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Electromagnetic property of SiO_2-coated carbonyl iron/polyimide composites as heat resistant microwave absorbing materials

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ABSTRACT

Heat resistant microwave absorbing materials were prepared by compression molding method, using polyimide resin as matrix and SiO_2 coated carbonyl iron (CI) as filler. The SiO_2 coated CI particles were prepared by Stober process. The microwave absorbing properties and the effect of heat treatment on the electromagnetic properties of SiO_2 coated CI/polyimide composites were investigated. When the content of SiO_2 coated CI is 60 wt%, the value of minimum reflection loss decreases from −25 dB to −33 dB with the thickness increases from 1.5 mm to 2.1 mm. According to the thermal-gravimetric analyses (TGA) curves, the polyimide matrix can be used at 300 °C for long time. The complex permittivity of the composites slightly increases while the complex permeability almost keeps constant after heat treatment at 300 °C for 10 h, which indicating that the composites can be used at elevated temperature as microwave absorbing materials at the same time have good heat resistance and microwave absorption.
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1. Introduction

With the fast advancement of wireless communication, the absorbers of electromagnetic waves are becoming increasingly important in both civil and military applications [1,2]. Typical microwave absorption materials are composed of resin matrix and magnetic absorbents. Carbonyl iron (CI) as a kind of conventional magnetic fillers with the advantages of high saturation magnetization, high Curie temperature and large value of magnetic permeability can be obtained in GHz above Sneok's limit had been studied by many researchers [3–5]. However, the using of CI particles is often limited by the oxidation sensitivity due to the high oxidation activity of iron at high temperature to form Fe oxides, which have worse magnetic properties [6]. Thus, some researchers have focused on using surface coating method to protect the CI particles from oxidation. Abshinova et al. [7] prepared CI particles coated with polyaniline (PANI) by using surfactant-stabilized PANI colloids in chloroform and improved the oxidation resistance of CI. Qing et al. [8] prepared SiO_2 coated flake CI and studied the effect of heat treatment on the electromagnetic properties of SiO_2 coated CI under argon and air atmosphere, the XRD results showed only a little amount of oxide were formed when the SiO_2 coated on the CI particles. Yan et al. [9] studied the microwave absorption of Fe nanoflakes after coating with SiO_2 nanoshell and found that Fe/SiO_2 composites have better absorption property compared with uncoated Fe nanoflakes owing to the combination of the proper electromagnetic impedance match. Although, the use of surface coating method can prevent the CI particles from oxidation, the heat resistance of resin matrix is another big problem. The commonly using resin matrices are epoxy [10], natural rubber [11], organic silicon resin [12] and other polymers [13], though these polymer matrices have the convenience to fabricate the composites, they cannot use at high temperature for long time. Therefore a kind of high performance polymer matrix which can use at high temperature is needed to fabricate absorbing materials.

Thermosetting polyimide resin was a good candidate for high temperature polymer matrix because of its high-temperature stability, excellent electrical and mechanical properties, and good chemical resistance [14,15], especially for phenylethynyl terminated polyimide (PETI). PETI can be thermally cured at temperature above 300 °C. After it was cured, the mechanical properties of polyimide were highly increased and also increased its resistance to temperature owing to the forming cross-linked molecular structures. Using the PETI as the polymer matrix can solve the problem of low heat resistance when compared with traditional polymer matrix.

Concerning the perfect performances of polyimide, the electromagnetic properties and microwave absorbing characteristics of polyimide/SiO_2 coated carbonyl iron composites were studied in this work. And also the influence of heat treatment on the electromagnetic properties of the composites was also discussed.

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It was reasonably expected that a kind of high temperature absorbing materials can be fabricated by integrating the advantages of the two phases.

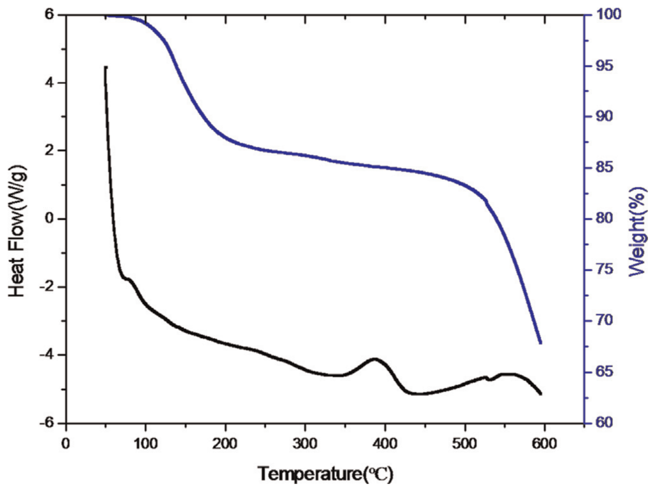

Fig. 1. The DSC–TGA analysis of pre-polyimide.

2. Experimental

2.1. Sample preparation

Carbonyl iron particles with average diameter of 1–5 μm were obtained from Xinghua Chemical Co. Ltd., Shaanxi Province, China, which were produced by decomposition of $Fe(CO)_5$, the content of α-iron > 99.5 wt%, and polycrystalline microstructure. The SiO_2-coated CI particles were prepared by the well known Stober process [8]. Firstly, the CI particles were dispersed into a mixture solution of anhydrous ethanol, water and tetraethyl orthosilicate. After mechanical stirring for 30 min, ammonia solution was added to the solution to adjust PH of the solution at 8–9. Secondly, the product was washed three time with anhydrous ethanol and dried in vacuum oven at 80 °C after the reaction was last for 4 h.

The composite was prepared by mixing SiO_2 coated CI with poly(amic acid) and the content of fillers was 60 wt%, 70 wt% and 80 wt%, respectively. In order to fabricate the composites, the mixture was first heat treated at 120 °C for 1 h and 240 °C for 2 h and then the mixture was ground into fine powders. The last, the powders were placed into a square metal die with a heating rate of 3–4 °C/min, a pressure of 5 MPa was applied on the die when the temperature reached 270 °C. After that the heating temperature

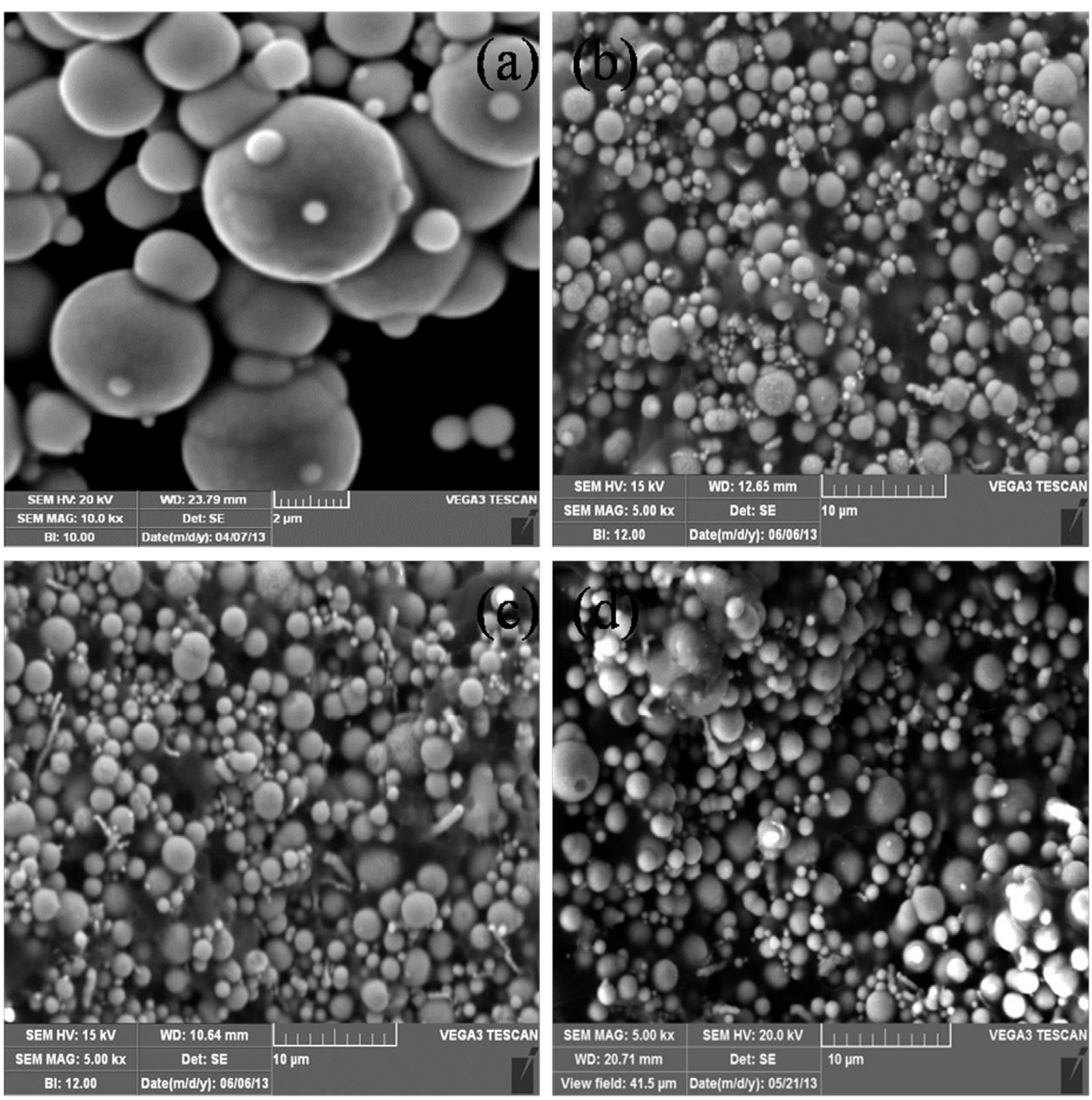

Fig. 2. Morphology of (a) SiO_2 coated carbonyl iron powder and fracture SEM image of polyimide/carbonyl iron composites (b) 60 wt%, (c) 70 wt% and (d) 80 wt%.

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