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DESIGN OF MAGNETIC PROPERTIES OF GLASS-COATED MICROWIRES FOR MAGNETIC SENSORS APPLICATIONS

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Summary: *We report on tailoring of magnetic properties of Fe-Co –rich glass-coated microwires. Main parameter that affects magnetic properties of glass-coated microwires is the magnetoelastic anisotropy that can be tailored either changing the composition of metallic nucleus or controlling the internal stresses. The internal stresses can be modified either by geometry of the microwires changing the* ρ*-ratio between the metallic nucleus diameter and total microwire diameter or by thermal treatment. Observed dependences of hysteresis loop on annealing conditions discussed considering stress relaxation and stress dependence of the magnetostriction coefficient.*

1 INTRODUCTION

Amorphous ferromagnetic materials have attracted considerable attention during last decades owing to excellent magnetic softness suitable for various industrial applications such as transformers, inductive devices, magnetic sensors [1,2]. Magnetic softness of amorphous materials is usually attributed to specific features of metallic glasses such as a lack of long-range atomic order (and consequently magnetocrystalline anisotropy), of microstructural inhomogeneities and defects typical for crystalline materials as well as to elevated electrical resistance. Therefore typically amorphous materials present extremely low coercivity, high magnetic permeability and low magnetic losses. The most common method employed for preparation of amorphous materials is a rapid quenching from the melt. Using different rapid quenching from the melt techniques amorphous materials can be prepared in the form of ribbons and wires [1,3]. Thermal conditions and geometrical characteristics of these materials are rather different. Consequently shape and magnetoelastic anisotropies and hence magnetic properties of amorphous ribbons and wires are rather different.

Amorphous wires known since the end of 80^{th} have present several magnetic properties quite attractive for applications in magnetic sensors, i.e. magnetic bistability and giant magnetoimpedance, GMI, effect [4-7]. These properties are closely related to specific magnetic domain structure of amorphous wires i.e. with core-shell domain structure and wither single inner axially domain typical for Fe-rich wires or high circular magnetic permeability of Co-rich amorphous wires with bamboo-like domain structure in the outer shell.

In fact both magnetic bistability and GMI effect can be also realized in amorphous ribbons after special thermal treatments [8,9]. But shape and magnetoelastic anisotropies of amorphous wires are more favorable for formation of aforementioned properties.

The origin of magnetoelastic anisotropy of amorphous materials is related to quenching stresses distribution arising during rapid solidification from the melt [10]. The outer she11 that first solidified induces strong and complex residual stresses inside the wire. The interplay between the shape and magnetoelastic anisotropies give rise to axial easy magnetization direction in the inner core and either radial or circular (depending on the magnetostriction constant sign) easy magnetization direction in the outer shell of wires [10,11].

Aforementioned GMI effect is one of most promising features for the magnetic sensors applications [5-7]. The origin of the GMI effect is related to the skin effect in soft magnetic conductor [6,7]. High circumferential permeability usually exhibited by amorphous wires with vanishing magnetostriction constant is essential for observation of high GMI effect [5-7].

The “in-rotating-water” quenching technique allows fabrication of amorphous wires with diameter between 70 and 200 μm [4-11]. The magnetic bistability of amorphous wire prepared using “in-rotating-water” quenching technique is usually observed in magnetostrictive compositions (either Fe-rich or Co-rich). The magnetic bistability is related to the presence of a single Large Barkhausen Jump interpreted as the magnetization reversal in a large single inner axially magnetized domain [10,11]. Although magnetic bistability is quite interesting for magnetic sensor applications, it restricted by the critical length that correlates well with the demagnetizing factor [12]. For magnetic wires prepared by the “in-rotating-water” quenching technique with typical diameter of 120 μm this critical length is about 4-7 cm (depending on chemical composition), which is quite inconvenient for applications [13]. Short magnetic wires with length below critical the hysteresis loop loses its squared shape. The origin of the critical length has been explained considering that the closure end domains penetrate from the wire ends inside the internal axially magnetized core destroying the single domain structure [12].

The way to avoid this inconvenient feature is the diameter reduction. Consequently either posterior cold drawing of cast wires or alternative rapid quenching technologies such as melt extraction or glass-coating techniques have been proposed for fabrication of thinner amorphous microwires [14-17].

The Taylor-Ulitovsky technique for preparation of glass-coated microwires allows preparation of thinnest microwires (with metallic nucleus diameters of 0.5- 100 μm) [17,18]. Consequently considerable reduction of the critical length has been observed for the case of glass-coated microwires: in microwires: for metallic nucleus diameter about 10 μm the critical length is about 2 mm [17,18]. Therefore these studies of magnetic glass-coated microwires attracted great interest considering it suitability for applications in microsensors

On the other hand Taylor-Ulitovsky technique involves simultaneous solidification of metallic nucleus inside the glass coating that results in elevated internal stresses arising from different thermal expansion coefficients of the metallic alloy and glass [5]. These internal stresses give rise to the additional magnetoelastic anisotropy. Therefore special efforts are needed for minimization of elevated magnetoelastic anisotropy and optimization of magnetic properties of glass-coated microwires. Different kinds of processing such as annealing or glass coating removal have been proposed[18-21].

Aforementioned magnetoelastic energy, K_{me}, plays the determining role in the formation of magnetic properties of amorphous microwires. As described elsewhere [18], the magnetoelastic energy is given by

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

where λ_s is the magnetostriction constant, and total stresses $\sigma = \sigma_{appl} + \sigma_i$, where σ_{appl} and σ_i are applied and internal stresses respectively.

The magnetostriction constant, λ_s, depends mostly on the chemical composition of amorphous metallic alloy. Vanishing λ_s values can be achieved in amorphous Fe-Co based alloys with Co/Fe $\approx$70/5 [18, 22,23].

On the other hand, the value of internal stresses inside the metallic nucleus, σ_i is determined by the ρ-ratio between the metallic nucleus diameter, *d*, and total microwire diameter, *D* ($\rho = d/D$).The strength of internal stresses increase with decreasing of the ρ-ratio [24,25].

The conventional way to relax internal stresses and stabilize magnetic properties is a heat treatment.

Consequently presently most promising applications of glass-coated magnetic microwires are related with optimization of the GMI effect and DW dynamics.

Therefore, the purpose of this paper is to present results on studies of the effect of magnetoelastic anisotropy on magnetic properties and GMI effect in amorphous magnetically soft microwires paying attention to find the conditions for observation of these two effects simultaneously.

2 EXPERIMENTAL METHOD

We studied glass-coated Co and Fe-rich microwires prepared by Taylor-Ulitovsky technique (also called in some publications as the drawing and quenching technique) described elsewhere [5,18]

Hysteresis loop of as-prepared and annealed microwires were measured by the induction method as described elsewhere [17]. We represent the normalized magnetization, M/M_o versus magnetic field, H, where M is the magnetic moment at given magnetic field and M_o is the magnetic moment of the sample at the maximum magnetic field amplitude, H_o.

We measured magnetic field dependences of impedance, *Z*, and GMI ratio, $\Delta Z/Z$. We used specially designed micro-strip sample holder placed inside a sufficiently long solenoid that creates a homogeneous magnetic field, H. There one wire end was connected to the inner conductor of a coaxial line through a matched microstrip line while the other was connected to the ground plane. This sample holder allows measuring the samples of 6 mm length. This sample length is sufficiently long allowing neglecting the effect of the demagnetizing factor [26]. We determined the impedance *Z* using the vector network analyzer from reflection coefficient S_{11}. Employed method allowed extending the frequency range up to GHz-range [26].

The magneto impedance ratio, $\Delta Z/Z$, has been defined as:

$$\Delta Z/Z = [Z(H) - Z(H_{max})] \cdot 100 / Z(H_{max}), \qquad (2)$$

an axial *DC*-field with maximum value, H_{max}, up to 20 kA/m was supplied by a magnetization coils.

The microwires have been annealed in conventional furnace at temperatures, T_{ann} of 200-300°C for duration, t_{ann}, from 5 and 60 minutes. After annealing the magnetic properties of microwires were studied again.

All samples (as-prepared and annealed microwires) present amorphous structure of metallic core similar to other recently studied Co-rich samples subjected to annealing at similar temperatures [26].

The technique allowing measuring the DW velocity in thin wires is well described elsewhere [26,27]. In order to activate DW propagation always from the other wire end in our experiment we placed one end of the sample outside the magnetization solenoid. We used 3 pick-up coils, mounted along the length of the wire and propagating DW induces electromotive force (*emf*) in the coils, as described in refs. [27,28]. These *emf* sharp peaks are picked up at an oscilloscope upon passing the propagating wall.

Then, DW velocity is estimated as:

$$v = \frac{l}{\Delta t} \qquad (3)$$

where l is the distance between pick-up coils and Δt is the time difference between the maximum in the induced *emf*.

3 TAILROING OF MAGNETIC PROPERTIES THRUOGH THE COMPOSITION AND FABRICATION PARAMETERS

Generally magnetic properties and overall shape of hysteresis loops of amorphous microwires depend on composition of the metallic nucleus as well as on composition and thickness of the glass coating. Usually Co-rich microwires present inclined hysteresis loops with low coercivity (Fig.1a) but magnetic permeability of Co-rich microwires is usually not high because of relatively high magnetic anisotropy field. As a rule, best soft magnetic properties are observed for nearly-zero magnetostrictive Co-rich compositions (Fig.1b). It is worth mentioning, that the magnetostriction constant, λ_s, in system $(Co_xFe_{1-x})_{75}Si_{15}B_{10}$ changes with x from -5x10^{-6} at x= 1, to $\lambda_s \approx 35$ x10^{-6} at x≈0.2, achieving nearly-zero values at Co/Fe about 70/5 [22,23].

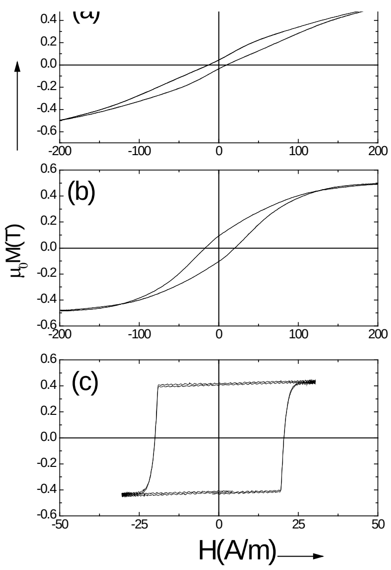


Figure 1. Typical hysteresis loops of Co-rich (a), Co-Fe-rich (b) and Fe-rich (c) microwires.

On the other hand microwires with Fe-rich metallic nucleus composition present completely different magnetic behavior exhibiting rectangular hysteresis loops related with large and single Barkhausen jump (Fig.1c).

On the other hand the composite character of glass-coated microwires affects magnetic properties mostly through the internal stresses induced by the glass coating [24,25]. As mentioned in the introduction the origin of these stresses is determined mostly by the difference in thermal expansion coefficients of the glass and metallic alloy and by rapid solidification of composite microwire. Moreover the strength of such internal stresses depends on the ρ-ratio between metallic nucleus diameter, d, and

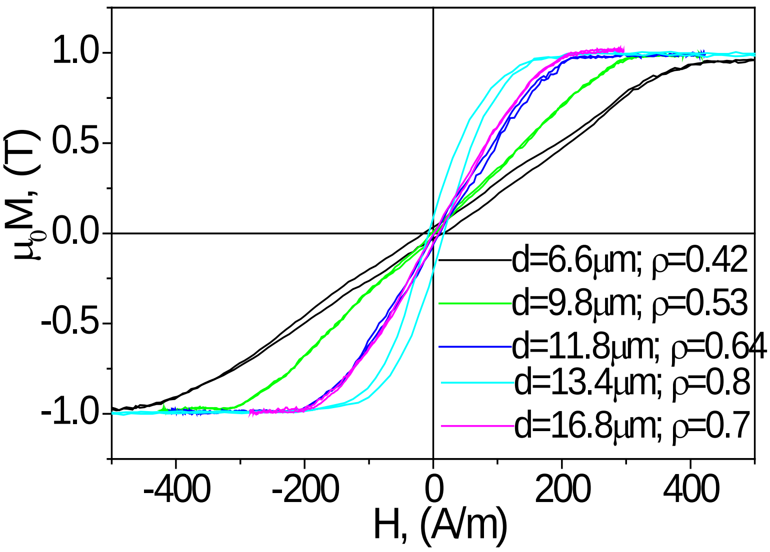


Figure 2. Hysteresis loops of $Co_{67.1}Fe_{3.8}Ni_{1.4}$ $Si_{14.5}$ $B_{11.5}$ $Mo_{1.7}$ microwires with different geometry.

total microwire diameter, D. Consequently the strength of these stresses can be controlled by the ρ-ratio: strength of internal stresses increases decreasing ρ-ratio, i.e. increases with increasing of the glass volume [5,18, 24].

Therefore within each group of aforementioned amorphous microwires compositions (Fe-rich with rectangular hysteresis loop or Co-Fe-rich with vanishing magnetostriction coefficient) considerable effect of ρ-ratio on hysteretic magnetic properties can be appreciated from Figs.2, 3.

Thus, increasing of the magnetic anisotropy field (Fig.2) and increasing of the coercivity (Fig.3) with decreasing of ρ-ratio is observed for Co-Ni- Fe and for Fe-based microwires respectively.

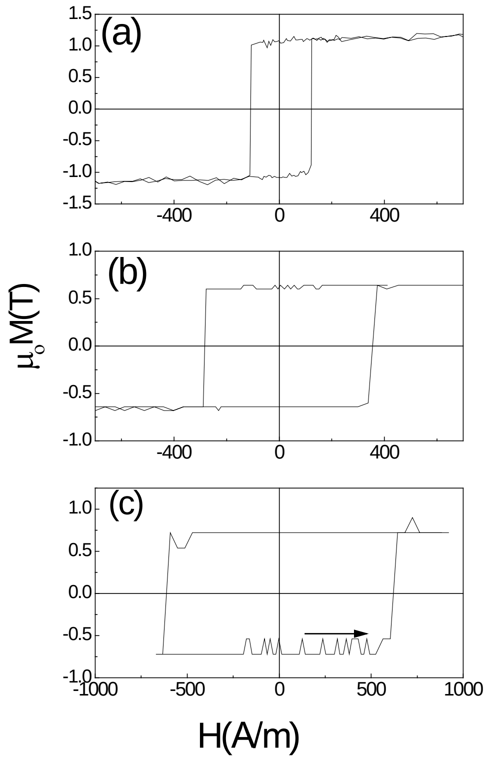


Figure 3. Hysteresis loops of $Fe_{70}B_{15}Si_{10}C_5$ amorphous microwires with the same sample length and different metallic nucleus diameter *d* and total diameters *D*: ρ= 0,48; *d*= 10,8 μm (a);ρ =0,26; *d*= 6 μm (b); ρ =0,16; *d*= 3 μm (c).

All Co-rich microwires exhibit quite low (4-5 A/m) coercivity (Fig.2). Magnetic anisotropy field, H_k, increases with increasing the ρ-ratio. Low coercivity remanent magnetization, magnetic anisotropy field and high magnetic permeability are usually attributed to the considerable magnetization rotation contribution. This denotes the existence of a circumferential magnetic anisotropy throughout the whole metallic nucleus volume, practically without inner core indicating that almost whole cross section should consist of a circumferentially magnetized shell, with a well-defined transverse magnetic anisotropy, K_φ, whose anisotropy field, $H_{\kappa\varphi}$, can be experimentally evaluated as the field required to reach saturation in the axial direction[18].

Magnetic bistability exhibited by amorphous glass-coated microwires with positive magnetostriction constant is associated with perfectly rectangular hysteresis loop exhibited by microwires related with axial orientation of magnetization in the most part of the metallic nucleus [18]. All hysteresis loops of presented in Fig.3 Fe-rich microwires exhibit perfectly rectangular hysteresis loops.

As can be observed in Fig 3 for the same Fe-rich composition of the metallic nucleus quite different coercivity values can be observed. The switching field, H_s, that is, the magnetic field required to reverse magnetization increases for the same metallic nucleus composition ($Fe_{65}B_{15}Si_{15}C_5$) with decreasing of the geometric ratio ρ. Such increasing of the switching field must be attributed to the increasing of the strength of internal stresses as increasing the glass coating thickness.

Additionally previously has been reported that in amorphous alloys the magnetostriction coefficient exhibits stress dependences that can be expressed as [29]:

$$\lambda_{s,\sigma} = \lambda_{s,0} - B\sigma \qquad (4)$$

where $\lambda_{s,\sigma}$ is the magnetostriction coefficient under stress, $\lambda_{s,0}$ is the zero-stress the magnetostriction coefficient and B is a positive coefficient of order 10^{-10} MPa. This stress dependence of the magnetostriction coefficient is expected to be relevant for nearly zero magnetostriction microwires compositions, i.e. for Co-rich microwires. Consequently in the case of Co-rich compositions one can expect considerable influence of the thermal treatment on magnetic properties of glass-coated microwires

4 TAILORIING OF MAGNETIC PROPERTIES BY ANNEALING

We studied the influence of annealing on hysteresis loops of Co-rich microwires that present inclined hysteresis loop with low coercivity typical for Co-rich microwires with low and negative magnetostriction coefficient before annealing (Fig. 4a). As we observed, annealing even for quite short time and at low temperature leads to significant changing of the magnetic properties (Fig 4b-d).

As mentioned above actually main technological interest in magnetically soft microwires is related to the excellent magnetic softness and GMI effect observed in nearly-zero magnetostriction composition [18]. This GMI effect consists in the large change of the electric impedance of a magnetic conductor when is subjected to an axial dc magnetic field. Usually GMI effect is characterized by the magnetoimpedance ratio, $\Delta Z/Z$, has been defined as:

$$\Delta Z/Z = [Z(H) - Z(H_{max})] / Z(H_{ma} \qquad (5)$$

where H_{max} is the maximum *DC* longitudinal magnetic field of the order of few kA/m, usually supplied by a long solenoid and/or Helholtz coils.

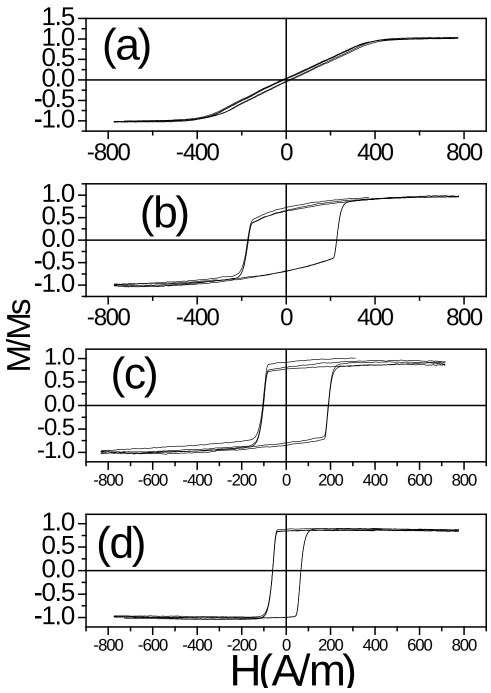


Figure 4. Hysteresis loops of as-prepared (a) and annealed for 5 min at 200^{o}C (b), 250^o C (c), and 300^{o}C (d) $Fe_{8,13}Co_{50,69}Ni_{17,55}B_{13,29}Si_{10,34}$ microwires

In spite of considerable magnetic hardening (increasing of coercivity from 4 to 200 A/m), both as-prepared and annealed microwires present considerable GMI effect as shown in Fig 5. The main difference of observed ΔZ/Z (H) dependences for as-prepared and annealed samples is the value of the magnetic field, H_m, at which ΔZ/Z maximum takes place: for annealed samples H_m –values are much lower than for as-prepared samples for all measured frequencies.

Increasing of the annealing time and temperature the hysteresis loop becomes more rectangular: remanent magnetization rises with increasing of T_{ann}, although coercivity, H_c, remains almost the same for all annealing conditions (Fig.6).

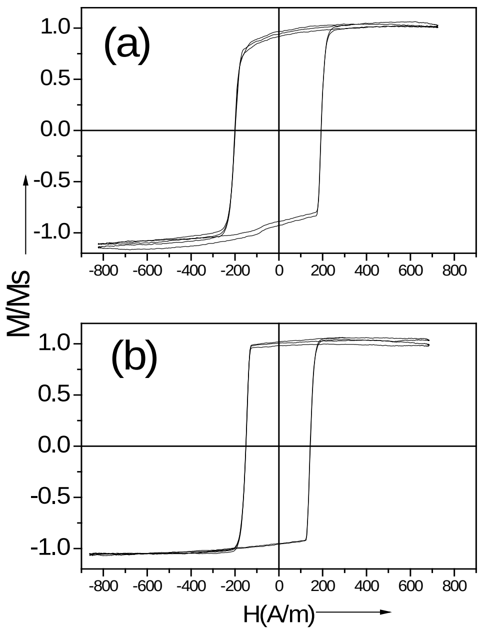


Figure 6. Hysteresis loops of $Fe_{8,13}Co_{50,69}Ni_{17,55}B_{13,29}Si_{10,34}$ microwires annealed at 250 °C for 60 min (a) and at 300 °C for 60 min (b)

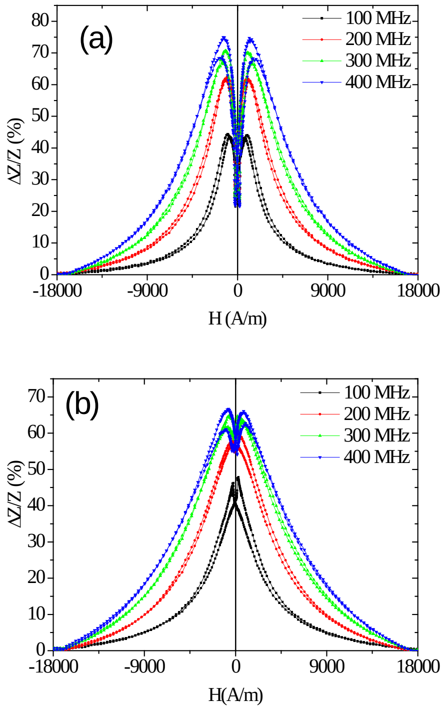
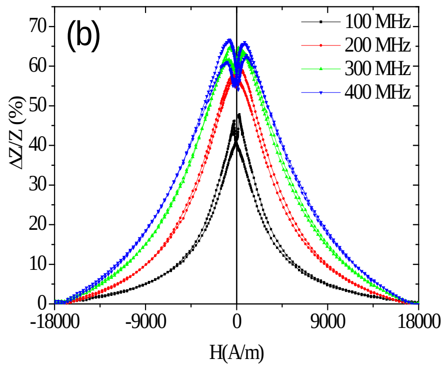


Figure 5. ΔZ/Z (H) dependences of as-prepared (a) and annealed at 200 °C for 5 min (b) $Fe_{8,13}Co_{50,69}Ni_{17,55}B_{13,29}Si_{10,34}$ microwires measured at different frequencies

On the other hand, all annealed $Fe_{8,13}Co_{50,69}Ni_{17,55}B_{13,29}Si_{10,34}$ samples present considerable GMI effect (Fig.7).

Similar results have been observed in other Co-rich $Co_{69.2}Fe_{4.1}B_{11.8}Si_{13.8}C_{1.1}$ microwires: annealing considerably affects magnetic properties of $Co_{69.2}Fe_{4.1}B_{11.8}Si_{13.8}C_{1.1}$ samples. Even after short (t_{ann}=5 min) at T_{ann} =250° C the hysteresis loops $Co_{69.2}Fe_{4.1}B_{11.8}Si_{13.8}C_{1.1}$ microwires become almost rectangular (Fig.8).

For an interpretation of the unusual magnetic hardening observed after annealing (Fig. 4) we must consider not only the stress relaxation (that usually results in coercivity decreasing) but also the change of the character of the remagnetization process induced by the heat treatment. The observed tendency allow us to assume that annealing induces an axial magnetic anisotropy, which is confirmed by the perfectly rectangular hysteresis loops exhibited by the annealed microwire and that are typical for microwires with a positive magnetostriction constant presenting an axial easy magnetization axis. To explain this unusual dependence of the magnetic properties upon the annealing temperature, we must consider that the stress relaxation affects the magnetostriction coefficient, as also recently described for other Co-rich microwires [30].



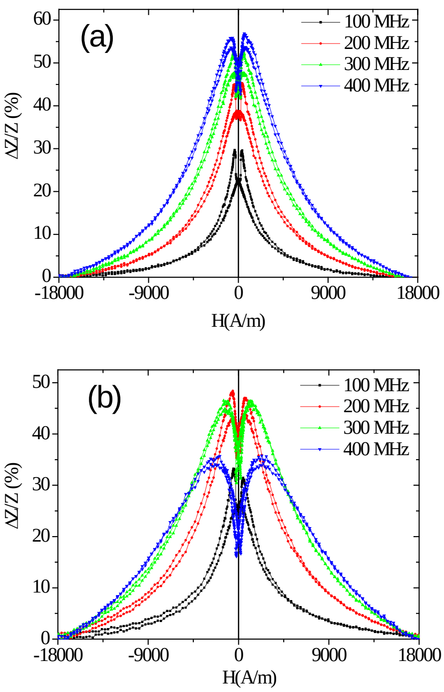


Figure 7. ΔZ/Z (H) dependences annealed for 60 min at 250 °C (a) and300 °C (b) $Fe_{8,13}Co_{50,69}Ni_{17,55}B_{13,29}Si_{10,34}$ microwires measured at different frequencies

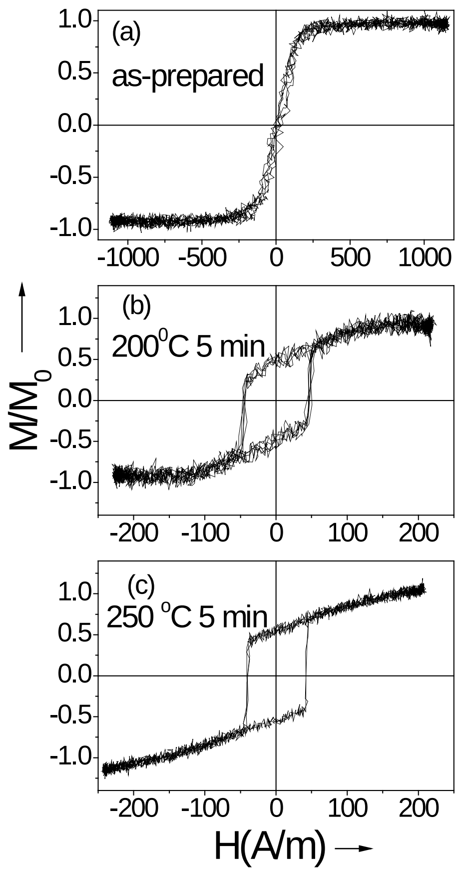


Figure 8. Hysteresis loops of as-prepared (a) and annealed for 5 min at different temperatures (b,c) $Co_{69.2}Fe_{4.1}B_{11.8}Si_{13.8}C_{1.1}$ microwires.

Similarly to recently observed changes induced by annealing we can assume that the stress relaxation induced by annealing can change the sign of the magnetostriction constant. Indeed reported internal stresses values inside the metallic nucleus are between 200 MPa and 5 GPa [24,25]. Experimentally measured *B* -coefficient values of expression (2) reported for similar $(Co_{0.94}Fe_{0.06})_{75}Si_{15}B_{10}$ amorphous alloy are about 1.8 10-10 MPa^{-1}[29] Moreover recently we confirmed this assumption by direct measurements of the magnetostriction coefficient in as-prepared and annealed Co-rich microwires [31].

We can also assume that the outer domain shell of the annealed Co-rich microwire that exhibits both rectangular hysteresis loop and a GMI effect has high circumferential magnetic permeability. This assumption is deduced by observing the much higher GMI ratio of Co-rich microwires that exhibit a rectangular hysteresis loop after annealing than that of the Fe-rich amorphous microwires also exhibiting similar bulk hysteresis loop characteristics but much lower GMI effect (usually about 1-5%).

5 CONCLUSIONS

Hysteresis loop of glass-coated microwires can be tailored either changing the composition of metallic nucleus or controlling the internal stresses. The internal stresses can be modified either by geometry of the microwires changing the ρ-ratio between the metallic nucleus diameter and total microwire diameter or by thermal treatment. Annealing considerably affects hysteresis loop and GMI effect of Co-rich microwires. Observed dependences of hysteresis loop on annealing conditions discussed considering stress relaxation and change of the magnetostriction after samples annealing. Understanding of the processes affecting formation of magnetic anisotropy and determining the remagnetization process of Co-Fe rich microwires with low magnetostriction constant allows us to tailor magnetic properties and to find the annealing conditions at which we can observe simultaneously magnetic bistability and considerable GMI effect.

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