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ORIGINAL PAPER

Preparation of silver/carbon fiber/polyaniline microwave absorption composite and its application in epoxy resin

Bo Cheng[1] · Jin Wang[1] · Fan Zhang[1] · Shuhua Qi[1]

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Abstract Microwave absorbing Ag/CF/polyaniline (PANI) composites with PANI particles evenly wrapped on the surface of the silver-plated carbon fiber, were prepared via in situ polymerization. The obtained composite was analyzed by SEM, XRD, TGA, four probes resistance tester and a vector network analyzer. The results indicated that silver nanoparticles were deposited on the surface of CF uniformly and Ag/CF was completely wrapped by PANI particles. Ag/CF/PANI composite with conductivity of 2.16 S/cm displayed better thermal stability than that of PANI. Meanwhile, Ag/CF/PANI composite with microwave reflection loss up to −13.2 dB at 9.3 GHz at thickness of 2.0 mm outperformed that of PANI matrix and CF/PANI composite. The epoxy matrix composite with Ag/CF/PANI dispersed inside homogenously performed −8 dB at 4.5 wt% loading content.

Keywords Microwave absorption · Silver · Carbon fiber · Conductive polymer · Polyaniline

Introduction

Microwave absorbing materials are attracting researchers due to their unique functions that can convert electrical microwaves into heat energy and other energy consumption when the electromagnetic wave is incident on the surface of the material. In view of the absorption mechanism of magnetic loss and electric loss, absorbing materials can be mainly divided into two categories: magnetic materials and conductive materials, such as ferrite and carbon fiber (CF) [1–3]. Recently, absorbing materials based on conductive materials and polymers have attracted

✉ Shuhua Qi
qishuhuanwpu@163.com

[1] Department of Applied Chemistry, School of Natural and Applied Sciences, Northwestern Polytechnical University, Xi'an 710129, People's Republic of China

much attention for their excellent electronic and excellent microwave absorption properties [4–6].

Conductive polymers, such as polyaniline (PANI), polypyrrole (PPY), polythiophene (PHT) and polyacetylene (PAC) are usually applied in the preparation of microwave absorbing composite materials due to their unique structures and properties [7–13]. As a conducting polymer, PANI possesses excellent properties such as cheap price, low weight and high conductivity. However, its microwave absorbing property is unsatisfactory [14–16]. Conductive fillers, such as carbon nanotube, CF and graphene, are always complexed with PANI to improve its microwave absorbing capacity [17–19].

Carbon fiber with carbon ratio being more that 95%, composed of graphite crystallites and other organic fibers, performs high modulus and excellent strength. The density of CF is lower than that of metal, while the strength is higher than that of steel. It also has good corrosion resistance and excellent electrical conductivity [3–5]. In addition, it also performs the inherent characteristics of carbon material, with the performance of high elastic modulus, fatigue resistance, low thermal expansion coefficient, electromagnetic shielding and other excellent properties [6, 20].

To further improve the electrical conductivity of the composites, some metals such as silver, nickel and cobalt were coated onto the surface of CF. Among the metals, Ag was an optimal option for its high electrical and thermal conductivity. A uniform silver layer can be easily wrapped on the surface of CF via electroless plating [21–23].

In this work, Ag/CF was prepared by electroless plating method, with silver nanoparticles deposited on the surface of CF. Subsequently, Ag/CF/PANI composite with low density and excellent absorption properties was prepared via in situ polymerization. The morphology and structure of CF, Ag/CF, PANI and Ag/CF/PANI were characterized by SEM and XRD. Properties such as electrical conductivity, thermal stability and microwave absorbing were measured. In addition, the Ag/CF/PANI composite was applied in the modification of epoxy resin, and the microwave absorbing properties of epoxy/CF hybrid composites were also studied.

Experimental

Materials

CF, T300B, was supplied by TORAY Chem. Co. (Japan). Stannous chloride ($SnCl_2{\cdot}2H_2O$) and palladium chloride ($PdCl_2$), analytically pure, were purchased from Xi'an Chemical Reagent Factory (China); Aniline, analytically pure, was obtained from Sinopharm Chemical Reagent Limited Company (China) and distilled at reduced pressure before use. Silver nitrate ($AgNO_3$), 40 wt% formaldehyde solution (HCHO), 28 wt% aqueous ammonia ($NH_3{\cdot}H_2O$), 36% ammonium persulphate (APS) and sodium hydroxide (NaOH) were commercially purchased from Sinopharm Chemical Reagent Co. Ltd and used as received. Epoxy resin

(E-51, epoxide value: 0.48–0.54) and curing agent were supplied by Bailing Petrochemical (China).

Preparation of Ag/CF

Electroless plating method was applied to prepare the silver-plated carbon fiber, as shown in Fig. 1; a certain amount of CF was taken and agitated in 0.5 mol/L of NaOH solution for 1 h, and then placed in concentrated nitric acid with ultrasonic treatment for extra 30 min. After vacuum filtration, the fibers were added into 0.5 mol/L of $SnCl_2{\cdot}2H_2O$ solution with ultrasonic treatment for 30 min until it was sensitized. Subsequently, the sensitized CF was further activated by $PdCl_2$, followed by being filtered and dried in vacuum oven at 80 °C. The sensitization and activation reaction formula are as follows:

$$SnCl_2 + H_2O \rightarrow Sn(OH)Cl + H^+ + Cl^-$$

$$SnCl_2 + 2H_2O \rightarrow Sn(OH)_2 + 2H^+ + 2Cl^-$$

$$Sn(OH)Cl + Sn(OH)_2 \rightarrow Sn_2(OH)_3Cl$$

$$Sn_2(OH)_3Cl + 2PdCl_2 \rightarrow Sn_2(OH)_3Cl_5 + 2Pd$$

$NH_3{\cdot}H_2O$ solution was added dropwise into the $AgNO_3$ solution to prepare a certain concentration of silver ammonia solution. The treated CF was then added to the silver ammonia solution. Subsequently, a certain concentration of HCHO solution was added into the mixture, and kept stirring for 30 min at room temperature. Ag/CF was obtained after washing and drying. The reaction formula is as follows:

$$HCHO + 4[Ag(NH_3)_2]OH \rightarrow 4Ag \downarrow + 6NH_3 + (NH_4)_2CO_3 + 2H_2O$$

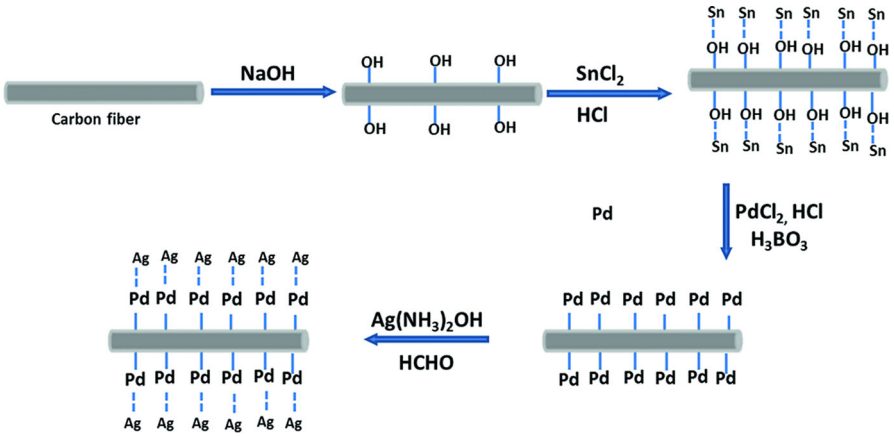

Fig. 1 Preparation process of Ag/CF

Synthesis of Ag/CF/PANI

Ag/CF/PANI composite was prepared via in situ polymerization. 1.0 g Ag/CF complex was added into the 30 ml of HCl solution (0.5 mol/L), the mixture was then placed in an ice water bath and kept stirring. Subsequently, 0.6 ml of aniline was added into the mixture. When the temperature of the mixture dropped to 2–4 °C, 40 ml of mixed solution containing APS (0.5 mol/L) and HCl (0.5 mol/L) was added into the mixture. The reaction temperature was kept at 2–4 °C for 8 h. After the reaction, the reaction liquid was filtered, and washed several times with HCl (0.5 mol/L). The solid products obtained were placed in a 90 °C vacuum for about 12 h. Finally, protic acid electrically conductive Ag/CF/PANI composite was obtained, as shown in Fig. 2.

Preparation of epoxy/Ag/CF/PANI hybrid composites

The Ag/CF/PANI composite was ground into powder before being mixed with epoxy resin. Epoxy resin and curing agent were mixed together at the weight ratio of 4:1, followed by the addition of Ag/CF/PANI. The mixture was kept stirring until the Ag/CF/PANI powder was dispersed homogenously into the epoxy resin. The epoxy/Ag/CF/PANI mixture obtained was then vacuumed to remove air bubbles at 60 °C for 4 h, and finally cured at 75 °C, for 5 h [7].

Measurement

The morphology and structure of CF, Ag/CF, PANI and Ag/CF/PANI was observed by scanning electron microscopy (SEM, JSM-6390, HITACHI, Japan) and X-ray diffraction (XRD, PANalytical, Holland). The thermal stabilities were analyzed using a thermogravimetric analyzer (TG, SDT-2960, USA) with a heating rate of 10 °C/min in the range from 40 to 800 °C under argon atmosphere. The electrical conductivity of the composites was measured using a SZ-82 digital four probes resistance tester (Suzhou Electronic Equipment Factory, China). For measurement of electrical properties, circle samples with a diameter of 15 mm and a thickness of

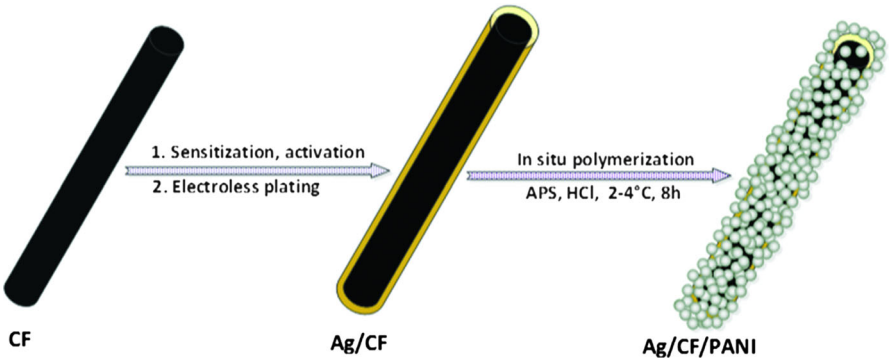

Fig. 2 Preparation process of Ag/CF/PANI composite

2 mm were prepared by casting them into stainless forms and cold-pressing. The microwave absorption properties of PANI, CF/PANI, Ag/CF/PANI and epoxy/Ag/CF/PANI were analyzed using a vector network analyzer (Agilent technologies, E8362B, China) in X band, the samples were prepared using the mix of the products (20 wt%) and paraffin (80 wt%) in a mould with the size of 22.86 mm × 10.16 mm × 2 mm.

Results and discussion

Morphology and microstructure

The morphology of the prepared CF, Ag/CF, Ag/CF/PANI and epoxy/Ag/CF/PANI composite was observed by SEM. As shown in Fig. 3a, the surface of pretreated CF was smooth and lustrous, after the process of alkali pickling, sensitization and activation. Figure 3b, c represented the SEM images of Ag/CF. It can be clearly observed that the surface of the carbon fibers was deposited by silver nanoparticles completely. Obviously, the Ag/CFs was further wrapped by PANI particles via in situ polymerization, as exhibited in Fig. 2d, e, indicating the successful preparation of Ag/CF/PANI ternary composite. Figure 2f revealed that the Ag/CF/PANI composite was dispersed in the epoxy matrix.

The CF, Ag/CF, PANI and Ag/CF/PANI were characterized by XRD from $2\theta = 10°$ to 80° with the scanning speed of 10 °/min, and the recorded diffractograms were displayed in Fig. 4. As shown in Fig. 4a, the peak at

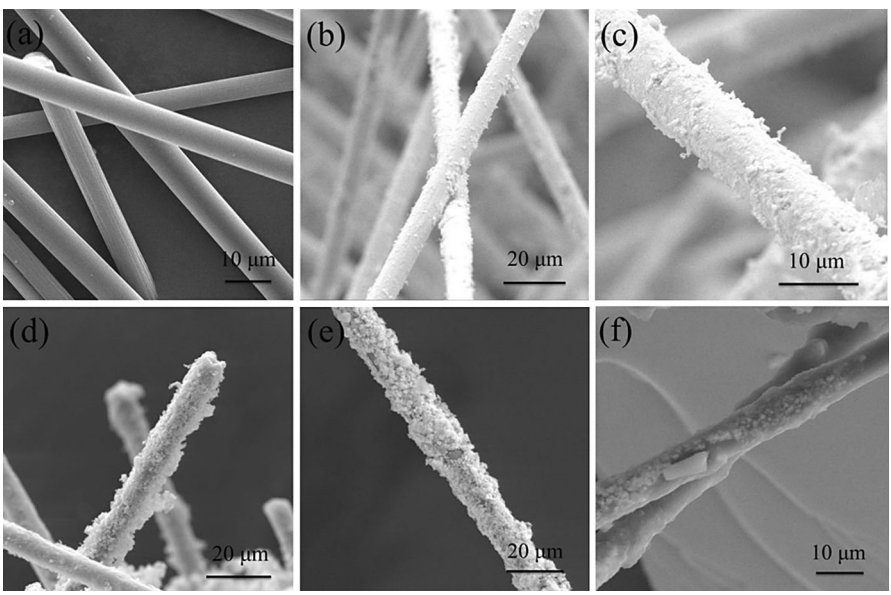

Fig. 3 SEM images of CF (**a**), Ag/CF (**b**, **c**), Ag/CF/PANI (**d**, **e**) and epoxy/Ag/CF/PANI composite (**f**)

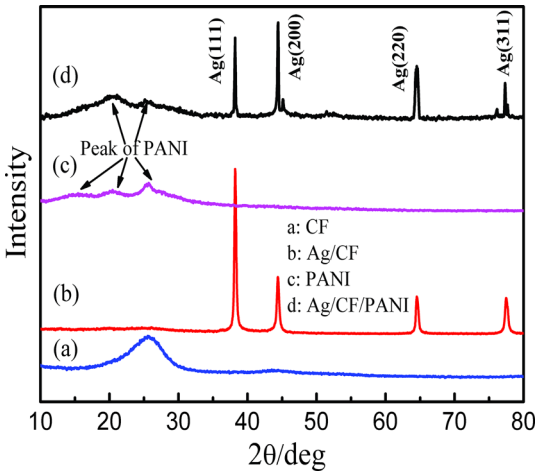

Fig. 4 The XRD spectrum of CF (*a*), Ag/CF (*b*), PANI (*c*) and PANI/Ag/CF (*d*)

$2\theta = 25.56°$ was corresponding to (0 0 2) typical diffraction peak of graphite. It was clear that the peak disappeared after silver plating, indicating that the surface of carbon fiber was completely covered by silver layer, which was consistent with the conclusions obtained by SEM images. From Fig. 4b, another four diffraction peaks can be observed at 38.22°, 44.4°, 64.56°, 77.44°, corresponding to the (1 1 1), (2 0 0), (2 2 0) and (3 1 1) planes of silver's cubic face-centered structure, respectively (JCPDS, File No. 04-0783). The three peaks before 30° can easily be observed in Fig. 4c, which referred to the characteristic peaks of PANI. Figure 4d represented Ag/CF/PANI composite, containing the characteristic peaks of PANI and Ag/CF, respectively. These results indicated that Ag/CF and Ag/CF/PANI were both successfully prepared [22, 23].

TG analysis

The thermal stabilities of PANI (a), CF/PANI (b) and Ag/CF/PANI (c) were characterized by TG analysis, and the curves were demonstrated in Fig. 5. The degradation temperature (T_d) of PANI is 511 °C due to the pyrolysis of main chain. Residual ratio (wt%) of PANI, CF/PANI and Ag/CF/PANI at different temperatures of 200, 400, 600 and 800 °C were shown in Table 1. As displayed in Fig. 5 and Table 1, it was clear that the residual ratios of CF/PANI and Ag/CF/PANI were relatively close at different temperatures, however, higher than that of the PANI, indicating better thermal stability. The phenomenon was attributed to the fact that carbon fiber imposed restriction on the pyrolysis of PANI chains and avoided heat concentration [24].

Electrical analysis

The conductivities of CF, Ag/CF, PANI, Ag/CF/PANI and CF/PANI were summarized in Table 2. Carbon fiber is a good conductor with a conductivity of

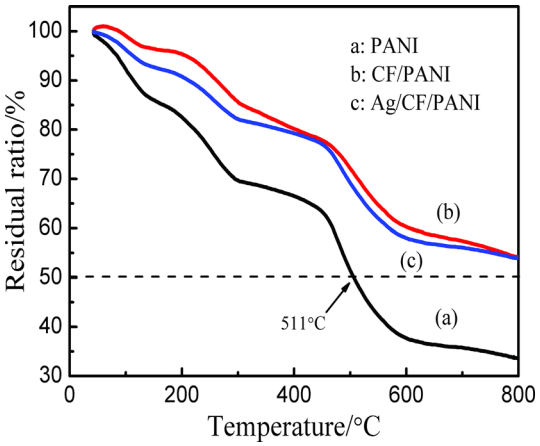

Fig. 5 TG curves of PANI (*a*), CF/PANI (*b*), and Ag/CF/PANI (*c*)

Table 1 Weight losses of PANI, CF/PANI and Ag/CF/PANI at different temperatures

Sample	200 °C	400 °C	600 °C	800 °C
PANI	82.5	66.5	37.7	33.6
CF/PANI	95.4	80.2	60.3	54.2
Ag/CF/PANI	90.8	79.2	58.0	53.9

Table 2 Electrical conductivity values of CF, Ag/CF, PANI, Ag/CF/PANI and CF/PANI (S/cm)

Sample	CF	Ag/CF	PANI	Ag/CF/PANI	CF/PANI
Conductivity	3.2	1.15×10^3	0.19	2.16	0.36

3.2 S/cm. However, when combined with silver, its conductivity could increase dramatically to 1.15×10^3 S/cm. This is mainly due to the silver particles depositing on the surface of CF, which allows electrons to move freely. As a conducting polymer, proton acid PANI has excellent electrical conductivity; the four probes suggest that its conductivity can reach 0.19 S/cm. When compounding with Ag/CF hybrid particles, its conductivity increased to 2.16 S/cm, which was approximately ten times higher than of PANI (0.19 S/cm) and five times higher than of CF/PANI (0.36 S/cm).

Microwave absorbing properties

The microwave absorbing properties of a material are closely related to its electromagnetic properties, and mainly characterized by reflection loss (RL). According to the transmit-line theory, when the electromagnetic wave vertically incidents upon the samples, the reflection loss can be calculated using the following equations [2, 24–26]:

$$R = 20\log\left|\frac{Z_{\text{in}} - Z_0}{Z_{\text{in}} + Z_0}\right| \tag{1}$$

$$Z_{\text{in}} = Z_0\left(\sqrt{\frac{\mu_r}{\varepsilon_r}}\right)\tanh\left[j\left(\frac{2\pi fd}{c}\right)\left(\sqrt{\mu_{\text{r}}\varepsilon_{\text{r}}}\right)\right], \tag{2}$$

where Z_0 is the characteristic impedance of vacuum, Z_{in} is the normalized input impedance of the absorber, c is the velocity of light in free space, $\varepsilon_{\text{r}} = \varepsilon' - j\varepsilon''$, $\mu_{\text{r}} = \mu' - j\mu''$ is the relative complex permeability and permittivity of the material, c is the velocity of the light, f and d is the frequency of the microwave and the thickness of the absorber, respectively. It indicates that the absorption capacity of the material at the same frequency mainly depends on the thickness of the material [27, 28].

CF, silver and PANI are all performed as non-magnetic material. Therefore, the magnetic parameters μ' and μ'' of the three materials (PANI, CF/PANI and Ag/CF/PANI) are 1 and 0, respectively. Their permittivities were displayed in Fig. 6, and the dielectric loss (tan $\delta_{\text{e}} = \varepsilon''/\varepsilon'$) was calculated. In the X band, the real part, imaginary part and tanδ_{e} values of CF/PANI (b) and Ag/CF/PANI (c) are pretty close, all higher than that of PANI (a). This is mainly due to the excellent electrical conductivity of CF/PANI and Ag/CF/PANI. The calculated frequency dependence RL curves of PANI, CF/PANI and Ag/CF/PANI in a frequency range of 8.2–12.4 GHz (X band) were exhibited in Fig. 7. The RL value of PANI was less than −10 dB (90% absorption), most located between −1 and −8 dB. When combined with CF, its microwave absorption performance was improved. The lowest value of the reflection loss could reach up to −10.2 dB. Furthermore, the curve representing the Ag/CF/PANI was below that of PANI/CF and that of PANI, and its lowest point appeared at $F = 9.3$ GHz, about −13.2 dB (more than 92.4% absorption). When the frequency was ranging from 8.4 to 10.6 GHz, its RL values were all higher than −10 dB. These results indicated that the microwave absorbing properties of Ag/CF/PANI were greatly improved compared with that of PANI and that of CF/PANI.

According to Eqs. (1) and (2), the absorption capacity of the material at the same frequency mainly depends on the thickness of the material. Therefore, it is necessary to simulate the thickness of absorbing materials [29, 30]. The simulated RL curves of Ag/CF/PANI at different thicknesses were shown in Fig. 8. It could be clearly seen that the thickness of the absorber had a great influence on the microwave absorbing properties. The peak of the reflection loss curve gradually shifted toward lower frequency with the increase of thickness, which matched the quarter-wavelength model [31, 32]. When the thickness was 1.5 mm, the RL value was less than −6 dB. However, when the thickness increased to 2.5 mm, its RL curve was ranging from −3.6 to −10.6 dB, and the maximum value appeared at $F = 8.2$ GHz. Obviously, its microwave absorbability was degraded compared with the 2.0 mm sample. When the thickness reached 3.0 and 3.5 mm, its RL value decreased with the increase of the thickness, which was less than the 2.5 mm sample. Thence, the conclusion that the best match thickness of Ag/CF/PANI is 2.0 mm in the X band with the RL value being lower both in low and high frequency could be drawn.

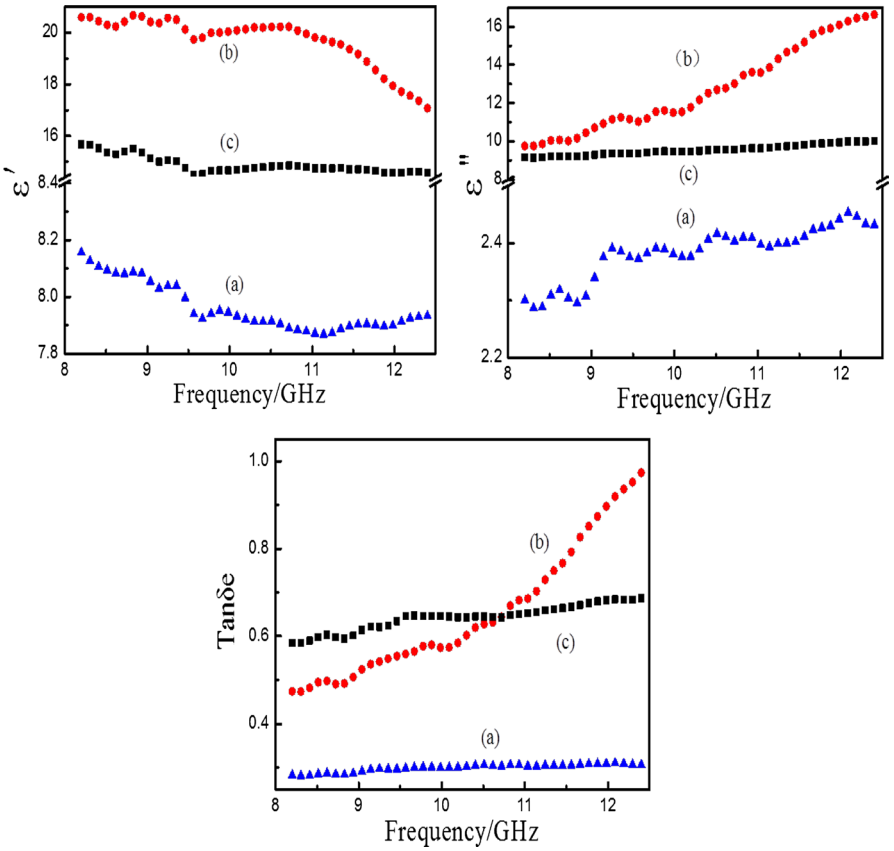

Fig. 6 The dielectric properties of PANI (*a*), CF/PANI (*b*) and Ag/CF/PANI (*c*)

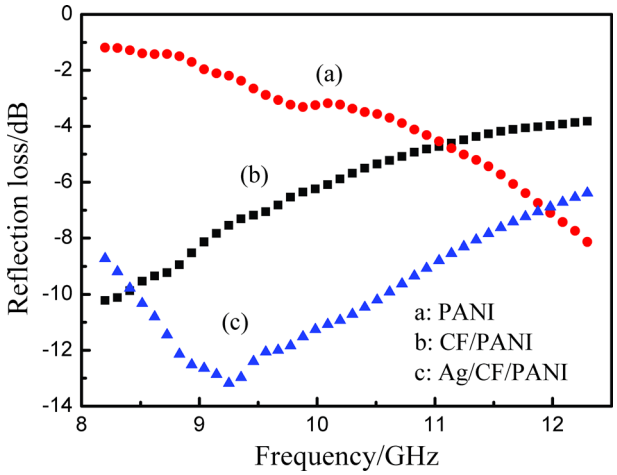

Fig. 7 RL curves of PANI (*a*), CF/PANI (*b*), and Ag/CF/PANI (*c*) in X band

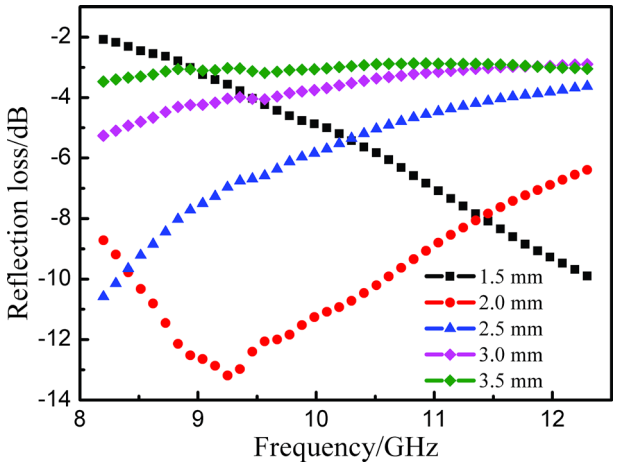

Fig. 8 The simulated RL curves of Ag/CF/PANI of different thicknesses

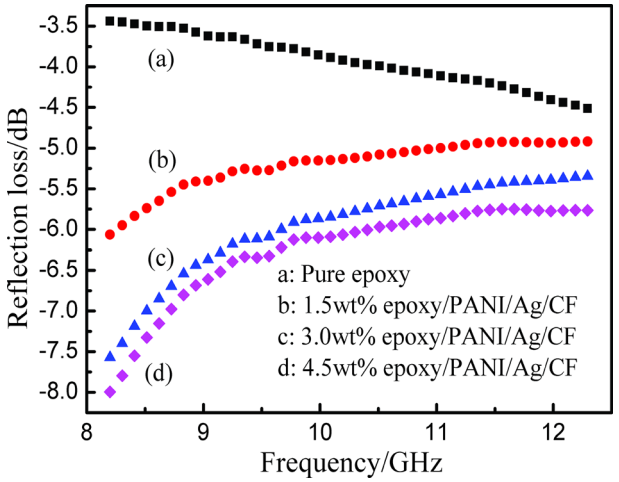

Fig. 9 RL curves of epoxy/Ag/CF/PANI composite of different mass percentages

The Ag/CF/PANI as a filler was applied to modify epoxy resin, and the RL curves of epoxy/Ag/CF/PANI composites with different mass percentage were displayed in Fig. 9. When the loading content was higher than 4.5%, the mechanical properties of epoxy composite became inferior significantly. Therefore, the 1.5, 3.0 and 4.5 wt% samples were selected as research samples. As shown in Fig. 9a, the microwave absorption property of pure epoxy was poor, and its RL value was about −4 dB. When the mass percentage rose to 1.5% (Fig. 9b) and 3.0% (Fig. 9c), the maximum reflection loss was about −6 and −7.5 dB, respectively, all higher than −5 dB (80% absorption). When the mass percentage increased to 4.5% (Fig. 9d), it could be observed that the curve was lower than curve (c) and curve (b); the

maximum RL value could reach −8 dB (87.5% absorption). The microwave absorbing properties were improved with the increase of Ag/CF/PANI mass percentage. Meanwhile, an epoxy resin matrix composite material with the microwave absorbing properties of higher than −5 dB in the entire X band was obtained.

Conclusions

In this study, the Ag/CF/PANI composite with excellent microwave absorption properties was prepared via a facile two-step method. Subsequently, the obtained Ag/CF/PANI composite as a modifier was applied in epoxy resin to enhance its absorption performance. SEM and XRD analysis showed that silver nanoparticles deposited densely on the surface of CF, the prepared Ag/CF was then wrapped with PANI particles. TG measurement indicated that thermal stability of PANI was improved by the addition of Ag/CF. The four-point probe test suggested that the conductivity of Ag/CF/PANI was 2.16 S/cm. Meanwhile, it also exhibited excellent microwave absorption properties, which could reach up to −13.2 dB at the thickness of 2.0 mm. When Ag/CF/PANI as modified filler was applied, the reflection loss of epoxy resin could be improved to −8 dB. This ternary composite can either be used as an attractive candidate for a new type of microwave adsorption materials, or a microwave adsorbing modified filler for epoxy or other resin.

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