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Structure and magnetic properties of FeCo–SiO_2 nanocomposite synthesized by a novel wet chemical method

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Abstract

A novel soft magnetic nanocomposite with FeCo particles encapsulated by amorphous SiO_2 was synthesized using a co-precipitation combined H_2 reduction method. The saturation magnetization of the $(Fe_{70}Co_{30})_{90}/(SiO_2)_{10}$ nanocomposite is as high as 200 emu/g, which is 4–5 times larger than that of traditional spinel ferrites. The frequency dependence of the complex initial permeability is intensely dependent upon the content of SiO_2 insulating phase. With increasing the content of SiO_2 to 10 wt.%, the cut-off frequency is drastically increased to over 1 GHz. The results show that a new high-frequency soft magnetic material with high saturation magnetization (M_s) can be achieved by introducing FeCo/SiO_2 nanocomposite.

Keywords: FeCo–SiO_2; Magnetic materials; Nanocomposites; Permeability

1. Introduction

The size reduction and high-frequency applications of electronic devices require soft magnetic materials that possess high saturation magnetization (M_s), high permeability (μ), high Curie temperature and low energy losses. Ferrites, which were conventionally used for high-frequency applications, have intrinsic disadvantage of small saturation magnetization (M_s) and low Curie temperature. The high-frequency performance of these ferrite cores is limited following Snoek's law [1,2]. It is highly desirable to develop novel magnetic materials having large magnetization and high resistivity to be applied in tens of megahertz to one gigahertz range. Metal/insulator nanocomposite, in which ferromagnetic nanoparticles of metal were coated by insulator phase, presents new opportunities to develop novel high-frequency soft magnetic materials [3,4]. Amongst the ferromagnetic metals, FeCo alloys, which have the highest value of M_s about 2.4 T in $Fe_{35}Co_{65}$ [5], are suitable for this purpose. FeCo alloys also have the highest Curie temperature, which make them suitable for high temperature applications. FeCo alloys have a low coercivity due to the low magnetocrystalline anisotropy of the body-centered cubic structure. Coating FeCo nanoparticles with an insulating phase can improve the electrical resistivity of the magnetic materials. Coating also hinders the diffusion or the grain growth of metallic particles during the formation or sintering of the nanoparticles. The possible exchange coupling between neighboring FeCo nanoparticles can overcome the anisotropy and demagnetization effect of the individual particles, resulting in much better soft magnetic properties [3,6].

Various methods have been explored to synthesize metal/insulator nanocomposites, including sol–gel [7], sputtering [8], electrodeposition [9], high energy ball milling [10], microemulsion and reverse micelle techniques [11]. In order to dramatically increase the electric resistivity of metallic magnetic alloys while retaining their excellent soft magnetic properties (high saturation magnetization, high permeability, high Curie temperature, etc.), SiO_2 was selected to be used as insulator phase. Up to now, no researches have been done for FeCo/SiO_2 magnetic nanocomposite. Herein, FeCo/SiO_2 nanocomposites were synthesized using a novel wet chemical approach. The structure and magnetic properties of the nanocomposite were investigated.

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2. Experimental procedure

Co-precipitation combined H_2 reduction method was employed to prepare $Fe_{70}Co_{30}/SiO_2$ nanocomposite particles. The detailed procedure was as follows: (1) Appropriate amount of $FeCl_2{\cdot}4H_2O$ and $CoCl_2{\cdot}6H_2O$ (the molar ratio of Fe^{2+} to Co^{2+} was 70:30) was dissolved into deionized water. (2) NaOH solution was introduced into the former solution with rapid stirring to keep the pH value in the range $12 \le pH \le 14$. The suspension was kept at 70 °C for 30 min. (3) After the suspension was cooled down to room temperature, sodium silicate ($Na_2SiO_3{\cdot}9H_2O$) solution was added into the suspension, controlling its pH value between $7 \le pH \le 9$. Then the suspension was warmed up to 80 °C for completing the hydrolyzation of sodium silicate and the precipitation of silicate. (4) The suspension was repeatedly washed, filtered for several times and dried at 100 °C in the air. The dried powder was then reduced by hydrogen in an electric heated quartz furnace at different temperatures.

The phase identification and structural analysis of the sample were examined by X-ray powder diffraction (XRD) with Cu K_α radiation. Morphology was analyzed using transmission electron microscopy (TEM). Magnetic properties were studied using a Lake Shore vibrating sample magnetometer (VSM) with a maximum applied magnetic field of 10,000 Oe. The composite particles were compacted into toroidal cores for the permeability measurement at the pressure 12 ton/cm^2. The toroidal size is φ3 mm in inside diameter, φ7 mm in outside diameter and 2 mm in height. Complex permeability spectra were measured in the frequency range 1 MHz to 1 GHz with a RF impedance/material analyzer (Agilent4291B + 16454A).

3. Results and discussion

Fig. 1 shows the XRD patterns for the $(Fe_{70}Co_{30})_{90}/(SiO_2)_{10}$ nanocomposite powders that were obtained by reducing the precursor in H_2 for 2 h at various temperatures. It was apparent from the X-ray diffraction data that three characteristic peaks for $Fe_{70}Co_{30}$ (2θ=44.760°, 65.157°, 82.499°), corresponding to Miller indices (110), (200), (211), were observed. All of the diffraction patterns match only the α-FeCo (bcc) structure and no peaks for Fe-oxide or Co-oxide were detected. This indicates that H_2 reduction effectively converted the oxide particles into FeCo. No crystalline SiO_2 was found in the reduced samples, which revealed that SiO_2 phase in the $FeCo/SiO_2$ nanocomposite is in amorphous state.

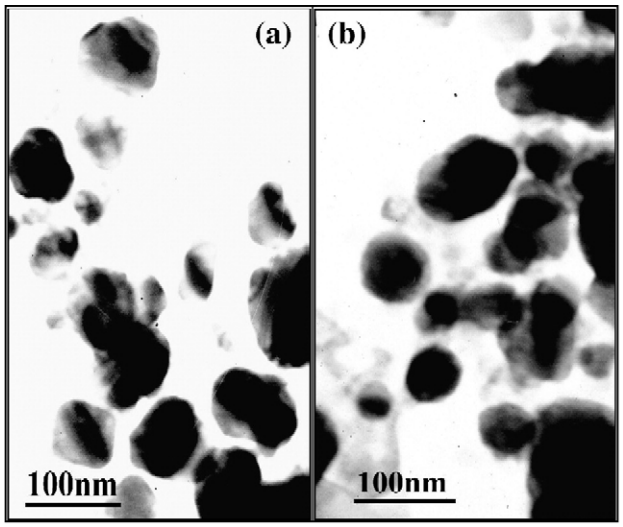

Fig. 2. TEM images for $(Fe_{70}Co_{30})_{90}/(SiO_2)_{10}$ samples reduced by H_2 for 2 h at (a) 500 °C and (b) 700 °C, respectively.

Fig. 2 shows the typical TEM micrographs for $(Fe_{70}Co_{30})_{90}/(SiO_2)_{10}$ samples which were heat-treated in hydrogen at 500 °C and 700 °C, respectively. The TEM images indicate that the synthetic $(FeCo)_{90}/(SiO_2)_{10}$ nanoparticles have the core/shell structure, in which FeCo nanoparticles (dark area) are coated by amorphous SiO_2 (semi-transparent layer). From the XRD pattern and TEM images, the size of $FeCo/SiO_2$ particles is estimated to be in the range about 30–70 nm and increases slightly with the increase of reduction temperature. This can be attributed to the SiO_2 coating which hinders the diffusion or the grain growth of FeCo nanoparticles during the calcinations of the nanoparticles at high temperatures.

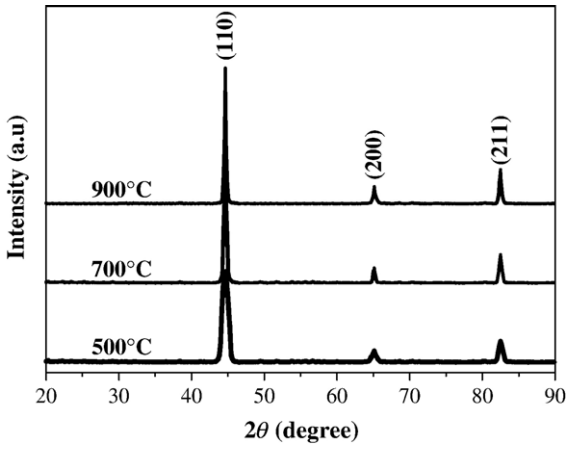

Fig. 1. X-ray diffraction patterns of $(FeCo)_{90}/(SiO_2)_{10}$ nanocomposite powders synthesized by H_2 reduction at different temperatures.

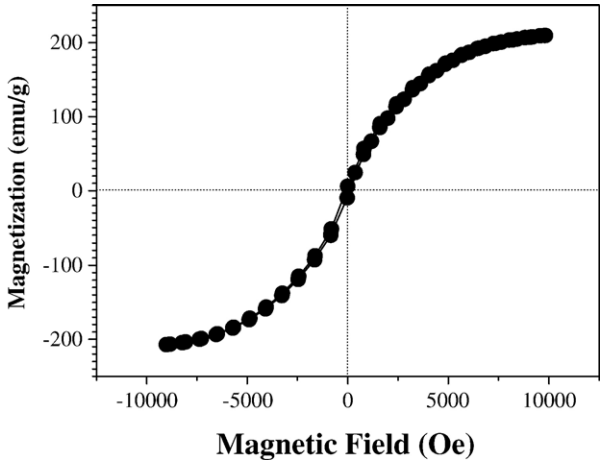

Fig. 3. Magnetic hysteresis loop for $(Fe_{70}Co_{30})_{90}/(SiO_2)_{10}$ nanoparticles synthesized by H_2 reduction at 500 °C for 2 h.