high infiltration efficiency of the BN matrix into the pores created during the pyrolysis process at high temperature. On the one hand, during the re-infiltration and pyrolysis, the pores in the composite were already sealed, decreasing the infiltration efficiency. Hence, the density increased only slightly in the subsequent cycles. On the other