



Facile synthesis and enhanced microwave absorption properties of multiferroic $\text{Ni}_{0.4}\text{Co}_{0.2}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4/\text{BaTiO}_3$ composite fibers

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ABSTRACT

Multiferroic composite fibers composed of $\text{Ni}_{0.4}\text{Co}_{0.2}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ (NCZFO) spinel ferrite and BaTiO_3 (BTO) have been successfully synthesized by a simple electrospinning and subsequent heat treatment. The effects of the BTO content on the structure, static magnetic performance, electromagnetic and microwave absorption properties of the as-prepared samples are thoroughly investigated. The electromagnetic parameters and microwave absorption performances can be readily and effectively tuned through controlling the molar ratio of the two phases. The sample of NCZFO/(40 mol%)BTO fibers shows the enhanced complex permittivity making it better in microwave absorption capability than the composite fibers with other compositions and the mixture of NCZFO fibers and BTO fibers. The microwave absorption values less than -10 dB can cover the entire 2–18 GHz frequency range over the absorber thickness of 2–7.5 mm, and the minimum reflection loss value reaches -65.6 dB at 15.7 GHz with a broad effective absorption bandwidth below -10 dB of 7.8 GHz ranging from 2.6 to 6.8 GHz and 13.7 to 17.3 GHz for a layer thickness of 5 mm. The enhanced absorbing ability for the optimal NCZFO/(40 mol%) BTO fibers arises from the good synergistic effect between magnetic and dielectric components within the quasi-one-dimensional space, the improved interfacial effect as well as the proper electromagnetic impedance matching.

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1. Introduction

In recent years, much effort has been devoted to developing new microwave absorbing materials with a high magnetic and electric loss not only for the sake of solving increasing electromagnetic (EM) interference due to the widespread applications of information technology and wireless communications but also for the stealth of military equipments [1–9]. It is well known that the microwave absorption performance is closely dependent on the relative complex permittivity/permeability, the EM impedance matching and the microstructure of the absorber [9,10]. The EM impedance matching is critical for the improvement of microwave absorption. According to dissipation mechanism, microwave absorption materials can be divided into dielectric absorbers and magnetic absorbers. It is hard to attain good impedance matching characteristic for unilateral dielectric loss or magnetic loss materials. It has been shown that the fabrication of hybrid composite

materials containing both magnetic and dielectric loss phases is an efficient strategy to improve the impedance matching and attenuation characteristics, and in turn to boost the microwave absorption properties due to the synergistic effect on dissipation of EM wave energy arising from both components. Therefore, the development of hybrid absorbents has attracted a lot of attention recently [3–13].

During the past decade, one-dimensional (1D) and quasi-one-dimensional (Q1D) nanostructures have received steadily growing interests as a result of their distinctive and fascinating properties superior to their bulk or particle counterparts. 1D and Q1D nanostructures have potential applications in many technological fields such as data storage, energy conversion and storage, environmental protection and treatment, biomedicine, catalysis, advanced sensors, micro/nano functional devices, electromagnetic wave absorption [14–19]. Among those 1D and Q1D nanostructures, 1D and Q1D magnetic nanocomposites show immense potential in EM wave attenuation and exhibit very good absorption performances resulting from the enhanced interfacial polarization, confinement effect, and effective complementarities between dielectric loss and

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magnetic loss [5,8,13,20,21]. For instance, the reflection loss can be up to more than -20 dB from the C-band to the Ku-band for magnetic carbon fibers containing Fe/Co/Ni particles at the absorber thicknesses in the range of 1.1–5.0 mm [5]. Ferromagnetic/ferroelectric composites, such as nickel-based spinel ferrite/BaTiO₃ composites [22–24], have been largely studied due to their excellent multiferroic properties and strong magnetoelectric coupling at the room temperature. On the other hand, this kind of composite material simultaneously possesses good magnetic and dielectric losses and also is a potential candidate for high-performance microwave absorbers. For the 1D and Q1D ferroelectric/ferromagnetic hybrid nanocomposites, the previous investigations were mainly focused on the magnetoelectric effect [25–30]. Recently, some research work on the microwave absorbers made of single 1D or Q1D ferroelectric or ferromagnetic nanostructure has been carried out [31–36]. However, to the best of our knowledge, there have been few studies on the microwave absorption properties of 1D or Q1D ferroelectric/ferromagnetic hybrid nanostructures so far.

BaTiO₃ (BTO) is an excellent ferroelectric material and widely applied in the electronic industry. Over recent years, BTO used as microwave absorber has been studied due to its good dielectric and ferroelectric properties [35–38]. However, single BTO absorbent is generally difficult to achieve strong microwave absorption in a broad frequency band. Ferrites, as conventional EM wave absorbing materials, have been extensively applied from megahertz to gigahertz range because of their large magnetic loss, strong absorbing ability, high electric resistivity, easy preparation, and low cost [39,40]. But some drawbacks such as narrow resonance frequency band and large matching thickness hinder their further application in stealth fields. For the Ni-Co-Zn spinel ferrite, it was observed that Ni_{0.4}Co_{0.2}Zn_{0.4}Fe₂O₄ has a promising microwave electromagnetic performance [41,42]. In the present work, we have prepared multiferroic Ni_{0.4}Co_{0.2}Zn_{0.4}Fe₂O₄/(x)BaTiO₃ [NCZFO/(x)BTO] composite fibers for the first time by a facile electrospinning process. The structure, micro-morphology, magnetic properties, electromagnetic and microwave absorption performances of the resultant products are investigated, and the possible microwave absorbing mechanisms are discussed with the help of real and imaginary parts of the relative complex permittivity and permeability.

2. Experimental

2.1. Preparation

NCZFO/(x)BTO fibers were synthesized by a combination of electrospinning with sol-gel process. The molar fraction x of BTO in the NCZFO/(x)BTO nanocomposites is set to be 10, 20, 30, 40 and 50 mol%. First, the required amount of ferric acetylacetonate, nickel acetate, cobalt acetate, zinc acetate, barium acetate and tetrabutyl titanate according to the stoichiometry were dissolved together into the mixture solvent of ethanol and acetic acid with a mass ration of 3:7, followed by magnetic stirring at room temperature for 1 h to ensure the complete dissolution of all metal salts. A certain amount of polyvinylpyrrolidone (PVP, $M_w = 1\,300\,000$, Sigma-Aldrich) was then added into the above solution and continuously stirred for about 3 h to form a homogeneous spinning solution, in which the mass fraction of PVP and metal salts is 8% and 12%, respectively. Using the home-made electrospinning apparatus with the applied positive voltage of 15 kV, receiving distance of 20 cm and feed rate of 0.5 mL/h, the obtained solutions were electrospun into PVP/metal salts precursor fibers. The collected precursor fibers were dried at 100 °C for about 24 h and finally calcined at 1050 °C for 2 h in air with a heating rate of 2 °C/min to produce the resulting products. For comparison, a simple physical

mixture of electrospun NCZFO fibers and BTO fibers with a molar ratio of 3:2 was also prepared denoted as the NCZFO-(40 mol%)BTO fiber mixture.

2.2. Characterization

The structure and phase composition of the as-synthesized samples were analyzed by powder X-ray diffraction (XRD) on a Shimadzu XRD-6000 diffractometer at a voltage of 40 kV and a current of 30 mA with graphite monochromatized Cu K α ($\lambda = 0.154056$ nm) radiation. The surface morphology of the products was observed using a JEOL JSM-7001F field-emission scanning electron microscopy (FE-SEM). The microstructure of the composite fibers was characterized using a JEOL JEM-2100 transmission electron microscopy (TEM) operating at an accelerating voltage of 200 kV. Room temperature magnetic hysteresis measurements were carried out by a HH-20 vibration sample magnetometer with a maximum applied field of 15 kOe. The EM parameters were measured using an Agilent PNA N5224A vector network analyzer based on the transmission/reflection method [43]. The measured frequency is from 2 to 18 GHz, and the measurement fixture is 7 mm coaxial air-lines. The composite samples used for EM properties measurement were prepared by homogeneously dispersing the resultant fibers into silicone (Dow Corning Sylgard 184) with a weight ratio of 7:3. The mixtures were then cured in a stainless steel mold at 120 °C for 1 h to form the toroidal-shaped specimens with an outer diameter of 7 mm, an inner diameter of 3 mm and a thickness of 2 mm. According to the transmission-line theory [44], the microwave absorption properties were evaluated from the measured complex permittivity and permeability at a given frequency and absorber thickness.

3. Results and discussion

3.1. Phase composition and morphology

Fig. 1 presents XRD patterns of the as-prepared NCZFO/(x)BTO composite fibers with $x = 10, 20, 30, 40$ and 50 mol%. The diffraction peaks appeared at $2\theta = 22.12^\circ, 31.50^\circ, 38.84^\circ, 45.16^\circ, 50.86^\circ, 56.12^\circ$,

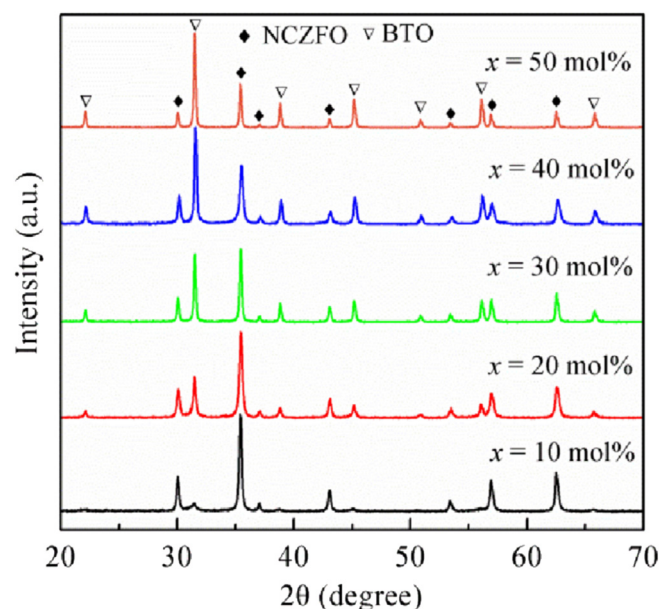


Fig. 1. XRD patterns of NCZFO/(x)BTO fibers.

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