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# Magnetic properties of “thick” glass-coated Fe-rich microwires

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## ABSTRACT

We report on preparation and magnetic properties of  $\text{Fe}_{71.7}\text{B}_{13.4}\text{Si}_{11}\text{Nb}_3\text{Ni}_{0.9}$  glass-coated microwire with metallic nucleus diameter  $d = 103 \mu\text{m}$  and total diameter  $D = 158 \mu\text{m}$  prepared by Taylor-Ulitovsky method. Amorphous structure of as-prepared microwires is confirmed by X-ray diffraction. As-prepared glass-coated microwires present relatively high GMI effect (about 50%) and relatively low coercivity (about 25 A/m). Additionally, as-prepared sample present rectangular hysteresis loop and fast single domain wall propagation with domain wall mobility of about  $11,9 \text{ m}^2/\text{As}$ . After annealing we observed considerable improvement of the GMI ratio (from 50% up to 100%). Observed GMI effect improvement has been attributed to the stresses relaxation. From aforementioned studies we can conclude that the Taylor-Ulitovsky technique allows us to obtain thick ferromagnetic microwires with good magnetic properties and GMI effect suitable for industrial applications.

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## I. INTRODUCTION

Amorphous and crystalline magnetically wires can present unique magnetic properties like the giant magnetoimpedance (GMI) effect or fast magnetization switching linked fast propagation of single domain wall (DW) through the wire.<sup>1-4</sup> However, amorphous wires are preferable for technical applications since they present superior mechanical and corrosive properties.<sup>5,6</sup> Accordingly, a number of prospective applications have been developed within recent few years.<sup>7-14</sup>

Glass-coated amorphous magnetic microwires prepared using the Taylor-Ulitovsky technique present a number of advantages related to a great extent to flexible, insulating and biocompatible glass coating allowing development of completely new applications.<sup>11,12</sup>

It is commonly believed that the Taylor-Ulitovsky technique allows preparation of only relatively thin magnetic microwires with metallic diameters ranging between 0.5-50  $\mu\text{m}$ .<sup>15,16</sup> However, for

certain applications, i.e., related to smart composite materials for non-contact stress and temperature monitoring or magnetic tags thicker microwires are demanded.<sup>13,14</sup>

There are only a few reports on studies (mostly of magnetic domain structure) of amorphous microwires with diameters thicker than 50  $\mu\text{m}$  in diameter.<sup>17,18</sup>

As regarding the chemical composition of magnetic wires: generally thin Co-rich microwires present better magnetic softness and higher GMI effect.<sup>19,20</sup> However, the spontaneous magnetic bistability and hence fast single domain wall (DW) propagation are generally observed in Fe-rich glass-coated microwires.<sup>21,22</sup> Furthermore, Co belongs to critical raw materials.<sup>23</sup> Although generally as-prepared Fe-rich microwires present much lower GMI effect than Co-rich microwires, recently a remarkable improvement of magnetic softness and GMI effect in Fe-rich microwires by appropriate post-processing is reported.<sup>24,25</sup> Therefore, less expensive Fe-based microwires presenting higher saturation magnetization are preferable for the applications.

Consequently, in this paper we present our recent experimental results on preparation and magnetic of “thick” amorphous Fe-rich magnetic microwires.

## II. EXPERIMENTAL METHOD

We have studied magnetic properties of  $\text{Fe}_{71.7}\text{B}_{13.4}\text{Si}_{11}\text{Nb}_3\text{Ni}_{0.9}$  glass-coated microwire with metallic nucleus diameter  $d = 103 \mu\text{m}$  and total diameter  $D = 158 \mu\text{m}$  prepared using the Taylor-Ulitovsky method described in details elsewhere.<sup>15,24-26</sup>

The Taylor-Ulitovsky method involves rapid quenching from the melt of glass-coated metallic microwire allowing preparation of homogeneous and rather long (up to a few km long) microwires.<sup>26</sup>

The ingot (of a few grams) of appropriate chemical composition is heated by a high frequency inductor inside a glass tube. After melting of the metallic alloy a glass tube softens covering the metallic droplet. Then a glass capillary is drawn from the softened glass and the molten metallic alloy fills the capillary. The formed glass-coated microwire is then drawn allowing preparation of homogeneous microwire completely covered by a continuous and flexible glass coating.<sup>26</sup>

Samples were annealed in conventional furnace at temperatures,  $T_{\text{ann}}$ , up to  $550^\circ\text{C}$  for annealing time,  $t_{\text{ann}}$ , up to 4 hours. The annealing temperature were chosen considering magnetic softening observed in Finemet-type (Fe-Cu-Nb-Si-B) microwires at  $T_{\text{ann}} \approx 300^\circ\text{C}$  and nanocrystallization at  $T_{\text{ann}} \approx 550^\circ\text{C}$ .<sup>25,26</sup>

Amorphous structure of as-prepared wires is confirmed by X-ray diffraction (XRD) using a BRUKER (D8 Advance) X-ray diffractometer with  $\text{Cu K}\alpha$  ( $\lambda = 1.54 \text{ \AA}$ ) radiation.

The fluxmetric method described in our previous publications is used for measurements of the hysteresis loops of studied microwires.<sup>4,23</sup> In order to compare as-prepared and heat treated samples we used the normalized magnetization,  $M/M_0$  (being  $M$  - the magnetic moment of the sample at given magnetic field,  $H$ , and  $M_0$  - the magnetic moment of the same sample at the maximum magnetic field amplitude) plotted versus magnetic field,  $H$ .

The sample impedance,  $Z$ , was evaluated from the reflection coefficient  $S_{11}$  measured using the vector network analyzer. For the evaluation of the impedance and its magnetic field dependence we used the micro-strip sample holder previously described elsewhere.<sup>27</sup> This technique allows measuring of the GMI effect up to GHz frequency range.

The GMI ratio,  $\Delta Z/Z$ , is defined as:

$$\Delta Z/Z = [Z(H) - Z(H_{\text{max}})]/Z(H_{\text{max}}) \quad (1)$$

where  $H_{\text{max}}$  is the maximum applied DC magnetic field.

The DW velocity,  $v$ , was evaluated by modified Sixtus-Tonks-like setup consisted of three pick-up coils mounted along the microwire placed inside the long solenoid providing the magnetic field.<sup>4</sup>

The propagating DW induces electromotive force ( $emf$ ) in the pick-up coils which can be detected by the digital oscilloscope. Accordingly, the DW velocity can be estimated as:

$$v = \frac{l}{\Delta t} \quad (2)$$

where  $l$  is the distance between a pair of pick-up coils and  $\Delta t$  is the time interval between the  $emf$  peaks.

This technique allows correct evaluation of the magnetic field dependence of the DW velocity.<sup>4</sup>

## III. EXPERIMENTAL RESULTS AND DISCUSSION

In the XRD pattern of as-prepared and annealed (at  $T_{\text{ann}} = 550^\circ\text{C}$  for  $t_{\text{ann}} = 1 \text{ h}$ ) microwires a wide halo at  $2\Theta \approx 45^\circ$  typical for amorphous materials is observed (see Fig. 1). Observed XRD pattern is quite similar to that of as-prepared and annealed prior the devitrification Finemet-type microwires.<sup>25,26</sup>

As discussed elsewhere an intensive wide halo is determined by the shortest distance between atoms.<sup>28</sup> Such XRD pattern is usually reported for the amorphous metal-metalloid alloys presenting significantly weaker scattering from metalloids as- compared to metallic atoms.<sup>28</sup>

However, in the amorphous alloys containing two or more metals with comparable scattering amplitude (e.g., Fe-Zr) double halo can be observed. The appearance of double halo is explained by existence of at least two types of the shortest distances between atoms, which naturally affect the X-ray diffraction pattern.<sup>29</sup>

Indeed, the chemical composition of studied samples (Fe-based with small amount of Ni and Nb) is quite similar to Fe-based Finemet-type microwires (with small amount of Nb and Cu) also presenting single main wide halo in the XRD patterns at  $2\Theta \approx 45^\circ$ .<sup>25,26</sup>

As-prepared glass-coated microwires present generally rectangular hysteresis with relatively low coercivity,  $H_c$ , (about 27 A/m, see Fig. 2a).

We selected two as-prepared samples (presenting the hysteresis loops shown in Fig. 2a) and then annealed each of them at  $T_{\text{ann}} = 300$  and  $550^\circ\text{C}$  respectively. The hysteresis loops of annealed at 300 and  $550^\circ\text{C}$  samples are shown in Figs. 2(b,c) respectively.

After annealing at  $T_{\text{ann}} = 300^\circ\text{C}$  (1h) we observed some magnetic softening:  $H_c$  decreasing from 27 to 12 A/m (see Fig. 2b). However, upon annealing at  $T_{\text{ann}} = 550^\circ\text{C}$  (1h) the opposite tendency (slight  $H_c$  rising from 27 to 41 A/m) is observed (Fig. 2c). The overall shape of the hysteresis loop does not change upon annealing. Such low coercivity values of annealed at  $T_{\text{ann}} = 300^\circ\text{C}$  samples are almost the same as  $H_c$  -values reported for amorphous Fe-rich wires prepared by conventional technique.<sup>30</sup>

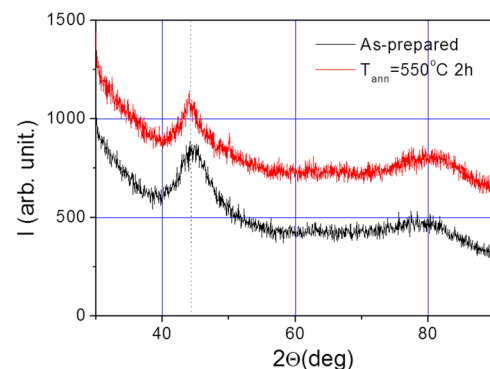
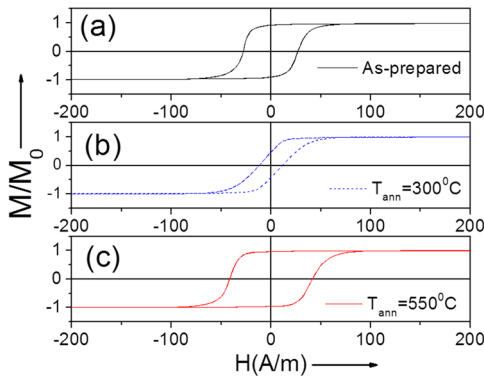


FIG. 1. XRD patterns of as-prepared (a) and annealed at  $550^\circ\text{C}$  for 2h (b) microwire.



**FIG. 2.** Hysteresis loops of as-prepared (a) and annealed at  $T_{ann} = 300$  °C for  $t_{ann} = 1$ h (b) and  $T_{ann} = 550$  °C for  $t_{ann} = 1$ h (c) microwires.

On the other hand, slight magnetic hardening observed upon annealing at  $T_{ann} = 550$  °C can be related either to the very beginning of crystallization or domain wall stabilization linked to pair ordering previously reported for amorphous materials containing two or more ferromagnetic elements.<sup>24,31,32</sup>

The  $\Delta Z/Z(H)$  dependencies of as-prepared and annealed samples measured at 30 MHz and 300 MHz are shown in Figs. 3a and 3b respectively. It is worth noting that as-prepared sample presents  $\Delta Z/Z(H)$  dependencies typical for magnetic wires with axial magnetic anisotropy,<sup>33</sup> i.e., a decay from  $H=0$  values (Figs. 3a, b). However, annealed samples present a double-peak  $\Delta Z/Z(H)$  dependencies (Figs. 3a, b). Consequently, in as-prepared sample maximum GMI ratio,  $\Delta Z/Z_{max}$  of about 50% at 30 MHz and 300 MHz is observed at  $H=0$  (Fig. 3). Observed  $\Delta Z/Z_{max}$ -values for as-prepared samples are clearly superior to  $\Delta Z/Z_{max}$ -values previously reported for thinner as-prepared Fe-rich microwires.<sup>24,31</sup>

In both annealed (at  $T_{ann} = 300$  °C and 550 °C) samples  $\Delta Z/Z_{max}$ -values at 300 MHz are observed at  $H \approx 1.5$  and 1.6 kA/m respectively (Fig. 3b).

Rising the frequency these double-peak dependencies of annealed samples become more evident (Fig. 3b). Such evolution of  $\Delta Z/Z(H)$  dependencies of annealed samples with frequency must be attributed to the magnetic anisotropy distribution within the metallic nucleus taking into account frequency dependence of the skin penetration depth,  $\delta$ , given by:

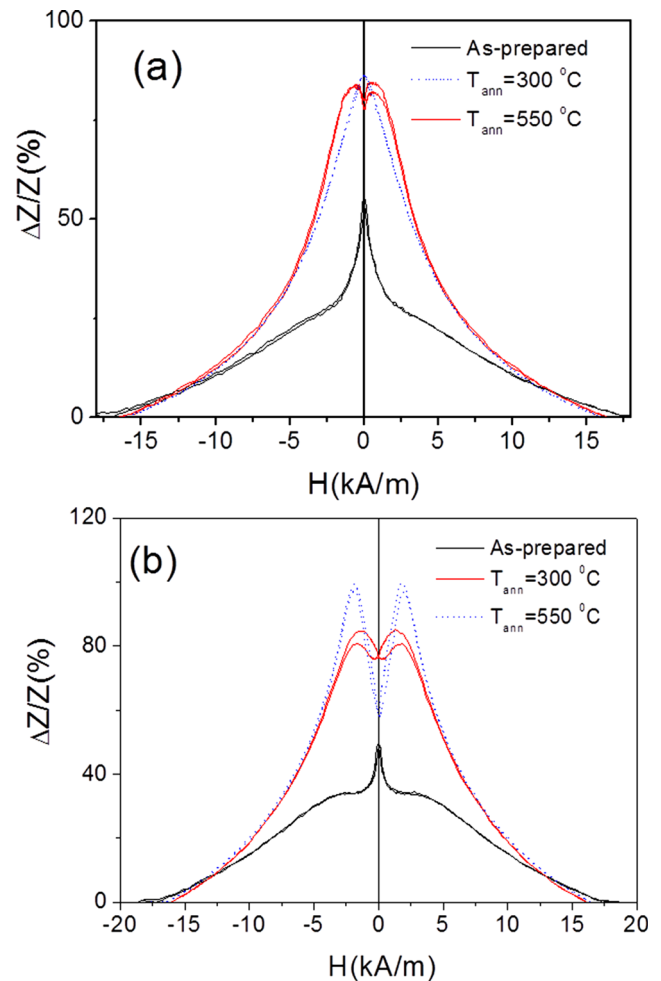
$$\delta = (\pi\sigma\mu_{\phi}f)^{-1/2} \quad (3)$$

where  $\sigma$  is the electrical conductivity and  $\mu_{\phi}$  - the circumferential magnetic permeability.<sup>3,34</sup>

At relatively low frequency (30 MHz), the skin depth is comparable to the microwire radius and the current is flowing through the most part of the ferromagnetic nucleus. However, rising the frequency the penetration skin depth decreases (as follows from eq. 3) and therefore the contribution of the metallic nucleus closer to the surface become more relevant.

Additionally, the double-peak  $\Delta Z/Z(H)$  dependence is predicted and reported in magnetic wires with transversal magnetic anisotropy.<sup>4,19,33</sup>

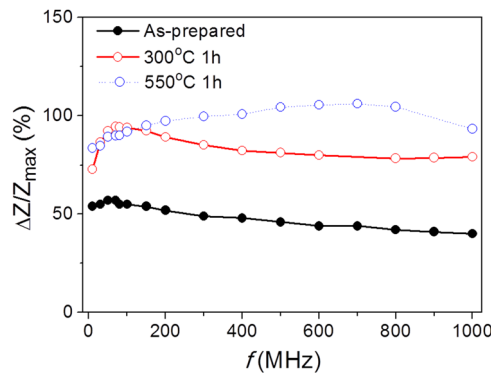
From aforementioned results on double-peak  $\Delta Z/Z(H)$  dependencies of annealed samples and discussion the transverse magnetic



**FIG. 3.**  $\Delta Z/Z(H)$  dependencies of as-prepared microwire and annealed at different  $T_{ann}$  microwires measured at 30 MHz (a) and 300 MHz (b).

anisotropy near the metallic nucleus surface in annealed samples can be assumed. One of the sources of such transverse magnetic anisotropy can be attributed to the formation of thin interface layer between the metallic nucleus and glass-coating. This interfacial layer with the thickness of 0.5  $\mu\text{m}$  has been previously observed for various Fe and Co-rich microwires.<sup>35,36</sup> The interface layer presents different magnetic composition and in consequence different GMI properties.<sup>35,36</sup> The origin of the interface layer was related to the preparation method involving melting of the metallic alloy inside the glass and consequent rapid quenching. However, the relaxation of internal stresses during annealing leads to the fact that the transverse anisotropy in the surface layer becomes more pronounced.

Annealing at  $T_{ann} = 300$  °C and 550 °C allows further GMI ratio improvement up to almost 100% (see Fig. 3). Previously such elevated GMI ratio values have been reported only either in nanocrystalline or stress-annealed Fe-rich microwires.<sup>24,25,31,37</sup>



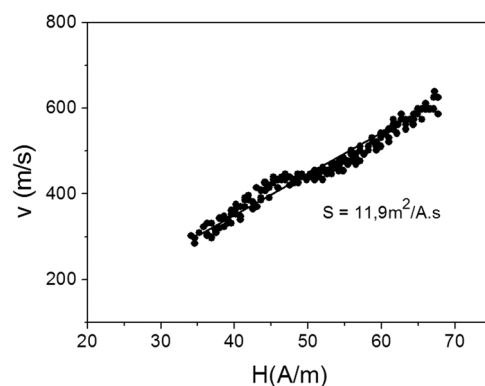
**FIG. 4.** Frequency dependence of maximum GMI ratio of as-prepared and annealed microwires.

Frequency dependencies of  $\Delta Z/Z_{\max}$  for as-prepared and annealed samples are shown in Fig. 4. From this dependence, higher  $\Delta Z/Z_{\max}$ -values in a wide frequency range in annealed samples are evident, as-compared to as-prepared sample.

Additionally, both annealed samples present high GMI ratio in a wide frequency range (see Fig. 4). In all studied samples the optimal frequency for obtaining of high  $\Delta Z/Z_{\max}$ -values is shifted to  $f \geq 100$  MHz, i.e. rather high frequency band as-compared to Co-rich conventional amorphous wires where the optimal frequency typically is about 1 MHz.<sup>38</sup>

This difference must be attributed to different frequency dependence of skin depth due to different domain structure and hence the magnetic anisotropy distribution of conventional and glass-coated microwires.<sup>19,24</sup>

Considering rectangular shape of hysteresis loop exhibited by studied sample, we evaluated the possibility to observe single DW propagation in prepared samples. As can be observed in Fig. 5, as-prepared microwire presents almost linear  $v(H)$  dependence with maximum DW velocity of about 600 m/s. Observed  $v$ -values are similar or even superior to those reported for Fe-rich amorphous wires with similar diameters obtained by in-rotating water technique.<sup>2,39</sup>



**FIG. 5.**  $v(H)$  dependencies of as-prepared microwire.

Almost linear  $v(H)$  dependence is commonly discussed as a viscous DW regime<sup>16,22</sup> given by:

$$V = S(H - H_0) \quad (4)$$

where  $S$  is the DW mobility,  $H$  is the axial magnetic field and  $H_0$  is the critical propagation field.

It is worth mentioning that the  $S$ -value obtained from the  $v(H)$  dependence using the expression (4) gives rather high values ( $S \approx 11,9 \text{ m}^2/\text{As}$ ).

It is remarkable that in the present case, the as-prepared sample presents considerable GMI ratio and single DW propagation. Previously simultaneous observation of GMI effect and DW propagation was reported only for Co-rich microwires with induced by annealing magnetic bistability.<sup>40,41</sup>

From aforementioned experimental results, we can conclude that the Taylor-Ulitovsky technique is suitable for preparation of “thick” magnetic microwires with amorphous structure that can present good magnetic softness, fast DW dynamics and GMI effect.

#### IV. CONCLUSIONS

We successfully prepared amorphous  $\text{Fe}_{71.7}\text{B}_{13.4}\text{Si}_{11}\text{Nb}_3\text{Ni}_{0.9}$  glass-coated microwire with metallic nucleus diameter  $d = 103 \mu\text{m}$  and total diameter  $D = 158 \mu\text{m}$  by the Taylor-Ulitovsky technique. Amorphous structure of as-prepared microwires is confirmed by X-ray diffraction.

As-prepared glass-coated microwires present relatively high GMI effect ( $\Delta Z/Z \approx 50\%$ ) in a wide frequency range and relatively low coercivity (about 25 A/m). Additionally, as-prepared sample present rectangular hysteresis loop and fast single domain wall propagation with the domain wall mobility of about  $11,9 \text{ m}^2/\text{As}$ . After annealing we achieved considerable improvement of the GMI ratio (from 50% up to 100 %) in a wide frequency range.

Observed GMI effect and DW dynamics improvement upon annealing has been attributed to the stresses relaxation.

Consequently, we demonstrated that the Taylor-Ulitovsky technique is suitable for preparation of “thick” Fe based amorphous microwires.

#### ACKNOWLEDGMENTS

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