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Ferromagnetic resonance of nonstoichiometric zinc ferrite and cobalt-doped zinc ferrite nanoparticles

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Abstract

Ferromagnetic resonance spectra of zinc ferrite and cobalt doped zinc ferrite nanoparticles, measured at various temperatures, exhibit an invariant point at a given field. This makes it possible to determine the equation relating the resonance field shift to the peak-to-peak linewidth. When particles are frozen in a matrix in a magnetic field, the anisotropy constant of the material can be derived from the angular variation of the resonance field. This procedure is useful to determine the thermal dependence of the anisotropy constant, but is shown to require various freezing temperatures experiments to estimate the accuracy of the deduced anisotropy constant values. It is also shown that the angular dependence of the resonance field is similar for a uniaxial (zinc ferrite) and cubic (zinc ferrite containing 40% cobalt ions) anisotropy. This unexpected result is explained by the weakness of the texturation, leading to a distribution in easy axes directions. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

In the last few years, ferromagnetic resonance has become a more widely used tool for studying the magnetic properties of nanoparticles. This is due to better theoretical understanding [1–4] and new experimental procedures, mainly using texturation to determine the anisotropy of nanoparticles [5–7]. For samples studied at various

temperatures, a simple model [8] proposes a power law of the form $H_R - H_0 \propto \Delta H_{pp}^3$ that interrelates the linewidth ΔH_{pp} and the resonance field shift $H_R - H_0$ (H_R being the resonance field and H_0 the limit resonance field at high temperature). For samples texturated in a magnetic field, another model [6,7] proposes an evaluation of the texturation parameter (leading to quantification of the anisotropy constant) for an assembly of nanoparticles, generally not completely oriented in the direction of the aligning field.

In this paper, we study the ferromagnetic resonance behavior of nonstoichiometric zinc ferrite nanocrystals doped or not with Co^{2+} ions.

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2. Experimental section

Sodium dodecyl sulfate, Na(DS), was from Fluka, ferrous chloride $\text{Fe}(\text{Cl})_2$ and cobalt acetate $\text{Co}(\text{CH}_3\text{CO}_2)_2$ from Merck, and zinc chloride $\text{Zn}(\text{Cl})_2$ from Prolabo. All the compounds were stated to be 99% (minimum) pure.

Ferromagnetic resonance (FMR) measurements were made with a Bruker ER 200 D X-band EPR spectrometer (9.43 GHz).

Texturation in PVA was performed with a commercial electromagnet generating a 2 T field.

In the last few years, we have developed a new technique for preparing ferrite nanoparticles using oil in water micelles [9,10] which provides control of the mean size of the nanoparticles. This procedure has been adapted to synthesize mixed ferrites.

Various relative amounts of divalent dodecylsulfate, $\text{X}(\text{DS})_2$, ($\text{X} = \text{Fe}, \text{Co}, \text{Zn}$) were solubilized in 10 ml of aqueous solution kept at 30°C. The iron dodecylsulfate concentration, $[\text{Fe}(\text{DS})_2]$, and the relative percentage $[\text{Fe}(\text{DS})_2]/\Sigma[\text{X}(\text{DS})_2]$ were $1.68 \times 10^{-2} \text{ M}$ and 0.75, respectively. The relative concentration of cobalt compared to zinc dodecylsulfate, $y = [\text{Co}(\text{DS})_2]/([\text{Co}(\text{DS})_2] + [\text{Zn}(\text{DS})_2])$, varied from 0 to 0.40. Methylamine was then added and particles collected after 2 h stirring. For any cobalt ion composition, the average particle size is 3.7 nm and the size distribution follows a lognormal law with a standard deviation $\sigma = 0.23$. The composition of particles measured by EDS is the same as that used for the synthesis and the existence of a solid solution is deduced from the reproducibility of EDS measurements in different zones. Hence the formula of the material can be written as $\text{Co}_{0.73y}\text{Zn}_{0.73(1-y)}\text{Fe}_{2.18}\square_{0.09}\text{O}_4$. Two procedures were used to prepare the samples.

(1) The particles were solubilized in aqueous solution containing 10 wt% polyvinyl alcohol, PVA. With time, the solvent evaporates and a slow polymerization process takes place. After total evaporation, the nanocrystals were dispersed in a disk-shaped substrate. The final concentration of particles in PVA was less than 1% weight. During the evaporation, the sample was subjected to a magnetic field and at the end of the evaporation the easy axes of particles are assumed

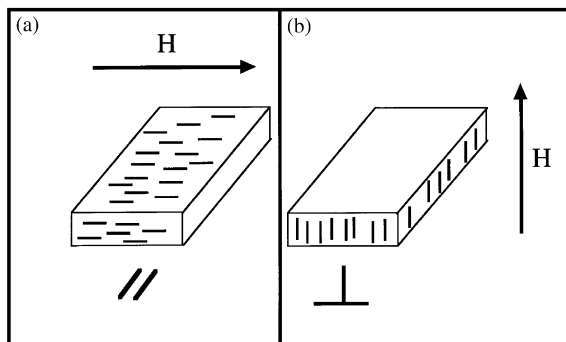


Fig. 1. Texturation of the PVA samples: (a) parallel to the substrate plane, (b) perpendicular to the plane.

to be aligned along the direction of the applied field. The material is called “texturated”. By changing the orientation of the applied field, the easy axes are either aligned parallel or perpendicular to the substrate (Fig. 1).

(2) The particles are coated with lauric acid and then dispersed in various non polar solvents such as isooctane and cyclohexane. This coating was obtained by dispersing nanocrystals in ethanol solution containing a large excess of lauric acid. After stirring the solution for a few hours, a precipitate appeared. This was washed with ethanol and the nanocrystals were then dispersed in non polar solvents.

3. Results and discussion

Fig. 2 shows the magnetization curves at 3 K obtained for 3.7 nm nanocrystals differing by the amount of cobalt ions included in the zinc ferrite matrix.

As shown in Fig. 2(a), there is low coercivity in absence of cobalt ($y = 0$). On increasing y , the coercivity increases (Fig. 2(b) and (c)). This is due to an increase in the anisotropy constant of the material when zinc ions are replaced by cobalt ions. Table 1 shows the increase in the ratio of remanence to saturation magnetization with increasing cobalt concentration. For $y = 0$, the M_R/M_S ratio is lower than 0.5. This indicates an axial anisotropy of nonstoichiometric zinc ferrite nanocrystals. Inclusion of cobalt induces an

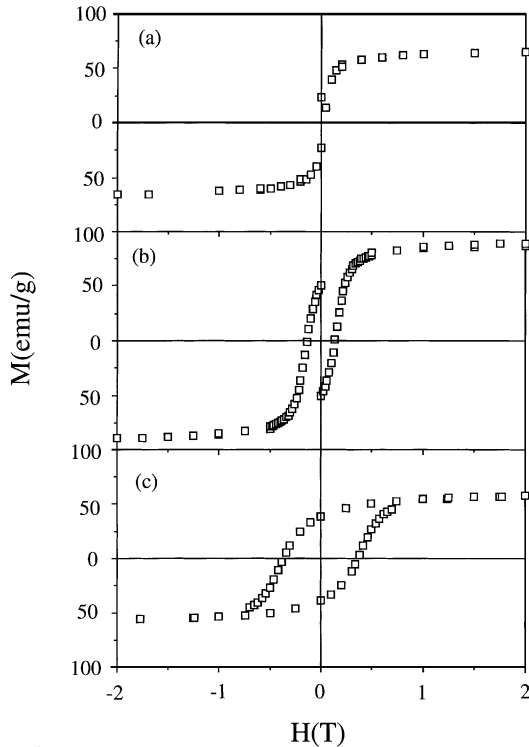


Fig. 2. Hysteresis loops at 3 K for 3.7 nm particles: $y = 0$ (a), $y = 0.10$ (b) and $y = 0.40$ (c).

Table 1

Coercive field H_C , ratio of remanence to saturation magnetizations M_R/M_S and saturation magnetization M_S , determined at 3 K for 3.7 nm diameter particles, at various y values

Y	0	0.10	0.40
H_C (Oe)	310	1390	3650
M_R/M_S	0.34	0.54	0.65
M_S	68.8	94.0	62.7

increase in M_R/M_S above 0.5 indicating a cubic anisotropy. Hence, even for a rather small amount of cobalt included in the zinc ferrite matrix the anisotropy of the material evolves from axial to cubic anisotropy. This is due to the strong cubic magnetocrystalline anisotropy of cobalt ions in octahedral sites.

The ferromagnetic resonance (FMR) spectra of 3.7 nm nanocrystals were recorded at various temperatures from 125 to 275 K. Fig. 3 shows a

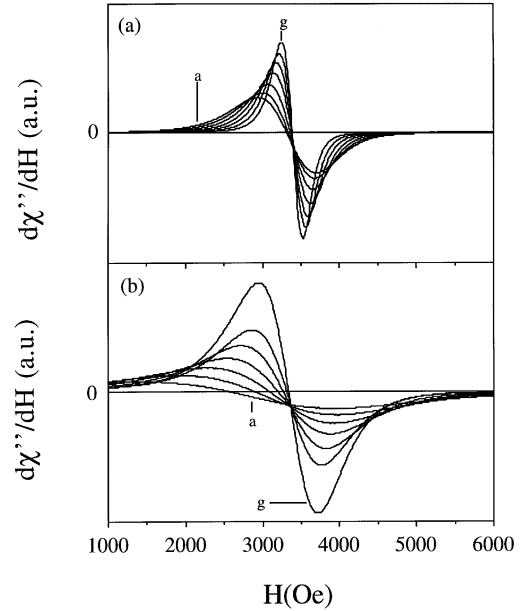


Fig. 3. FMR spectra of 3.7 nm diameter ferrite nanoparticles dispersed in PVA, at various temperatures a to g : 125–275 K with 25 K steps, for two compositions: $y = 0$ (a) and $y = 0.10$ (b).

drastic change in the derived FMR spectra for nanocrystals in the absence of cobalt ions (Fig. 3(a)) or in presence of 10% cobalt ions ($y = 0.10$) (Fig. 3(b)) in the zinc ferrite matrix. Similar behavior is observed with a linewidth broadening and shift in the resonance toward lower fields with decreasing temperature. These behaviors markedly increase with zinc ferrite nanocrystals containing 10% cobalt ions. From the data shown in Fig. 3, an invariant point is observed at a given field called H_0 . The derived FMR spectra can be fitted by

$$\frac{\partial \chi''}{\partial H} = -\frac{C(H - H_R)}{\Delta H_{PP}^3} f\left(\frac{H - H_R}{\Delta H_{PP}}\right) \quad (1)$$

where χ'' is the imaginary part of the susceptibility and C a factor directly related to the spectrum intensity. ΔH_{PP} and H_R are the experimental peak-to-peak linewidth and resonance field, respectively. $f(x)$ is a combination of functions $f_L(x)$ and $f_G(x)$ resulting from Lorentzian and Gaussian shapes

$$f_L(x) = -\frac{8}{3}\left(1 + \frac{4}{3}x^2\right)^{-2},$$

and

$$f_G(x) = -4 \exp(-2x^2). \quad (2)$$

The spectra are normalized to $\chi''(0) = C/\Delta H_{PP}$ for the Lorentzian and Gaussian shapes. In this case the combination is verified to be 50% Lorentzian and 50% Gaussian for all temperatures.

Because $H_0 - H_R$ is too small with zinc ferrite nanocrystals, in the following calculation we take into account data obtained when 10% cobalt ions were included in the zinc ferrite matrix (Fig. 3(b)).

The C value is derived, using Eq. 1, from the best fit of the FMR spectrum. This value is obtained at various temperatures. Fig. 4(a) shows the variation in the log–log scale of C versus ΔH_{PP} . Assuming $H_0 = 3380$ Oe, the ratio $C(H_0 - H_R)/\Delta H_{PP}^3$ is constant. Since $f((H_0 - H_R)/\Delta H_{PP}) = 1$ for $H_0 - H_R \ll \Delta H_{PP}$, H_0 is an invariant point. The H_0 value (3380 Oe) is approximately the value at the invariant point observed in Fig. 3(b). Of course, the H_0 value determined from Fig. 3(b) has some

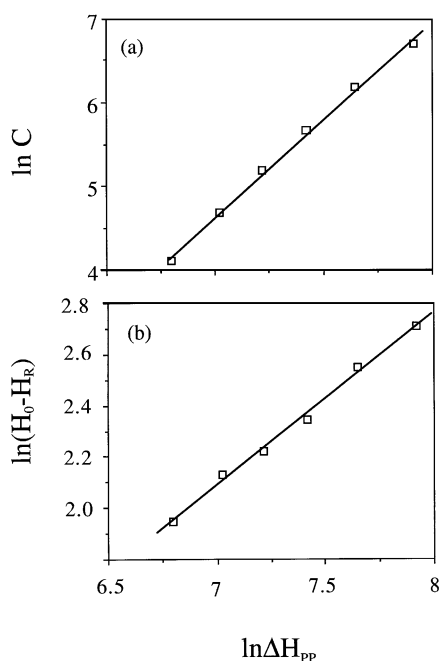


Fig. 4. $\ln C$ versus $\ln \Delta H_{PP}$ (a), $\ln(H_0 - H_R)$ versus $\ln \Delta H_{PP}$ (b).

uncertainty (around 30 Oe). This is in the experimental error.

The log–log plot of $H_0 - H_R$ versus ΔH_{PP} , in Fig. 4(b) shows linear behavior with a slope of 2.32. This exponent differs from that determined by Nagata and Ishihara [8]. They claimed that the exponent is 3 when nanocrystals are randomly dispersed and 2 under texturation (particles submitted to a magnetic field during the sample preparation). This difference in the exponent for randomly dispersed particles can be attributed to the value of the limiting resonance field that was chosen. In fact from our experimental data, the ΔH_{PP} exponent is 2.63 and 2.21 for limiting resonance fields of 3360 and 3390 Oe, respectively. In any case, whatever the exact H_0 value determined in the experimental error range, the exponent values are always smaller than that determined by Nagata & Ishihara [8]. This is due to the resonance shift linked to the strength of the material anisotropy, whereas Nagata considered only the influence of demagnetizing fields due to the shape of the particles.

The presence of an invariant point in the set of spectra for the two samples (with and without cobalt ions in the zinc ferrite matrix) indicates a universal behavior.

Zinc ferrite nanocrystals coated with lauric acid were dispersed in non polar solvent as isoctane ($T_g = 166$ K). The solution was frozen down to 100 K in an applied magnetic field of 1.2 T. FMR spectra were recorded at various temperatures and Θ values (Θ being the angle between the direction of the supposed aligned axes and the freezing field). As expected for a uniaxial anisotropy [5–7], Fig. 5 shows that the resonance field varies with $\sin^2 \Theta$.

At temperature T , the anisotropy constant K is derived from the equation [6]

$$\begin{aligned} H_R(90^\circ) - H_R(0^\circ) &= \frac{3K(T)}{M_s(T)} P_2(\cos \theta) \\ &= \frac{3K(T)}{M_s(T)} f(K(T_g), T_g), \end{aligned} \quad (3)$$

where $H_R(90^\circ)$ and $H_R(0^\circ)$ are the resonance fields for $\Theta = 90^\circ$ and 0° , respectively. θ is the angle between the easy axis of a particle and the direction of the cooling field. $M_s(T)$ is

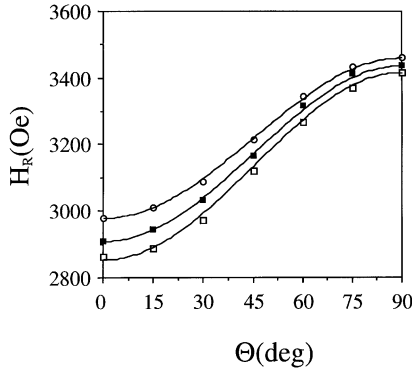


Fig. 5. Angle dependence of resonance field for textured $y = 0$ nanoparticles. \square : 100 K, \blacksquare : 120 K, \circ : 150 K. line: fit using a $\sin^2 \theta$ dependence.

the saturation magnetization at temperature T . The variation of saturation magnetization with temperature was recorded at 5 T. It follows Bloch's law as shown in Fig. 6.

$$M_s(T) = M_s(0)(1 - (T/T_0)^{1.5}),$$

with $T_0 = 400$ K and $M_s(0) = 68.8$ emu/g. Hence the saturation magnetization at the freezing temperature, T_g , is deduced from Fig. 6. $f(K(T_g), T_g)$ is the calculated disorder parameter. It is due to the fact that the particles are not perfectly aligned. It depends on the solvent freezing temperature, T_g , on the anisotropy constant at T_g and on the field used to texture the sample.

Taking into account the Saenger et al. calculation [7] and by correcting $\langle \cos^2 \theta \rangle$ into $\langle (3\cos^2 \theta - 1)/2 \rangle$, the disorder factor is

$$f(K(T_g), T_g) = \frac{3}{2} \left[\frac{L(b)}{b} - \left(\frac{3L(b)}{b} - 1 \right) \times \left(\frac{\exp(a)}{aI(a)} - \frac{1}{2a} \right) \right] - \frac{1}{2} \quad (4)$$

with $a = K(T_g)\langle V \rangle / k_B T_g$, $b = M_s(T_g)\langle V \rangle H / k_B T_g$, $L(b) = \coth(b) - 1/b$ and

$$I(a) = \int_{-1}^1 \exp(ax^2) dx.$$

As observed in Eq. (4), to calculate the disorder factor we need to know the anisotropy constant at the freezing temperature. To determine this value, $H_R(90^\circ) - H_R(0^\circ)$ was determined experimentally

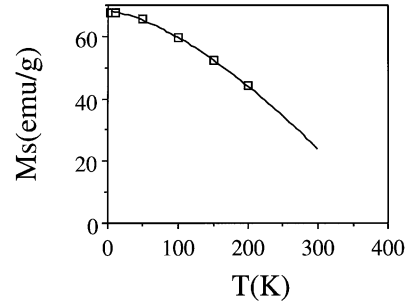


Fig. 6. Thermal variation of saturation magnetization for 3.7 nm particles ($y = 0$).

at T_g . The saturation magnetization was determined from Bloch's law (see above). The anisotropy constant $K(T_g)$ and the disorder factor $f(K(T_g), T_g)$ were deduced from the best fit of Eq. (3). The variation of the anisotropy constant with temperature (Eq. 3) can be derived from the constant value of the disorder parameter $f(K(T_g), T_g)$.

Fig. 7(a) shows the variation of the anisotropy constant with temperature of 3.7 nm nanocrystals dispersed in isooctane. It suggests a linear dependence of the anisotropy constant with temperature. However because of the low freezing temperature of isooctane, the temperature interval is narrow. This prevents from establishing a general behavior. Cyclohexane is characterized by a high freezing temperature (277 K). So similar experiments as described above are performed in cyclohexane in order to investigate a broader temperature range. However, because of the superparamagnetic behavior of the nanocrystals the texturation is very weak. The evaluation of the texturation parameter is not as reliable as in isooctane. This explains the difference in the anisotropy constant calculated values when replacing isooctane by cyclohexane. Similar behavior is observed with nonane (Fig. 7(a)). The relative variation $(H_R(90^\circ) - H_R(0^\circ))M_s(T)$ is considered. The extrapolated $K(0)$ values are normalized to that obtained for isooctane. Fig. 7(b) shows a linear decrease of the anisotropy constant with temperature, in the range 100–300 K

$$K(T) = K(0)(1 - T/T_A),$$

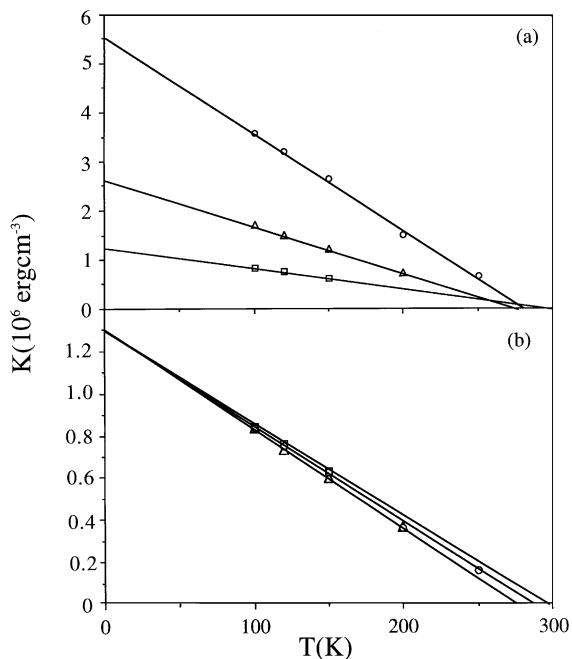


Fig. 7. Calculated anisotropy density versus temperature for $y = 0$ particles, from FMR measurements. (a) \square : particles ($\langle V \rangle = 36 \text{ nm}^3$) in isooctane; \circ : cyclohexane; \triangle : nonane. (b) Curves normalized with $K(0)$ deduced from the experiment in isooctane.

where $K(0)$ and T_A are the extrapolated anisotropy constant at 0 K and the temperature corresponding to a zero anisotropy.

T_A is found close to 280 K. This low value is attributed to a decrease in the magnetic interactions between magnetic ions when nonmagnetic ones are included in the spinel structure of ferrite nanocrystals.

The good agreement between the relative thermal variations in different solvents confirms a rather sharp decrease of anisotropy constant with temperature. This has to be considered when determining anisotropy constants with other techniques (Table 2). However, it is rather difficult to get an accurate value of the anisotropy constant, found around $1.3 \times 10^6 \text{ erg/cm}^3$ by the experiment in isooctane. This is attributed to the fact, the evaluation of a disorder parameter is not accurate for poorly textured samples, and experimental causes as interactions between

Table 2

Anisotropy constants extrapolated at 0 K (in 10^6 erg/cm^3) determined by ZFC magnetization, thermal variation of hyperfine field and ferromagnetic resonance measurements, for 3.7 nm diameter zinc ferrite particles

	FMR	ZFC	Mössbauer
$K(0)$	1.3	3.5	2.0

particles, the mechanism of solvent freezing, distribution of anisotropy constants and saturation magnetizations of the particles in the same sample. Hence, we conclude FMR measurements on textured samples give reliable information about the relative thermal variation of the anisotropy constant, and the anisotropy constant can be roughly evaluated. The order of magnitude found by this technique is the same as found by ZFC or Mössbauer experiments (Table 2). In fact experiments using various solvent freezing temperatures are a crucial test for the procedure. Other groups generally use only one solvent to deduce the anisotropy constant [5–7]. No comparisons are performed and the accuracy of the obtained value is not questioned. Here, we claim the procedure needs comparisons between various freezing temperatures to estimate the accuracy of the model.

For zinc ferrite nanocrystals containing 40% cobalt ions ($y = 0.4$) the ratio of remanence over saturation magnetizations is 0.65 (Fig. 2(c)). This high value is characteristic of a cubic anisotropy. For a cubic anisotropy the resonance is, for an applied field oriented at angles (θ, ϕ) with respect to the cubic crystal axes [11,12].

$$H_R(\theta, \phi) = \frac{\omega}{\gamma} - H_A \left(1 - \frac{5}{4} \sin^2(2\theta) - \frac{5}{4} \sin^4 \theta \sin^2(2\phi) \right),$$

where ϕ and H_A are the azimuth and the anisotropy field respectively, ω is the pulsation of the spectrometer and γ the gyromagnetic ratio. After averaging the resonant field is

$$H_R(\theta) = \frac{\omega}{\gamma} - H_A \left(1 - \frac{5}{4} \sin^2(2\theta) - \frac{5}{8} \sin^4 \theta \right). \quad (5)$$

If the easy axes are well oriented by the field (efficient texturation) there is no θ distribution in the sample and $\theta = \Theta$.

The resonance field variation with Θ angles is recorded for samples subjected during the preparation either to a parallel or a perpendicular field with respect to the sample plane (see details above). Good agreement between the experimental data and a simulated $\sin^2\Theta$ dependence is observed (Fig. 8). This is rather surprising because the $\sin^2\Theta$ dependence was always linked to a uniaxial anisotropy [5–7]. From these data it can be concluded that the $\sin^2\Theta$ dependence is not a signature of an axial anisotropy.

A marked change in the resonance field with the orientation of the applied field imposed during the sample preparation (texturing field) is observed. This can be attributed to a demagnetizing field in the perpendicular configuration. However, a difference in the efficiency of the alignment of easy axis cannot be excluded.

The fact the law expected for cubic anisotropy is not found experimentally is attributed to orientation disorder of easy axes. To support such claim, a simple model is used. It is considered that:

- (i) the individual signal is Gaussian with width ΔH_{pp} .
- (ii) after texturation (at $\Theta = 0$) we have a two-dimensional orientation distribution of z -axes: $f(\theta) = 2/\alpha$ for $-\alpha \leq \theta \leq \alpha$ and $f(\theta) = 0$ for $|\theta| > \alpha$.

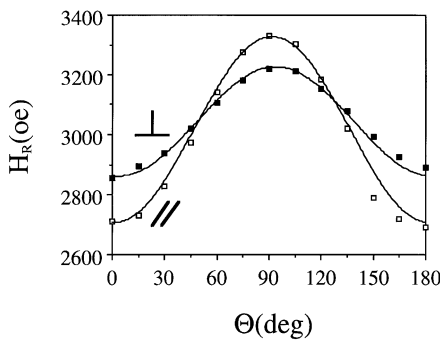


Fig. 8. Angle dependence of resonance field for texturated $\gamma = 0.40$ nanoparticles at room temperature, for texturation parallel and perpendicular to the disk plane.

Of course, the real distribution is quite different since the distribution of z -axes is in a cone following a Boltzmann statistics, but this complication is not required for the demonstration. If $S(\Theta)$ is the spectrum at angle Θ , we have

$$S(\Theta) = \int_{-\alpha}^{\alpha} f(\theta)s(\Theta + \theta) d\theta$$

with in the case of Gaussian shape

$$s((\Theta + \theta), H) = -C \frac{H - H_R(\Theta + \theta)}{\Delta H_{pp}^2} \times \exp\left(-2\left(\frac{H - H_R(\Theta + \theta)}{\Delta H_{pp}}\right)^2\right). \tag{6}$$

From simulation the resonance field of spectrum $S(\Theta)$ is deduced. The H_A and ΔH_{pp} values are chosen as 500 and 1600 G, respectively. These values are close to the experimental data. ω/γ is chosen as 2800 G. This has no consequence on Θ dependence. Fig. 9 shows the evolution of the resonance field H_R versus Θ with $\alpha = 30^\circ$ and 45° as compared to the function expected for perfect alignment. With $\alpha = 30^\circ$ the function $H_R(\Theta)$ still exhibits the same behavior as for perfect alignment, whereas for $\alpha = 45^\circ$ it fits with a $\sin^2\Theta$ law. This clearly shows that for nanoparticles a $\sin^2\Theta$ law is not necessarily the signature of a uniaxial anisotropy. Angular disorder of easy axes after

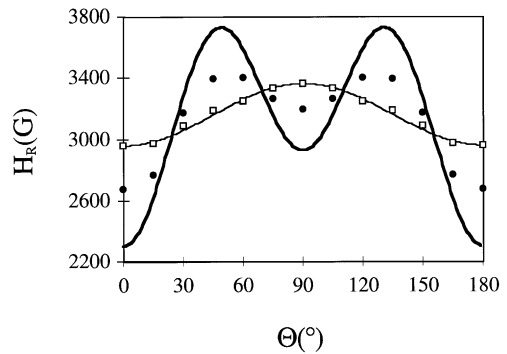


Fig. 9. Simulated angular dependence of resonance field for a cubic anisotropy: thick line: case of perfect alignment of easy axes; \bullet : distribution of easy axes with $\alpha = 30^\circ$; \square : $\alpha = 45^\circ$, fitted with a $\sin^2\theta$ law.

texturation induces a significant change in the angular dependence of the resonance field and leads to uniaxial-like behavior. Hence the evaluation of the anisotropy constant of poorly textured samples is still an open question, since our simple model is only sufficient to illustrate the effect of the orientation disorder as far as the angular dependence of the resonance field is concerned.

In this paper, we demonstrate that the derived FMR spectra of zinc ferrite with or without cobalt ions are characterized by an invariant point at a given field, H_0 . This makes it possible to establish an equation relating the resonance shift and the linewidth: $H_0 - H_R$ is proportional to $(\Delta H_{PP})^{2.3}$.

Like several groups, we use the angular dependence of FMR spectra of textured samples to measure the anisotropy constant of zinc ferrite nanoparticles (3.7 nm diameter). We show that the anisotropy constant varies linearly with temperature (in the range 100–300 K) and vanishes around 300 K. Nevertheless, we claim the evaluation of the anisotropy constant using the procedure of Gazeau [6] requires experiments at various freezing temperatures, in order to estimate the accuracy of the deduced values.

We demonstrated that the $\sin^2\theta$ variation, for the angular dependence of the resonance field of textured samples, can be obtained for poorly textured cubic anisotropy nanocrystals and is not linked, contrary to what was claimed before [5–7], to the uniaxial anisotropy of the material.

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