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Synthesis of polypyrrole decorated FeCo@SiO₂ as a high-performance electromagnetic absorption material



Suping Li^{a, b}, Ying Huang^{a, *}, Na Zhang^a, Meng Zong^a, Panbo Liu^a

^a The MOE Key Laboratory of Material Physics and Chemistry Under Extraordinary Conditions, Ministry of Education, School of Science, Northwestern Polytechnical University, Xi'an 710129, PR China

^b Jiaozuo Coal Industry (Group) Kaiyuan Chemical Co., Ltd., Jiaozuo 454000, PR China

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ABSTRACT

A ternary composite material FeCo@SiO₂@PPy with high-performance electromagnetic absorption have been successfully synthesized and characterized. In this paper, the components, structure and morphology of the FeCo@SiO₂@PPy have subsequently tested by XRD, TEM and SEM. The electromagnetic wave parameters of the composite was detected by the vector network analyzer (VNA). The reflection loss (R_L) of FeCo@SiO₂@PPy composites reaches -65.17 dB with the thickness of 2.1 mm at 16.48 GHz and the absorption frequency bandwidth R_L -10 dB was up to 6.8 GHz (10.4 GHz–17.2 GHz) with the layer of 2.5 mm. The absorption frequency bandwidth lower than -10 dB is up to 13.12 GHz with the range of thickness from 2.1 to 5.0 mm.

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1. Introduction

Nowadays, the rapidly developed electronic devices generated electromagnetic interference (EMI) has given rise to seriously disadvantageous affects the health of human beings. So much electromagnetic pollution is more and more attention [1-3]. In order to reduce the EMI of the electronic devices, many teams have made efforts in electromagnetic shielding materials, which can convert electromagnetic energy into heat energy or other forms of energy and dissipate it through interference [3-5]. So that the application of electromagnetic shielding materials become popular in the field of military and civil life. The key factors of a high-performance electromagnetic absorbing materials include broad width of absorption, ultralight weight, strong attenuation characteristics and thinner thickness [6-8]. According to the process of energy loss inside the absorbers, there are two types of the loss mechanism, namely magnetic loss and dielectric loss [9,10]. In recent years, because of the single-component dielectric materials or magnetic material cannot achieve ideal absorbing performance. Therefore, the more and more people begin to composite dielectric materials and magnetic materials to obtain excellent microwave absorbers.

Iron-cobalt alloy (Fe-Co) is a well-known soft magnetic material with cubic spinel structure, with high coercivity (Hc) and higher saturation magnetization (Ms) [11]. It has been widely used in ferrofluids and magnetic drug delivery. Because of its high magnetic hysteresis loss and magnetic loss, the magnetic nanoparticles perform excellent microwave absorbing ability [12,13]. However, the application of were restricted by high density and their narrow frequency range that requires light weight and the absorption of wide bandwidth. By introducing of the dielectric loss materials, it can reduce the weight of the material and can greatly improve the electromagnetic properties of materials [14–16].

As far as we know, pyrrole is a five-membered aromatic heterocycle containing both carbon and nitrogen. It is easily oxidized under the influence of oxidant and electric fields, generating polypyrrole (PPy) by a polymerization reaction [17]. PPy as typical conductive polymers attracted great attention with good characteristic, such as lightweight, high conductivity, oxidation resistance, softness and ease synthesis routes [18,19]. Therefore, PPy has been used in the field of in chemical and biological sensors, battery, anticorrosion, electromagnetic absorption, electrode materials, membrane separation and so on [20,21]. However, when PPy is used alone as microwave absorbing materials, the wave absorption performance of PPy is very poor [22]. But it as a coating of conducting polymer material to improve electromagnetic microwave absorption characteristics of material in many composites systems,





Corresponding author.
E-mail addresses: yingh@nwpu.edu.cn, lsp2008lst@163.com (Y. Huang).

which reinforced materials microwave absorbing performance [23,24].

In our work, we have successfully prepared a ternary composite FeCo@SiO₂@PPy via two step reaction of liquid-phase reduction reaction and in situ polymerization. The practical production programing of the samples is demonstrated in Fig. 1. The electromagnetic wave absorption characteristics of FeCo@SiO₂@PPy composite were investigated. When the thicknesses of samples are increasing, the position of the absorption peak moves to lower frequency. The R_L can reach – 65.17 dB at 16.48 GHz with a layer of 2.1 mm and the effective frequency width of absorption is 6.8 GHz. Thus FeCo@SiO₂@PPy composites can be expected to have wide application as an electromagnetic shielding material in the future.

2. Experimental part

2.1. Raw and preparation of materials

All the chemicals and reagents used in the laboratory were of analytical grade without any purification or preparation. The aqueous solutions were freshly produced in our laboratory and was used for all experiments.

2.2. The preparation process of FeCo@SiO₂

A certain amount of Fe-Co alloy which was synthesized using liquid-phase reduction reaction [25] added into a mixture which made up of water and alcohol and mechanical stirring for 30 min. And then added dropwise the ammonia (3 ml) and TEOS (0.4 mL) in turn. The above mixture was stirring intensely for 12 h at 24 °C. The obtained samples were handled with washing six times by water

and dried in a vacuum drying oven for 5 h at 40 °C.

2.3. The preparation process of FeCo@SiO₂@PPy

The samples (FeCo@SiO₂, 0.18 g) was dispersed in 130 ml aqueous and mechanical stirred for 30 min in the reactor of low temperature, and the range of temperature is 0-5 °C, then the pyrrole (0.3 ml) and the methyl benzene sulfonic acid (0.076 g) was dropped into the mixture solution and stirred for 30 min at the same reaction conditions. Afterward, (NH₄)₂S₂O₈ (APS), as an oxidant, was dissolved in 30 mL aqueous and drop into the above mixture solution and vigorous stirred for 24 h at the same temperature. The obtained products were washed by water six times and by alcohol one time, and then dried in a vacuum drying oven for 8 h at 60 °C.

2.4. Characterization

The crystal structural of products were conducted by XRD (Rigaku, model D/max-2500). Transmission electron microscopy (TEM, Tecnai F30 G2, FEI, USA) and scanning electron microscopy (SEM, Verios G4, FEI) were used to examine size and shape distributions of the particles. Element analysis was performed by X-ray photoelectron spectroscopy (Thermal Scientific K-Alpha, New York, USA). The magnetic moments of the products were tested by a VSM (Riken Denshi, BHV-525). For the measurement of electromagnetic parameter (ε' , ε'' , μ' , μ'') were measured by a vector network analyzer (VNA, 3672A/B/C-S), in which the products were melted in paraffin with weight ratio of 1:2 (products/paraffin) at 60 °C and then the mixture was made to be toroidal samples (φ_{out} : 7 mm, φ_{in} : 3.04 mm, H: 3 mm).



Fig. 1. Schematic demonstration of the FeCo@SiO2@PPy ternary composites.

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