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Microwave characteristics of low density flaky magnetic particles

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ABSTRACT

Diatomite coated with thin Fe films were obtained by the Chemical Vapor Deposition process. The resultant Fe-coated flaky diatomite particles had low densities (2.7–4.0 g/cm^3) and high saturation magnetization (93–157 emu/g). Annealing treatment led to grain growth and an increased saturation magnetization. The high frequency properties of the composites consisting of Fe-coated flaky diatomite particles and wax were investigated. The permittivity and permeability increased with increasing flaky magnetic particles content in the composite and increasing the Fe weight percentage of the particles. The reflection loss of the composite was found dependent on the absorber material thickness, wax:flaky magnetic particles ratios, the Fe content, as well as the annealing treatment. At a thickness of 1 mm, the composite records a minimum reflection loss of −18 dB at 6 GHz.

1. Introduction

With rapid development of wireless communication technology, in order to reduce the threat to the environment, human health, and military security caused by electromagnetic wave (EM-wave), the electromagnetic absorbing material is emerged [1,2]. Fine magnetic materials, such as powders of iron [3] and ferrites [4], embedded in an insulating matrix are widely used as microwave absorbers. However, the conventional absorptive materials have difficulties in increasing the permeability in gigahertz region because of Snoek's limit for ferrites [5] or eddy current loss for magnetic metals [6]. Also, those materials are quite heavy, which restricts their usefulness in applications requiring light weight mass [7,8]. The use of metal thin films coated on microspheres or micro-organisms of low density may be one of the ways to overcome these problems [9].

The flake is considered to be one of the best shapes of EM-wave absorbing particles, which would improve their electromagnetic parameters [10] and increase the strength of EM-wave absorbing materials. Currently, the flake electromagnetic wave absorbing particles are generally obtained by milling. However, no study has ever been carried out on the fabrication of disk flake shape iron. The diatomite mineral is formed by *Coscinodiscus* sp., which fixes and concentrates silicon [11]. The main chemical composition of flaky diatomite particles is SiO_2. It is a good base material for synthesizing the EM-wave absorbing materials for its low density, chemical stability, disk flake shape and rich pore-structure.

In the present investigation, we propose Fe metal thin film as the coating layer on the flaky diatomite particles and we have conducted a preliminary work on the processing and properties of these Fe-coated flaky diatomite particles. The microstructure, static magnetic properties, and dynamic electric and magnetic properties of Fe-coated flaky diatomite particles of low density have been reported in this paper.

2. Experimental section

2.1. Materials

Commercially available diatomite C292 were obtained from Celite Corporation. The flaky diatomite particles are mainly composed of SiO_2 (70–90%), with a diameter range of 20–60 μm, thickness range of 2–10 μm and bulk density of 0.4–0.9 g/cm^3.

2.2. Preparation of flaky magnetic particles

Coating the flaky diatomite was conducted by the Chemical Vapor Deposition (CVD) process. The preparation was started by adding diatomite to the reactor with N_2 atmosphere, followed by heating the reactor to 300 °C and keeping the temperature constant. Then the diatomite was stirred at 100 r/min, and $Fe(CO)_5$ steam was added into the reactor until the reaction is completed.

After completion of the reaction, cool down the sample in the reactor to room temperature, and remove it for use.

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Put some sample into the muffle furnace with N_2 atmosphere, and annealed it at different temperatures (550 °C, 700 °C) for 1 h. After that, cool down the sample in the furnace to room temperature, and remove it for use.

2.3. Characterization

The phase structure analysis of products was identified (within 2θ range of 20–85°) using a X-ray diffractometer (XRD, Rigaku D/max-3) utilizing Cu-Kα X-radiation of wavelength 1.5418 Å. A scanning electron microscopy (SEM, CS3400) equipped with an energy dispersion X-ray spectroscopy (EDS, INCA X-sight 7421) was used to analyze the surface morphology and the size distribution of the flaky particles, while EDS analysis was done to clarify their chemical makeup. Their magnetic properties were then evaluated on a vibrating sample magnetometer (VSM, JDM-13), and the field reached up to 1.5×10^4 Oe. A vector network analyzer was used to measure the microwave parameters of the samples over the frequency range of 2–18 GHz. The cylindrical toroidal measurement samples were composed of the developed ferrite flakes randomly dispersed in wax with various weight ratios (40 wt%, 50 wt%, and 55 wt%). The sample had an inner diameter of 3 mm, an outer diameter of 7 mm and a thickness of 2 mm. Microwave absorption properties were evaluated by the reflection loss (RL), which was derived from the following formulae [12]: where Z_{in} is the input impedance of absorber, Z_0 is the impedance of air and c the velocity of light.

$$RL = 20\log\left|\frac{(Z_{in}-Z_0)}{(Z_{in}+Z_0)}\right| \quad (1)$$

$$Z_{in} = Z_0\sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left\{j\frac{2\pi f d}{c}\sqrt{\mu_r\varepsilon_r}\right\} \quad (2)$$

3. Results and discussion

Fig. 1 shows the SEM images of flaky diatomite particles and Fe-coated flaky diatomite particles by the CVD process in various coating time (1 h, 2 h, and 3 h). Fig. 1(a) shows the appearance of flaky diatomite particles without coating, whose surfaces are very smooth. It could obviously be seen that the diatomite possesses a porous arrays structure and a disk flake shape. Fig. 1(b)–(d) shows the appearance of Fe-coated flaky diatomite particles, demonstrating that all of the flakes are coated with Fe nanoparticles. The surfaces of flakes are very coarse, and their porous arrays disappear. It indicates that Fe nanoparticles continuously deposit on the surfaces of flakes. It was found that with the reaction time increases, the Fe film thickness and grain size becomes larger and larger. In current investigation, we could coat the flaky diatomite particles with various Fe film thicknesses by controlling the coating time.

The EDS patterns of flaky diatomite particles and flaky diatomite particles with Fe coating are presented in Fig. 2. It is clear that the flaky diatomite particles consist of Si and O elements (Fig. 2(a)). As to flaky diatomite particles with Fe coating (Fig. 2(b)), elements Fe emerge apart from the elements of flake diatomite particles (Si and O elements) due to Fe nanoparticles coating flaky diatomite particles. The X-ray diffraction (XRD) pattern for as-plated Fe-coated flaky diatomite particles (plated for 1 h) is shown as curve (a) in Fig. 3, indicating an amorphous or nanocrystalline structure. The strong background intensity and unclear peak in the XRD spectrum result from the amorphous substrate (flaky diatomite particles) due to the rather thin coating layer. To investigate the effect of crystallization on the properties, part of as-plated diatomite particles was annealed in the muffle furnace with N_2 atmosphere at different temperatures (550 °C, 700 °C) for 1 h. Fig. 3(b) and (c) shows the XRD patterns of annealed flaky particles at 550 °C and 700 °C, respectively. From Fig. 3(a) well crystallized structure has been demonstrated.

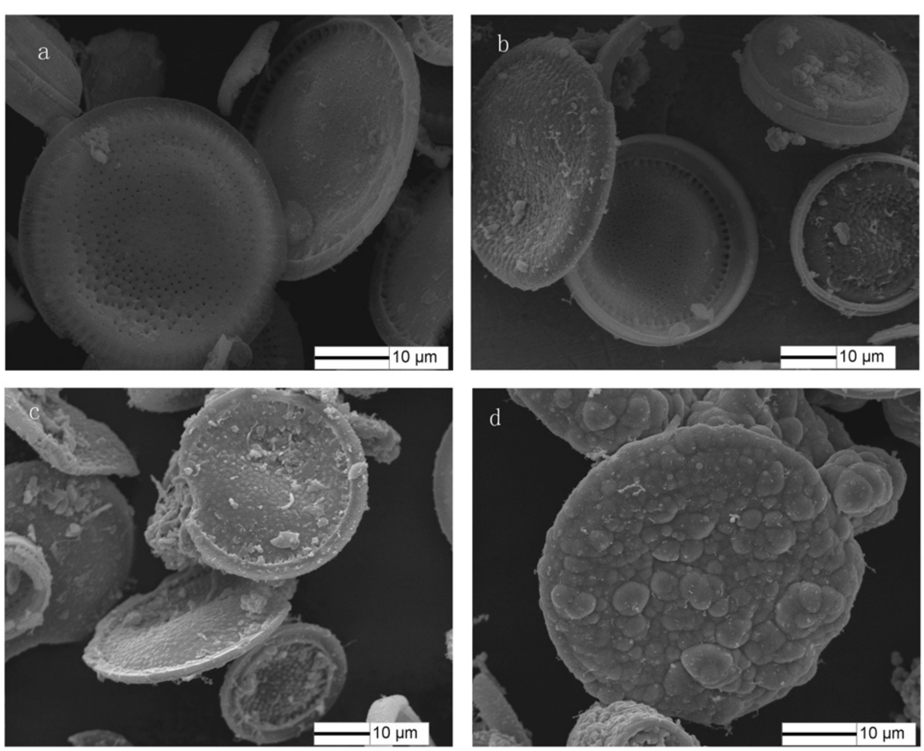

Fig. 1. SEM images of: (a) flaky diatomite particles, (b) Fe-coated flaky particles (plated for 1 h), (c) Fe-coated flaky particles (plated for 2 h), and (d) Fe-coated flaky particles (plated for 3 h).