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Research articles

Enhanced antioxidation and microwave absorbing properties of SiO_2-coated flaky carbonyl iron particles

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ABSTRACT

SiO_2 was successfully coated on the surface of flaky carbonyl iron particles using a chemical bath deposition method in the presence of 3-aminopropyl triethoxysilane (APTES). The morphologies, composition, valence states of elements, as well as antioxidation and electromagnetic properties of the samples were characterized by scanning electron microscope (SEM), energy dispersive spectrometer (EDS), X-ray photoelectron spectroscopy (XPS), thermogravimetric (TG) and microwave network analyzer. TG curve shows the obvious weight gain of carbonyl iron was deferred to 360 °C after SiO_2-coated, which can be ascribed to the exits of SiO_2 overlayer. Compared with the raw carbonyl iron, SiO_2-coated sample shows good wave absorption performance due to its impedance matching. The electromagnetic properties of raw and SiO_2-coated carbonyl iron particles were characterized in X band before and after heat treatment at 250 °C for 10 h. It was established that SiO_2-coated carbonyl iron demonstrate good thermal stability, indicating SiO_2-coating is useful in the usage of microwave absorbers operating at temperature up to 250 °C.

1. Introduction

With the rapid development of wireless communications and high-frequency circuit devices in the gigahertz range, electromagnetic interference (EMI) problems have attracted increasing attention recently. In order to provide solution to EMI and microwave absorption, the microwave absorption materials (MAMs) are becoming very important, which have attracted much attention of many scientists [1–3]. Among all MAMs, metallic magnetic materials are especially focused on because their permeability remains high in the gigahertz range due to high saturation magnetization [4].

Carbonyl iron, as well as ferrites, has been extensively studied for a long time as magnetic components of polymeric composites for the application of electromagnetic wave absorbers [5]. However, ferrites cannot be used in higher frequency range due to their Snoke's limit, so the electromagnetic wave absorbers filled with ferrites can only play a good role in a narrow band. In contrast, carbonyl iron has larger values of saturation magnetization and its Snoke's limit is located at a higher frequency [6]. So carbonyl iron is more suitable to be applied in a broad frequency range.

For carbonyl iron particles, the oxidation-prone property usually limits their usages at a higher temperature, especially for the particles with flaky shape [7]. A good way to conquer the limitations is to encapsulate them with other materials, which could effectively protect them from contacting with oxygen. Therefore, various iron based nanocomposites with core-shell structures were fabricated, such as Al/iron [8], PANI (polyaniline)/carbonyl iron [9], Ni/carbonyl iron [10] and Co/carbonyl iron [11]. However, iron particles in these composites were mostly spherical, and particles other than spherical were seldom seen. Generally, nonspherical shaped grains have higher levels of permittivity due to the enhancement of polarization [12,13]. It was reported that the flake-shaped iron particles had large values of microwave permeability due to the advantages of higher saturation magnetization and low eddy current loss coming from the particles shape effect [14]. On the other hand, few works were devoted to the variation of electromagnetic properties/microwave absorption after heat treatment.

Among the various materials for candidate coatings of carbonyl iron, reasons of easy control of the deposition process, good adiabatic performance and optical transparency, make silica an ideal, low-cost material to tailor the surface properties of carbonyl iron,

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while basically maintaining the physical integrity of the underlying core [15]. At present, there have been many researchers using tetraethoxysilane (TEOS) as silicon source, however, the preparation of SiO_2 layer is commonly villous [16], owing to the exclusion of strong polar groups, such as –OH, $–NH_2$, –COOH. In this work, TEOS was substituted by 3-aminopropyl triethoxysilane (APTES) to forming a dense SiO_2 layer.

In this manuscript, we described a simple solution method for the preparation of SiO_2 coated carbonyl iron particles. The synthetic procedures, morphological details, thermal stability and electromagnetic properties of the SiO_2-coated carbonyl iron were discussed in detail, showing the improved oxidation resistance and microwave absorption performance.

2. Experimental and characterization

2.1. Preparation

The flaky-shaped carbonyl iron powder, purchased from Xinghua chemical Co. Ltd, Shanxi province, China, was used as the raw material. SiO_2-coated carbonyl iron particles were prepared by a chemical bath deposition method. Firstly, the raw carbonyl iron particles (1 g) were added into deionized water (100 ml). After sonicating in an ultrasonic washer for 10 min, a mixture solution of anhydrous ethanol (100 ml) and 3-aminopropyl triethoxysilane (APTES) (10 ml) were added to the solution. Secondly, the reaction solution was mechanical stirred (340 r/min) at 80 °C for 2 h. At last, the product was washed three times with anhydrous ethanol and dried in a vacuum drying oven at 50 °C.

For convenience, the raw carbonyl iron particles and SiO_2-coated carbonyl iron particles were denoted as Sample A and Sample B, respectively.

2.2. Characterization

The size-distribution, morphology and surface composition were characterized by scanning electron microscope (SEM; JEOL JSM-5800 LV SKANNING) attached with a Links Systems energy dispersive spectrometer (EDS). XPS analyses were performed using KRATOS Axis Ultra (Kratos Analytical, Shimadzu, Japan). The phase compositions were identified by a Cu Ka radiation (XPert Diffractometer, Philips, Netherlands). Magnetic measurement was conducted at room temperature using a vibrating sample magnetometer (VSM, Riken Denshi, BHV-525). In addition, the antioxidation property of the samples was characterized using a thermal analyzer (Netzsch STA 449 TG-DTA/DSC) in air with the heating rate 10 °C/min up to 800 °C.

For measurement of the microwave properties, the samples were dispersed in paraffin homogeneously with a sample-to-paraffin weight ratio of 7:3. The effective complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and permeability ($\mu_r = \mu' - j\mu''$) of the samples were measured using a network analyzer (Agilent technologies E8362B: 10 MHz–20 GHz). The dimension of the samples for electromagnetic measurement was 22.86 mm (length) × 10.16 mm (width) × 2 mm (thickness), which were based on the measurements of the reflection and transmission module between 8.2 and 12.4 GHz in the fundamental wave-guide method.

3. Results and discussion

3.1. Morphologies and XPS analysis

Fig. 1 shows the SEM and EDS of Sample A and Sample B. As seen from Fig. 1(a), the Sample A has thin flakes with diameters ranging in 1–8 μm and thickness below 1 μm. In addition, there

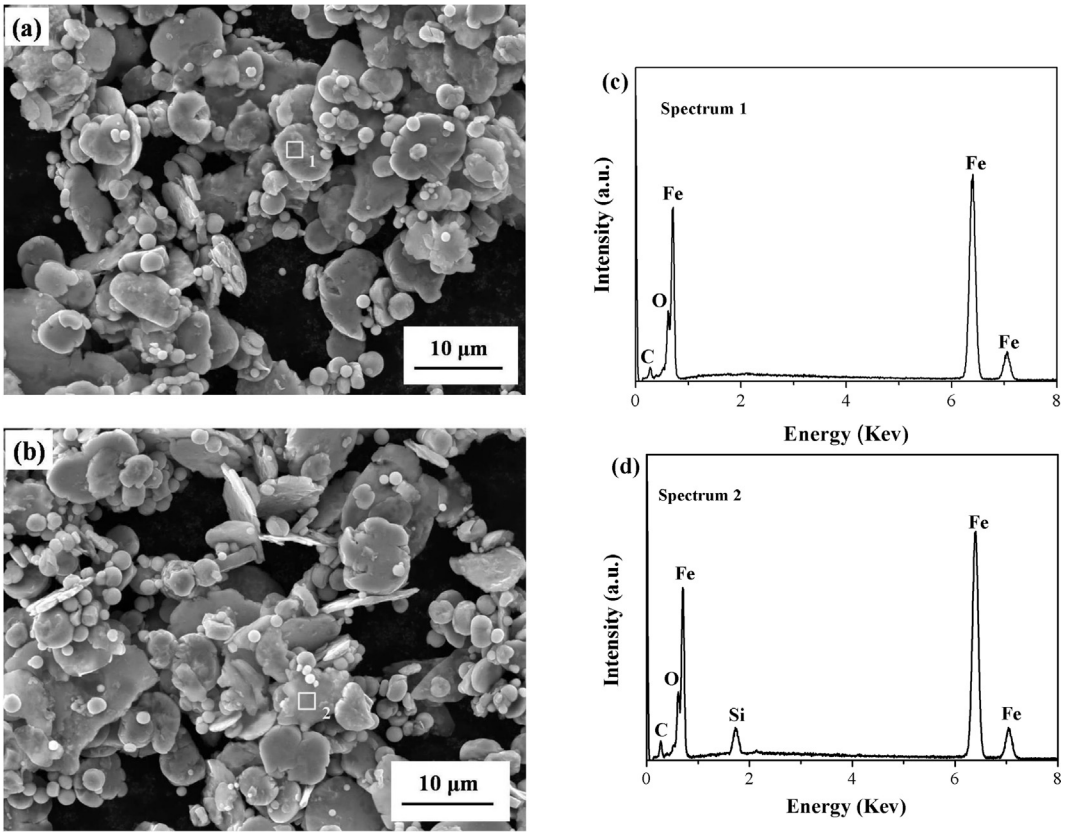

Fig. 1. SEM images of: (a) raw carbonyl iron powders; (b) SiO_2-coated carbonyl iron powders and EDS analysis of: (c) spectrum 1; (d) spectrum 2.