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Facile synthesis of $rGO/SmFe_5O_{12}/CoFe_2O_4$ ternary nanocomposites: Composition control for superior broadband microwave absorption performance



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ABSTRACT

It is still a challenge to design and synthesize ferrite-based composites with strong absorption capability and broad frequency bandwidth. In this work, firstly, the binary Sm₃Fe₅O₁₂/CoFe₂O₄ composites with different mass ratio of Sm₃Fe₅O₁₂ to CoFe₂O₄ are synthesized by a facile hydrothermal process. Next, adding 10 mg graphite oxide (GO) into Sm₃Fe₅O₁₂/CoFe₂O₄ solution, series of novel reduced graphene oxide (rGO)/Sm₃Fe₅O₁₂/ CoFe₂O₄ ternary nanocomposites are fabricated using a one-pot hydrothermal technique. The microstructure, morphology, chemical composition and magnetic property of as-synthesized nanocomposites are characterized by XRD, Raman, TEM, SEM, XPS and VSM. The magnetic performance of Sm₃Fe₅O₁₂ changes with the addition of CoFe₂O₄ and rGO. The microwave absorption measurement is carried out by vector network analyzer, where the as-synthesized samples exhibit excellent and tunable microwave adsorption performance. Herein, introducing $CoFe_2O_4$ and rGO into $Sm_2Fe_5O_{12}$ can effectively tune its dielectric and magnetic loss. Encouragingly, when the mass ratio of Sm₃Fe₅O₁₂ to CoFe₂O₄ is 1:3, the rGO/Sm₃Fe₅O₁₂/CoFe₂O₄ ternary nanocomposite possesses the maximum reflection loss (RL) of -73.71 dB at 14.88 GHz with a thickness of 2.09 mm. The effective bandwidth (RL < -10 dB) of the as-synthesized sample is 7.12 GHz (from 10.80 to 17.92 GHz) with a thickness of 2.18 mm, which is higher than that of most microwave absorbers reported previously. Noticeably, the superior microwave adsorption performance of as-synthesized samples can be mainly attributed to the synergistic effect of dipole polarization, interfacial polarization, natural resonance, eddy current loss, multiple scattering and reflection. These results show that the as-synthesized rGO/Sm₃Fe₅O₁₂/CoFe₂O₄ ternary composites are promising candidates for the application in microwave adsorption with high reflection loss, broad bandwidth, lightweight and thin thickness.

1. Introduction

Nowadays, a large amount of electromagnetic wave (EMW) existing in our environments radiates from the digital communication facilities and modern electronic devices, which is seriously threatening human health and severely interfering with the normal operation of electronic equipments [1–3]. Electromagnetic interference, together with wastewater and air pollution, has become the tricky global pollution questions [4–6]. According to electromagnetic theory, the reflection of material surface, the absorption and the multiple internal reflections from the material itself may successfully attenuate the energy of electromagnetic wave [7,8]. That is, the EMW absorbing materials can absorb electromagnetic wave and then dissipate the wave or convert

electromagnetic energy into thermal energy. Therefore, developing the special materials to absorb electromagnetic wave has been considered as an effective and feasible approach to solve the above electromagnetic interference problem [9–11]. In particular, recently, the design of novel EMW absorbing materials with thin thickness, lightweight, broad bandwidth and strong absorption is a hot research topic in this field

Various experiments have demonstrated that EMW absorbing materials with single component usually exhibit narrow absorption band, weak reflection attenuation and high density, which is disadvantageous to their practical applications [13]. Therefore, at present, much attention has been paid to design new multiple composites with excellent EMW absorption properties. Generally, the EMW absorption

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performance of an absorber is mainly determined by its dielectric loss, magnetic loss and effective complementarities of the two loss mechanisms. In addition, improving impedance matching and enhancing electromagnetic attenuation are two significant aspects on the design of multiple composite materials [14]. That is, fabricating multiple composites with both magnetic and dielectric loss phases is a credible strategy to improve impedance matching and attenuation characteristics, and then, due to the synergistic effect on dissipation of EMW energy from both components, the microwave absorption properties of the composites can be immensely enhanced.

Ferrite, as a traditional magnetic material, is a noticeable EMW absorber, owing to its moderate saturation magnetization, good chemical stability and remarkable microwave absorption property [15.16]. Generally, ferrites contain three types: spinel, garnet and magnetoplumbite type. In fact, a number of ferrite EMW absorbers have been reported in the previous papers. Torkian et al. reported the synthesis and their microwave absorption of spinel Co2+ substituted nickel ferrites, where the maximum reflection loss of the sample was -26 dB and the effective bandwidth is 4 GHz [17]. Sharma et al. prepared yttrium iron garnet (YIG) nanoparticles via sol-gel and solid-state reaction methods and analyzed their excellent microwave absorption properties [18]. Chen et al. synthesized urchin-like SrFe₁₂O₁₉ magnetoplumbite type ferrite using a one-step hydrothermal technique and pointed out their minimum reflection loss (RL) of -22.8 dB at 15.1 GHz with a thickness of 3 mm [19]. Even though all the three types of ferrites exhibit EMW absorption performance at different levels, the single ferrite particles exhibit weak microwave absorption property, meanwhile, the application of single ferrite particles as an EMW absorber is limited owing to its high density, easy agglomeration and low magnetic loss at high frequency [20]. So, to improve this situation, the fabrication of novel ferrite-based magnetic composites as the EMW absorbers with dielectric fillers is becoming pressing and desirable.

Two-dimensional single atomic layer graphite, especially reduced graphene oxide (rGO), due to its low density, special surface properties, residual defects, high dielectric loss and unique microstructure, has been regarded as a promising microwave absorber. However, owing to its high conductivity, bad impedance matching characteristics and weak magnetic loss property, the microwave absorption performance of single graphene is very poor [21]. Hence, incorporating magnetic ferrite particles into graphene nanosheet has been reported to be an advisable proposal to enhance the EMW absorption property, for it can obtain good impedance matching of graphene/ferrite composites. In the same way, the introduction of graphene can effectively prevent the agglomeration of magnetic ferrite particles, which is advantage to the EMW absorption property of their composites [22]. Meanwhile, the high electrical conductivity of graphene can produce skin effect and additional reflection at the interface between air and composites, which helps to attenuate the EMW effectively [23]. Consequently, the preparation and microwave absorption properties of graphene/ferrite composites have been intensively studied in the past decades [24-27].

Notably, some recent reports revealed that new multiple composites composed of two or three kinds of ferrites show better EMW absorption property than that of single ferrites. Feng et al. presented that, comparing to $CoFe_2O_4$ and $NiFe_2O_4$ nanoparticles, the finite-size $CoFe_2O_4/NiFe_2O_4$ nanocapsules have an obvious increase in dielectric permittivity and loss in the whole S-Ku bands of microwaves of 2–18 GHz [28]. Pahwa et al. reported the superior reflection loss of $BaFe_{12}O_{19}/NiFe_2O_4$ nanocomposites [29]. Of course, the EMW absorption property of these binary composites should be further improved if continuing to be combined with graphene. Wang et al. demonstrated that graphene/ $CoFe_2O_4/Y_3Fe_5O_{12}$ nanocomposite exhibits evidently enhanced microwave absorbing performance in comparison with $CoFe_2O_4/Y_3Fe_5O_{12}$ nanocomposite [30]. Even though these nanocomposites exhibit excellent electromagnetic attenuation ability, their broadband performance is unsatisfied.

Now, in this work, using a hydrothermal method, we will firstly

synthesize the binary $Sm_3Fe_5O_{12}/CoFe_2O_4$ composites with different mass ratio of $Sm_3Fe_5O_{12}$ to $CoFe_2O_4$, where the effect of mass ratio of $Sm_3Fe_5O_{12}$ to $CoFe_2O_4$, where the effect of mass ratio of $Sm_3Fe_5O_{12}$ to $CoFe_2O_4$ on the morphology, microstructure and magnetic properties is discussed. Next, the corresponding $Sm_3Fe_5O_{12}/CoFe_2O_4$ composites are combined with GO, and then the ternary $rGO/Sm_3Fe_5O_{12}/CoFe_2O_4$ composites are obtained. Herein, the EMW absorption performance of different $rGO/Sm_3Fe_5O_{12}/CoFe_2O_4$ samples is detailedly analyzed. The possible microwave adsorption mechanism is presented. The main aim of this study is to find the new method on tuning EMW absorption property of rGO/ferrite composites and to explore the new route of fabricating high-performance microwave absorber.

2. Experimental

2.1. Materials

Ferric(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O), cobaltic(II) nitrate hexahydrate (Co(NO₃)₂·6H₂O) and sodium borohydride (NaBH₄) were utilized to synthesize cobalt ferrite. Samarium(III) nitrate hexahydrate (Sm(NO₃)₃·6H₂O), ferric(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O) and potassium hydroxide (KOH) were used to prepare samarium garnet. Graphite flakes (325 mesh), concentrated sulfuric acid (H₂SO₄), hydrochloric acid (HCl), hydrogen peroxide (H₂O₂) and potassium permanganate (KMnO₄) were used to prepare graphene oxide. All the chemicals, purchased from Sinopharm Chemical Reagent Co., Ltd. China, were of analytical grade without any further treatment. Deionized water was used throughout the experiments.

2.2. Preparation of Sm₃Fe₅O₁₂ powders

The reaction materials used in this work to prepare $Sm_3Fe_5O_{12}$ powders were samarium nitrate hexahydrate ($Sm(NO_3)_3\cdot 6H_2O$), ferric nitrate nonahydrate ($Fe(NO_3)_3\cdot 9H_2O$) and potassium hydroxide (KOH). In a typical hydrothermal reaction, 6 mL of $0.4\,mol/L\,Sm(NO_3)_3$ and $10\,mL$ of $0.4\,mol/L\,Fe(NO_3)_3$ solutions were mixed and stirred for 30 min to form a finely dispersed solution. Then, $18\,g\,KOH$ was added into this solution with continuously stirring for another 30 min to form slurry. Next, the slurry was transferred into a Teflon-lined stainless steel autoclave ($50\,mL$). After a reaction at $240\,^{\circ}C$ for $15\,h$, the product was cooled to room temperature. Finally, the obtained yellow products were washed with deionized water for several times and finally dried at $60\,^{\circ}C$ for $12\,h$. Thus, the $Sm_3Fe_5O_{12}$ powders were successfully synthesized.

2.3. Preparation of Sm₃Fe₅O₁₂/CoFe₂O₄ binary composites

In order to gain 100 mg CoFe₂O₄ ferrite after the reaction, in our experiment, 0.852 mmol ferric nitrate nonahydrate (Fe(NO₃)₃·9H₂O) and 0.426 mmol cobalt nitrate hexahydrate (Co(NO₃)₂·6H₂O) were regarded as the raw materials, and then, they were dissolved in water to form solution 1. Secondly, 100 mg samarium garnet (Sm₃Fe₅O₁₂) particles were added into solution 1 and continued to disperse for 30 min. Next, sodium borohydride solution (NaBH₄) was added into the prepared metal ions slurry dropwise with a simultaneous ultrasonic treatment for 30 min at room temperature to obtain a dispersed suspension. Then, the suspension was transferred into a Teflon-lined stainless steel autoclave (100 mL) and heated at 200 °C for 2 h. After that, the obtained products were centrifuged and washed several times with deionized water, and finally dried at 60 °C for 12 h. Now, the mass ratio of Sm₃Fe₅O₁₂ to CoFe₂O₄ is 1:1 in the Sm₃Fe₅O₁₂/CoFe₂O₄ binary composites, and the sample is named as S1. In the same way, the mass ratio Sm₃Fe₅O₁₂ to CoFe₂O₄ in the Sm₃Fe₅O₁₂/CoFe₂O₄ composites is selected as 1:2, 1:3, 1:4, 1:5 and 1:6, the corresponding products are named as S2, S3, S4, S5 and S6, respectively.

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