



Evolution of double magnetic resonance behavior and electromagnetic properties of flake carbonyl iron and multi-walled carbon nanotubes filled epoxy-silicone



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ABSTRACT

The complex permittivity and permeability, reflection loss of the flake carbonyl iron (FCI) particles and multi-walled carbon nanotubes (MWCNTs) filled epoxy-silicone resin were investigated in the frequency range of 2–18 GHz. Both the values of complex permittivity and permeability were enhanced with increasing FCI content. The magnetic resonance behavior of such composites was mainly depended on the content of FCI particles, and the μ'' - f spectra changed from no visible peak to double resonance as the FCI content increased from 10 to 50 wt%. Single-layer and multi-layer microwave absorbing coatings were also designed based on the transmission line theory and electromagnetic properties of the absorber. By changing the filler content and/or optimizing the absorber structure, the measured reflection loss below –10 dB can be obtained in the frequency range of 2–16.9 GHz for FCI particles and MWCNTs filled four-layer absorber.

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1. Introduction

Electromagnetic (EM) waves absorbing materials in the frequency range of 2–18 GHz are important for both commercial and military applications, for example, protect the electronic devices and components, reduce the RCS for stealth systems [1,2]. It is also well known that the absorption of EM waves was worked by dissipating the EM energy into heat via magnetic and/or dielectric losses. Commonly, the effectiveness of the EM wave absorbers were mainly depended on the EM properties of the absorber (such as the complex permittivity and permeability). Therefore, the control of the EM properties of absorbers is the key point for design the EM wave absorbers, and which can be satisfied by incorporating different dielectric and/or magnetic loss fillers into an insulating binder or matrix [3–5].

Carbon nanotubes (CNTs) have been received considerable interest for EM absorption composites due to their high dielectric loss, unique mechanical and excellent electrical properties [6]. Most of the prior works have been concerned with the EM properties and microwave absorption of the CNTs filled composites by changing the structure and content of the CNTs [7–9]. Recent studies have shown that carbonyl iron (CI) particles filled composites possess high real (μ') and imaginary (μ'') part of complex permeability in the frequency range of 2–18 GHz, indicating that such composites could be used as the EM waves absorbing material

[10–13]. Recently, EM wave absorbing materials fabricated by multi-walled carbon nanotubes (MWCNTs)/magnetic particles composites have been intensively investigated [14–16]. For the both conductive MWCNTs and magnetic particles filled composites, the EM properties of such composites were depended on the fillers (MWCNTs and/or magnetic particles) content as well as the boundary conditions of the MWCNTs/magnetic particles/polymer interfaces. The reflection loss (RL) results also showed that such composites with both dielectric loss (owning to MWCNTs) and magnetic loss (owning to magnetic particles) were suitable to fabricated EM waves absorbing materials.

In our previous publication [13], two peaks were found in the μ'' - f spectra of the composites containing MWCNTs and flake carbonyl iron (FCI) particles. Such result was mainly resulted from the interactions between the MWCNTs and FCI particles, and the mechanism of double magnetic resonance behavior is still need to further investigate. Furthermore, very few data have been published on the unusual double magnetic resonance behavior of the composites filled with MWCNTs and FCI particles. Therefore, the present investigation was undertaken to studying the EM property of the absorber under a fixed amount of MWCNTs (as dielectric absorber) by changing the FCI content (as magnetic absorber). The evolution of double magnetic resonance behavior was also investigated in details. In order to further improve the RL of MWCNTs and FCI particles filled composites, multi-layer microwave absorbing coatings were also designed and measured by varying the filler content and the thickness of each layer.

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2. Materials and methods

2.1. Materials and fabrication of samples

The epoxy-silicone resin used as matrix and the polyamide resin used as curing agent were supplied by XI AN Leao Technological Co. Ltd., Shaanxi province, China. The MWCNTs used as dielectric absorber were supplied by Shenzhen Nanotech port Co. Ltd., China. The MWCNTs with a length of 2–5 μm , a diameter of 60–100 nm and the purity is 95%. The FCI particles used in this study was fabricated by the decomposition of $\text{Fe}(\text{CO})_5$ and purchased from Xinghua chemical Co. Ltd., Shaanxi province, China. The main characteristics of the FCI particles are: the content of α -iron >99.5 wt%, thin flakes of 1–5 μm in diameter and below 1 μm in thickness, and polycrystalline microstructure. In order to prevent entanglement of the MWCNTs, sodium dodecyl benzene sulfonate (SDS) (Sinopharm Chemical Reagent Co., Ltd. China) with a content of 0.5 wt% was employed as a surfactant for all of the composites to obtain better dispersion of the MWCNTs.

The specimens of C2CI0, C2CI1, C2CI2, C2CI3, C2CI4 and C2CI5 contained a fixed concentration of 2 wt% MWCNTs, and 0, 10, 20, 30, 40 and 50 wt% FCI particles, respectively. These specimens allowed us to investigate the change of EM property resulting from the variation of the content of FCI particles under a fixed amount of MWCNTs. First, a fixed content of 2 wt% MWCNTs and 0.5 wt% SDS were dispersed in ethanol by an ultrasonic bath at room temperature for 1 h. Then, FCI particles with different content were added into the ethanol-based solution. After that, the suspensions were stirred at 2000 rpm for 10 min. Then, the mixtures were placed in an oven at 80 $^{\circ}\text{C}$ to evaporate the ethanol completely. After adding the epoxy-silicone and polyamide resin, the mixtures were stirred at 2000 rpm for 10 min. The curing process of the hybrid mixtures was precured at 90 $^{\circ}\text{C}$ for 30 min and the postcured at 120 $^{\circ}\text{C}$ for 2 h.

2.2. Characterization of samples

The dispersion of both MWCNTs and FCI particles in the matrix was observed using scanning electron microscopy (SEM) (Model JSM-6360, JEOL, Tokyo, Japan). The complex permittivity $\epsilon(f)$ and permeability $\mu(f)$ of the composites were measured using the T/R coaxial line method in the frequency range of 2–18 GHz by a network analyzer (Agilent technologies E8362B: 10 MHz–20 GHz). The testing specimens had a cylindrical toroidal specimen: outer diameter of 7.0 mm and inner diameter of 3.03 mm. The RL of the samples was measured using a network analyzer (Agilent Technologies E8362B) by comparing the signals transmitted by the samples and those reflected from its input. The antennas used for the measurements were ridged wideband horns covering the microwave region of 2–18 GHz. The sample with desired thickness for RL measurement was mounted on an aluminum substrate (microwave reflector) with the same size (180 mm \times 180 mm).

3. Results and discussion

3.1. The microstructures of FCI particles and MWCNTs filled composites

Fig. 1 shows the typical SEM images of the fractured surface of the composites contained FCI particles and MWCNTs. As seen from Fig. 1b, the MWCNTs and FCI particles were dispersed uniformly in the matrix and the FCI particles were connected and/or surrounded with MWCNTs. It can be concluded that the exposure of the MWCNTs in ethanol solution with ultrasonic treatment and then subsequent intense stirring process of the mixture could decrease the van der Waals force as well as the hydrogen bonding between the MWCNTs [13], leading to a uniform dispersion of the FCI particles and MWCNTs.

3.2. The complex permittivity and complex permeability of the absorber

Fig. 2 shows the complex permittivity of the FCI particles and MWCNTs filled composites. For the absorber filled only with MWCNTs, the average values of real (ϵ') and imaginary (ϵ'') part of complex permittivity were 5 and 0.5 in the measured frequency range, respectively. Both the ϵ' and ϵ'' increased in the whole measured frequency range as the FCI particles content increased from 10 to 50 wt%. The ϵ' of the composites filled with higher FCI particles content decreased with increasing frequency and showed frequency dependence in the whole frequency range [17,18]. The ϵ'' showed some fluctuation in the measured frequency range,

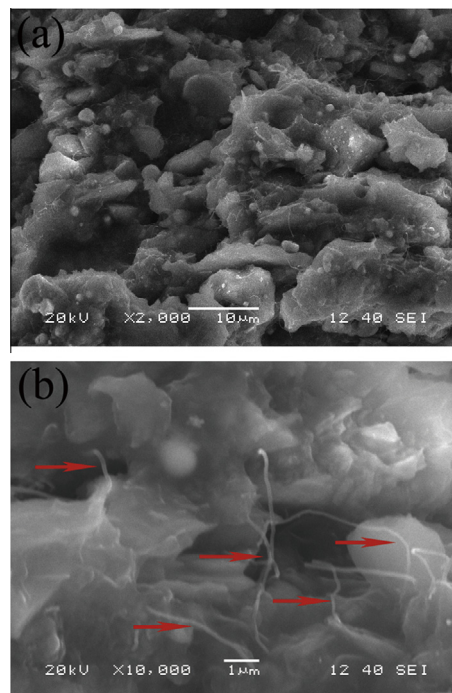


Fig. 1. Typical SEM images of the fractured surface for CI particles and MWCNTs filled epoxy-silicone composites (the red arrows indicate the MWCNTs). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

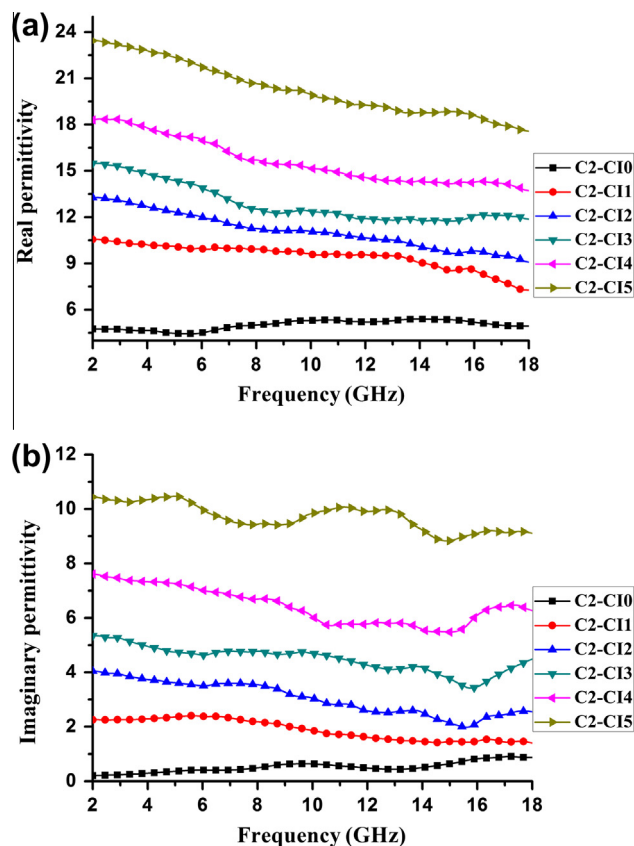


Fig. 2. (a) Real part and (b) imaginary part of complex permittivity spectra of the epoxy-silicone resin filled with FCI particles and MWCNTs in the frequency range of 2–18 GHz.

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