

Short Communication

Investigation on the Microwave Absorbing Property of Fe₃O₄/CNFs Synthesized by Chemical Co-precipitation

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Received: 27 March 2015 / Accepted: 1 August 2015 / Published: 26 August 2015

In this paper, Fe₃O₄ nanoparticles have been coated on the surface of Carbon nanofibers (CNFs) by the method of chemical co-precipitation. This Fe₃O₄/coated CNFs exhibit remarkably improved microwave absorption property as compared to the bare CNFs. Our research provides an economical and highly productive approach for fabricating Fe₃O₄/CNFs nanostructures which is suitable for the application of electromagnetic wave absorption and shielding.

Keywords: Carbon nanofiber; Fe₃O₄ nanoparticles; chemical co-precipitation; Microwave absorption

1. INTRODUCTION

Carbon nanofiber (CNF) is an excellent kind of microwave absorption material that has great carrying capacity, low radar cross section, high strength and modulus [1]. It can be used for structural maintenance and multiple functions material. As compared to many other functional materials, carbon nanofibers exhibit more excellent performances in the aspects of conductivity [2,3] and electronic countermeasure [4]. It also can be adopted as a kind of electromagnetic shielding material [5,6]. The distinct advantages of CNFs, for instance, light weight, large surface area, excellent thermal and electrical conductivity property [5,7] when used as microwave absorption material, make it more competitive in practical application. However, CNFs mainly take the dielectric loss and very low magnetic loss, which limits their performance. It has been reported that introducing Fe₃O₄ coating on the carbon can improve its microwave absorption performance, but Fe₃O₄ nanoparticle possess high

surface energy and magnetic property which would cause them to aggregate with each other and not uniformly coated on the carbon surface [8-10]. Therefore, to promote the high resistivity [11] and good magnetic [12] Fe₃O₄ nanoparticles uniform coating, we use fenton reagent to treat CNFs. The Fe₃O₄ coating adjusts the electric-magnetic parameters, so that make the dielectric loss and magnetic loss combined, making the composite a potential microwave absorption material.

A variety of techniques were used to fabricate Fe₃O₄/carbon composite, such as electrochemical deposition [13], magnetron sputtering [14], sol-gel [15] and chemical precipitation [16]. For these techniques, the chemical co-deposition is broadly adopted owing to its simple operation and device [17]. Furthermore, the Fe₃O₄ coating produced by this method displays great continuity and homogeneity. In this research, we have successfully prepared Fe₃O₄ coating on the surface of CNFs by chemical co-precipitation method, leading to the formation of Fe₃O₄/CNFs composite. The structure, morphology, microwave absorption properties of bare CNFs and Fe₃O₄/CNFs composite were investigated and compared with each other.

2. EXPERIMENT

Some researchers reported the electrochemical preparation and microwave absorption properties of magnetic carbon fibers coated with Fe₃O₄ films [13]. However, the procedure of electrochemical deposition is relatively tedious and the orientation of Fe₃O₄ electrochemical growth is very strong, which easily lead to the generation of large particles. Besides, Fe₃O₄ particles generated electrochemically are difficult to evenly disperse around the carbon nanofibers. Chemical co-precipitation preparation reported here is facile and scalable. CNFs (0.5 g) treated with fenton reagent were dispersed in 100 ml deionized water, then FeSO₄ (0.8 g) and FeCl₃ (0.7 g) were added to the above suspension, respectively. Subsequently, the suspension was transferred to the water bath and maintained at 90 °C, then NH₃·H₂O (30 ml) was added to the mixture solution and kept 30 min. Finally, the fresh CNFs/Fe₃O₄ composite was washed with deionized water and dried at 60 °C for 12 h. To test the microwave absorption property, bare CNFs (20wt%) and Fe₃O₄/CNFs composite (20wt%) were uniformly mixed with paraffin (80wt%) and pressed into wafer with the thickness of 2 mm.

The crystalline structure of the samples was detected by X-ray diffractometer (XRD, Rigaku TTR III) using Cu K α radiation ($\lambda=1.5406 \text{ \AA}$). The morphology of the composite was examined by scanning electron microscopy (SEM, FEI QUANTA 200). The relative complex permittivity and permeability of the composites were tested in the frequency range of 1~18 GHz by an HP8722ES vector network analyzer.

3. RESULTS AND DISCUSSION

Fig.1a shows the XRD patterns for bare CNFs. Fig.1b gives the XRD patterns of the sample, which coincides well with the standard date of Fe₃O₄(magnetite, JCPDS 10-716337). And the diffraction peak appearing at $2\theta=25$ degree is due to the presence of CNFs in the composite. The

strong and sharp Fe_3O_4 diffraction peaks at XRD patterns confirmed the formation of well-crystallized Fe_3O_4 . The average crystal size was estimated to be about 10 nm by scherrer formula for the generated Fe_3O_4 nanoparticles.

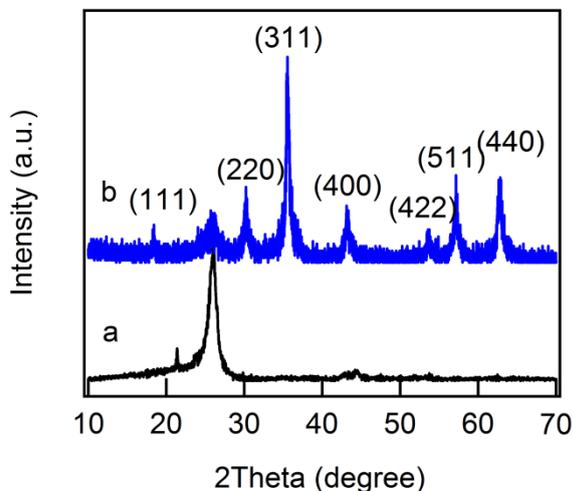


Figure 1. XRD patterns of (a) bare CNFs and (b) $\text{Fe}_3\text{O}_4/\text{CNFs}$ prepared by chemical co-deposition

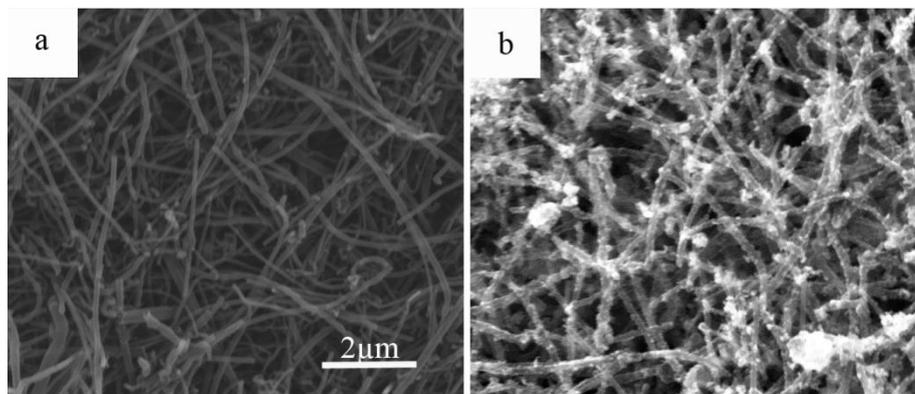


Figure 2. SEM images of (a) bare CNFs and (b) $\text{Fe}_3\text{O}_4/\text{CNFs}$ prepared by chemical co-deposition

The surface morphologies of bare CNFs and $\text{Fe}_3\text{O}_4/\text{CNFs}$ composite were shown in Fig.2. Fig. 2a presents the morphology and structure of the CNFs, in which CNFs have smooth surface with diameters ranging from 50 nm to 200 nm. Fe_3O_4 deposited on the CNFs has been investigated by SEM as shown in Fig.2b. We could see that the Fe_3O_4 nanoparticles anchored on the surface of CNFs uniformly.

For researching the microwave absorption property, the relative complex permittivity and permeability of bare CNFs and $\text{Fe}_3\text{O}_4/\text{CNFs}$ composite were measured at the range from 1 GHz to 18 GHz. With the increase of the frequency, the real part of complex permittivity keeps constant below 4 GHz and then decreases until 15 GHz as shown in Fig.3a. And the imaginary part decreases to 100

GHz. The real part of complex permeability decreases to about 0.45 at 5 GHz and then increases slightly. The imaginary part increases slightly and then decreases to about 0.1. The dielectric loss and magnetic loss factors are presented in Fig.3c. The magnetic loss factor is close to 0, it indicates that the main attribution to the dielectric loss is the reflection loss (RL) of CNFs.

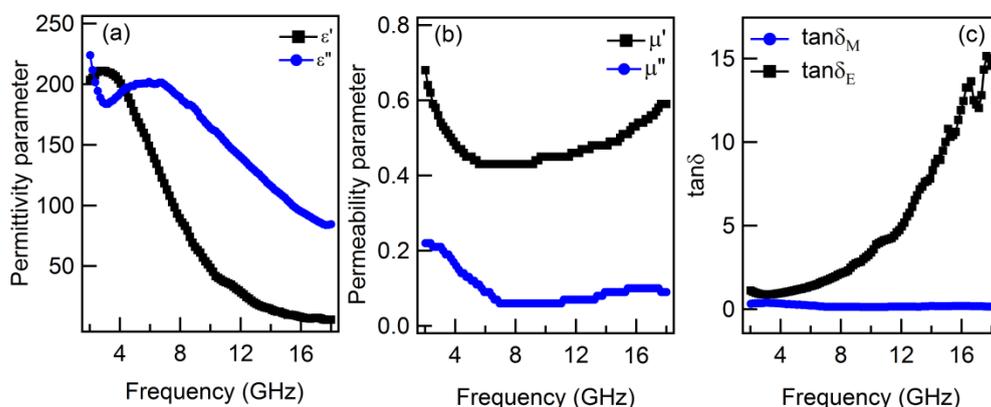


Figure 3. Measurements of electromagnetic parameters for blank CNFs/paraffin(20wt%): (a) Permittivity spectra, (b) permeability spectra, (c) loss factor.

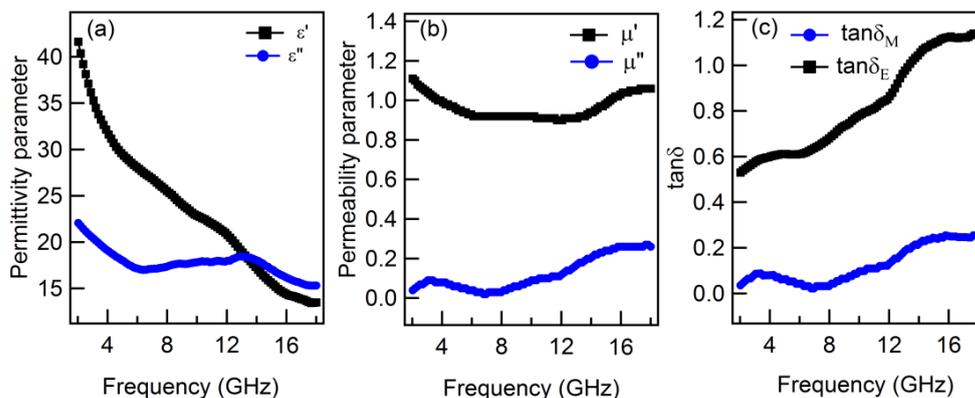


Figure 4. Measurements of electromagnetic parameters for Fe₃O₄- CNFs/paraffin(20wt%): (a) Permittivity spectra, (b) permeability spectra, (c) loss factor.

In the Fig.4, the fact $\tan \delta_E$ is higher than $\tan \delta_M$ suggests that the RL of Fe₃O₄/CNFs composite is also due to the dielectric loss. Comparing the Fig.3c and 4c, it is obvious that $\tan \delta_M$ of Fe₃O₄/CNFs composite is much larger than that of bare CNFs. Thus, Fe₃O₄/CNFs composite will exhibit better microwave absorption performance.

Fig.5 shows the plots of microwave RL for bare CNFs and Fe₃O₄/CNFs composite. The results confirm that bare CNFs and Fe₃O₄/CNFs composite have different absorption properties. Bare CNFs almost exhibit no absorption property because its RL is -1.5 dB at 2.5 GHz. By contrast, the absorption peak of Fe₃O₄/CNFs composite material shifts to the higher frequency at 7 GHz. Meanwhile, the

frequency bandwidth of 6 GHz and the RL values of -11 dB have been achieved. It indicates that this composite material is more suitable as EM wave absorber. The reflection loss of Fe₃O₄/carbon composite material is less than -10 dB [18]. Various methods are proposed to prepare the different structural composites and adjust the thickness of the absorber to achieve the better absorbing performance. Many of the preparation methods are tedious and involve in severe experimental conditions as well as sophisticated equipments [19, 20]. In comparison, chemical co-precipitation method has the advantages of easy operation and mass production. Furthermore, Fe₃O₄ nanoparticles obtained by chemical co-precipitation can be well dispersed on the surface of carbon nanofibers. We can also achieve better microwave absorbing performance through controlling the amount of Fe₃O₄ in the composite material.

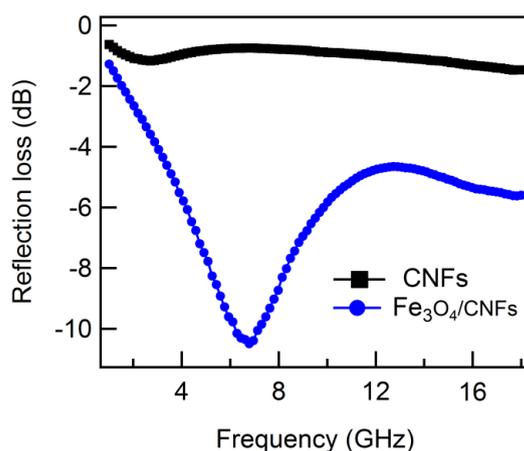


Figure 5. Microwave absorbing characteristics of both samples.

4. CONCLUSIONS

In this study, CNFs/Fe₃O₄ composite was synthesized by chemical co-precipitation method. A large number of function groups were introduced to the surface of CNFs through the treatment of fenten reagent, which made Fe₃O₄ coating grown on the CNFs more uniform. The fabricated Fe₃O₄/CNFs composite shows lower RL and broader absorption frequency domain compared with bare CNFs. For optimizing the microwave absorption property of Fe₃O₄/CNFs nanocomposite, we could control the quantity of coated Fe₃O₄ or adjust the thickness of product/paraffin composite. Such kind of Fe₃O₄/CNFs composite, which is light-weighted and flexible, is expected to become a promising microwave absorbing material in the future.

ACKNOWLEDGMENTS

We greatly appreciate the financial support for our work from the National Nature Science Foundation of China (grant No. 61166006, 61366002 and U1137602), the Program for Overseas High-level talents of Yunnan Province and the Introduction Program of high-end scientific and technological talents in Yunnan Province (Grant No. 2013HA019). The Project is also funded by the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry.

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