Tuning of Magnetic Properties of Magnetic Microwires by Post-Processing

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***Abstract*—The influence of post-processing conditions on the magnetic properties of amorphous and nanocrystalline microwires has been analyzed, paying attention on the effect of magnetoelastic and magnetocrystalline anisotropies on the hysteresis loops of Fe-, Ni- and Co-rich microwires. We demonstrated that the selection of appropriate chemical composition and geometry of as-prepared microwires and appropriate post-processing consisting of annealing or glass-coated removal allow tuning of magnetic properties of glass-coated microwires. Magnetic hardening of the microwires can be achieved by the devitrification of Fe-Pt-Si microwires.**

Keywords- magnetic microwires; magnetic softness; hysteresis loops; internal stresses; magnetic anisotropy.

I. INTRODUCTION

A family of amorphous and nanocrystalline materials prepared using rapid melt quenching is one of the most promising families of soft magnetic materials with a number of advantages, such as excellent magnetic softness, fast and inexpensive manufacturing process, dimensionality suitable for various sensor applications, and good mechanical properties [1]-[6]. Such excellent physical properties have been reported in amorphous materials with either planar (ribbons) [1]-[3] or cylindrical (wires) geometry [4]-[6].

Amorphous and nanocrystalline wires can present quite peculiar magnetic properties, such as spontaneous magnetic bistability associated with single and large Barkhausen jump [7]-[9] or Giant Magneto-Impedance, GMI, effect [10]-[13]. In spite that both large Barkhausen and GMI effect have been also observed in crystalline wires [14][15], as well as in properly heat treated amorphous ribbons [3][12]. However, fast single domain wall, DW, propagation and high GMI effect have been observed in amorphous magnetic wires even without any post-processing [7]-[13].

These features of amorphous and nanocrystalline microwires can be attributed to the combination of cylindrical geometry together with the specific magnetoelastic anisotropy. The latter is linked with the internal stresses distribution characteristic of rapid melt quenching, allowing to obtain a core-shell domain structure either with a high circumferential magnetic permeability (in negative magnetostrictive Co-rich compositions), or with the presence of an axially magnetized single inner domain responsible for the observation of a single and the large Barkhausen jump and the associated single DW propagation [16][17].

The main interest in GMI effect is justified by extremely high impedance sensitivity to an external magnetic field (up to 10 %/A/m) reported for magnetic microwires [18]-[20]. Several sensor applications, such as magnetic compass and acceleration sensors integrated in Complementary Metal-Oxide-Semiconductor (CMOS) circuits, reduced-sized magnetometer suitable for magnetic field mapping, detection of a biomagnetic field with the pico-Tesla sensitivity, magnetoelastic and temperature sensors have been developed [10][12][20]-[22].

On the other hand, magnetically bistable microwires are suitable various applications. Single and controllable DW propagation is suitable for magnetic logic, magnetic memory and electronic surveillance [23]-[26]. The former application is based on use of magnetic tags containing several microwires with well-defined coercivities (characteristic for magnetically bistable microwire) [26]. This application requires a variety of coercivity, H_c, values that can be achieved by the H_c tunability (either by compositional H_c dependence or influence of heat treatments on H_c -values). Accordingly, microwires with rectangular hysteresis loops and plurality of coercivities are requested in electronic surveillance applications.

The additional advantages of glass-coated microwires are excellent corrosion and mechanical properties, biocompatibility, fast and simple fabrication method and

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reduced dimensionality [4][5][27]-[30]. Such features allow to extend the applications possibilities.

As reported elsewhere, magnetic properties of amorphous microwires are affected by the fabrication conditions (like quenching rate or glass-coating thickness), chemical composition of the metallic alloy and post-processing conditions [9][12][15]. Accordingly, we will analyze the influence of various factors on the magnetic properties of glass-coated microwires and provide the guideline for selection of appropriate post-processing for optimization of properties of magnetic microwires.

In section 2, we present the description of the experimental techniques, while in section 3 we describe the results on effect of fabrication and post-processing conditions on hysteresis loops of the microwires.

II. EXPERIMENTAL DETAILS

We prepared and analyzed amorphous glass-coated microwires based on Fe-, Co- and Ni- alloys with minor metalloid additions (Si, B, C) necessary for preparation of amorphous alloys [9][15]. The employed Taylor-Ulitovsky technique is described earlier elsewhere [15].

We studied as-prepared and annealed samples. The annealing temperature, T_{ann}, was between 200 °C to 700 °C for annealing time, t_{ann}, up to 256 min. Typically, the crystallization of amorphous microwires was reported for $T_{ann} \geq 490$ °C [31].

Hysteresis loops of single microwires have been measured using the fluxmetric method previously described in details elsewhere [32]. In order to compare the samples with different compositions and subjected to different post-processing, we represent the hysteresis loops as the normalized magnetization, M/M_0, versus magnetic field, H, where M is the magnetic moment at a given magnetic field and M_0 is the magnetic moment of the sample at the maximum magnetic field amplitude, H_m.

In the case of magnetically hard microwires, the hysteresis loops have been measured using vibrating sample magnetometer by Physical Properties Measurements System by Quantum design.

The magnetostriction coefficient, λ_s, of the studied microwire, was evaluated by the Small Angle Magnetization Rotation (SAMR) method recently adapted for microwire [33].

III. EXPERIMENTAL RESULTS AND DISCUSSION

The magnetostriction coefficient, λ_s, sign and value affects the hysteresis loops of amorphous microwires (see Figure 1). The character of hysteresis loops of amorphous microwires with positive and negative λ_s-values is quite different: microwires with positive λ_s-values present rectangular hysteresis loops, while hysteresis loops of microwires with negative λ_s-values are almost non-hysteretic with low coercitivity, H_c, values. Such influence must be attributed to the fact that the magnetoelastic anisotropy is the main source of magnetic anisotropy in amorphous materials [33].

The rectangular hysteresis loop observed in Fe-rich microwires with positive λ_s-values was interpreted in terms of axial magnetic anisotropy intrinsically related to a peculiar domain structure consisting of inner axially magnetized single domain and the outer domain shell with radial magnetization orientation [17].

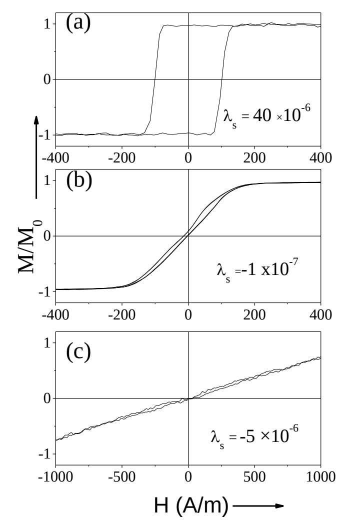

Figure 1. Hysteresis loops of magnetic microwires $Fe_{75}B_9Si_{12}C_4$ with positive (a) $Co_{67.1}Fe_{3.8}Ni_{1.4}Si_{14.5}B_{11.5}Mo_{1.7}$ with vanishing (b) and $Co_{77.5}Si_{15}B_{7.5}$ with negative (c) λ_s values.

The remagnetization of such microwires is running by the single and large Barkhausen jump within the inner domain [8][17]. Perfectly rectangular hysteresis loop character is related to an extremely fast magnetization switching by single domain wall propagation. Such microwires with rectangular hysteresis loops are suitable for the electronic surveillance applications. However, plurality of H_c-values is required for such applications. One of the possibilities to tune the H_c-values is the control of the internal stresses.

There are several factors responsible for the internal stresses value and distribution: (i) the difference in the thermal expansion coefficients of metallic alloy nucleus solidifying simultaneously with the glass coating surrounding it; (ii) the quenching stresses itself related to the rapid solidification of the metallic alloy nucleus from the surface inside the wire axis; and (iii) the drawing stresses [34]-[37].

Most theoretical evaluations of the internal stresses value and distribution show that the largest internal stresses are associated with the difference in the thermal expansion coefficients of the metallic alloy and the glass coating [34]-[37]. Accordingly, we must assume that the internal stresses value inside the metallic nucleus can be tuned by the ρ -ratio between the metallic nucleus diameter, d, and the total microwire diameter, D ($\rho = d/D$) [34]-[37].

Provided below hysteresis loops of $Fe_{70}B_{15}Si_{10}C_5$ microwires with different ρ–ratio experimentally confirm such assumption (see Figure 2). The difference of the hysteresis loops of microwires with the same chemical composition must be related to different internal stresses, σ_i, values. Indeed, the magnetoelastic anisotropy, K_{me}, is given by [8][9][34][38]:

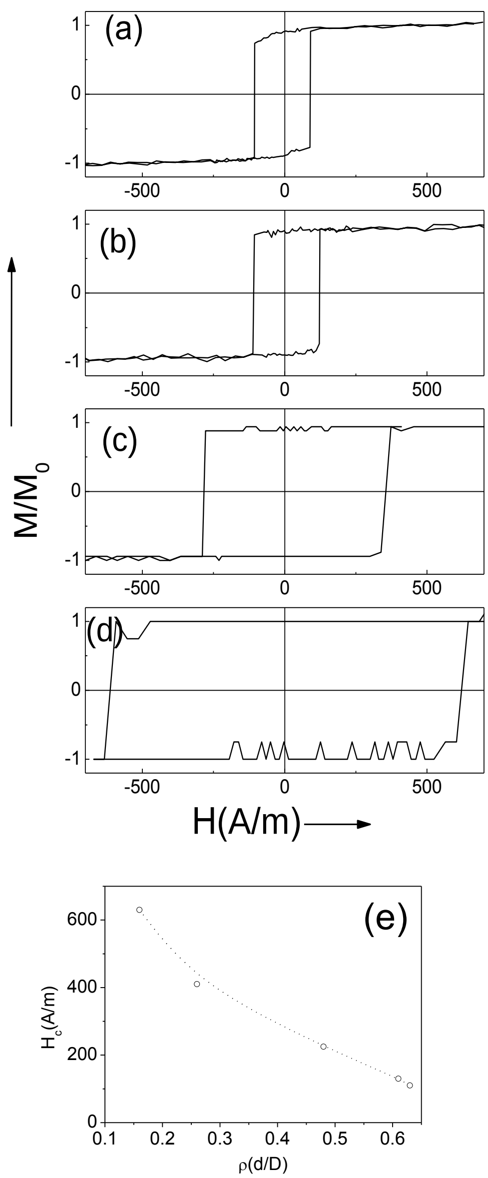

Figure 2. Hysteresis loops of $Fe_{70}B_{15}Si_{10}C_5$ amorphous microwires with different metallic nucleus diameter d and total diameters D: with $\rho = 0.63$; $d = 15$ µm (a); $\rho = 0.48$; $d = 10.8$ µm (b); $\rho = 0.26$; $d = 6$ µm (c); $\rho = 0.16$; $d = 3$ µm (d) and $H_c(\rho)$ dependence of the same microwires(e).

$$K_{me}=3/2\ \lambda_s\sigma_i \qquad (1)$$

Naturally, controllable glass-coating removal is the other route to tune hysteresis loops of glass-coated microwires.

The chemical etching of the glass-coating of $Co_{68.5}Si_{14.5}B_{14.5}Y_{2.5}$ microwire with higher negative λ_s-values allows transformation of linear hysteresis loop into rectangular (see Figure 3). As previously reported, after etching in 10% HF for 50 min, the glass-coating thickness decreases from 8.5 to 4 µm [39]. Accordingly, chemical etching of the glass-coating makes Co-rich microwires suitable for the electronic surveillance applications. Such remarkable influence of chemical etching must be associated with the relaxation of the internal stresses related

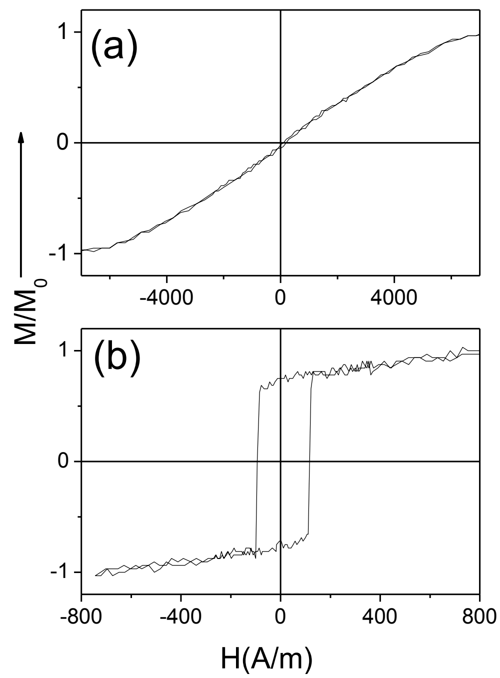

Figure 3. Hysteresis loops of as-prepared (a), and subjected to chemical etching for 50 min (b) $Co_{68.5}Si_{14.5}B_{14.5}Y_{2.5}$ microwires, adapted from [38].

to different thermal expansion coefficients of glass coating and metallic alloy. Accordingly, one can assume that the annealing allowing internal stresses relaxation must be the other route for adapting of glass-coated microwires for the electronic surveillance applications. Furthermore, such processing allows to keep the flexible and insulating glass coating.

In the case of $Fe_{70}B_{15}Si_{10}C_5$ amorphous microwires, annealing allows a slight coercivity decrease, however the shape of the hysteresis loops remains the same (see Figure 4).

More remarkable and complex annealing influence is observed in Fe-Ni based microwires. As-prepared $Fe_{62}Ni_{15.5}Si_{7.5}B_{15}$ microwires present rectangular hysteresis loops (see Figure 5a) as expected for microwires with

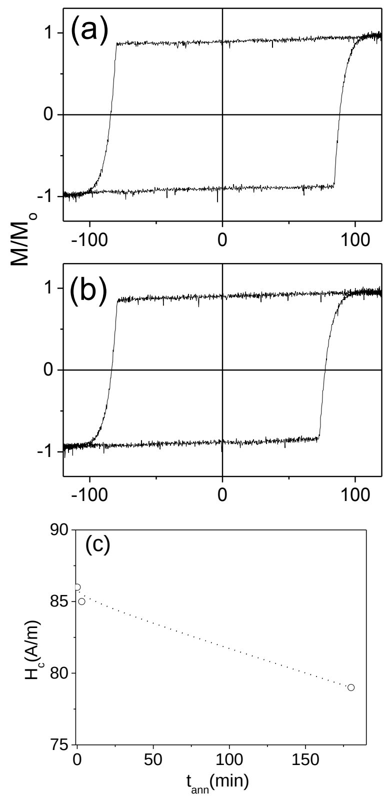

Figure 4. Hysteresis loops of as-prepared (a), and annealed at T_{ann} = 400 °C for 180 min (b) $Fe_{75}B_9Si_{12}C_4$ microwires and dependence of coercivity on annealing time (c).

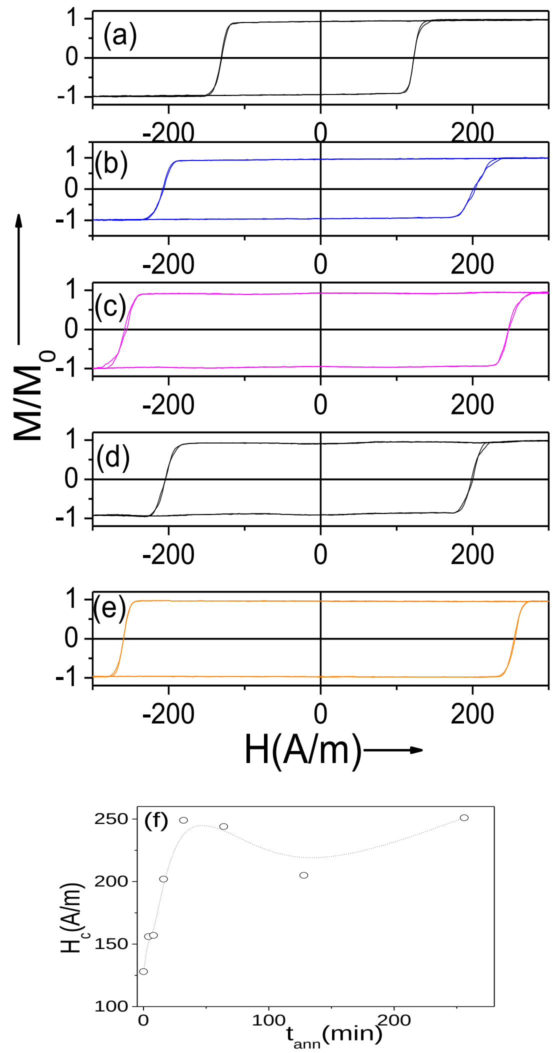

Figure 5. Hysteresis loops of as-prepared (a) and annealed at T_{ann} = 410 °C for 16 min (b) 32 min (c), 128 min (d), and 256 min (e) $Fe_{62}Ni_{15.5}Si_{7.5}B_{15}$ microwires and $H_c(t_{ann})$ dependence of the same microwire.

positive λ_s-values (about 27 × 10^{-6}). After annealing of $Fe_{62}Ni_{15.5}Si_{7.5}B_{15}$ microwires, a remarkable increase in coercivity, H_c, is generally observed (see Figures 5b–f). The hysteresis loop character remains unchanged: all hysteresis loops present rectangular shape.

One of the origins of a rather different effect of annealing on coercivity of Fe and Fe-Ni based microwires can be a domain wall stabilization due to directional ordering of atomic pairs being considered [41]–[44]. However, local nano-sized precipitations and clustering observed in annealed Fe-Ni based microwires by the atom probe tomography can be the other origin of remarkable magnetic hardening and complex $H_c(t_{ann})$ dependence [42]. Furthermore, atom pair ordering and hence DW stabilization is reported for Fe-Ni and Fe-Co amorphous alloys [41]–[44]. Such DW stabilization is considered as the main origin of the H_c rising upon annealing observed in amorphous materials containing two or more ferromagnetic elements [43][44]. Accordingly, annealing of Fe-Ni based microwires allows considerable variation of coercivity in Fe-Ni based microwire with rectangular hysteresis loops.

Even more remarkable hardening upon conventional annealing has been reported in a variety of Co-rich microwires with vanishing λ_s-values [31][32]. Thus, transformation of linear hysteresis loop with low coercivity (H_c ≈ 4 A/m) into rectangular with H_c ≈ 90 A/m and considerable magnetic hardening are observed in $Fe_{3.6}Co_{69.2}Ni_1B_{12.5}Si_{11}Mo_{1.5}C_{1.2}$ microwire upon annealing without stress (see Figure 6).

Observed annealing influence must be attributed to the internal stresses relaxation and rising of the inner axially magnetized inner core diameter. Similar evolution of hysteresis loops upon annealing is confirmed in various Co-based microwires with low and negative λ_s-values [9][45]. Consequently, similarly to glass-coating removal, annealing of Co-rich microwires allows for obtaining magnetically bistable Co-rich microwires.

On the other hand, magnetically hard and semi-hard wires are desirable for various applications like the electronic article surveillance, compass needles, motors, tachometers, magnetic tips for magnetic force microscopy,

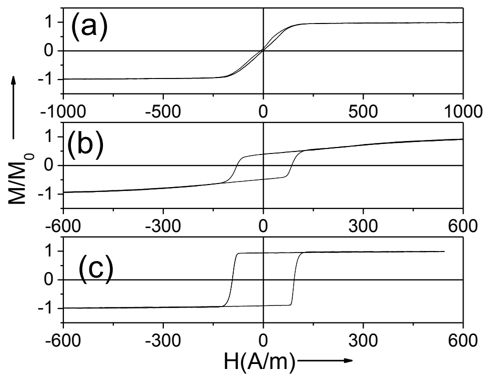

Figure 6. Hysteresis loop of as-prepared (a) and annealed at T_{ann} = 250 °C (b) and T_{ann} = 300 ºC (c) $Fe_{3.6}Co_{69.2}Ni_1B_{12.5}Si_{11}Mo_{1.5}C_{1.2}$ microwires.

or dentistry [46]. For this purpose, $Fe_{50}Pt_{40}Si_{10}$ microwires have been prepared. As-prepared $Fe_{50}Pt_{40}Si_{10}$ microwires present amorphous structure. Magnetic hardening in $Fe_{50}Pt_{40}Si_{10}$ microwires has been observed after annealing (see Figure 7). In this case, after devitrification of amorphous $Fe_{50}Pt_{40}Si_{10}$ microwire, H_c ≈ 40 kA/m is observed. Such magnetic hardening has been attributed to the formation of L10-type superstructure after crystallization of as-prepared amorphous precursor. As can be appreciated, magnetic hardening is observed in a wide temperatures range.

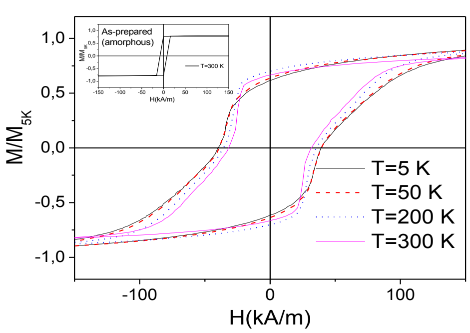

Figure 7. Hysteresis loops of annealed at 500 °C for 1 h $Fe_{50}Pt_{40}Si_{10}$ microwires measured at different temperatures. Hysteresis loops of as-prepared are provided in the inset Adapted from Ref. [46].

IV. CONCLUSIONS

We showed that the magnetic properties of amorphous magnetic microwires can be tuned either in as-prepared state or by controlling the magnetoelastic anisotropy through the magnetostriction coefficient value and by the internal stresses values related to the fabrication conditions and geometry of microwires. Furthermore, appropriate post-processing (including either conventional heat treatment, heat treatment in the presence of applied stress or magnetic field, or glass-coating removal) allows further tuning of magnetic properties of magnetic microwires. We showed that the microwires with coercivities from 1 A/m to 40 kA/m can be prepared. Future work will focus on the influence of external stimuli (temperature, stresses) on the magnetic properties of magnetic microwires.

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