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Microwave Absorbance of Polymer Composites Containing SiC Fibers Coated with Ni-Fe Thin Films

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Abstract Conductive and dielectric SiC are fabricated using electroless plating of Ni–Fe films on SiC chopped fibers to obtain lightweight and high-strength microwave absorbers. The electroless plating of Ni–Fe films is achieved using a two-step process of surface sensitizing and metal plating. The complex permeability and permittivity are measured for the composite specimens with the metalized SiC chopped fibers dispersed in a silicone rubber matrix. The original non-coated SiC fibers exhibit considerable dielectric losses. The complex permeability spectrum does not change significantly with the Ni–Fe coating. Moreover, dielectric constant is sensitively increased with Ni–Fe coating, owing to the increase of the space charge polarization. The improvements in absorption capability (lower reflection loss and small matching thickness) are evident with Ni–Fe coating on SiC fibers. For the composite SiC fibers coated with Ni–Fe thin films, a -35 dB reflection loss is predicted at 7.6 GHz with a matching thickness of 4 mm.

Keywords: Microwave absorbers, Silicon carbide fibers, Nickel-iron plating

1. Introduction

Silicon carbide (SiC) is a dielectric structural ceramic material having high strength and hardness, low density, good resistance to oxidation, high thermal stability and high thermal conductivity at elevated temperatures [1-3]. Additionally, SiC is a good potential microwave absorbing materials due to its high dielectric loss in microwave radiation [4-6]. Compared to glass fibers, polymer-derived SiC fibers have higher modulus and wide range of electrical resistivity by tailoring the compositions or surface characteristics, which can develop their microwave absorbing applications [7-11]. It was reported that the ion jump and dipole relaxation through the reorientation of lattice defect pairs is the dominant factor for the high dielectric loss [6]. However, the absorption capability of SiC is not satisfactory as a single absorber, due to the difficulty in precise control of its dielectric property leading to impedance matching. It is generally utilized as an auxiliary absorbent in other materials or after surface treatments to enhance the absorption capability [12-14].

In resonant absorbers of quarter wavelength, zero-reflection can be obtained by access to wave impedance mating at the surface of the absorbing layer, which requires a proper combination of magnetic permeability and dielectric permittivity at a given thickness and frequency. Those material parameters in high frequencies can be controlled by use of magnetic metals (permittivity control by electrical property and permeability control by magnetic property) in a single-layered microwave absorber [15]. In this study, Ni-Fe films are coated on SiC chopped fibers through electroless plating for the control of high-frequency electromagnetic properties and its effect on microwave absorbance is investigated.

2. Experimental Procedure

Commercially available SiC fibers (DACC Carbon Co. Ltd., Korea) were used as absorbent filler materials. The SiC fibers with a diameter of 5 μm have a low density of

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Table 1. Chemical agents used in the electroless plating of Ni-Fe on SiC chopped fibers.

	Metal salts	Reducing agent	Complexing agent	pH modifier
Chemical agent	$NiCl_2 \cdot 6H_2O$ $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$	$NaPH_2O_2 \cdot H_2O$	$KNaC_4H_4O_6 \cdot 4H_2O$	NH_4OH

3.2 g/cc. The fibers were chopped into about 0.5 mm length. Ni-Fe (10 mol%) films were coated on the fibers in a two-step operation: surface sensitizing and metal plating. The surface sensitization was conducted in a catalyst solution of $PdCl_2$, $SnCl_2 \cdot 2H_2O$, and HCl. The surface-sensitized microspheres were placed in plating solution and the mixture was tumbled at 70-80°C for 1 h. Table 1 shows the chemical agents (metal salts, reducing agent, complexing agent, and pH modifier) used in electroless plating of Ni-Fe films. The microspheres were gravity filtered, washed with distilled water and air dried at 60°C. The batch was repeated two times for the complete coating of the metal film.

The coating morphology Ni-Fe film and its crystal structure were verified using a scanning electron microscope (SEM: Carl Zeis/LEO-1530) and X-ray diffraction (XRD: PANAnalytical/X'Pert PRO MRD). The complex permeability and permittivity was measured in the composite specimens with the metalized SiC chopped fibers dispersed in a silicone rubber matrix. Composite specimens were prepared using RTV (room temperature vulcanization) silicone rubber as a matrix material. The mixing ratio of SiC fibers to rubber was 1 by weight. The mixture was molded into a coaxial die with a 3-mm inner diameter, 7-mm outer diameter, and approximately 2-mm thickness for microwave measurement. Curing at room temperature for about 10 h under a pressure of 0.1 ton/cm^2 produced the flexible and dense composite specimens.

Toroid samples were inserted in a standard coaxial sample holder, and the reflection coefficient (S_{11} parameter) and transmission coefficient (S_{21} parameter) were measured using a network analyzer (HP 8722D). The complex permittivity and permeability was calculated from the S_{11} and S_{21} parameters. The reflection loss was determined through measuring the S_{11} parameter after the rear face of the sample was terminated by metal.

3. Results and Discussion

Fig. 1 shows the SEM observation of the non-coated and Ni-Fe coated SiC chopped fibers. Cylindrical SiC particles with an average diameter of about 5 μm and length of 0.5 mm were observed (Fig. 1(a)). Fig. 1(b) shows the microstructure of the SiC fibers coated with Ni-Fe films. Non-uniform but nearly complete coating of Ni-Fe film is observed. The XRD patterns of Ni-Fe coated SiC fibers further confirmed the incorporation of Ni-Fe grains, as depicted in Fig. 2. A broad peak at $2\theta = 35°$, which corresponds to Bragg's diffraction from SiC (111) plane, reveals the amorphous nature of SiC fibers. Another sharp diffraction peak at $2\theta = 44°$ corresponds to the reflections from the (111) planes of Ni-Fe alloy.

Fig. 3 shows the complex permeability ($\mu_r = \mu_r' - j\mu_r''$) and complex permittivity ($\varepsilon_r = \varepsilon_r' - j\varepsilon_r''$) determined in the composite specimen containing Ni-Fe coated SiC chopped fibers. The amount of SiC fibers in rubber matrix (defined by F/R ratio in weight) was 1. For the composite of non-coated SiC fibers, dielectric constant is

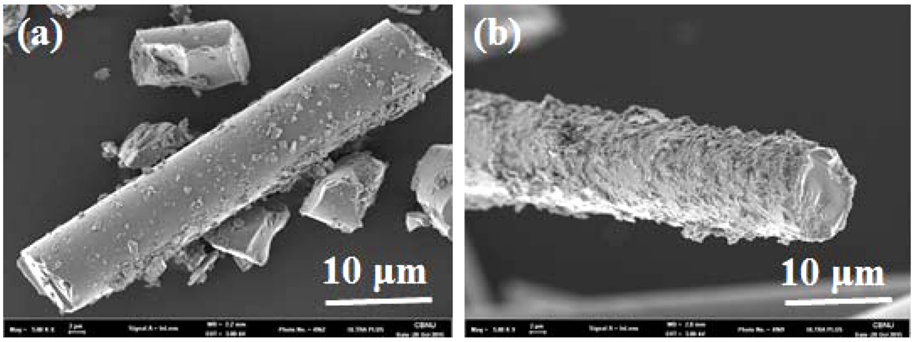

Fig. 1. SEM morphology of (a) non-coated SiC chopped fiber, (b) Ni-Fe coated SiC fiber.

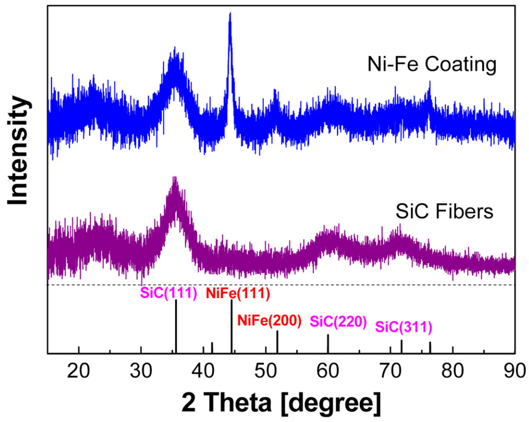

Fig. 2. XRD patterns determined from the original and Ni-Fe coated SiC fibers.

$\varepsilon_r' = 4.5$ and a considerable value of dielectric loss ($\varepsilon_r'' \cong 0.5$) were estimated (Fig. 3(a)), which are almost constant (not dispersive) with frequency. It was reported that the ion jump and dipole relaxation through the reorientation of lattice defect pairs is the major factor for the high dielectric loss [6]. Because of non-magnetic property of the constituent materials, the constant values of complex permeability were determined to be $\mu_r' \cong 1.0$ and $\mu_r'' \cong 0.05$ (Fig. 3(b)).

With coating of Ni-Fe films, the dielectric constant increases gradually from $\varepsilon_r' = 4.5$ (non-coated SiC) to $\varepsilon_r' = 6.0$ (at 10 GHz), as depicted in Fig. 3(a). Higher value of dielectric constant with Ni-Fe coating is attributed to the electrically conductive nature of films. The space charge polarization between adjacent conductive particles (separated by insulating rubber matrix) gives rise to a higher value of dielectric constant. However, the dielectric loss is not greatly increased with Ni-Fe coating. It is believed that the main source of dielectric loss is the SiC fibers having a loss mechanism of dipole relaxation or dipole reorientation [6]. The increase in magnetic permeability and magnetic loss is not so great (Fig. 3(b)), which is due to small thickness of Ni-Fe film to respond to external magnetic field.

For the composite layer terminated by metal, the input impedance at the layer surface (Z_{in}) is given by the following equation:

$$Z_{in} = Z_0\sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left[\frac{j2\pi d}{\lambda}\sqrt{\varepsilon_r\mu_r}\right], \tag{1}$$

where Z_o is the wave impedance of free space (= 377 Ω),

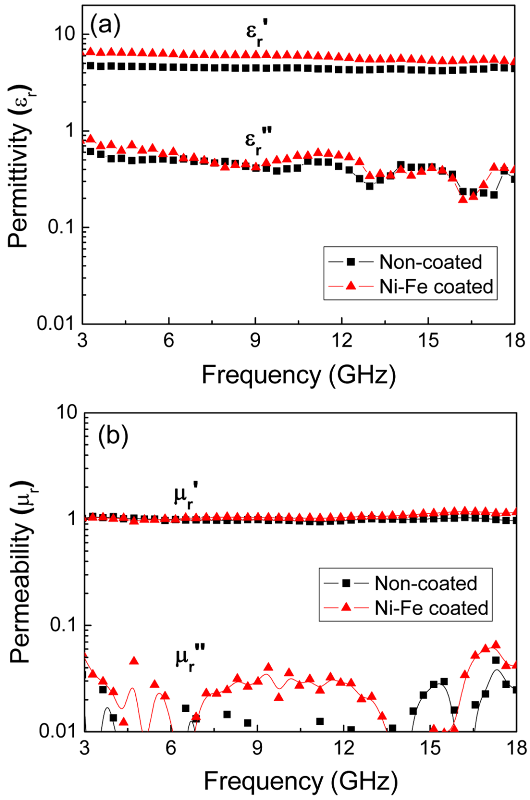

Fig. 3. Material parameters measured from the composites of SiC chopped fibers (non-coated and plated with Ni-Fe films): (a) complex permittivity and (b) complex permeability.

λ is the wavelength in free space, and d is the layer thickness. Since the reflection coefficient Γ is proportional to the difference between Z_{in} and Z_o as expressed in Eq. (2), the reflection loss (RL) can be calculated from the measured material parameters (μ_r and ε_r) as a function of frequency and thickness.

$$\Gamma = \frac{Z_{in} - Z_0}{Z_{in} + Z_0}, \tag{2}$$

$$RL = 20\log|\Gamma| \tag{3}$$

Fig. 4 presents the improvement of microwave absorbance by coating the SiC chopped fibers with Ni-Fe films. For the composite of non-coated SiC fibers, -25 dB reflection loss (99.6% power absorption) is predicted at 7.5 GHz with a layer thickness of 5 mm, as presented in Fig. 4(a). For the composite of SiC fibers plated with Ni-Fe film, a smaller matching thickness is predicted. At the thickness of 4 mm, -35 dB reflection loss (99.99% power absorption) is determined at 7.6 GHz, as depicted in Fig. 4(b). The improvement in absorption capability (lower

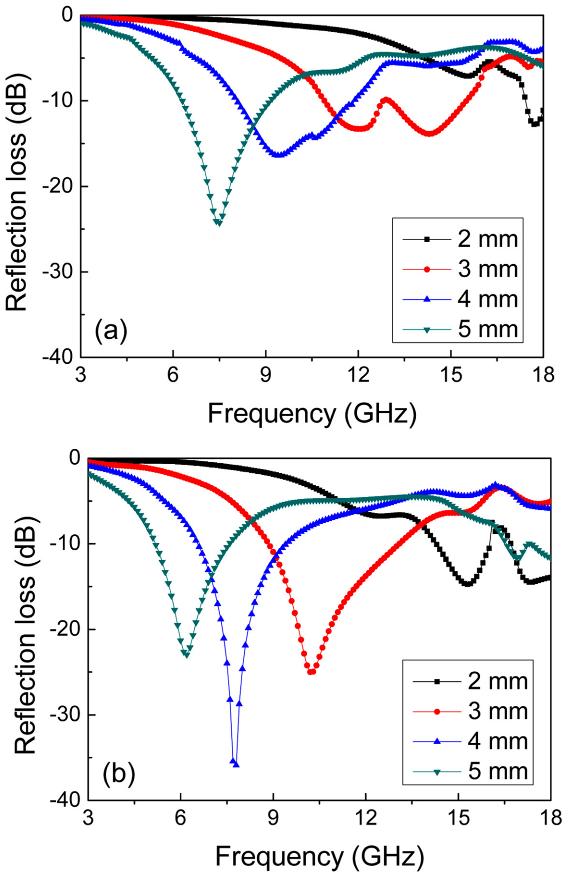

Fig. 4. Reflection loss determined in the composites of SiC chopped fibers: (a) non-coated and (b) plated with Ni-Fe films.

reflection loss and small matching thickness) is evident with Ni-Fe coating on SiC fibers, which is mainly due to the increase in the dielectric constant of the composite samples.

4. Conclusions

The most significant result of this study is that the lightweight and thin microwave absorbers with high absorption capability can be designed by using the SiC chopped fibers coated with Ni-Fe thin films. The original non-coated SiC fibers exhibited a considerable value of dielectric loss. The complex permeability spectrum is not greatly changed with Ni-Fe coating. On the while, dielectric constant is sensitively increased with Ni-Fe coating, due to the enhancement of space charge polarization. For the composite of SiC fibers coated with Ni-Fe thin film, the improvement in absorption capability (lower reflection loss and small matching thickness) is evident with Ni-Fe coating on SiC fibers. Reflection loss less than -30 dB was predicted at 7.6 GHz with a matching thickness of 4 mm.

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