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# Microwave absorbing properties of flake-shaped carbonyl iron/reduced graphene oxide/epoxy composites



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#### ABSTRACT

Microwave absorbers are used in a wide range of fields such as radar-cross section (RCS) reduction for stealth aircrafts, electromagnetic interference (EMI) reduction for electronic products and health protection for human. In this study, the flake-shaped carbonyl iron (FCI) powder was obtained from raw spherical carbonyl iron (SCI) powder by ball milling for 60 h. The SCI powder was mixed with reduced graphene oxide (rGO) and then ball-milled for 60 h to obtain rGO/FCI composite particles. Flake-shaped rGO/FCI particles have length of 1–20  $\mu$ m, thickness of 0.3–1  $\mu$ m, and aspect ratio of 1–60. The electromagnetic properties ( $\varepsilon'$ ,  $\varepsilon''$ ,  $\mu'$ ,  $\mu''$ ) of rGO/FCI/epoxy composite absorbers were measured by the transmission/reflection method in the frequency range of 2–18 GHz for three samples with 50 wt% of SCI, FCI and rGO/FCI particles, respectively. The results show that the values of permittivity and permeability both increase after ball-milling treatment. Compared with FCI/epoxy composite, the rGO/FCI/epoxy composite absorber exhibits higher dielectric loss and lower reflection loss. For absorbers with the same thickness, the rGO/FCI/epoxy composite shows better reflection loss with the target frequency shifting to the lower frequency region. For 50 wt% of rGO/FCI/epoxy absorber with a thickness of 2 mm, the minimum reflection loss reaches –32.3 dB at matching frequency of 11.0 GHz.

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#### 1. Introduction

The electromagnetic pollution as a result of electromagnetic radiation has raised a serious concern for human health and environmental safety. Hence, to develop microwave absorbers or radar absorbing materials (RAM) that can reduce the reflection or transmission of electromagnetic radiation has important implications not only for the protection of human health but for the elimination of electromagnetic interference of high-speed wireless electronic devices. RAM can significantly reduce the radar cross section of the targets in specific radar frequencies and can be used in the area of stealth technology [1]. Carbonyl iron (CI) particles possessing relatively low electrical conductivity, low coercivity, high Curie temperature, and high specific saturation magnetization intensity ( $4\pi$  Ms) can be used as microwave absorbing fillers which are embedded in an insulating polymer matrix to become microwave composite absorbers for higher frequencies ranging

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http://dx.doi.org/10.1016/j.materresbull.2017.01.045 0025-5408/© 2017 Elsevier Ltd. All rights reserved. from 2 to 18 GHz [2]. However, these isotropic magnetic materials have small values of permeability in gigahertz region due to Snoek's limit as a result of their low eddy current loss originating from the particle shape effects. To increase the values of complex permeability of soft magnetic particles, one can use the ball-milling technique to obtain flake-shaped anisotropic magnetic particles [3,4].

Graphene comprises of a single layer of carbon atoms which are compactly bonded in a two-dimensional hexagonal lattice. The unique properties of graphene such as lightweight, flexibility, high mechanical strength, high thermal conductivity, high specific surface area, high corrosion-resistance, high temperature-resistance and extraordinary electric conductivity [5] have received much attention as a promising material for liquid crystal devices, super capacitor [6], paper-like material and microwave absorbers [7]. In this study, we have prepared chemically reduced graphene oxide (rGO) due to its scalability, high yield, and the easiness of chemical functionalization.

The planar anisotropy obtained by material processing and combining CI as magnetic absorbent fillers with rGO as an electrically conductive material can produce microwave composite absorbers with both high magnetic and dielectric losses which are crucial and tunable by changing the ratio of rGO to CI in the composites. In the present study, for the development of thinner and better microwave absorbers in GHz range, raw spherical carbonyl iron (SCI) powder was mixed with rGO and then ball-milled for 60 h to obtain rGO and flake-shaped CI composite particles which were then added to an epoxy resin to prepare composite samples with different composition of fillers. A vector network analyzer and the coaxial waveguide method are used to characterize the complex permittivity and permeability of the composite absorbers at the frequency range of 2–18 GHz.

#### 2. Experimental procedure

The flake-shaped carbonyl iron (FCI) powder was obtained from raw spherical carbonyl iron (SCI) powder (Micropowder Iron S-3700, ISP, DE, USA) by ball milling for 60 h. The raw SCI powder was mixed with rGO and then ball-milled for 60 h to obtain rGO/FCI composite particles. The morphology of SCI with particle size of 2–  $5\,\mu\text{m}$  and the achieved FCI were characterized by scanning electron microscopy (SEM) as shown in Fig. 1. The thickness of the rGO/FCI particles was ~0.3-1  $\mu$ m with a diameter of ~1-20  $\mu$ m.

The graphene oxides (GO) were synthesized by the modified Hummers' method [8]. The as-fabricated GO were then put into a nitrogen-filled quartz tube furnace for thermal annealing at 800 °C for 1 h in order to remove oxygen functional groups. A representative SEM image of rGO powder in Fig. 1a exhibits that the sheets are highly agglomerated powder with a "fluffy" structure. The dimensions of rGO sheets are from submicron to several micrometers. The XRD patterns of rGO, SCI, FCI, and rGO/FCI powders are presented in Fig. 2. It is observed that the intensity of CI peaks decreases with a slightly shifting position compared to that of rGO/FCI which means that the wrapping of rGO decreases the crystallinity of FCI and also indicates good interfacial interaction between the components of the composites of rGO/FCI. No graphitic carbon was observed in the XRD patterns. This can be attributed to the strong signals of the iron oxides which tend to



Fig. 1. SEM micrographs of (a) rGO, (b) SCI, (c) FCI by ball milling for 60 h, (d) rGO/FCI by ball milling for 60 h, EDX of (e) SCI, and (f) rGO/FCI.

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