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Functionalization of FeCo alloy nanoparticles with highly dielectric amorphous oxide coatings

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FeCo alloy nanoparticles have been prepared by using a two step modified polyol process using Fe(II) chloride and Co acetate tetrahydrate as Fe and Co metal precursors. Tetraethyl silicate, aluminum isopropoxide, and zirconium(IV) acetyl acetonate were used to make amorphous SiO₂, Al₂O₃, and ZrO₂ coatings, respectively. X-ray diffraction studies showed that there are no crystalline peaks corresponding to SiO₂, Al₂O₃, and ZrO₂ because the oxide coatings of the FeCo core are amorphous in nature. The scanning electron micrograph analysis depicted the cubic nature of the particles with mean particle size of about 45 nm. The maximum saturation magnetization of 205 emu/g was achieved at 300 and 4 K. FeCo nanocomposites were screen printed as films and aligned by using an external magnetic field of 10 kOe. The microwave properties measured by in-plane ferromagnetic resonance at various frequencies indicate a minimum linewidth of ≈ 3700 Oe. © 2008 American Institute of Physics. [DOI: [10.1063/1.2839593](https://doi.org/10.1063/1.2839593)]

INTRODUCTION

Today's electronics industry requires improved alloys for use in thin film inductors which should possess a high permeability, high saturation magnetization, and low coercivity.¹⁻⁴ Thin films of these materials can be engineered for applications in high frequency inductors. Magnetic dielectric nanocomposites have also attracted sustained interest due to their unusual combined magnetic and electric properties. In fact, the metallic nature, eddy currents, limits the application of pure nanoparticles at high frequency. The coating by an insulating shell on the surface of soft magnetic nanoparticle cores such as FeNi and FeCo confers to the material for applications in the microwave frequency range. Such materials are typically used for telecommunication applications.⁵ The high saturation magnetic moment, Curie temperature, and high permeability properties of FeCo alloys make its potential from magnetic recording head to drug delivery applications. There are few reports using the bottom-up approach to prepare the magnetic metal alloy core and crystalline oxide shell particles.⁶⁻⁸ Here, we prepared the amorphous oxide shell coated FeCo alloy nanoparticles by the two step polyol process followed by screen printing. The films of these materials can be engineered for applications in high frequency inductors. The structural, magnetic, and microwave properties were studied in detail.

EXPERIMENT

Fe(II) chloride and cobalt acetate tetrahydrate with 0.01M were dissolved in ethylene glycol with appropriate

amounts of NaOH. The solution was placed in an oil bath and heated to 120 °C with constant mechanical stirring and allowed to reflux for a maximum period of 2 h at this temperature. After refluxing, the suspension was allowed to cool to RT. The black precipitated particles were isolated by centrifuging and were then washed three times with ethanol to remove the by-products. In order to make the oxide coating on the surface of FeCo nanoparticles, the following procedures have been developed. The as-prepared FeCo alloy nanoparticles were transferred to the diethylene glycol media and a suitable oxide precursor was added with definite amount of deionized water and NaOH. Zirconium(IV) acetylacetonate and tetraethyl orthosilicate were added as the metal oxide precursors. The mixed solution was heated to 180 °C for 2 h. The reaction temperature will help to stabilize the oxide phase over the FeCo alloy core nanoparticles. The ratio of FeCo nanoparticles and oxide precursor was varied to find the optimal condition. The oxide coated FeCo alloy nanoparticles were suspended in an epoxy and hardener and screen printed onto a dielectric (Al₂O₃) substrate with a thickness of about 1–2 mm. Particle orientation was performed under an external dc magnetic field of 10 kOe.

The crystallographic phase of the particles was analyzed using θ -2 θ x-ray powder diffraction (XRD) (Rigaku, Cu K α radiation, $\lambda=1.54506$ Å). The surface morphology of the particles was examined by scanning electron microscopy (SEM) (Zeiss). Chemical analyses have been carried out using an induction coupled plasma spectrophotometer (ICP 20P VG Elemental Plasma Quad2) as well as the SEM-EDAX facility. The magnetic properties were measured using a superconducting quantum interference device (SQUID) magnetometer. Ferromagnetic resonance (FMR) measurements

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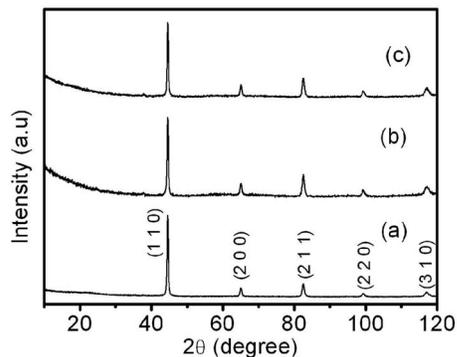


FIG. 1. X-ray diffractograms of the (a) FeCo, (b) amorphous ZrO₂, and (c) SiO₂ coated FeCo.

were performed in both out-of-plane and in-plane FMR conditions by using a TE₀₁ rectangular waveguide at room temperature in *K_a*-band frequency. The FMR data allow us to calculate the effective magnetization, anisotropy field, and FMR linewidth (ΔH_{FMR}).

RESULTS AND DISCUSSION

For the practical applications, the nanoparticles need to be controlled to have identical size, shape, and chemical composition and free from agglomeration. The polyol process is an efficient technique, in which the solvent acts as a reducing and oxidation preventing agent in addition to the molecular or atomic level control. The detailed reduction chemistry of the polyol process could be found elsewhere.^{9–11} XRD studies in Fig. 1 demonstrated that there are no crystalline peaks corresponding to SiO₂, Al₂O₃, and ZrO₂ because the oxide coatings of the bcc-FeCo core are

amorphous in nature. Figure 2 showed the scanning electron micrograph of the FeCo, FeCo–Al₂O₃, FeCo–ZrO₂, and FeCo–SiO₂ nanocomposites. The particles are in cubic nature with a mean particle size of about 45 nm. When the FeCo and oxide precursor was changed from 10:1 to 10: 3, a very clear oxide layer has been observed on the surface of the metal nanoparticles, as shown in Fig. 2(b). When the FeCo particles, were coated with oxides, the edges of the particles were not, clear as in Figs. 2(b)–2(d). The structural evolution study suggested that the silica oxide layers grow without affecting the core of the nanoparticles. SEM-EDX analysis proved that the particles have metal as well as oxide nanoparticles (insets of Fig. 2). The hysteresis loops are measured at 300 and 4 K for the FeCo nanoparticles and showed a saturation magnetization value of about 205 emu/g [Fig. 3(a)], which is in good agreement with the reported values. The coating quality is determined from the saturation magnetization values of the nanoparticles. For example, the ZrO₂ coated FeCo nanoparticles show a saturation magnetization of 170 emu/g, as shown in Fig. 3(b). The nonmagnetic contribution of the zirconia, alumina, and silica leads to a lower magnetization than the as-prepared FeCo alloy nanoparticles.

For the high frequency applications such as thin film inductors, it is important to control the thickness of the oxide coating to control the ferromagnetic resonance properties.

FMR measurements were performed by applying a swept dc magnetic field parallel to the film plane, i.e., parallel FMR configuration. The frequency was fixed during each field sweep and the measurements were taken for a wide range from 27 to 40 GHz. When H_{ext} is parallel to the film, the FMR condition is given as follows:⁶

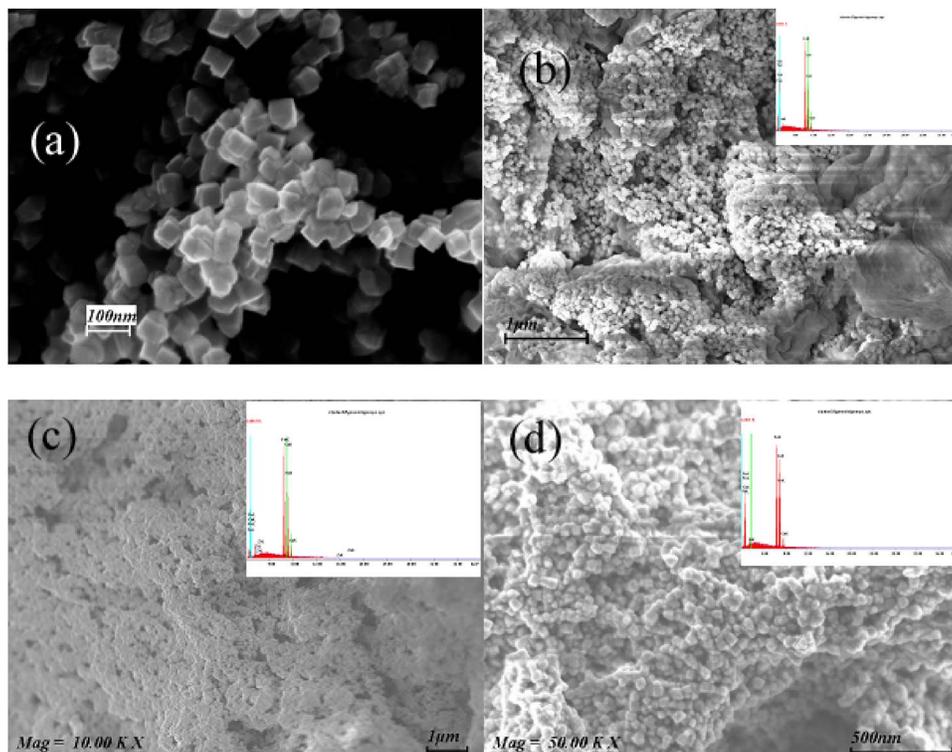


FIG. 2. (Color online) Scanning electron micrograph of the (a) FeCo, (b) Al₂O₃, (c) ZrO₂, and (d) SiO₂ coated FeCo nanoparticles. The inset figure shows the corresponding EDAX spectrum.

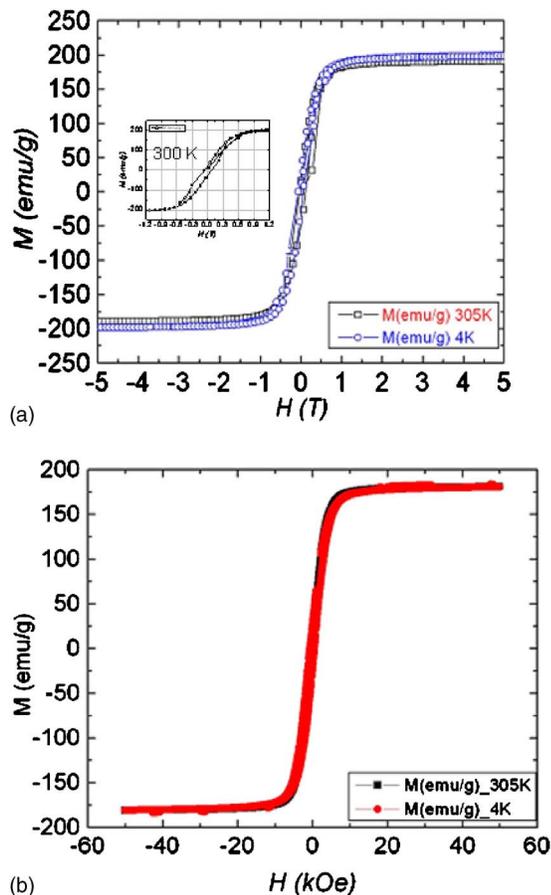


FIG. 3. (Color online) Magnetic properties of (a) FeCo alloy nanoparticles and (b) FeCo-ZrO₂ coated nanoparticles measured at 305 and 4 K by the SQUID magnetometer.

$$\frac{\omega}{\gamma} = \sqrt{(H_{\text{ext}} + H_A)(H_{\text{ext}} + H_A + 4\pi M_S)},$$

where $\omega = 2\pi f$ and $\gamma = 2\pi(g \times 1.4 \times 10^6)$ Hz/Oe. Figure 4 shows the variation of the FMR derivative linewidth (ΔH) as a function of frequency over a range of 27–40 GHz for the zirconia and silica coated FeCo films. Minimum linewidths of 2900 and 3700 Oe were realized for zirconia and silica coated FeCo films, respectively. The linewidth can be further reduced by improving the in-plane orientation and density of the film.

CONCLUSIONS

Amorphous oxide coated FeCo alloy nanoparticles were prepared using the two step polyol process. The maximum saturation magnetization of about 205 emu/g was achieved at room temperature for the 50 nm FeCo alloy nanoparticles and it decreases with the oxide content. The microwave properties measured by in-plane ferromagnetic resonance at various frequencies indicate a minimum linewidth of ~ 2900 Oe. The insulating amorphous silica, alumina, and zirconia coatings may be effective in reducing eddy current losses at high frequencies.

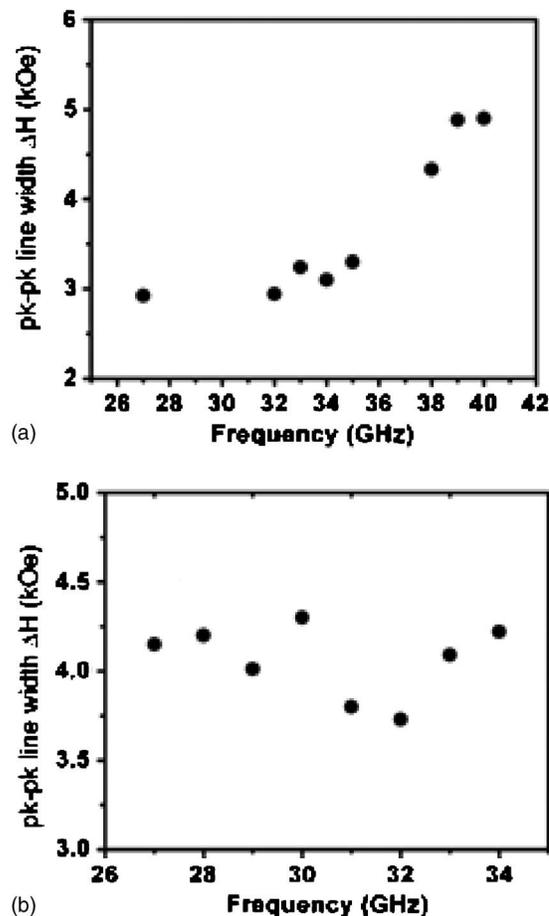


FIG. 4. Ferromagnetic resonance peak-to-peak derivative linewidths (ΔH) vs magnetic field for the (a) ZrO₂ coated and (b) SiO₂ coated FeCo alloy nanoparticles.

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