Research Article

Preparation and Study of Electromagnetic Interference Shielding Materials Comprised of Ni-Co Coated on Web-Like Biocarbon Nanofibers via Electroless Deposition

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Received 22 September 2014; Revised 11 December 2014; Accepted 11 December 2014

Academic Editor: Myoung-Woon Moon

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Electromagnetic interference (EMI) shielding materials made of Ni-Co coated on web-like biocarbon nanofibers were successfully prepared by electroless plating. Biocarbon nanofibers (CF) with a novel web-like structure comprised of entangled and interconnected carbon nanoribbons were obtained using bacterial cellulose pyrolyzed at 1200°C. Paraffin wax matrix composites filled with different loadings (10, 20, and 30 wt%, resp.) of CF and Ni-Co coated CF (NCCF) were prepared. The electrical conductivities and electromagnetic parameters of the composites were investigated by the four-probe method and vector network analysis. From these results, the EMI shielding efficiencies (SE) of NCCF composites were shown to be significantly higher than that of CF at the same mass fraction. The paraffin wax composites containing 30 wt% NCCF showed the highest EMI SE of 41.2 dB (99.99% attenuation), which are attributed to the higher electrical conductivity and permittivity of the NCCF composites than the CF composites. Additionally, EMI SE increased with an increase in CF and NCCF loading and the absorption was determined to be the primary factor governing EMI shielding. This study conclusively reveals that NCCF composites have potential applications as EMI shielding materials.

1. Introduction

In recent years electromagnetic interference (EMI) has become a critical problem for electric devices because it may lead to malfunctions such as in airplane control panels, mobile phones, and many computers. In order to address these problems the development of EMI shielding materials received increasing attention [1–3]. Carbon composites have outstanding potential for use in EMI shielding [4–9] due to their excellent electrical properties, low density, high aspect ratio, and high strength and modulus. However the electrical conductivity of carbon materials is considerably lower than that of metals and therefore a greater amount of carbon material is needed to achieve the same shielding effect. Although satisfactory shielding capabilities can be obtained by simply adding more carbon materials, it is difficult to produce composites with high carbon volume fractions when using extrusion or injection molding. In addition, a high carbon volume fraction increases the composites cost and thus limits its commercial use. To optimize the conductivity and EMI shielding effectiveness (SE) of composites, it is necessary to attach metal particles to the surface of carbon materials [10–12]. The introduction of metal particles onto carbon materials has previously been shown to lead to good wettability between the fillers and matrices [13], which implies that the dispersion of carbon materials can improve in the matrices. Amongst the common metal materials, nickel and cobalt are suitable for EMI shielding because these metals exhibit good conductivity and magnetic properties, as well as antioxidant properties. Such good conductivity and magnetic materials can produce a large induced current under perturbation by electromagnetic waves, and, according to Lenz’s law, these currents will be effective at weakening the penetration of electromagnetic waves [14]. Therefore, it is expected that carbon materials coated with a layer of metal will have the advantages of good conductivity, magnetic, high intensity, and low density and they combine the properties of carbon materials and the magnetic metal together. Although
a variety of techniques are available for altering the chemical and physical properties of the surface of carbon materials, electroless plating has been found to be the most suitable method because of its ability to provide a uniform surface and the cost effectiveness of the process [15–17]. Thus, electroless plating is a convenient approach to produce Ni-Co coated carbon materials. However, the application of traditional metal-coated carbon materials for use as EMI shielding composites is hindered by a major obstacle: the cost and limited supply of carbon materials.

Recently, we developed a novel method of fabricating biocarbon nanofiber (CF) with a web-like structure consisting of interconnected carbon nanoribbons using low-cost natural materials such as bacterial cellulose as carbonaceous sources. In this work, we report the preparation process of CF, and the electroless plating method was applied to deposit a layer of Ni-Co coating on the surface of CF. The microstructure, magnetic properties, electrical conductivity, electromagnetic parameters, and EMI SE of the composites were investigated.

2. Experimental Procedures

2.1. Preparation of CF. Web-like structure CF was obtained by heat treatment of bacterial cellulose in a high-temperature tube furnace for 4 h under a nitrogen atmosphere at 1200°C. Afterwards, dried CF was dispersed in absolute ethanol and mechanically milled into a slurry. The resulting CF was dried using a vacuum at 60°C. The bacterial cellulose was kindly provided by Hainan Ye Guo Foods Co., Ltd., Hainan, China.

2.2. Pretreatments and Electroless Plating of CF with Ni-Co. For metal-coated CF/polymer matrix composites, the most important factor affecting the SE of composites is the adhesion between the metal coating and CF. Good adhesion can reduce the delamination of metal deposits by shear stress during compounding and can maintain the conductive network. In order to enhance the interfacial adhesion between the CF and the coating, CF was subjected to an oxidation treatment before surface activation. The CF was immersed in an aqueous solution of 200 g/L (NH₄)₂S₂O₈ and 100 mL/L of H₂SO₄ (the acid solution readily etches the surface of CF) by sonication at 35°C for 80 min, then was filtered, and thoroughly washed with distilled water until the washings reached pH = 7. This procedure promotes the coarsening of the carbon fiber, which can form different functional groups on the CF surface. These surface functional groups can form anchoring sites for metallic nanoparticles, thereby improving the adhesion between the CF and the nanoparticles [18]. The coating procedure consisted of three stages: sensitization, activation, and metallization. The CF and the optimum operating conditions of the sensitized, activated, and plating solutions were listed in Table 1. Firstly, coarsened CF was sensitized for 30 min at 25°C to adsorb Sn²⁺ ions. The CF was then filtered and washed with distilled water, and the sensitized CF was placed in an activated solution at 25°C for 30 min to adsorb Pd nuclei on the CF surface. Sonication was applied during the sensitization and activation treatment to promote uniform coating of the surface of CF. Finally, the activated CF was washed with distilled water and then added to a bath for metallization, which was carried out at 35°C for 15 min with magnetic stirring. Ammonia solution was used as a buffer to maintain a pH value of 9. Chemicals used in the experiments were all of analytical grade and were purchased from Kelong Reagent Co., Ltd., Chengdu, China. After plating, the Ni-Co coated CF (NCCF) was washed with distilled water and then heat-treated at 450°C in a nitrogen atmosphere for 2 h in a high-temperature tube furnace. After heat treatment, the electrical conductivity of NCCF will improve due to the phase transformation of NCCF [19].

2.3. Fabrication of Test Samples. CF and NCCF were dispersed in dimethylbenzene with paraffin wax by sonication at 60°C for 50 min, respectively. Both CF and NCCF dispersed efficiently in the paraffin wax matrix. Test samples with different weight percentages of CF and NCCF (10, 20, and 30 wt%) were fabricated according to the above methods. For electrical conductivity characterization, rectangular plates (10 mm × 10 mm × 1.5 mm) of samples were molded under 7 MPa pressure. For EMI SE characterization, samples were pressed into cylindrical dies with 7.0 mm outer diameter, 3.0 mm inner diameter, and approximately 2.5 mm height at 2 MPa.

2.4. Characterization. The morphologies of CF and NCCF were analyzed by transmission electron microscopy (TEM, Libra 200FE, Carl Zeiss SMT Pte Ltd., Germany) coupled with energy dispersive spectroscopy (EDS). Raman spectra were recorded on a Raman spectrometer (Invia, Renishaw, UK), with an excitation laser wavelength of 514.5 nm. An X-ray diffractometer (XRD, X’pert PRO, PANalytical, Netherlands) equipped with a rotating anode and Cu Kα radiation (λ = 0.15406 nm) over an incident angle (2θ) range of 10°–80° was used to identify the crystalline phases of the coating. The static magnetic properties were determined using a vibration sample magnetometer (BKT-4500Z, Beijing Ze Tian Wei Ye Science and Technology Co., Ltd., China). The sheet resistance (Rₛ) of the samples was measured by the four-probe method using a Keithley 2400 multimeter (Cleveland, OH, USA). The electromagnetic and scattering parameters of the samples were measured using a vector network analyzer (E5071C, Agilent Science and Technology Co., Ltd., USA) in the 8.2–12.4 GHz (X-band) frequency range.

### Table 1: Composition and operating conditions for the different stages in nickel-cobalt electroless coating.

| Stage       | Solution                                                                 |
|-------------|---------------------------------------------------------------------------|
| Sensitization | 23 g/L SnCl₂·2H₂O, 10 mL/L HCl                                           |
| Activation  | 0.25 g/L PdCl₂, 30 mL/L HCl                                              |
| Metallization | 17 g/L NiSO₄·6H₂O, 9 g/L CoSO₄·7H₂O, 80 g/L Na₃C₆H₅O₇·H₂O, 35 g/L Na₃H₂PO₄·H₂O, 49 g/L (NH₄)₂SO₄ |
3. Results and Discussions

The morphology of nanofibers is an important factor affecting the SE of fiber composites [20]. Figure 1(a) shows the TEM image of the obtained CF, which reveals that the CF are web-like and the diameters can be up to 20 nm. A higher magnification shows that a small amount of graphitic multilayer structure was seen as shown in the inset figure, showing that the CF is slightly graphitized. Figure 1(b) shows the Raman spectra of the obtained CF. In the Raman spectra, two fundamental vibrations are observed at 1340 cm\(^{-1}\) and 1585 cm\(^{-1}\) for CF, which are attributed to the D and G bands, respectively. The D peak can be assigned to the disorder-activated Raman mode, while the G peak arises from the sp\(^2\) hybridized carbon [21]. Figure 2 is a representative TEM image of NCCF, which clearly indicates that nickel-cobalt spherical grains were formed on the CF surface. The coating layer tended to form as spherical grains, perhaps because the surface exhibits high curvature and on a curved surface the normal growth rate is higher than the lateral growth rate. Obviously, the diameter of NCCF is wider than the obtained CF after electroless plating. These results indicate that the wider NCCF in the diameter is highly beneficial to the improvement of electrical conductivity of the CF. The EDS spectrum showed that the major components in NCCF are carbon, nickel, cobalt, and oxygen.

The phase structures of both CF and NCCF have been determined by XRD. Figure 3 shows the XRD patterns of (a) the pristine CF and (b) NCCF. It suggests that the CF and NCCF display amorphous structures. In the diffraction pattern (Figure 3(a)) of pristine CF, a broad and weak peak at 2\(\theta\) = 24.35° is observed, which is assigned to the (002) plane of graphitic carbon, which is in good agreement with Figure 1(b). In the case of NCCF, a new broad peak other than the (002) diffraction peak of graphitized CF is clearly
observed at around 44.57° as shown in Figure 3(b), which originates from the deposited Ni-Co coating layer. The results of the XRD analyses indicate that the Ni-Co nanoparticles were successfully deposited on the surface of the CF.

Figure 4 shows the magnetic hysteresis loops of CF and NCCF. The CF exhibited very little magnetization or coercivity, while the NCCF exhibited obvious magnetization. The saturation magnetization and coercivity of NCCF were 312 Oe and 5.58 emu/g, respectively. The magnetic properties of NCCF are significantly higher than those of CF, which indicates that the CF has been covered successfully by a layer of ferromagnetic Ni-Co nanoparticles.

Conductivity is another factor affecting the shielding properties of a material [22]. Electrical conductivity is calculated according to the following equation:

$$\sigma = \frac{1}{R_s t},$$  \hspace{1cm} (1)

where $\sigma$, $R_s$, and $t$ represent the direct current conductivity, sheet resistance, and sheet thickness, respectively. The relationship between electrical conductivity and nanofibers (CF and NCCF) loading is illustrated in Figure 5, which shows that the electrical conductivity of NCCF is bigger than that of pure CF at the same mass fraction. The composites containing 30 wt% NCCF exhibit the highest electrical conductivity of 1313 S/m, suggesting that the addition of electrical Ni-Co nanoparticles onto CF is highly beneficial to the electrical conductivity of the composites. The electrical conductivity increases with nanofibers loading because the interfacial affinity between nanofibers and the paraffin wax matrix increases the number of conductive pathways for electron transfer. The excellent electrical conductivity of the composites can improve the electromagnetic energy absorption and dissipation capabilities, resulting in enhancement of microwave shielding effectiveness [23].

To evaluate the EMI performance of CF and the NCCF composites, we thus measured the permittivity ($\epsilon = \epsilon' - j\epsilon''$) constitutes the real part ($\epsilon'$) and imaginary part ($\epsilon''$), resp.) of our composites in the frequency range of 8.2–12.4 GHz.

Figure 6 shows the room temperature permittivity of the pure CF and the NCCF composites. The results indicate that the real part and imaginary part of the permittivity of NCCF are bigger than those of pure CF at the same mass fraction. According to the theory of permittivity, when electromagnetic radiation is incident on metallic surface the electric field induces two types of electrical currents within the material (displacement currents and conduction). The former arises from bound charges, that is, polarization effects (real part of permittivity) which mainly involves unpaired point defects, and the latter arises due to free electrons giving rise to electric loss (imaginary part of permittivity) [24]. In other words, the real part of the composites permittivity is a measure of the number of the polarization centers inside the material originating from defects in the nanofibers structure. Therefore, the NCCF nanofibers increase the number of structural defects. The increase of the imaginary part of the permittivity can be attributed to the enhanced electrical conductivity of the composites. Both the real and imaginary parts of permittivity increase as the loading of the CF or NCCF was increased, which reflects an increase in both the number of polarization centers and dissipating mobile charge carriers, leading to higher permittivity. While this explanation implies that more structural defects and high conductivity result in high permittivity, nevertheless, it is noted that the permittivity decreases with increasing frequency. This behavior is due to the lag of the induced electric field in the composites in response to the reversing external E-field, which leads to a reduction in electronic oscillation at high frequency [25].

The EMI SE of a material is represented by the following equation:

$$SE_T = 10 \log \left( \frac{P_m}{P_{out}} \right) = SE_R + SE_A + SE_M,$$  \hspace{1cm} (2)
where $P_{in}$ and $P_{out}$ are the power (electric or magnetic field) of incident and transmitted EM waves, respectively, and $S_E$, $S_A$, and $S_M$ are the SE due to reflection, absorption, and multiple reflections, respectively [26]. Reflection occurs as a result of mobile charge carriers (electrons or holes) in the shield material which interact with the electromagnetic field in the radiation; the absorption is due to the energy dissipation when electromagnetic wave interacts with electric or magnetic dipoles in the shield material; the multiple reflections refer to the reflection at different surfaces or interfaces in the shield material [27].

$S_{11}$ (or $S_{12}$) and $S_{21}$ (or $S_{22}$) are the scattering parameters (S-parameters) of the two-port vector network analyzer system and can be related to reflectance and transmittance as $R = |S_{11}|^2 = |S_{22}|^2$ and $T = |S_{21}|^2 = |S_{12}|^2$. The absorbance ($A$) can be written as $A = (1 - R - T)$. When $S_{11}$ is higher than 10 dB, $S_{11}$ can be ignored [28]. Therefore, $S_E$ can be conveniently expressed as $S_E = S_R + S_A$. The EMI SE was calculated based on the S-parameters as follows:

$$S_E = 10 \log \frac{1}{|S_{12}|^2} = 10 \log \frac{1}{|S_{21}|^2},$$

$$S_R = 10 \log \left( \frac{1}{1 - |S_{11}|^2} \right),$$

$$S_A = 10 \log \left( \frac{1 - |S_{11}|^2}{|S_{12}|^2} \right),$$

where $|S_{ij}|^2$ represents the power transmitted from port $i$ to port $j$ [29].

The influence of mass fraction on the $S_E$ values of the composites at 10 GHz is shown in Figure 7. The experimental trend is similar to the trend of the electric conductivity of the CF and NCCF composites. In line with the above data concerning conductivity and permittivity, the $S_E$ values of the CF and NCCF composites increased with the weight percentage. This may be ascribed to the increase of electrical conductivity of the composites. As was observed for the conductivity of composites, the $S_E$ of the NCCF composite is significantly higher than that of CF at the same mass fraction. The Ni-Co coating on the CF can lead to a decrease in the contact loss between CF due to the formation of metal-metal interfaces. This behavior can improve the electrical conductivity and subsequently the $S_E$ of the NCCF composites.

To clarify the EMI shielding mechanism of the CF and NCCF composites, the EMI $S_E$, $S_A$, and $S_M$ of the composites with 30 wt% conductive nanofibers in the 8.2–12.4 GHz range are shown in Figure 8. From the figure, $S_{11}$ was shown to be ca. 34.3 and 37.3 dB for CF and NCCF composites at 12.4 GHz, respectively. For the reflection component, $S_R$ was ca. 2.6 and 3.9 dB for CF and NCCF composites at 12.4 GHz, respectively. These results demonstrate that the $S_E$ of the NCCF composites can be up to 41.2 dB (99.99% attenuation), which is higher than that of the pristine CF composites [30]. It has been observed that, for conduct CF and NCCF composites, the $S_E$ is mainly dominated by absorption while the $S_R$ contributes little.

### 4. Conclusions

In conclusion, NCCF was successfully manufactured by electroless plating. This work demonstrates that both the electrical conductivity and the EMI SE increase with nanofibers loading and the paraffin wax composite containing 30 wt% NCCF exhibited the highest electrical conductivity of
1313 S/m and EMI SE value of 41.2 dB (99.99% attenuation). This result may be attributed to the presence of Ni-Co coated on the CF, which can lead to decrease in the contact loss between CF due to the formation of metal-metal interfaces. This behavior can improve the electrical conductivity and permittivity and subsequently improve the EMI SE. The SE of composites increases with nanofibers loading and the absorption is the primary factor governing EMI shielding. This study reveals that the NCCF composites have potential applications as EMI shielding materials.

**Conflict of Interests**

The authors declare that there is no conflict of interests regarding the publication of this paper.

**Acknowledgment**

This work was supported by the Program for New Century Excellent Talents in university (no. MCET-11-1061).

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