Article

A Novel Processing for CNT-Reinforced Mg-Matrix Laminated Composites to Enhance the Electromagnetic Shielding Property

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Abstract: The microstructure, electrical conductivity, and electromagnetic interference (EMI) shielding effectiveness (SE) of CNTs/Mg Matrix composites prepared by accumulative roll bonding (ARB) were systematically investigated to understand the effects of CNTs on the electromagnetic interference shielding effectiveness property of magnesium. A model based on the shielding of the electromagnetic plane wave was used to theoretically discuss the EMI shielding mechanisms of ARB-processed composites. The experimental results indicated that the methods were feasible to prepare laminated composites. The SE of the material increased gradually with the increase of electrophoretic deposition time. When the electrophoretic deposition time reached 8 min, the value of SE remained 87–95 dB in the frequency range of 8.2–12.4 GHz. The increase in SE was mainly attributed to the improvement in the reflection and multiple reflection losses of incident electromagnetic wave due to the increased amounts of CNTs and interfaces. The methods provided an efficient strategy to produce laminated metal matrix composites with high electromagnetic shielding properties.

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1. Introduction

With the rapid popularization of electronic equipment and the continuous improvement of the utilization rate, electronic noise, electromagnetic interference (EMI) and radio frequency interference, other electronic pollutions increase sharply [1,2], which will lead to the abnormal operation of electronic equipment and harm human health [3–5]. Therefore, EMI has become a matter of concern, especially in the field of 3C and electrical apparatuses [6]. In order to ease the pollution of electromagnetic interference, it is imperative to develop EMI shielding materials [7]. However, traditional metal materials, such as copper, nickel, and permalloy have the drawback of their heavy weight [8,9]; polymer composites are light in weight, but they suffer in terms of their cost and low strength [10–13], so they cannot be used as structural materials; and coats materials are easy to shed. Therefore, it is urgent to develop light weight and cost-effective shielding materials, especially in portable electronics and aerospace industries [8].

Magnesium possess low density, high specific stiffness, high specific strength, excellent damping capacity, recyclability, and easy fabrication [14–16] while also possessing good electromagnetic shielding property [5]. At present, most of the researches on the electromagnetic shielding properties of magnesium with rare earth (RE) have already been carried out [3,5,9,17,18]. For example, Chen et al. explored the different Nb contents on the electromagnetic interference (EMI) shielding properties of an Mg–Y–Zr–Nd alloy, and it was found that the EMI shielding effectiveness of the alloy could be improved by the precipitated second phases of Mg₂₄Y₅, Mg₄₁Nd₅, and β phase with a Mg₄₁Nd₅Y composition [3]; Liu et al. reported that Ce addition induced the formation of the Mn–Zn–Ce

phase, which was beneficial to improve the SE [19]. However, the addition of RE increased the cost and density.

As is known to all, Carbon nanotubes (CNTs) are ideal metal-matrix reinforcers, which possess extremely high elastic modulus and strength, as well as thermal, electrical and absorbed properties [20–24]. CNTs could be deposited on substrates by the electrophoretic deposition (EPD) process, which is an effective method; as well, the thickness of CNTs can be controlled [25–27]. The accumulative rolling bonding (ARB) process is not only a traditional rolling deformation method, but also a metallurgical diffusion welding process [28,29]. The process can manufacture ultra-fine grain materials and is a low cost, simple process. In the experiments, we produced the magnesium matrix composites with good electromagnetic shielding by the method; it could weld the adjacent layers and disperse CNTs evenly, resulting in a layered structure with the increase of rolling passes. When this occurs, the number of interface layers will increase rapidly.

In this paper, CNTs/Mg matrix laminated composite materials were designed and produced by the ARB process. The EMI SE of composites were investigated in the X-band frequency range. In general, the reaches were expected to lead to new insights into design a lightweight shielding material providing radiation protection.

2. Experimental Methods

The fabrication process of the CNT-reinforced Mg-matrix laminated composites was shown in Figure 1. The five main procedures were included. (1) The CNTs was acidizing treatment; (2) the CNTs were deposited on Mg plates by electrophoretic deposition (EPD); (3) the plates were stacked in the order; (4) the stacked plates were warmly rolled; and (5) the rolled plates were cut into equal parts and then rolled again.

Figure 1. Schematic illustration for the preparation of composite materials with layered structure.

Commercially pure magnesium (99.95 wt.%), with a thickness of 0.5 mm, and multi-walled CNTs (inner diameter: 2–10 nm, outer diameter: 20–40 nm, length: 10–30 μm) were purchased from the Shanghai Aladdin Biochemical Technology. The specifications of all the chemicals are analytical pure. Van der Waals forces exist between the carbon nanotubes, which could cause aggregation of carbon nanotubes. Therefore, CNTs were added to the mixture of HNO_3 and H_2SO_4 (1:3 volume ratio), after ultrasonic treated for 30 min, the slurry was stirred for 6 h at 70 $^\circ\text{C}$, then place into the dialysis bag which was dialyzed by distilled water until the PH reached 7. Subsequently, the slurry was dried at 110 $^\circ\text{C}$. 0.04 g of surfaced treated CNTs and 0.08 g of $\text{Al}(\text{NO}_3)_3$ were dispersed into 1000 mL alcohol and then ultrasonic treated for 4 h. Finally, the CNTs aqueous dispersion were obtained. CNTs were deposited onto Mg sheets by EPD process and the layer elements of the composites were prepared. Mg sheets 110 $\times$ 60 $\times$ 0.5 mm, which were cleaned by the steel ball, washed by alcohol. Then the sheets were served as the cathode. The stainless-steel sheet was adopted as the anode. The distance of the two electrodes was arranged as 50 mm, and the

voltage was 60 V. By changing the deposition time, the amount of CNTs deposited on the magnesium plate was varied, in this experiment, the time of EPD was arranged as 0 min, 4 min, and 8 min (named the materials I–III) respectively. In the ARB process, the reduction was 50–60% per pass, with the rolling temperature of 270 °C, in the middle of each pass, the materials were annealed at 300 °C for 5 min. After the initial bonding, the materials were rolled three times by ARB. The schematic of ARB process was shown as Figure 1. Finally, CNTs/Mg matrix materials with laminated structure were produced.

Microstructures were observed by optical microscopy (OM) (ZEISS, Jena, Germany). The microstructures and fracture surfaces of Mg and composites were performed by scanning electron microscopy (SEM) (ZEISS, Jena, Germany). Raman spectra were utilized to analyze the structure characteristics of CNTs with a 532 nm wavelength laser (JOBIN YVON, Paris, France). The phase determination was carried out with X-ray diffractometer (XRD) (PANalytical B.V, The Netherlands). The electrical conductivity of the composites was evaluated utilizing Sigma 2008B conductivity meter (Tianyan, Xiamen, China), and each conductivity value was the average of five tests. EMI SE was determined in the X-band (8.2–12.4 GHz), which using the standard coaxial cable method in accordance with ASTM D4934-2010 (Dinrong, Beijing, China) [7], the samples in a square form with 41.4×41.4 mm, and 0.5 mm in thickness.

3. Results and Discussion

3.1. CNTs Dispersion

As shown in Figure 2. The raw CNTs agglomerated in large clusters. After acidification, the process was conducive to dispersion, and it is known that the acidification process could overcome the Van der Waals force between CNTs [30–33], which was conducive to dispersion. However, the multilayer structure of CNTs could be damaged during acidification process. It was well known that the Raman spectra provided information regarding the quality of the internal CNTs [34,35]. From the Raman spectra results in Figure 3, the intensity ratio (I_D/I_G) of disordered-band (D-band) near 1350 cm^{-1} to the graphite-band (G-band) near 1590 cm^{-1} was known to provide information on the structure of CNTs, the minor difference between these I_D/I_G values indicated that the CNT structure was hardly damaged during the fabrication process, the values indicated that the process was attributed to the introduction of the functional groups which was beneficial to EPD.

Figure 2. SEM images of the CNTs. (a) pristine CNTs, (b) acid treated CNTs.

Figure 3. Raman spectra of CNTs. (a) pristine CNTs and (b) surface treated CNTs.

3.2. Microstructure of Laminated Composite

Microstructure of the pure magnesium plate after three times of the ARB process was showed in Figure 4. In the RD-ND cross section, the lamellar structure was inapparent, which illustrated that the subsequent rolling was beneficial to the previous combination.

Figure 4. SEM micrographs of the pure magnesium sheet after three times.

Figure 5 illustrated the OM images of composites with different EPD. Through the EPD process and ARB process, the composites were formed into the layered structure. The thickness of the CNTs increased with EPD time, and it could be seen that the ARB process could promote the distribution of CNTs by the way of the shear stress. Figure 6 illustrated the microstructure of EPD 8 min and corresponding magnified figure. From Figure 6b, it could be seen that carbon nanotubes and magnesium matrix were closely combined, and there were no obvious holes at the interfaces. At the same time, the rolling process could promote dispersion secondarily, in the interfaces, there were some sites where CNTs gathered and some were rare, however, the overall trend was relatively uniform, as shown in Figure 6a.

Figure 5. OM micrographs of the ARB process composites microstructures. (a) The EPD 4 min and (b) the EPD 8 min.

Figure 6. SEM micrographs of the EPD 8 min composites (a,b) corresponding magnified figure.

Figure 7 displayed the typical XRD patterns of CNTs/Mg matrix composites with different EPD time, the profile for composite included peak which corresponded to the Mg phase. The peaks of Mg–C could not be clearly identified, which indicated that there was no reaction between Mg and C.

Figure 7. XRD pattern of the ARB process materials.

From Figure 8, the lamellar structure could be seen at the fracture surface, which indicated that the lamellar structure was formed in the process of ARB. When the electromagnetic wave propagates in the composite materials, these layered structures will provide multilayers reflection in the process of electromagnetic shielding attenuation.

Figure 8. Fracture Surface of the composites. (a) EPD 4 min and (b) EPD 8 min.

3.3. Electromagnetic Shielding Properties

The electromagnetic shielding effectiveness of the CNTs/Mg matrix composites with different EPD times in the X-band were shown in Figure 9. The curves were wavy, which did not show a decreasing trend with the increase of the frequency. The reasons were that the composites were prepared into layered structure and there were the oxidation hardening layer, CNTs in the middle of the composites. The SE value of the pure magnesium after ARB process was 82–92 dB; with the EPD 4 min, the value was 86–94 dB; with increasing EPD 8 min, the composite showed that the value was 87–95 dB. With the increase of the EPD time, the overall EMI shielding performance increased. The EPD process played an obvious effect on the electromagnetic shielding performance. The ARB process can form layered structure and improve electromagnetic shielding performance [7,11]. In this experiment, the materials possessed the good shielding performance.

Figure 9. EMI shielding effectiveness of the different EPD time during 8.2–12.4 GHz.

The layered composites were constructed by the ARB process, and there were some interfaces in the composites. The electromagnetic shielding mechanisms were illustrated in Figure 10. The shielding mechanisms of the composite were special owing to the CNTs between adjacent magnesium layers. Impedance value of magnesium was different from CNTs, so it was necessary to make a detailed analysis of its electromagnetic radiation attenuations.

Figure 10. The electromagnetic shielding mechanism of the layered composites.

In the primary, the CNTs in the middle of pure magnesium, with the increase of ARB process, the layered structure with magnesium on both sides and CNTs in the middle was formed. In such a structure, the impedance of adjacent layers was different. When the electromagnetic wave passed through the composites, the special structure provided the multilayer reflection, which contributed to increase the reflection attenuation. At the same time, the interface had a positive effect on the EMI shielding property, which was like “wavy”, as shown in Figure 6. The interface could enhance the multiple reflection loss. As is known, the ARB process could refine the grain [28,29], which could introduce the grain boundary in the plates and could also increase the multiple reflection loss, scattering the incident electromagnetic wave.

When the electromagnetic wave incidented on the composites and propagated in the multilayer shielding materials, the shielding property could be enhanced under the synergistic effect of the above factors. The electromagnetic shielding mechanisms of CNTs/Mg matrix composites were more complex than that of the magnesium alloy, which were attributed to multilayer-reflection, CNTs-reflection, CNTs-absorption, and so on.

According to the Schelkunoff formula [6], the transmission line analogy is suitable for a conductor flat-type shielding material [8,11,17]. When the electromagnetic wave propagates in the prepared materials, we regard the composites as a transmission line, when the electromagnetic wave incident on the material surface from the medium air, owing to the mismatch impedance between the materials and the air, the reflected electromagnetic wave occurred. At the same time, when the transmitted wave propagates in the primary layer of magnesium matrix, there will be absorption loss. Meanwhile, when the transmitted electromagnetic wave reaches the CNTs layer, due to the different impedance between the magnesium and the CNTs, the reflection loss will occur again. Next, the absorption loss will occur in the CNTs. According to this mode, the electromagnetic wave propagates in the composites until it passes through the composites. In order to quantitatively describe

the shielding performance of the materials, the shielding effectiveness (SE) is used. The total SE value can be expressed by the following formula:

$$SE(dB) = R + A + B \quad (1)$$

$$SE_R = 20 \lg \frac{|1+k|^2}{4|k|}, k = \frac{Z_m}{Z_0} \quad (2)$$

$$SE_A = 20 \lg(e^{\frac{t}{\delta_m}}) \quad (3)$$

$$SE_B = 20 \lg(1 - e^{\frac{-2t}{\delta_m}}) \quad (4)$$

$$Z_m = 3.68 \times 10^{-7} \sqrt{\frac{f \mu_m}{\sigma_m}} \quad (5)$$

$$\delta_m = \sqrt{\frac{1}{\pi \mu_m \sigma_m f}} \quad (6)$$

where $R, A, B, Z_m, B, Z_0, t, f, \delta_m, \sigma_m, \mu_m$ are reflection loss at the two interface of the shielding material, absorption loss in the body of the shielding material, multiple reflection loss, shield impedance, air impedance (377Ω), thickness, electromagnetic radiation frequency, skin depth, electrical conductivity, and magnetic permeability, respectively.

Based on Equations (1), (2) and (5), the EMI shielding capacity of CNTs/Mg depended on the electrical conductivity mainly.

Figure 11 shows the electrical conductivity of the composites with different content of CNTs; the value is $22.07, 21.96$ and $21.89 \times 10^6 \text{ S/m}$, respectively. As we have learned, in the electromagnetic shielding alloys, the high electrical conductivity converts to high vortex electrical current and the abundant energy of incident wave dissipate in the form of ohmic electrothermal heat, at the same time producing a high reverse electromagnetic field [9]. From Figure 11, the conductivity decreased slightly, but the overall change was not significant. The electrical conductivity declined slightly with the increase of electrophoresis time. The ARB process could introduce oxide layers and other contaminants, which could reduce the conductivity of the materials. However the existence of the CNTs could improve the conductivity, so the change of the conductivity was slight.

Figure 11. Electrical conductivity of the layered composites with different EPD time.

4. Conclusions

1. A new CNTs/Mg matrix laminated composite was prepared by ARB method, which was applied to the field of electromagnetic shielding.

2. When the EPD time was 8 min, in the testing frequency of 8.2–12.4 GHz, the SE value was 85–96 dB. The main shielding mechanism was closely related to the layered structure. Since the impedance of adjacent layers was inconsistent, the layered structure increased the multilayer reflection ability.
3. Oxide layers and other contaminants could be introduced into the interlayer during EPD and ARB process. Due to the existence of CNTs, the electrical conductivity declined slightly, and the value was 22.07, 21.96 and 21.89×10^6 S/m, respectively.

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