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Magnetic properties of $Fe_{90}Zr_7B_3$ ribbons studied by FMR and magnetization

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Abstract

The amorphous alloy $Fe_{90}Zr_7B_3$ containing alpha-iron nanocrystallites (known commercially as NANOPERM®) is produced in ribbons by melt spinning and presents magnetic properties that allow its wide applicability in magnetic shielding, low loss RF transformers, choke coils, magnetic sensors and flux-gate magnetometers. In this work, we present a study of the effect of the annealing process realized at different temperatures on the magnetic properties of this material. Comparisons of experimental results of ferromagnetic resonance (FMR) and magnetization measurements show that the precipitation of alpha-iron phase causes the reduction of the coercive field and the in-plane magnetic anisotropy by about 70% in ribbons annealed for 1 h at temperatures between 250 and 350 °C.

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Keywords: Nanoperm; FeZrB; FMR; Anisotropy field; Coercive field

1. Experimental and discussion

In this paper we present the recent results of our analysis of $Fe_{90}Zr_7B_3$ amorphous alloy produced by the meltspinning [1]. The effect of annealing on the precipitation of a crystalline phase in this material was studied using ferromagnetic resonance (FMR) and magnetization.

Nine different samples were prepared starting from a $Fe_{90}Zr_7B_3$ ribbon (thickness ~25 μm). Eight of these samples were annealed during 1 h at 200, 250, 300, 350, 400, 450, 550 and 650 °C in an argon atmosphere (~0.3 bar) to avoid contamination and the last one was left in the received state to be used as a reference.

In order to determine the composition of the samples, X-ray diffraction (XRD) experiments were performed using a HZG 4 diffractometer equipped with a Seifert ID 3000 X-ray generator (Cu anode, 40 kV, 40 mA) and the

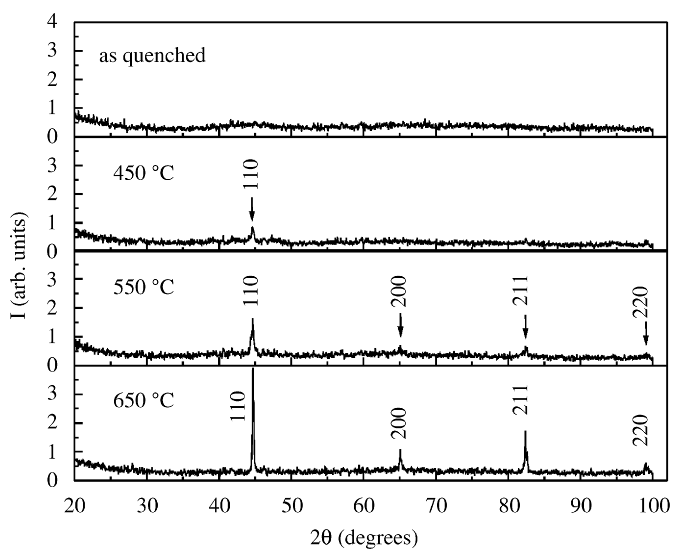

Fig. 1. The X-ray diffractogram of as quenched sample does not reveal any crystallinity. Otherwise, the diffractograms of samples annealed at 450, 550 and 650 °C only reveal the presence of α-Fe phase.

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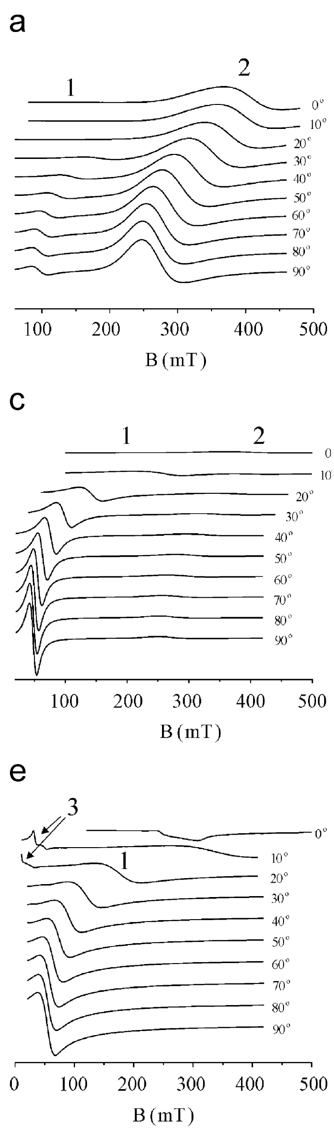

Fig. 2. Out-of-plane FMR spectra of as quenched (a) and samples annealed at 250 (b), 350 (c), 450 (d), 550 (e), 650 °C (f). Some samples were omitted to not overcharge the figure. The orientation angles were measured between the normal vector of the samples and the field direction. The spectral line marked with 1 is attributed to α-Fe phase and the line marked with 2 is due to the amorphous phase. For samples annealed at 550 °C the spectra present DARMA peaks (marked with 3).

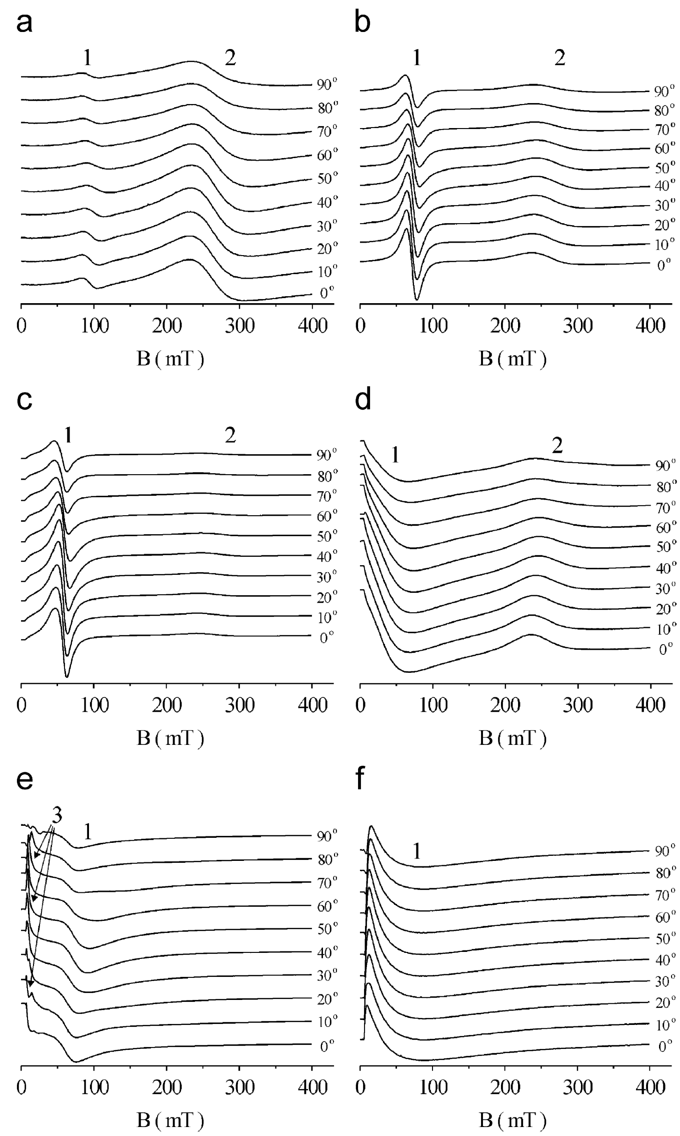

Fig. 3. In-plane FMR spectra of as quenched (a) and samples annealed at 250 (b), 350 (c), 450 (d), 550 (e) and 650 °C (f). Some samples were omitted to not overcharge the figure. The angles were measured between the ribbon axis and the field direction. Note the presence of DARMA peaks (marked with 3) in the spectra of sample annealed at 550 °C.

diffractograms were analyzed with the help of programs DRX-Win and PCPDF-Win. As shown in Fig. 1, α-Fe ($a = 2.886\,\text{Å}$) is the only crystalline phase observed in the diffractograms, even those corresponding to the samples annealed at higher temperatures (550 and 650 °C). The diffractogram corresponding to the as-quenched sample do not reveals any crystallinity.

The magnetic behavior of material was analyzed by FMR in the X-band (9.52 GHz) using a Varian E-9 spectrometer. Figs. 2 and 3 show the FMR spectra (first-derivative of RF absorption with respect to the field) in arbitrary units recorded for different samples and orientations. All FMR measurements were performed at room temperature using the following parameters: RF power = 2 mW, modulation frequency = 10 kHz, modulation amplitude = 2.5 G, scan time = 120 s and time constant = 300 ms. We can note that the FMR spectra of the samples are the superposition of two main spectral lines, which occur at different resonance fields. At lower fields we observe the spectral line attributed to the α-Fe precipitates (marked with one), while at higher fields we observe the line due to amorphous matrix (marked with two). As expected, the ratio of relative intensities of these two lines increases with the annealing temperature, confirming the results of XRD. We also observe DARMA peaks (marked with three) in the spectra of the sample annealed at 550 °C, confirming the results obtained for other authors, which studied a similar alloy [2]. These peaks are due to the initial magnetization process of the sample.

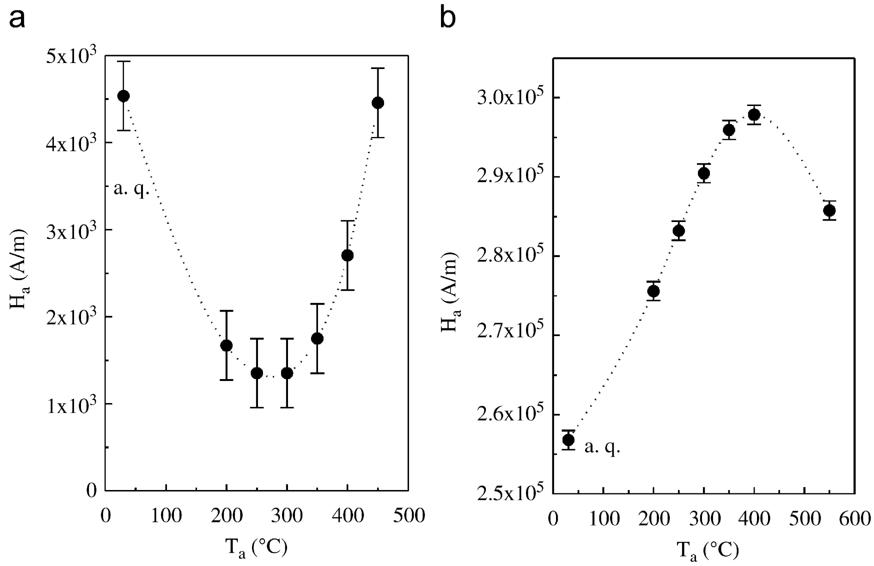

Fig. 4. In-plane (a) and out-of-plane (b) anisotropy fields obtained from the angular variation of FMR spectra corresponding to the crystalline phase as function of annealing temperature. In both these figures a. q. denotes the as-quenched sample and the dashed curves are only guides for the eyes.

As the microwave penetration depth in Fe-based alloys is about 1 μm in both sides of ribbon, the magnetic anisotropy (and other physical properties) observed by FMR are more influenced by the compressive mechanical stress present in the sample's superficial layer than tensile stress that occurs in its central layer [3]. This feature should be kept in mind when comparisons of these data with those obtained by DC magnetization are made.

The anisotropy field was obtained from FMR spectra by angular variation of the sample position [2] in two different ways: with the magnetic field applied out of sample's plane and with the field lying in the sample's plane (see Figs. 2 and 3). In both cases, this quantity was calculated by taking the difference between the maximum and minimum value of resonance field of spectral line attributed to α-Fe phase. Due to the low degree of orientation of crystallites, the macroscopic effect of its magnetocrystalline anisotropy is very small in comparison with the shape anisotropy. This feature may be clearly noted in Figs. 2 and 3 by comparing the spectra measured at different orientation angles.

As shown in Figs. 2 and 4b, the out-of-plane shape anisotropy achieves its maximum value for samples annealed at temperatures around 400 °C. For higher annealing temperatures, the anisotropy starts to decrease. This behavior may be explained by considering the combined effects of the critical size of crystallites in which they become multi-domain (about 50 nm for α-Fe) and the clustering effects. Both these phenomena may contribute to decrease the demagnetizing field and therefore, the shape anisotropy. The average crystallites diameter calculated using the Scherrer relation for samples annealed at 550 and 650 °C were 20 and 45 nm, respectively, thus, we may suppose that the greater crystallites may easily overcome the critical size.

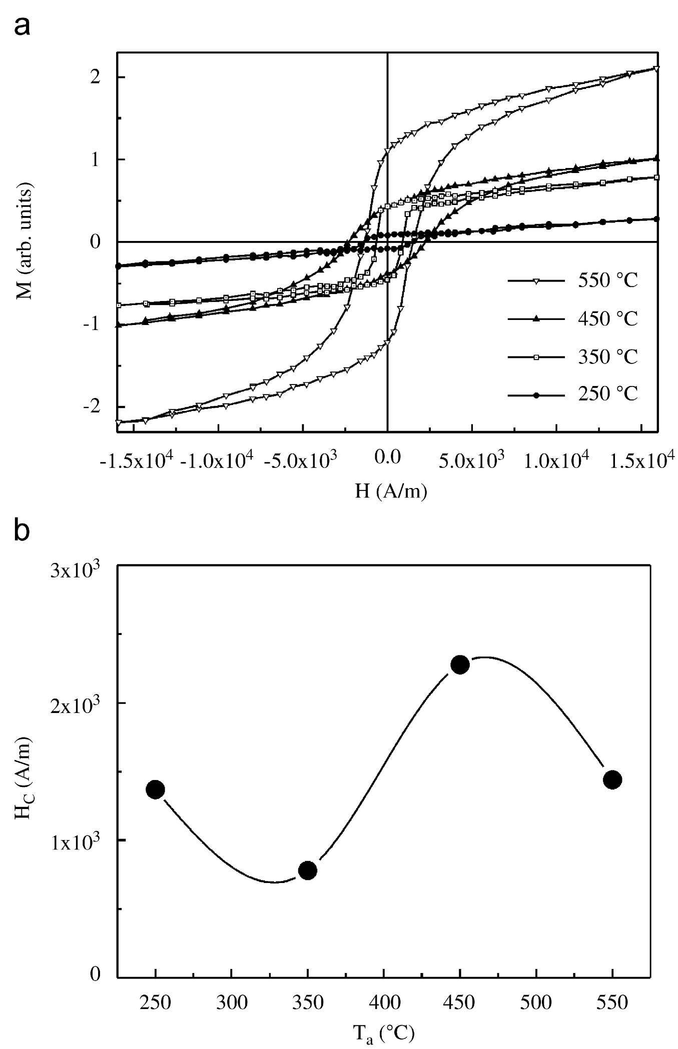

Fig. 5. In-plane hysteresis loops (a) and coercive fields (b) obtained for samples annealed at different temperatures. The continuous curves are only guide for the eyes.

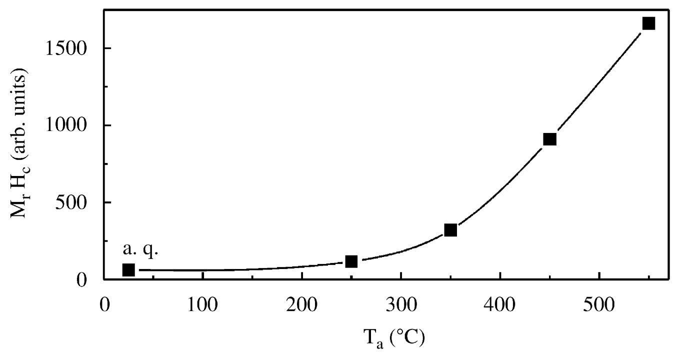

Fig. 6. The product M_rH_c presents a remarkable growth for annealing temperatures higher than 300 °C.

The analysis of the in-plane angular variation data shows that the resonance field is minimum at 0° (parallel to ribbon axis) and 90° (perpendicular to ribbon axis) and maximum at 45°. This feature reveals that the very tiny α-Fe crystallites (cubic magnetocrystalline anisotropy) that precipitate during the quenching process present, on the average, their ⟨100⟩ crystalline directions oriented along the ribbon axis [4]. This behavior may be due to heat flow direction that occurs during the quenching process of the molten $Fe_{90}Zr_7B_3$ alloy.

The in-plane DC magnetization of samples versus the applied field (hysteresis loops) was measured at room temperature using a vibrating sample magnetometer (VSM).

The hysteresis loops plotted in the Fig. 5a show that, while the saturation values of magnetization increase with the annealing temperature, the coercive field (Fig. 5b) presents a minimum in the annealing temperature range between 250 and 350 °C and a maximum at approximately 450 °C. These behaviors are in agreement with the observed minimum of in-plane anisotropy field obtained from FMR spectra (see Fig. 4a). In addition, Fig. 6 shows that the product MrHc starts to increase for annealing temperatures higher than 300 °C.

2. Conclusion

The FMR spectra recorded at different sample orientations reveal that the out-of-plane and the in-plane magnetic anisotropies are strongly affected by the annealing, even when it is performed at relatively low temperatures. Our results show that the coercive field (from DC magnetization data) and the in-plane anisotropy field (from FMR spectra) present a minimum at the same range of annealing temperature. However, these results do not agree with the alpha-iron crystallization temperature reported by other authors [5]. This may be explained considering that the annealing process only promote the coarsening of preexistent alpha-iron nanocrystallites that nucleate during the quenching process.

In order to determine the optimum annealing conditions to minimize the coercive field of the system and to allow the study of the precipitation kinetics, an accurate analysis alpha-iron precipitation process should be made at different period of time in a constant annealing temperature.

Acknowledgments

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