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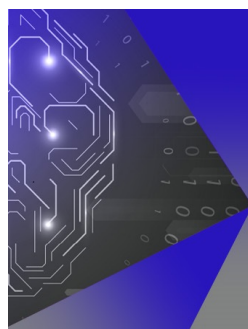
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ABSTRACT

Giant magnetoimpedance, GMI, effect and magnetic properties upon temperature influence of as-prepared and stress-annealed amorphous Fe₇₅B₉Si₁₂C₄ glass-coated microwires produced by the Taylor-Ulitovsky technique are analyzed. Remarkable change in the hysteresis loops and GMI effect is observed for both samples upon heating. Tuning of the stress-annealing conditions allows one to vary the temperature dependence. Furthermore, it is observed almost complete reversibility of the changes induced by the temperature. Observed dependences are explained by the heating effect on the internal stresses relaxation, by the modification of the thermal expansion coefficients of the metallic nucleus and the glass coating, and by the Hopkinson effect.

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I. INTRODUCTION

Magnetic materials are nowadays crucial for a vast range of different applications.^{1–10}

Magnetically soft, amorphous and nanocrystalline microwires among the magnetic materials obtained by rapid melt quenching have great potentiality in terms of mentioned applications and industry adoption owing to their outstanding and adjustable magnetic properties together with their economical and relatively simple fabrication technology.^{5,6} Thus, continuous investigation on magnetic wires has been developed since the 70s.^{11–13}

Taylor Ulitovsky fabrication technique allows the fabrication of glass-coated microwires that can present exceptional magnetic softness, high Giant magnetoimpedance, GMI, effect^{2,5,13} and fast magnetization switching,^{5,14} which are joined with the reduced

dimensions, satisfactory mechanical performance, enhanced corrosion resistance¹⁵ and biocompatibility^{6,7,13} that the glass coating confers. Such physical properties combination makes glass-coated microwires appropriate for the optimization of current and emerging applications.^{6–9,15}

Hysteresis loops, GMI effect and domain wall dynamics dependencies on the external stimuli such as stress, temperature or magnetic field allow the possibility of sensing remotely for example in structure health monitoring, manufacturing processes control, magnetoelastic sensorics or in biomedicine, etc.^{6–10,15,16}

Recently, multifunctional composite materials with magnetic wires inclusions have been proposed for nondestructive and contactless stress and temperature monitoring.¹⁷ As a consequence, studies of temperature dependence of the GMI effect become essentially

pertinent. Up to now, there have been very few studies on temperature dependence of GMI and most of them restricted to thicker amorphous wires without glass coating and to rather limited temperature range.^{18–20} As a rule, the highest GMI effect is reported in Co-rich magnetic wires.^{5,12} However, it has recently been found that in stress-annealed Fe-rich glass-coated microwires the magnetic softness and GMI effect can be substantially improved.^{21–23} The main advantages of Fe-rich microwires are their lower price and higher saturation magnetization. As a result, investigation on Fe-rich microwires with high GMI effect has attracted newly significant emphasis.^{10,21–23}

In consequence, in this paper we provide our most recent experimental results on the influence of temperature on the magnetic properties and GMI effect of as-prepared and stress-annealed Fe₇₅B₉Si₁₂C₄ microwires.

II. EXPERIMENTAL DETAILS

Amorphous Fe₇₅B₉Si₁₂C₄ glass-coated microwires with metallic nucleus diameter $d = 15.2 \mu\text{m}$ and total diameter $D = 17.2 \mu\text{m}$, were investigated. Fe-rich glass coated microwires are characterized by positive magnetostriction coefficient, λ_s . λ_s value of about 38×10^{-6} was earlier on reported for the microwires studied.^{22,24}

The Taylor-Ulitovsky method, known since the 60s^{25,26} and substantially modified during last decades^{10,27} has been used to prepare samples. This preparation technique involves the melting of the metallic alloy inside a Duran glass tube using a high frequency inductor heater, drawing out a soften glass capillary filled with the molten metal alloy, and wining of the composite microwires onto a rotating spool. The recent technological development of this technique made it possible to expand the diameter range of the microwires from 0.1 up to 100 μm ^{27,28} together with new and improved functional properties, such as the GMI effect.^{11–13}

Fe₇₅B₉Si₁₂C₄ microwires were stress-annealed in a standard furnace at 325 °C during 15 minutes applying a mechanical load of 190 MPa during the annealing.^{21,22,29} The temperature inside the furnace during the treatment was monitored by a commercial thermocouple (NiCr-Ni).²⁹

Using X-ray diffraction (XRD) and differential scanning calorimetry (DSC), the amorphous structure of the as-prepared and stress-annealed up to 400 °C samples was confirmed.³¹

Hysteresis loops of the microwires were measured by means of vibration sample magnetometer (VSM) MicroSense EV9 and by fluxmetric method. For the measurements microwire samples of 5 mm in length were employed. The measurements were performed from ambient temperature up to temperature $T = 300 \text{ }^\circ\text{C}$, as previously described in detail.³²

For the GMI effect evaluation the GMI ratio, $\Delta Z/Z$, was used, determined as:^{11–13}

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

being Z the impedance of the microwire, H and H_{max} , the applied DC magnetic field and the maximum field, respectively.

Specially designed experimental system for impedance measurements of the microwire and evaluation of the GMI ratio in a temperature, T , range from ambient temperature up to 300 °C at frequencies up to 110 MHz.³³ The GMI effect measurements were performed at a frequency range from 5 MHz up to 110 MHz. For evaluation of the effect of temperature on the GMI effect intermediate frequency of 50 MHz was selected.

III. RESULTS AND DISCUSSION

Hysteresis loops of as-prepared Fe₇₅B₉Si₁₂C₄ microwires measured at high field under the temperature influence are presented in Fig. 1(a). Similar to what was reported earlier, magnetic bistability, characterized by a near-perfect rectangular hysteresis loop, is observed in as-prepared Fe₇₅B₉Si₁₂C₄ microwires.²¹ Rectangular hysteresis loop transforms gradually into inclined one with the temperature increase. Such a transformation can be clearly seen from Fig. 2, where low field hysteresis loops measured at ambient temperature and at $T=100 \text{ }^\circ\text{C}$ are shown. Anisotropy field, H_k , evaluated from the curvature change in the hysteresis loop exactly before approaching the magnetization saturation, M_s , shows an increase until $T = 250 \text{ }^\circ\text{C}$, where it starts decreasing.

The axial magnetic anisotropy of Fe-rich microwires with positive λ_s values is due to their singular domain structure, consisting of an internal single domain with axial magnetic anisotropy and an external domain shell with a radially oriented magnetization.²³ This particular domain configuration explicates the rectangular character of the hysteresis loops, related to an exceptionally fast magnetization switching by single domain wall propagation.

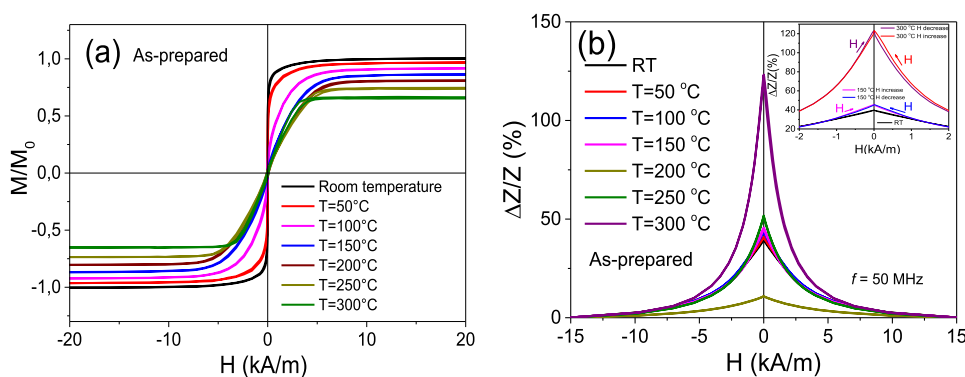


FIG. 1. Hysteresis loops (a) and GMI ratio at $f = 50 \text{ MHz}$ (b) of as-prepared Fe₇₅B₉Si₁₂C₄ microwires measured at different temperature.

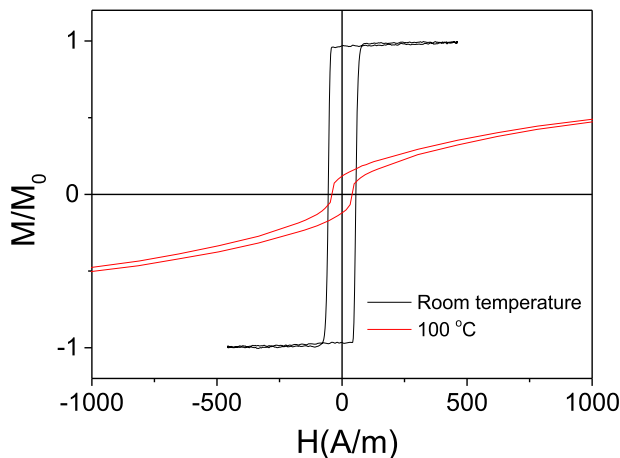


FIG. 2. Low field hysteresis loops of as-prepared $\text{Fe}_{75}\text{B}_9\text{Si}_{12}\text{C}_4$ microwires measured at ambient temperature and at $T = 100^\circ\text{C}$.

The axial magnetic anisotropy in Fe-rich microwires correlates with the internal stresses that arise during the manufacturing process. The main contribution to the internal stress is due to the difference in the thermal expansion coefficients of the glass cover and the metallic nucleus.^{34,35} The axial component of the internal stresses is predominant in the main part of the metallic nucleus of the microwire.^{23,34,35} For this reason, the temperature dependence of the hysteresis loops can be explained by the decrease in the internal stresses due to the decrease in the difference between the thermal expansion coefficients as well as by the internal stresses relieve derived by the heating.

The consequent effect of temperature on the GMI ratio dependencies can also be observed in Fig. 1(b). GMI ratio dependence measured at frequency $f = 50$ MHz exhibits low values and a single peak shape dependence at ambient temperature and low heating temperatures associated with axial anisotropy that starts to transform into double peak dependence, typically related to circumferential anisotropy and relatively high maximum GMI ratio, $\Delta Z/Z_{\text{max}}$, values. The beginning of such transformation can be appreciated from slight splitting of the single peak at the highest temperature of 300°C

and by a slight increase in the GMI hysteresis with T increasing [see Fig. 1(b)]. The origin of the GMI hysteresis is heretofore discussed in terms of the helical magnetic anisotropy contribution as well as by the magnetostatic interaction of the inner axially magnetized core with the outer domain shell.²² Maximum GMI ratio values have an appreciable increase from the values obtained at ambient temperature up to $\Delta Z/Z_{\text{max}} \approx 125\%$ for $T = 300^\circ\text{C}$. Although there is an exception in this tendency for 200°C , where maximum GMI ratio values are lower than those of as-prepared sample without applying temperature. The transformation of the hysteresis loops from near-perfect rectangular to a sloping one correlates with the GMI effect improvement.

The temperature dependence of hysteresis loops of stress-annealed $\text{Fe}_{75}\text{B}_9\text{Si}_{12}\text{C}_4$ microwire show the same tendency as for as-prepared sample [see Fig. 3(a)]. The microwires were stress annealed at annealing temperature $T_{\text{ann}} = 325^\circ\text{C}$ for annealing time $t_{\text{ann}} = 15$ min and applied mechanical stress, $\sigma = 190$ MPa. Induced transverse anisotropy strongly depends on the annealing conditions.^{21–23}

A similar tendency, as for as-prepared samples, was also observed in the GMI ratio measurements at 50 MHz for stress-annealed samples [Fig. 3(b)]. Consequently, with the hysteresis loops modification an improvement in the GMI ratio values is achieved. Even higher improvement in the maximum GMI ratio values (up to $\Delta Z/Z_{\text{max}} \approx 220\%$) was observed at $T = 300^\circ\text{C}$. The induction of transverse anisotropy by stress annealing, as earlier on reported, results in noticeable GMI effect enhancement.^{21–23,30}

For explanation of the temperature evolution of the hysteresis loops and GMI effect, a superposition of several effects is considered, including the Hopkinson effect, change of internal stresses with temperature and partial stresses relaxation.

To study the change in the internal stresses a comparison of the GMI ratio dependencies for the sample stress-annealed measured before and after heating up to 300°C is presented in Fig. 4, as well as the related change in the hysteresis loop (inset of Fig. 4). Slight hysteresis loop and GMI effect modification can be observed. However, as recently reported for the same FeBSiC microwires,³³ partial reversibility of the stress-annealing induced magnetic anisotropy is confirmed.

The partially reversible behavior of the stress-annealing induced magnetic anisotropy indicates that the origin of such induced anisotropy lies in the so-called “back stresses” as well as

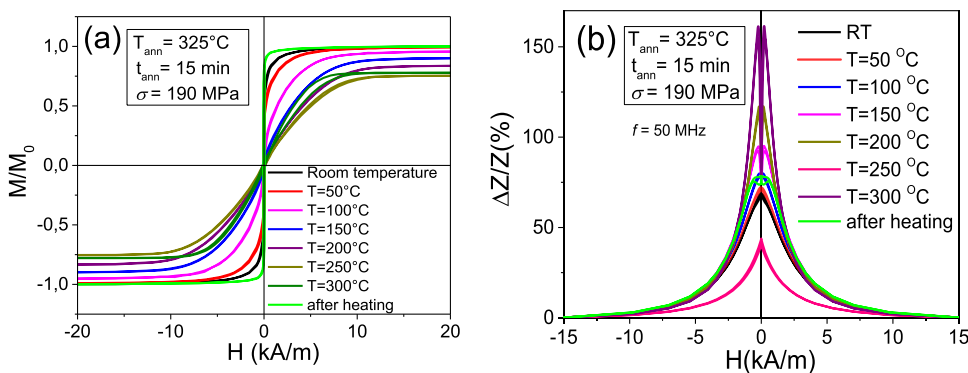


FIG. 3. Hysteresis loops (a) and GMI ratio at $f = 50$ MHz (b) of stress annealed sample at 325°C during 15 min (applied stress $\sigma = 190$ MPa), measured at different temperature.

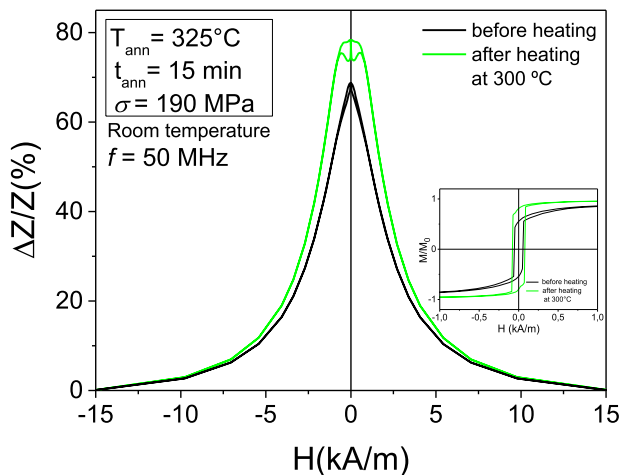


FIG. 4. $\Delta ZZ(H)$ dependencies of stress-annealed sample ($t_{ann} = 15$ min) before and after heating at 300 °C and hysteresis loops of the same samples (inset).

the structural magnetic anisotropy related to topological short range ordering.^{23,33}

An increase in the magnetic susceptibility observed below the Curie temperature, T_c is commonly explained in terms of the Hopkinson effect,^{36–38} associated with a faster magnetic anisotropy constant decrease with temperature comparing with the magnetization. In our case, although $T_c \approx 410$ °C,^{21–23} the comparison of the hysteresis loops of as-prepared and stress-annealed samples [Figs. 1(a) and 3(a), respectively] at high temperatures (particularly from 200 °C up to 300 °C) shows a change in the slope of the hysteresis loops (that must attributed to magnetic permeability) upon heating at $T > 250$ °C.

Comparably, GMI effect enhancement upon heating below T_c was recently reported in Co-Fe-Ni-rich microwires with low T_c .³⁹

Despite the fact that our experimental set-up allows heating temperatures only up to 300 °C, Hopkinson effect contribution in the observed GMI effect evolution seems feasible.

The observed temperature dependencies of the GMI effect in as-prepared and stress-annealed $Fe_{75}B_9Si_{12}C_4$ microwires open up new possibilities in temperature monitorization applications.

IV. CONCLUSIONS

Hysteresis loops and GMI ratio measurements of as-prepared and stress-annealed $Fe_{75}B_9Si_{12}C_4$ amorphous microwire showed substantial temperature dependence of the magnetic properties and GMI effect upon temperature. Magnetic softening upon heating of as-prepared $Fe_{75}B_9Si_{12}C_4$ microwire and consequent GMI ratio improvement are observed. Such magnetic softening and increase in the GMI ratio present almost complete reversibility. For the case of stress-annealed microwires despite the improvement observed in the GMI ratio after the stress annealing GMI ratio values decrease with temperature increase. Adjustment of the stress annealing conditions allows to modify the temperature dependence of the hysteresis loops and GMI ratio values.

The origin of observed temperature dependences is explained taking into account contributions from the Hopkinson effect, the relaxation of the internal stresses and temperature dependence of the named internal stresses.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

P. Corte-Leon: Data curation (equal); Formal analysis (equal); Investigation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal). **I. Skorvanek:** Conceptualization (equal); Formal analysis (equal); Funding acquisition (equal); Investigation (equal); Methodology (equal); Resources (equal); Supervision (equal); Validation (equal). **F. Andrejka:** Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal). **V. Zhukova:** Conceptualization (equal); Investigation (equal); Methodology (equal); Supervision (equal); Validation (equal). **J. M. Blanco:** Conceptualization (equal); Investigation (equal); Methodology (equal); Supervision (equal); Validation (equal). **M. Ipatov:** Investigation (equal); Supervision (equal); Validation (equal). **A. Zhukov:** Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Methodology (equal); Resources (equal); Validation (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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