Preparation, electromagnetic and enhanced microwave absorption properties of Fe nanoparticles encapsulated in carbon nanotubes

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Abstract: Using hollow Fe₂O₃ particles as the catalyst, the core/shell structured Fe/carbon nanotube (CNT) hybrid could be synthesized by a chemical vapor deposition method without the hydrogen reduction process. Based on the obtained results, a possible growth mechanism of the Fe/CNT hybrid was discussed. And the investigations of electromagnetic and microwave absorption performances indicate that a minimum reflection loss (RL) value of the obtained sample is ca. -40.15 dB at 17.15 GHz with a matching thickness of 1.5 mm, and the RL value below -20 dB can be obtained in the whole frequency range (1.0-18 GHz) with the sample thickness varies from 1.3 to 10.0 mm. The results demonstrate that a simple and environment-friendly route has been proposed for the production of core/shell structured carbon nanohybrid. The obtained Fe/CNT hybrid exhibits excellent microwave absorption properties and has potential applications in thin thickness and light weight microwave absorbers.

Keywords: Core/shell structures; Fe/CNT hybrid; Chemical vapor deposition; Microwave absorption properties

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1. Introduction

In order to solve the expanded electromagnetic interference (EMI) problems in recent years, considerable theoretical and experimental investigations have been focused on designing and fabricating the cost-efficient, light-weight and effective microwave absorption materials (MAMs) with promising applications in electronic devices for commercial, industrial and military affairs [1-3]. It is well known that the complex permittivity ($\varepsilon_r = \varepsilon'_r - j\varepsilon''_r$) and permeability ($\mu_r = \mu'_r - j\mu''_r$) of MAMs are the crucial factors for their microwave absorption performances. Therefore, MAMs can be divided into dielectric absorbers and magnetic absorbers [4]. However, unilateral dielectric materials or magnetic absorbers are difficult to achieve impedance match conditions [5]. Moreover, these MAMs are also restricted because of their high density and/or instability at room temperature (RT).

Nowdays, because of their synergistic properties, coating of carbon nanotubes (CNTs) with conductive polymers, functionalization of CNTs and core/shell structured nanohybrids have been studied intensively as the novel high-performance microwave absorbers and optical sensors [6-14]. Among them, coating magnetic loss materials with carbon nanotubes (CNTs) for tunable dielectric loss and/or magnetic loss attract extensive interest due to their advantages with respect to light weight, low cost, design flexibility and good stability [14-17]. For example, Kim et al. reported the excellent EMI shielding of multiwalled CNT hybrids containing Fe catalyst [18]. Che et al. found that Fe encapsulated within CNTs presented an improved ability for microwave absorption [19]. Su et al. and Zhao et al. studied the microwave absorbing properties of Fe filled CNTs in details, respectively [20,21]. Generally, the methods for the synthesis of core/shell nanohybrids usually involve the catalytic deposition of carbon source over metal nanoparticles or the opening of CNTs and depositing the

foreign material. However, the electromagnetic (EM) absorbing performances of Fe/CNTs are not very good such as the obtained optimal reflection loss (RL) of the obtained MAMs is usually above -30 dB. Moreover, the growth of core/shell nanohybrids directly over metal oxide was seldom reported.

In the previous work, we have employed the chemical vapor deposition technique for the synthesis of core/shell structured carbon nanohybrids over Ni or Fe nanoparticles [22-25]. What is common among these routes is the reduction of metal oxide in hydrogen. In this paper, we report the synthesis of core/shell structured Fe/CNTs directly over the hollow Fe₂O₃ particles. The microstructure, complex permittivity and permeability, and microwave absorbing performances of Fe/CNTs were investigated in details. Because of the effective complementarities between the dielectric loss and magnetic loss, the obtained Fe/CNTs are superior to those Fe/CNTs reported before in terms of microwave absorption ability [26,27].

2. Experimental

2.1. Material preparation

Similar to the method as described elsewhere [28], the hollow Fe₂O₃ particles were synthesized by the hydrothermal method. In a typical procedure, 30 ml of aqueous NH₄H₂PO₄ solution (0.05 M) was added to 30 ml of aqueous Fe(NO₃)₃ solution (0.3 M) under vigorous stirring at RT. The obtained solution was transferred into a Teflon-sealed autoclave for hydrothermal treatment at 220 °C for 48 h. After the vessel cooling to RT, the product was collected after centrifugation, washed with deionized water and absolute ethanol, and dried at 80 °C for 8 h. Then, the obtained product was dispersed on a ceramic plate and placed inside a quartz tube, and acetylene was introduced into the tube at 600 °C for 2 h. After cooling to RT, a certain amount of sample (black in color) was collected on the plate.

2.2. Characterization

The phases and morphologies of sample were examined over an X-ray powder diffractometer (XRD), a transmission electron microscope (TEM) and a field-emission scanning electron microscope (FE-SEM), respectively. Fourier transform infrared (FTIR) spectroscopy of sample was recorded using a Nicolet 510P spectrometer. For microwave measurement, 30 wt% of the as-prepared sample was mixed with paraffin and pressed into coaxial clapper in a dimension of outer diameter of 7.0 mm, inner diameter of 3.0 mm, respectively. The complex permittivity and permeability of the composite were measured in a frequency range of 1.0-18 GHz over an Agilent E8363B vector network analyzer. The frequency dependence of RL and attenuation constant α were calculated by the following equations [29,30]:

$$\alpha = \frac{\sqrt{2}\pi f}{c} \sqrt{(\mu''\epsilon'' - \mu'\epsilon') + \sqrt{(\mu''\epsilon'' - \mu'\epsilon')^2 + (\epsilon'\mu'' + \epsilon''\mu')^2}} \quad (1)$$

$$Z_{in} = \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh\left(j \frac{2\pi f d \sqrt{\mu_r \epsilon_r}}{c}\right) \quad (2)$$

$$RL = 20 \log \left| \frac{Z_{in} - 1}{Z_{in} + 1} \right| \quad (3)$$

where f is the frequency of EM wave, d is the thickness of an absorber, c is the velocity of light and Z_{in} is the input impedance of the absorber.

3. Results and discussion

3.1. Phase, morphology and discussion on the possible growth mechanism of the core/shell structured Fe/CNT hybrid

Figure 1 shows the XRD pattern and microstructure images of the catalyst. As shown in Figure 1a, the XRD pattern of the catalyst indicates that all the peaks correspond to Fe_2O_3 , which is very close to the literature value (JCPDS No.86-0550).

No other peak of impurity can be detected. The FE-SEM investigation of the catalyst (as shown in Figure 1b) shows that the size-uniform (average diameter: ca. 200 nm) hollow Fe_2O_3 particles (as indicated by the arrows) is the majority in the obtained catalyst, and the content of hollow particles in the obtained sample is ca. 88%. Overall, the results confirm that the used catalyst is the hollow Fe_2O_3 particles.

Figure 2 displays the phase and microstructure of the as-synthesized sample. As shown in Figure 2a, the XRD pattern indicates that the diffraction peaks (as indicated by the symbols of round and rhombus) can be assignable to graphite (JCPDS No.75-1621) and $\alpha\text{-Fe}$ (JCPDS No.01-1262) can be observed clearly, respectively. And the other peaks can be indexed to the orthorhombic phase with the lattice constants $a = 5.091 \text{ \AA}$, $b = 6.744 \text{ \AA}$ and $c = 4.527 \text{ \AA}$ (JCPDS No.34-0001). Figure 2b shows the FTIR spectrum of the obtained sample. No infrared signals such as $-\text{CH}=\text{CH}-$ or $-\text{CH}_3$ entities can be detected over the sample. Accordingly, we deduce that C_2H_2 is decomposed completely over the hollow Fe_2O_3 particles, which is consistent with the sharp peak of graphite in the XRD characterization. As shown in Figure 2c, the FE-SEM observation of the as-synthesized sample reveals that a large quantity of size-uniform carbon nanomaterials (CNMs) have been obtained, exceeding 93% of the entire content. The diameters of the obtained CNMs are in the 80-120 nm range. Figure 1d shows a typical TEM image of the obtained CNMs. A closer TEM observation reveals that the catalyst particles are enwrapped tightly by CNT and the sizes of encapsulated particles are in the range of 50-90 nm. All the results indicate that the obtained sample is the core/shell structured Fe/CNT hybrid. Compared to the previously reported Fe-encapsulated CNTs [20,26], more and larger nanoparticles (as indicated by the arrows) are formed inside the CNT product here. Most important of all, the Fe/CNT hybrid can be synthesized directly over Fe_2O_3

particles without the hydrogen reduction process. Briefly, in our experimental process, the formation of core/shell carbon nanohybrids can be explained by the following reactions:

Through the aforementioned reactions, the element of Fe can form through the reduction of Fe_2O_3 by C_2H_2 , and Fe particle is carbonized easily to give the formation of Fe_3C . It is well known that the formation of Fe_3C is very crucial to the catalytic growth of core/shell carbon nanohybrids effectively [31]. Moreover, according to the previous models for CNM growth and reported experimental results [32-36], the catalyst nanoparticles can appear at one end/both ends of CNMs or inside the obtained CNMs for the growth of CNMs in the CVD process, which can also be seen clearly in our experiment. Generally, based on the previous models and obtained experimental results, the growth of Fe/CNTs can be understand fully in the paper.

3.2. EM and microwave absorbing properties of the core/shell structured Fe/CNT hybrid

The EM properties of the composite containing 30 wt% of Fe/CNTs have been investigated, which are the vital importance to its microwave absorbing performances. As illustrated in Figure 3a, the real (ϵ') part of relative complex permittivity of the composite declines from 15.5 to 7.1. And the imaginary (ϵ'') part of relative complex permittivity of the composite almost remains constant, indicating that it has a stable dielectric loss property. Figure 3b shows the real (μ') and imaginary (μ'') part of complex permeability obtained over the composite as a function of frequency. One can see that the former is close to 1.08 while the latter to 0. And two resonance peaks at ca. 7.5 and 16.0 GHz can be observed clearly. It is well known that the magnetic

loss of magnetic materials originates mainly from the natural resonance, eddy current loss and exchange resonance in the microwave region. And the eddy current loss can be expressed by the theory, $\mu'' = 2\pi\mu_0(\mu')^2 d^2 f \sigma$, where μ_0 is the permeability of vacuum and σ is the electric conductivity. If magnetic loss only results from the eddy current loss, the value of $\mu''(\mu')^{-2} f^{-1} = 2\pi\mu_0 d^2 \sigma$ should be constant when the frequency varies. Figure 3c shows the value of $\mu''(\mu')^{-2} f^{-1}$ as a function of frequency. One can find that the value increases at the low frequency range and remains nearly constant at the high frequency. Therefore, the magnetic loss is mainly caused by the natural resonance at the low frequency and the eddy current loss should contribute to magnetic loss at the relatively high frequency. And the eddy current can produce an induced magnetic field which opposes the applied field and in turn radiates EM wave, leading to a negative μ'' at the high frequency (as shown in Figure 3b) [37]. Based on the data in Figures 3a and b, the dielectric tangent loss ($\tan \delta_E = \epsilon''/\epsilon'$) and magnetic tangent loss ($\tan \delta_m = \mu''/\mu'$) were calculated, and the results are shown in Figure 3c. The $\tan \delta_E$ value is much larger than $\tan \delta_m$ in the whole frequency range, indicating that the dielectric loss plays the main role in the EM absorption. Besides, one can observe clearly that the change trend of $\tan \delta_E$ inverses to that of $\tan \delta_m$. Such complementarities between the dielectric loss and magnetic loss favor the strong EM attenuation, which implying that the obtained Fe/CNT hybrid has the excellent EM absorption properties.

Based on the measured EM parameters and aforementioned equations, the microwave absorption characteristics of the Fe/CNT composite as a function of frequency can be obtained, which are shown in Figure 4. As shown in Figure 4a, the Fe/CNT composite exhibits a big value of α in the 1.0-18 GHz frequency range.

Compared to those core/shell structured carbon nanohybrids reported elsewhere [26,38], the as-synthesized Fe/CNT composite shows the higher α values, indicating the excellent attenuation or EM absorption. From the color map of RL, as shown in Figure 4b, it is obvious that the minimum RL value moves toward to the lower frequency region with an increasing thickness. The RL values below -20 dB (99% of EM wave attenuation) can be obtained in the whole frequency range with the sample thickness varies from 1.3 to 10.0 mm. Figures 4c and d depict the RL of the obtained composite in the 1.5-8.0 mm thickness range. It indicates that the minimum RL value reaches -40.15 dB at 17.15 GHz with a matching thickness (d_m) of 1.5 mm, and double absorption features can be observed clearly in the thickness range of 4.0-8.0 mm, which are helpful to broaden the bandwidth of microwave absorption. Compared with those representative Fe/CNTs reported before, as shown in Table 1, the obtained Fe/CNTs of the present study exhibit enhanced microwave absorption ability. Generally, the obtained results indicate that the core/shell Fe/CNTs are very promising candidates for lightweight MAMs. Based on the obtained results and compared to the previous results [24,26], the enhanced microwave absorption ability of the obtained Fe/CNTs should be related to the more magnetic nanoparticles inside the CNT product and lower value of ϵ' . It is well known that the high content of magnetic nanoparticles in CNTs is conducive to achieve the good complementarities between the dielectric loss and magnetic loss, which can promote their EM absorption ability effectively. Moreover, in terms of magnetic nanoparticle/CNT hybrids, it has been reported that a relatively low ϵ' is favorable to strike a balance between the permeability and permittivity, which favors the penetration of EM wave and results in the enhanced EM absorption [40-42].

4. Conclusions

In summary, the core/shell structured Fe/CNT hybrid has been synthesized in high yield directly over the hollow Fe₂O₃ particles. Because of the high attenuation constant and good complementarities between the dielectric loss and magnetic loss, the excellent microwave absorption performances have been obtained. A minimum RL value of ca. -40.15 dB at 17.15 GHz with a matching thickness (d_m) of 1.5 mm was observed. The RL value below -20 dB could be obtained in the whole frequency range. Compared to those of representative Fe/CNTs, the obtained core/shell structured Fe/ CNT hybrid exhibits the enhanced microwave absorbing performances and can be used as the light-weight EM wave absorber.

Acknowledgments

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Figure Captions

Figure 1. (a) XRD pattern, and (b) a typical FE-SEM image of the obtained catalyst.

Figure 2. (a) XRD pattern, (b) IR spectrum, (c) FE-SEM image, and (d) TEM image of the Fe/CNT hybrid.

Figure 3. (a) Complex permittivity, (b) complex permeability, (c) values of $\mu''(\mu')^{-2} \text{ f}^{-1}$, and (d) loss tangent versus frequency for the Fe/CNT composite.

Figure 4. Microwave absorption characteristics of the Fe/CNT composite: (a) Attenuation constant, (b) Two-dimensional representation of RL, and (c,d) absorption properties of composite with different thickness.

Table 1. EM wave absorption properties of the representative Fe/CNT hybrids.

Sample	Optimal RL value (dB)	d_m (mm) (RL<-20 dB)	Frequency range (GHz) (RL<-10 dB)	Frequency range (GHz) (RL<-20 dB)	Reference
Fe/CNTs	-25	1.2	2-18	10.2-11.6	19
Fe/CNTs	-25	2.0	2-10.1	9.6-10.6	20
Fe/CNTs	-31.71	1.0	11.8-14.7	12.2-13.8	21
Fe/CNTs	-26.39	2-3	4-18	7.5-10.7	24
Fe/CNTs	-39	3.36-5.57	2.04-7.5	2.04-3.47	26
Fe/L-HCNTs ^a	-39.40	1.5-3.0	0.5-18	7.0-15.2	27
Fe/CNTs	-22.73	3.5	11.8-18	15-16	39
Fe/CNTs	-40.15	1.5-8.0	0.5-18	2.0-17.9	This work

^aFe/low-helicity carbon nanotubes

Figure. 3 (a) Complex permittivity, (b) complex permeability, (c) values of $\mu''(\mu')^{-2} f^{-1}$, and (d) loss tangent versus frequency for the Fe/CNT composite.

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- The paper reports the synthesis of Fe/CNT hybrid directly over Fe₂O₃ particles.
- The RL value below -20 dB can be obtained in the 1.0-18 GHz frequency range.
- The obtained Fe/CNT hybrid exhibits enhanced microwave absorption property.