Rev.Adv.Mater.Sci. 14(2007) 57-60

FMR STUDY OF γ-Fe_2O_3 AGGLOMERATED NANOPARTICLES DISPERSED IN GLUES

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Received: December 08, 2006

Abstract. Four samples of γ-Fe_2O_3 magnetic nanoparticles (size 10 nm) in an agglomerates state (size 100 μm) dispersed in different glues: TEC 4185H modified acrylic (sample 1), TEC 4185 acrylic (sample 2), Durotak 280-122A acrylic PSA (sample 3), and Aroset 8084 acrylic PSA (sample 4), have been investigated by ferromagnetic resonance (FMR) method at room temperature. To study the influence of agglomerate size on the FMR spectrum, two additional samples have been prepared with aggregates of 2 mm and 100 nm in diameter. Very intense and broad FMR Lorentzian lines have been recorded for all six studied samples. The values of the resonance lines are quite similar for all samples; they were characterized by g_{eff}=2, indicating that the internal magnetic field is almost zero. This is in contrast with agglomerates embedded in harder matrices, for which a larger shift had been observed.

1. INTRODUCTION

One of the interesting problems in the physics of magnetic ordering processes is the shift of resonance lines produced by nanoparticles and observed by resonance methods, like electron paramagnetic resonance (EPR), spin-muon paramagnetic resonance (SPR) or ferromagnetic resonance (FMR) [1-5]. For separated atoms or ions, at temperatures higher then magnetic ordering, the resonance field is changing linearly with decreasing temperature while for magnetic nanoparticles the temperature ratio of the line shift $\Delta B/\Delta T$ could be greater over one order of magnitude [1,5]. Magnetic nanoparticles dispersed at low concentration in a non-magnetic matrix could provide valuable information about magnetic interactions between magnetic moments and additionally offer the insight into dynamical processes in the matrix. This way the idea has arisen to characterize different materials by using low concentration of magnetic nanoparticle or agglomerates at room temperatures. The FMR measurements of hard segment of a copolymer have shown that the resonance field and the linewidth strongly depended on the concentration as well as on the state of used agglomerates [5]. It would be very interesting to study the behavior of these agglomerates in an environment mechanically softer than previously used matrixes.

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Glues would ideally suit these requirements because they are multiphase and very soft at room temperature.

The aim of this report is the FMR investigation of different types of glues filled by γ-Fe_2O_3 nanoparticles in agglomerated state. The used glues were: TEC 4185H modified acrylic, TEC 4185 acrylic, Durotak 280-122A acrylic PSA, and Aroset 8084 acrylic PSA.

2. EXPERIMENTAL

Four samples of magnetic γ-Fe_2O_3 nanoparticles (size 10 nm) in agglomerated state (size 100 μm) mixed in different glues: TEC 4185H modified acrylic (sample 1), TEC 4185 acrylic (sample 2), Durotak 280-122A acrylic PSA (sample 3), and Aroset 8084 acrylic PSA (sample 4), have been synthesized. Moreover, two additional samples for studying the effect of agglomerate size have been prepared: one with a large agglomerate ~2 mm (sample 1'), the other with small agglomerates ~100 nm (sample 1"). The concentration of magnetic particles in glues was about 2%. The process of the samples preparation was presented in previous works [6,7]. The following four different glues have been synthesized:

– TEC 4185 H modified acrylic PSA with the following content: 51.0 wt.% 2-EHA, 45.5 wt.% BA, 3.5 wt.% AA and added 30.0 wt.% polyterphene phenolic resin to the polymer mass (sample 1);
– TEC 4185 acrylic PSA with the content: 51.0 wt.% 2-EHA, 45.5 wt.% BA, 3.5 wt.% AA (sample 2);
– Durotak 280-122A (ICE) acrylic (pressure-sensitive) consisting of 66.5 wt.% 2-ethylhexyl (2-EHA), 33.0 wt.% methyl acrylate (MA), 0.5 wt.% acrylic acid (sample 3);
– Aroset 8084 acrylic PSA containing 45 wt.% 2-EHA, 30 wt.% butyl acrylate (BA), 20 wt.% MA, 5.0 wt.% AA (sample 4).

Ferromagnetic resonance measurements were carried out on a conventional X-band (ν=9.43 GHz) Bruker E 500 spectrometer with 100 kHz magnetic field modulation. The measurements were performed at room temperature. A square-shaped sample of 3.0$\times$1.0 mm^2 cut out from a polymer sheet was attached to a sample holder made of a quartz rod 4 mm in diameter. The sample holder was accommodated in the center of the TE_{102} cavity, i.e., at the local maximum of the microwave magnetic component H_1 and in the nodal plane of the electric component E_1. Prior to measurements, samples have been magnetized by a steady magnetic field of 1.6 T to saturate any domain structure.

3. RESULTS AND DISCUSSION

Fig. 1 presents the FMR spectra at room temperature of four samples containing different glue ma-

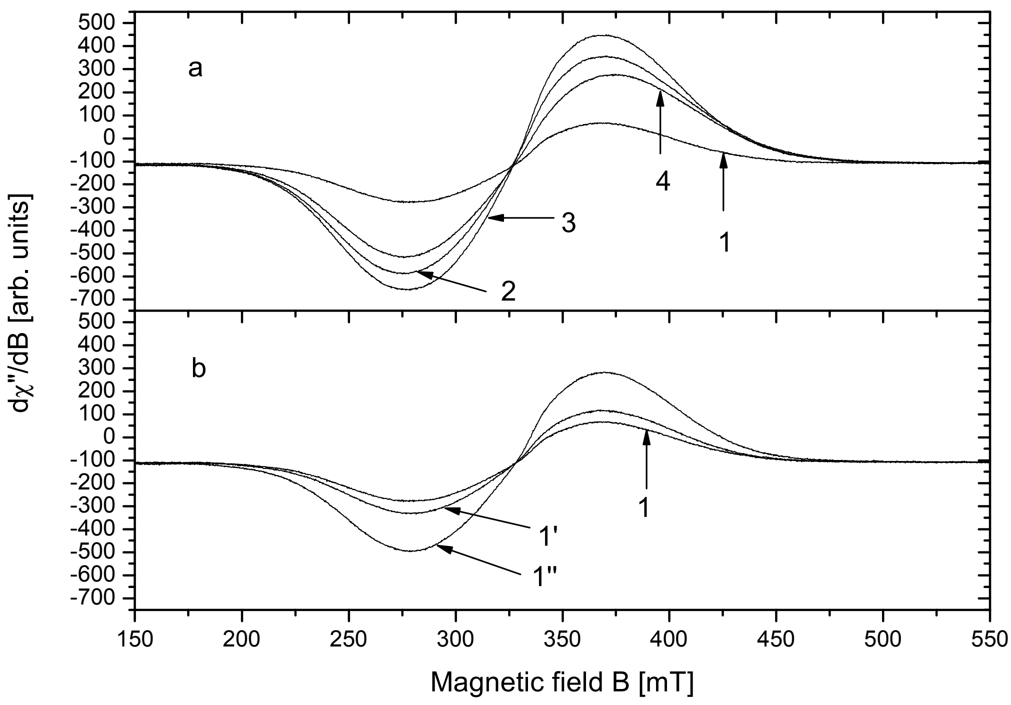

Fig. 1. The FMR spectra of samples (a) 1, 2, 3, 4, and (b) 1, 1’, 1”.

Table 1. The values of the FMR parameters for all six investigated samples at room temperature and, for comparison, for samples I and II. Sample I contains 0.1 wt.%, and sample II 0.3 wt.% of γ-Fe_2O_3 nanoparticles dispersed in a poly(ether-ester) multiblock copolymer ((PEN and PTT)- block- PTMO) [8].

Sample designation	Resonance field B_{res} [mT]	Effective *g*-factor g_{eff}	Linewdth ΔB [mT]	Relative intensity I_{int}	Line shift ΔB_{res} [mT]
1	326.2	2.044	86.3	1.0	4.8
1’	326.4	2.043	86.7	1.3	4.6
1”	330.7	2.016	87.1	2.3	0.3
2	325.1	2.051	91.1	3.3	5.9
3	325.4	2.049	89.0	2.9	5.6
4	326.9	2.040	93.7	3.0	4.1
I	299	2.256	121.3	1.0	36.8
II	307	2.199	137.6	3.0	29.9

$B_{res}(g=2)$ = 331.0 mT; $\Delta B_{res}=B_{g=2}-B_{res}$

trixes (Fig. 1a) and three samples with different agglomerate sizes (Fig. 1b). A slightly asymmetrical and very intense FMR line is recorded for all these samples. The registered FMR spectra differ slightly and depend on magnetic nanoparticle agglomerate size as well as on kind of glue. The experimental FMR spectra have been fitted reasonably well by a single Lorentzian function (Fig. 2) and the obtained spectral parameters are given in Table 1. The following spectral parameters have been introduced: B_L, B_R, and $B_{res}=(B_L+B_R)/2$, where they are the left, the right extremes of the derivative resonance line, and the effective resonance field, respectively [2]. Table 1 shows the values of the resonance field B_{res}, the peak-to-peak linewidth $\Delta B=B_R-B_L$ and the integrated intensities $I_{int}=I\cdot\Delta B^2$ (where I is the derivative line amplitude) for all samples. Comparison of the resonance fields demonstrates that they are almost similar in value and centered near g_{eff}=2 ($B_{res}(g=2)$ =331 mT). Almost

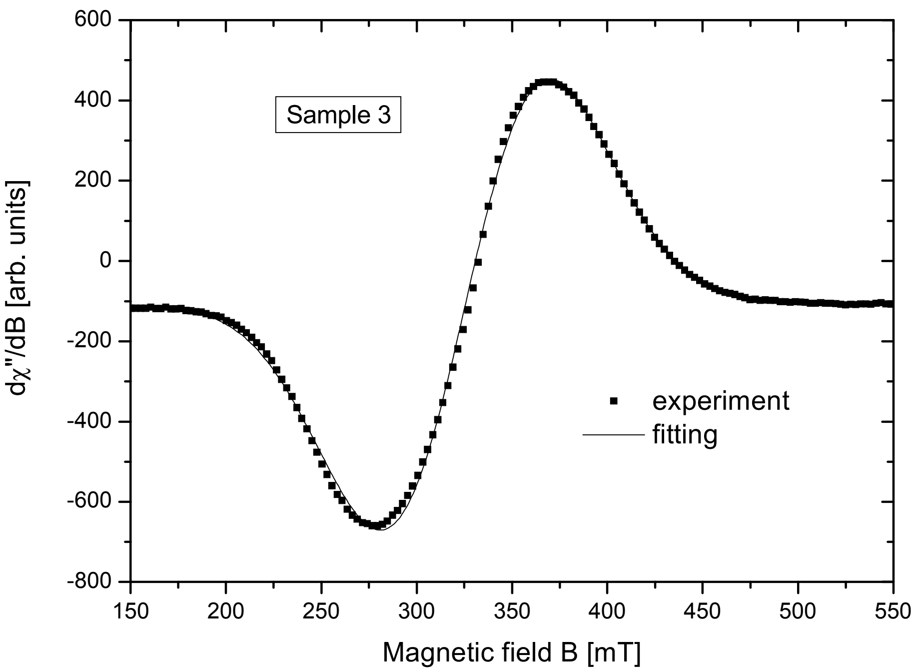

Fig. 2. Fitting of the FMR spectrum of sample 3 by Lorentzian function.

the same value of the resonance field is obtained for samples 1 and 1', which differ greatly in the size of agglomerates (100 μm and 2 mm, respectively). The value of an average internal magnetic field inside agglomerate is calculated as $\Delta B_{res}=B_{g=2}-B_{res}$, what in that case amounts to 4.7 mT. For smaller agglomerates (sample 1") the internal field is even smaller (0.3 mT) (Table 1). Slightly larger internal fields are measured for samples 2 and 3 (5.9 mT and 5.6 mT, respectively). For sample 4 the field is also in the same range. It is a very interesting result which could be compared with previously studied samples I and II, constituting γ-Fe_2O_3 nanofillers in a polymer matrix [2-5,8]. For the latter case, the internal field was significantly larger (~30 mT). This indicates that for a softer matrix (glue) the average internal magnetic field is smaller, probably due to lower disorder of magnetic particles. Antiparallel arrangement of magnetic dipoles forming agglomerates causes cancellation of internal magnetic fields. As the size of agglomerates is concerned, it seems that below a certain threshold value (~1 mm) the internal field drops significantly as a result of more systematic field cancellation. Slight differences in the values of the FMR resonance line positions for various kinds of investigated matrices could be related to the variations of their crystallization temperatures, which is the lowest for sample 2 and the highest for sample 4.

4. CONCLUSIONS

It has been shown that in a softer matrix (glue) the internal magnetic field, as seen by the FMR spectroscopy, is significantly weaker than in harder matrix (polymer). This might be due to the cancellation of the dipole fields produced by the agglomerates. The size of agglomerates is also important in this cancellation as it is more effective for agglomerates smaller than 1 mm. The crystallization temperature of the matrix could influence spectral parameters of the FMR spectrum of agglomerates.

ACKNOWLEDGEMENTS

This work was partially supported by grant ΓΓΕΤ 165ε.

REFERENCES

[1] H.Gamari-Seale, N.Guskos, A.Koufoudakis, I.Kruk, C.Mitros, V.Likodimos, D.Niarchos and V.Psycharis // *Phil. Mag.* **B 65** (1992) 1381.
[2] Yu. A. Koksharov, S. P. Gubin, I. D. Kosobudsky, G. Yu. Yurkov, D. A. Pankratov, L. A. Ponomarenko, M. G. Mikheev, M. Beltran, Y. Khodorkovsky and A. M. Tishin // *Phys. Rev. B* **63** (2000) 012407.
[3] R. Berger, J. C. Bissey, J. Klava, H. Daubric and C. Estournes // *J. Magn. Magn. Mater.* **234** (2001) 535.
[4] N. Guskos, J. Typek, M. Maryniak, Z. Roslaniec, D. Petridis and M. Kwiatkowska // *Mat. Science-Poland* **23** (2005) 971.
[5] N. Guskos, V. Likodimos, S. Glenis, J. Typek, M. Maryniak, Z. Roslaniec, M. Baran, R. Szymczak, D. Petridis and M. Kwiatkowska // *J. Appl. Phys.* **99** (2006) 084307.
[6] Z. Czech // *Eur. Coating J.* **5** (2005) 31.
[7] Z. Czech // *J. Appl. Polym. Sci.* **97** (2005) 886.
[8] N. Guskos, J. Typek, M. Maryniak, Z. Roslaniec, D. Petridis and M. Kwiatkowska // *Mat. Science-Poland* **23** (2005) 971.