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Microwave non-resonant absorption in fine cobalt ferrite particles

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Abstract

Cobalt ferrite particles of average crystallite size of 11 nm were obtained by a sol–-gel process at 400 °C. The powders were annealed at temperatures of 500, 600, 700 and 800 °C in air. Derivative microwave power absorption (dP/dH) measurements were carried out as a function of magnetic field (H_{DC}) at X band (9.4 GHz), in the field range −80–796 kA/m for all annealed temperatures. In order to compare the response of saturation magnetization measurements with high frequency measurements, we calculated the areas inside both the magnetization (A_M) and the absorption hysteresis loops (A_{LFS}). The dependence of these areas as a function of crystallite size is remarkably similar in both experiments.

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Keywords: Microwave absorption; Hysteresis; Cobalt ferrite

1. Introduction

There is an increasing interest in magnetic ferrite nanoparticles because of their broad applications in several technological fields including magnetic fluids, microwave devices, and high density information storage [1,2]. The spinel type ferrites are frequently used in the microwave region, in the 3–30 GHz band. In this work, we report measurements of microwave absorption of cobalt ferrite powders as a function of magnetic field, which were carried out around zero magnetic field. The most novel aspect of these microwave absorption signal at low field (LFS) is the presence of hysteresis loops. Soft magnetic materials as amorphous ribbons [3] and soft ferrites (Ni–Fe, Ni–Zn) [4] present this signal, clearly distinct from ferromagnetic resonance (FMR). We make a comparison between the areas inside both hysteresis loops (magnetization measurements and LFS), and found a remarkable agreement. On the other hand, the saturation magnetization and the coercive field are strongly dependent on the annealing temperatures and can be directly related to variation of particle size [5,6].

2. Experimental

Stoichiometric amounts of Co $(CH_3CO_2)_2 \cdot 4H_2O$ (1/200 mol) and $Fe(NO_3)_3 \cdot 9H_2O$ (1/100 mol) were first dissolved in 2-metoxyethanol (100 mL) and water (15 mL) for 30 min with the help of ultrasonic vibration. The solution was refluxed for 24 h at 80 °C to allow gel formation, and then dried at 60 °C in air. The dried amorphous precursor powder was ground and heated at temperatures ranging from 400–800 °C with intervals of 100 °C, at rates of 10 °C/min in a flux of air (50 mL/min). The XRD pattern of the as prepared sample and the patterns for samples heated at various temperatures were recorded in the $2\theta = 10$–90° range in steps of $0.04^\circ\,min^{-1}$ on a Siemens D-5000 diffractometer using Cu K_α radiation. Derivative microwave power absorption (dP/dH) measurements were carried out as a function of magnetic field (H_{DC}), in a JEOL JES-RE3X spectrometer at X band

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(9.4 GHz) in the range −80–796 kA/m. For this purpose a JEOL ES-ZCS2 zero cross sweep unit was used, to digitally compensate for any remanence in the electro-magnet, allowing precise measurements in the low field range −80–80 kA/m (LFS measurements). Magnetization measurements were carried out using a LDJ 9600 vibrating sample magnetometer, VSM, to obtain the *M*–*H* loop at room temperature.

3. Results and discussion

The XRD powder pattern of the precursor (sol–gel) showed an amorphous state. For the sol–gel powders annealed at different temperatures, XRD patterns indicated that the spinel structure of cobalt ferrite first appears at 400 °C (JCPDF file, No. 26-1086). The diffraction peaks were broadened, due to reduced particle size. The full-width at half-maximum (FWHM) of (220), (311), (400), (511), and (440) diffraction peaks were used for the estimation of crystallite size by means of Scherrer's equation, $D = k\lambda/\beta_{1/2}\ \cos\theta$. The average values of the crystallite diameters are given in Table 1. The increase in the annealing temperature yields a sharpness increase of the mayor peaks, in agreement with the expected growth of the grain size.

The saturation magnetization and the coercive field are strongly dependent on the annealing temperatures and therefore they can be directly related to variations of cobalt ferrite particle size. Fig. 1 shows the behavior saturation magnetization and coercive field as a function of the average crystallite size at a maximal external field of ±796 kA/m. The magnetization increases with annealing temperature from 19.3 to 75 emu/g, while, the coercive field goes through a maximum for 500 °C and an average particle size of 20 nm. A further increase in crystallite size above 30 nm decreases the coercive field. Fig. 2 shows derivative microwave power absorption (DMPA) measurements with a large hysteresis; this feature is indicative that the signal has no resonant character, and therefore, does not correspond to ferromagnetic resonance.

Table 1
Average crystallite size (*D*) estimated from the FWHMs of X-ray diffraction peaks

Annealing temp. (°C)	220	311	400	511	440	*D* (nm)
400	9	8	15	13	8	11
500	24	22	24	22	21	23
600	28	29	29	27	27	28
700	57	58	51	39	37	48
800	110	87	85	67	65	83

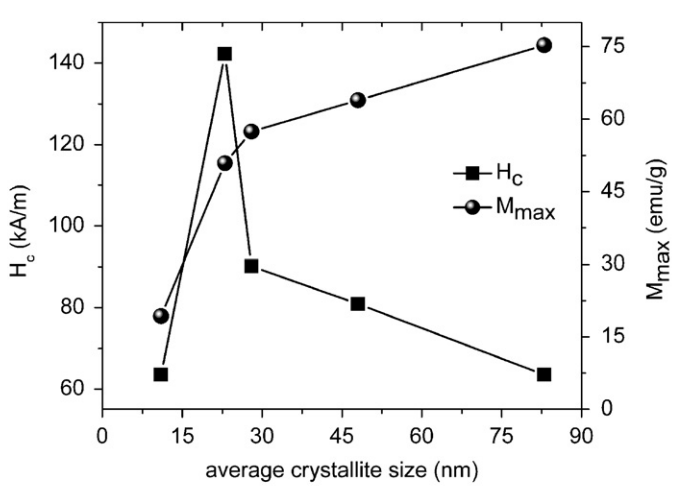

Fig. 1. Variation of saturation magnetization and coercive field against average crystallite size of cobalt ferrite.

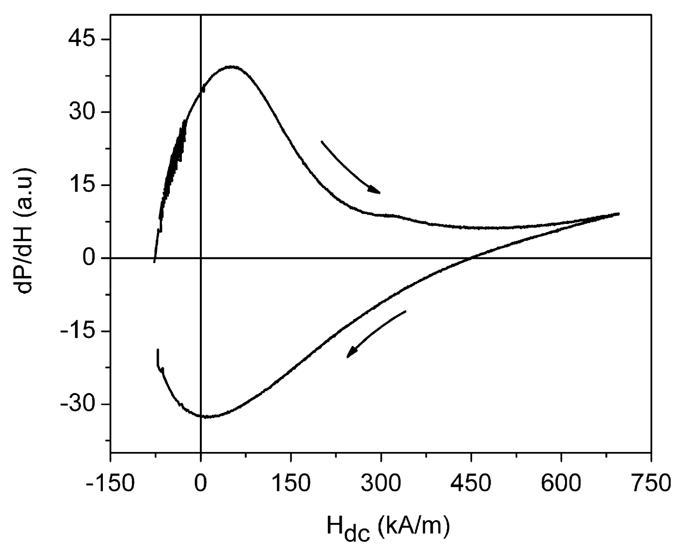

Fig. 2. X-band (9.4 GHz) derivative microwave power absorption (d*P*/d*H*) measurements the cobalt ferrite powder annealing temperature 700 °C. The signals corresponds to the cycling the DC magnetic field from −80 at 716 kA/m.

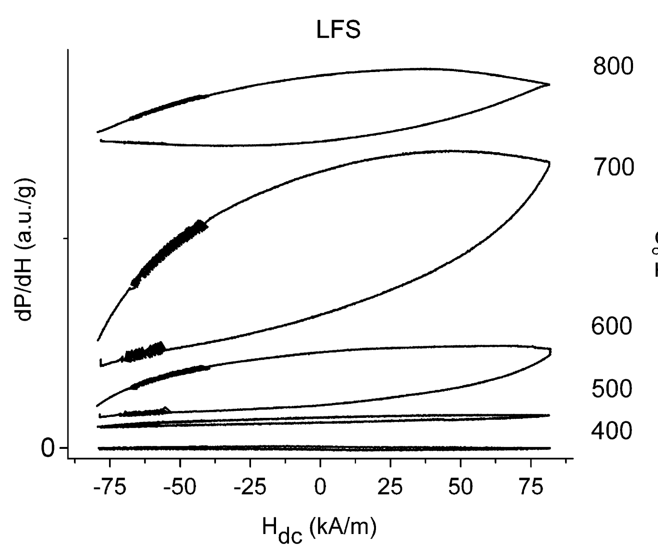

Fig. 3. X-band (9.4 GHz) derivative microwave power absorption (d*P*/d*H*) measurements of the cobalt ferrite at different annealing temperatures. The signals corresponds to the cycling of the DC magnetic field from −80 at 80 kA/m (microwave absorption signal at low field, LFS).

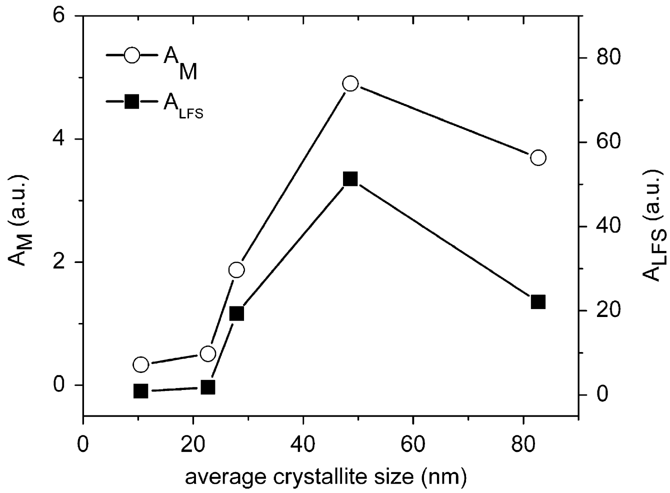

Fig. 4. The relationship between the average crystallite size and the areas inside the magnetization (A_M) and the absorption hysteresis loops (A_{LFS}) carried out cycling the DC magnetic field from −80 at 80 kA/m.
H. Montiel, G. Alvarez, I. Betancourt, R. Zamorano and R. Valenzuela.

In order to compare the DMPA measurements with vibrating sample magnetometer (VSM) measurements, we calculated the areas inside hysteresis loops in both experiments: the magnetization (A_M) and the absorption loops ($A_{LFS = \text{low field signal}}$), cycling the DC magnetic field in the interval of −80–80 kA/m. Fig. 3 shows that at 400 °C a minimum of the absorption hysteresis loop area is observed, and then it increases with annealing temperature, reaching a maximum at 700 °C and finally decreases for 800 °C. The curves in Fig. 4 show the areas calculated versus average crystallite size. We observe that the dependence of these areas as a function of crystallite size (temperature) is very similar in both experiments. This behavior suggests that the areas of the derivative microwave power absorption curves (power absorption) associated with the magnetization dynamic process are similar to used energy in the magnetization process, at least within the interval of magnetic field used in this experiment.

We have recently carried out similar measurements in soft ferromagnetic materials [4]. The DMPA in these materials exhibit common features with the magnetization processes and the giant magnetoimpedance effect (GMI). A general result of these investigations is that the magnetization processes have a strong dependence with the anisotropy field. In contrast, we have found no evidence of such dependence on anisotropy field in the present work. We believe that this is due to the fact that nanometric cobalt ferrite is significantly harder than soft materials previously studied (amorphous ribbons and microwires), and the magnetic fields used here are not enough to attain the anisotropy field of cobalt ferrite. The coercive fields observed are between 64 and 140 kA/m for applied fields of ±790 kA/m, and our DMPA system can scan only between −80 at 719 kA/m.

4. Conclusion

In this paper we have shown a novel aspect of microwave power absorption (dP/dH) measurements in nanoparticles of cobalt ferrite. The observed hysteresis in the spectra is dependent of crystallite size in very good agreement with those observed in magnetization measurements. The power absorption associated with the magnetization dynamic process is similar to used energy in the magnetization process, at least within the interval of magnetic field used in this experiment.

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