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FORC analysis of ferro-ferromagnetic exchange bias in nanocrystalline ribbons

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ABSTRACT

Horizontal shift and distortion of the hysteresis loops can be induced in some Co-based nanocrystalline systems in which soft and hard ferromagnetic phases coexist. As all the aspects of the phenomenon can be well explained in terms of the exchange interaction between the two phases, it has been identified as an induced ferro-ferromagnetic exchange bias. In this work we use the differential analysis based on first-order reversal curves to analyse this particular kind of exchange bias, through the comparison of the FORC diagrams corresponding to samples with different crystallization degrees. A detailed study of the evolution of such diagrams is presented, pointing in each case to the more outstanding features of the spots corresponding to the different phases as well as to their interactions.

1. Introduction

Grain size and intergrain distance mainly determine the magnetic properties of nanocrystalline ferromagnets obtained by devitrifying amorphous alloys. Soft magnetic behaviour is exhibited when they present densely compacted agglomerations of nanoparticles with sizes smaller than the exchange correlation length [1,2]. Larger agglomerations (typically above 20 nm) give rise to magnetic hardening with respect to the as-quenched amorphous precursor [3]. An intermediate kind of nanocrystalline systems may be achieved by an adequate devitrification resulting in a low concentration of non-agglomerated nanoparticles embedded in the residual amorphous matrix. Depending on the nature of the crystalline phase and the magnetic character of the matrix, the material can be a combination of ferromagnetic hard and soft phases presenting distorted and biased hysteresis loops (HL) [4–6].

Similar anomalous hysteretic behaviour has been reported in other hard/soft ferro/ferro multiphase composite systems, often being attributed to the magnetostatic interaction between the magnetic hard crystallites precipitated during the annealing and the still soft residual matrix [7–11]. Contrarily, in a recent paper [12], the authors demonstrated that, except for samples with very specific geometries, the exchange interaction is mainly responsible for this phenomenon which has been so identified as an induced ferro-ferromagnetic exchange bias.

On the other hand, the experimental measurement of First-Order Reversal Curves (FORC), performed to obtain magnetization as a function of the reversal field H_r and the applied field H, is turning into an essential tool to characterize the hysteretical behaviour of ferromagnets as well as to investigate their magnetic intrinsic interactions [13–18]. The corresponding Switching Field Distributions (SFD) are obtained by partial differentiation of the FORC with respect to the applied field H, and the so-called FORC diagram is calculated by second partial differentiation with respect to the reversal field H_r. In this work we use the differential FORC analysis to study this particular kind of exchange bias.

2. Experimental procedure

The study has been carried out on samples obtained by isothermal annealing of amorphous ribbons of $Co_{70}Fe_5Si_{15}B_{10}$, prepared by melt-spinning in ultrahigh vacuum. Differential Scanning Calorimetry measurement revealed that the first crystallization peak of the as-quenched precursor is centred at $T_1 = 520$ °C.

According to this, samples 40 mm long, 2 mm wide and 20 μm thick were annealed at T_1 for different durations in order to produce series with different crystallization degrees. Table 1 shows a list of studied samples, their annealing conditions and an estimation of their crystallization degree (in percentage of magnetization). The thermal treatments were performed in argon atmosphere inside a preheated quartz-tube furnace automatically controlled with a temperature ripple of ± 1 °C.

Structural analysis of the devitrified ribbons by X-Ray Diffraction and Transmission Electron Microscopy pointed to orthorhombic Co_3B as the most probable crystalline phase [19].

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Table 1
Annealing conditions of the studied samples with an estimation of the crystallization degree (magnetization ratio of the crystallized phase).

Sample	Temperature (°C)	Duration (min)	Degree
A	–	0 min	As-quenched
B	520	15	<1%
C	520	30	≃60%
D	520	60	>99%

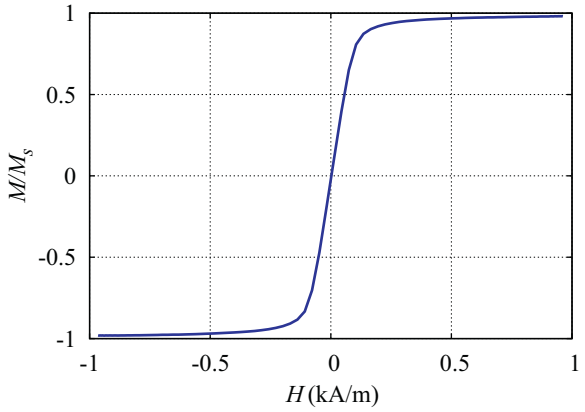

Fig. 1. Hysteresis loop of sample A, of composition $Co_{70}Fe_5Si_{15}B_{10}$ in as-quenched state, produced by a quasistatic alternating magnetic field of amplitude 0.95 kA/m.

The HL and FORC were measured by means of a computer-controlled inductive hysteresis meter in quasi-static regime. An alternating external field of 40 kA/m of maximum amplitude and 0.5 Hz of frequency is applied to the samples to measure a set of 100 FORC. The values of the applied field H, and the electromotive force induced in the pickup coil, are digitized with a sample rate of 2500 Sa/s. Thus, after integrating the magnetization M, around 2500 data pairs (H,M) per FORC are available. The SFD are calculated by performing the derivatives of the FORC with respect to the applied field H by means of a Fourier analysis of the magnetization signal. The inverse Fourier transform is used to reduce the number of points (removing the highest frequencies) and to get a set of interpolated values corresponding to equidistant applied fields. Then, the second mixed derivative is calculated again by Fourier analysis. No smoothing procedure is performed to avoid losing essential information.

3. Results and discussion

The as-quenched sample A presents, for an amplitude of the driving magnetic field of only 0.95 kA/m (=12 Oe), the hysteresis loop showed in Fig. 1: coercivity and remanence are negligible and, in consequence, its quasi-reversible magnetic behaviour produces a non-significant FORC diagram in which the null values become masked by the noise introduced by the numerical differentiation.

Let us focus our attention on sample D: annealed at 520 °C for an hour, it is almost totally crystallized, as can be observed in the HL as well as in the SFD of Fig. 2. A deep look at the FORC diagram in the same figure allows us to point out three features: (i) there is only one spot, split in a red-positive and a blue-negative parts, corresponding to the crystalline phase (and so there is no appreciable soft phase), (ii) the boundary between these two parts points, in the limit of large negative return fields, to the coercive field of the crystals which reaches a value of about 22 kA/m (as can also be concluded on the basis of the peak position in the SFD and the width of the HL), and (iii) the order of the colors in the spot (going horizontally in the direction of increasing applied fields), first blue and then red, reveals an internal interaction clearly dominated by the ferromagnetic exchange. Another evidence of this last conclusion is the position of the spot, below the diagonal $H = -H_r$ [20–23].

Regarding sample C, annealed at 520 °C for 30 min, it can be inferred from its HL as well as from its SFD (Fig. 3) that it has an approximate crystallization degree of 60% in ratio of magnetization. The position of the peaks in its SFD allows also to estimate the coercive fields of the two phases, respectively, H_c^{soft} = 0.7 kA/m and H_c^{hard} = 21.7 kA/m. A trained look at the FORC diagram of Fig. 3 leads to the following observations. (i) Two magnetic phases can be clearly distinguished: one softer, corresponding to the residual amorphous matrix, and another one harder, matching the crystallized phase. The first one appears in the diagram as a couple of blue/red linear spots, whose mutual frontier points to a value (in the H-axis) of around $H_c^{soft} \simeq 2$ kA/m; the second one as a red boomerang-shaped spot with a slight blue shadow at its concave side, whose mutual border points to $H_c^{hard} \simeq 20$ kA/m. (ii) The horizontal order of the colors in the spots (first blue and then red, in both cases) as well as their position in the region below the diagonal $H = -H_r$ allows stating the positive nature of the internal magnetic interaction. (iii) The tilt of the soft spot and the yellow spreading of its positive half towards the boomerang reveal the mutual positive interaction (exchange) between both phases [20–23].

Finally, Fig. 4 shows the HL, the SFD and the FORC diagram of sample B. This sample, annealed at the same temperature of 520 °C for 15 min, presents a negligible degree of crystallization. In fact, both its HL and its SFD seem to correspond to a material with only one magnetic phase, with coercive field H_c=0.8 kA/m. However, its FORC diagram discloses the presence of crystallites by sharing with that of sample C some features which have been associated to the exchange interaction between the crystalline phase and the amorphous residual matrix: the tilt of the soft spot and the yellow spreading of its positive half towards the position

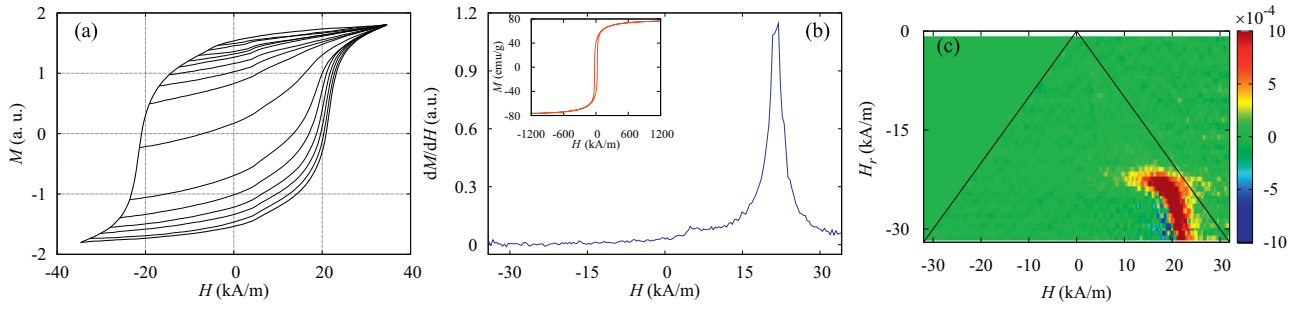

Fig. 2. (a) Hysteresis loop and some FORC, (b) SFD and (c) FORC diagram of sample D, annealed at 520 °C for one hour. (For interpretation of the references to colour in this figure caption, the reader is referred to the web version of this paper.)