Heat and Current Annealing Effects on Magnetic Properties of Fe-rich Glass-Coated Amorphous Microwires with Different Radius

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***Abstract*—For Fe-rich glass-coated microwires to be a competitive option for magnetic technologies, the enhancing and tuning of their properties is necessary, and therefore, a better understanding of these processes is also needed. In this work, two Fe microwires with different geometries are subjected to both conventional and current annealings to study their effects. Results show a general enhancement of magnetic softness and domain wall dynamics after both treatments, also suggesting an earlier onset of crystallization in thin samples.**

Keywords- Amorphous alloys; Annealing; Glass-coated microwires; Magnetic characterization.

I. INTRODUCTION

Glass-Coated amorphous microwires are wires with radius sizes of µm, consisting of an amorphous metallic alloy core enveloped by a glass coating. The main interest in this kind of materials comes from their wide range of magnetic properties, such as: bi-stability, ultra-fast magnetic switching, magnetic softness [1] and the general modification of most of these properties under different situations, like the application of high temperatures and/or electrical currents to a microwire sample. As magnetic properties of these microwires are dependent on their chemical composition and geometric parameters, so are the resulting changes on them after annealing, thus the same process parameters may produce different results on different kinds of samples.

While Fe-rich microwires properties are generally considered inferior (mainly lower magnetic sensitivity and magnetic softness) to those possessed by Co microwires, they represent a cheaper, more obtainable alternative [1]. Thus, annealing might prove to be an important asset in producing microwires with properties tuned to their desired use. As such, the main goal of this work is to measure some of the magnetic properties of two different types of glass-coated microwires under the same annealing conditions, to obverse and better understand the resulting changes and the differences between them.

Henceforth, this paper is structured as follows: Section II focuses on the physical techniques used to procure the experimental data to be studied in this paper. As such, Section III consists of a series of subsets, each focusing on the observation and study of data, each related to a relevant property of the microwires and their change with different parameters. After exposing the results, Section IV conducts a briefer explanation and discussion on them, and the conclusions that can be drawn from this work.

II. EXPERIMENTAL

In this section we introduce and describe the experimental procedures applied in the obtention of our studied data, consisting of both the fabrication technique of our microwires and the measurement experiments conducted to study their magnetic properties, as well as their most relevant parameters to their results.

A. Fabrication and Parameters of the Microwires

While some different fabrication processes for amorphous magnetic microwires may be used, we focus on the Taylor-Ulitovsky technique, explained in further detail in [2], as it allows to produce up to 103 m of a single microwire, while simultaneously coating it with Duran type glass. The importance of this glass coating comes from its mechanical interaction with the metallic core. As the alloy lacks crystalline ordering, magnetoelastic anisotropy is one of the main parameters to define its magnetic structure. Therefore, the presence of a glass coating and the stresses that it brings to the core strongly contribute to its magnetic behavior. As such, chemical composition of the core is also relevant, as it defines the magnetostriction constant, λ. By this criteria, two general compositions are usually described and studied: Co-rich with $\lambda<0$, and Fe-rich with $\lambda>0$ [1].

A notable characteristic of microwires, originated from their λ values and the internal stresses originated from the shell-core interactions, are magnetic domain structures: an inner and outer domain arranged in a cylindrical symmetry, with the inner domain presenting axial magnetic anisotropy, pararel to the wire's axis for both Co and Fe based microwires. However, while the former's outer domain has circular magnetic anisotropy, the latter's anisotropy is radial,

 ISBN: 978-1-68558-091-9

TABLE I. TABLE TYPE STYLES

Sample name	*Composition*	*Inner diameter d (μm)*	*Total Diameter D (μm)*	*d/D*
"Thick"	$Fe_{71.8}B_{13.27}Si_{11.02}Nb_{2.99}Ni_{0.92}$	47.9	53.2	0.9
"Thin"	$Fe_{74.87}B_{9.06}Si_{11.99}C_{4.08}$	15.2	17.2	0.88

Composition and geometries of studied microwires. The inner diameter, d, corresponds to only the metallic core, while the total diameter, D, includes both core and glass coating.

usually with a proportionally thinner inner domain [1]. These differences in magnetic domains are related to magnetic properties of microwires: while Co-based microwires typically attract more attention, as they show higher magnetic softness, Giant Magneto-Impedance (GMI) effect and sensitivity to stimuli (like stress or magnetic fields), Fe is a more common, cheaper material, allowing for the production of bigger quantities of microwire for less costs, making it an attractive alternative for the long run [1].

On the other hand, treatment of microwires has shown observable changes of their magnetic properties, depending on both treatment conditions and microwire parameters [3]. Therefore, a correct combination of these two elements may be used to tune properties into the most advantageous for desired applications. With all of this in mind, two Fe-rich glass coated microwires have been fabricated and characterized, as shown in Table I. From these microwires, several 24 cm samples were then cut off, as to allow their proper handling for annealing and experimentation.

B. Magnetic Characterization and Annealing

For this work, we focus on the measurement of the hysteresis loop, Domain Wall (DW) velocity and GMI efficiency of microwire samples before and after specific annealing treatments. Measurements of hysteresis loops are carried out via a simple fluxmetric method at 114Hz, with a 13cm long inductive coil, and 2 cm pickup and compensator coils. DW dynamics is studied with a modified Sixtus-Tonks method [4], using three pickup coils to detect the change in magnetic flux produced by the change in the sample magnetization as domain walls move through them.

Determination of the GMI efficiency is carried out by using a network vector analyser, as described in [5], which allows measuring the impedance Z of a ~5mm sample under the action of both an alternate current at fixed frequencies and homogeneous magnetic fields. Thus, for each AC frequency we can evaluate the GMI ratio as a function of the applied magnetic field, H [6]:

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

Hmax being the max value of the applied magnetic field in the used experimental setup. Measurements were carried out on samples subjected to at least one of two different types of annealing: conventional annealing, by simply introducing them into a furnace at temperatures of 300, 400 and 500 ºC, for 1 and 3 hours; and current annealing, by making a direct current pass through them. Current intensities and annealing times presented in this work were chosen as it had been previously observed that they produce the most noticeable changes in these microwires [7].

III. RESULTS

Having produced our microwire samples and experimented on their magnetic behavior, we now proceed to study the obtained data on their magnetic hysteresis, domain wall dynamics and giant magneto-impedance effect, comparing the results depending on measurement and annealing conditions, as to ascertain the effect of said conditions and their possible combination to tune the microwire's properties for desired applications.

A. Magnetic Hysteresis

Some examples of the measured loops are presented in Figure 1. As usual for Fe based microwires with positive λ (~10-6), their shapes are fairly rectangular, with a single Barkhausen jump and magnetic bi-stability [8]. It can be seen that, the thicker the sample, the bigger its coercive field, HC. It is also worth noting that the effect of current annealing seems to be greater on both samples, as it brings HC to lower values.

To better compare the effects of annealing, Figure 2 shows the measured HC obtained for all realized treatments, except for the "Thin" wire annealed with 500ºC. It is obvious that for "Thick" samples, the higher the annealing temperature, the bigger its reduction of HC as a result of the relaxation of their inner stresses. Opposite to this, "Thin" samples show a raise after long enough annealing times, as their higher surface to volume ratio allow for higher thermal energy densities, and thus, for an earlier onset of the alloy's crystallization. On the other hand, current annealing of all samples shows a consistent reduction of HC, even for long annealing times.

The reason for excluding "Thin" samples annealed with 500ºC is that they exhibited rather different results, as can be seen in Figure 3. Annealing at high temperatures of "Thin" samples resulted on an observable magnetic hardening, most likely as a result of crystallization of the metallic core [1].

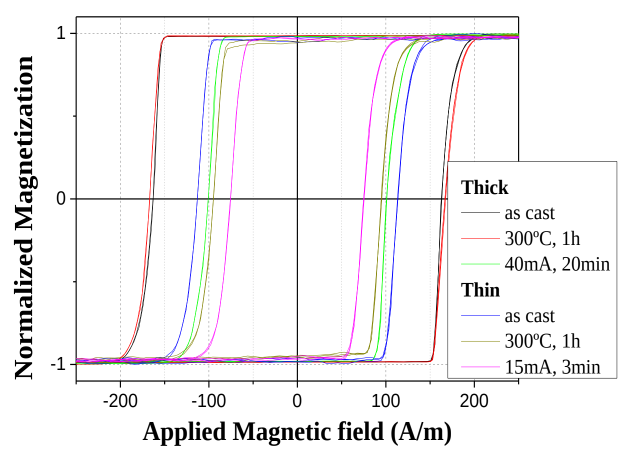

Figure 1. Hysteresis loops of samples before and after different treatments.

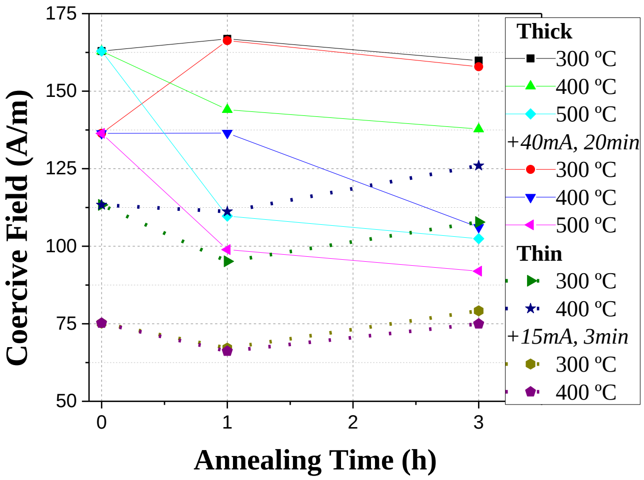

Figure 2. H_C dependece on annealing times. The “+” indicates the application of current annealing after conventional annealing.

Additionally, as the inset in Figure 3 shows, a small metastable transition between two Barkhausen jumps is taking place at around 0 magnetization, which could be hinting at the presence of two distinct crystalline phases in the metallic core upon its devitrification, with the magnetization of one phase beginning shortly after the other one is completely done. Furthermore, current annealing of these samples may be resulting in a change in the proportion of said crystalline phases, as the position of this metastable step is modified upon the application of current.

B. Domain Wall Dynamics

After measuring DW velocities of all samples under applied field, a consistent pattern, briefly represented in Figure 4, was observed. Firstly, “Thin” microwires possess higher DW mobility (slope of the field dependence of DW velocity [4]), than “Thick” ones, reaching higher DW velocities for the same applied field. Conventional annealing of both types of samples yields a boost on DW mobility, especially on the “Thin” ones.

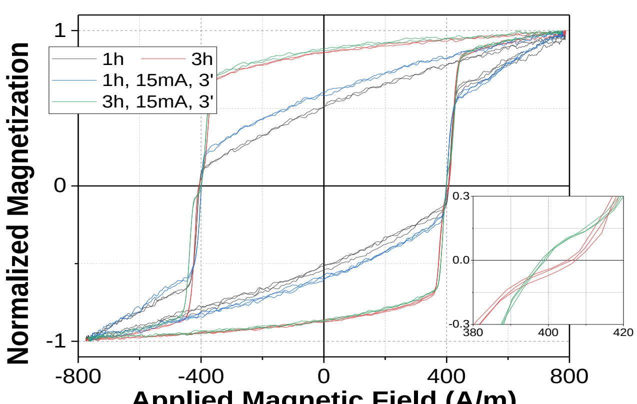

Figure 3. Hysteresis loops of “Thin” microwires annealed with 500˚C for different times and with posterior current annealing.

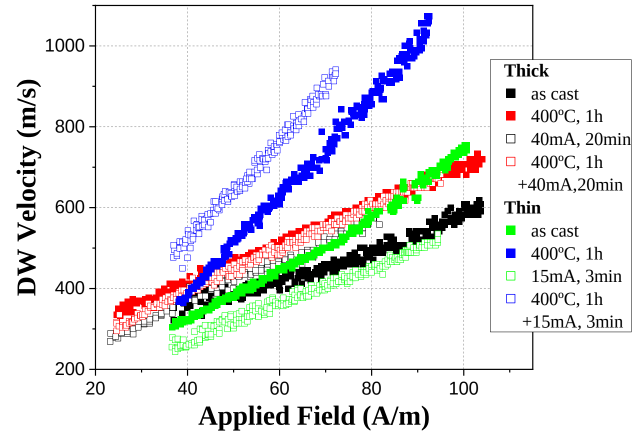

Figure 4. DW velocities of samples after annealing.

DW velocities of “Thin” samples annealed at 500º could not be measured, most probably due to their magnetic hardening, as shown in Figure 3, which would make them require a higher magnetic field than those produced by our system for their DW to star propagating. Secondly, current annealing results on different effects depending on the sample and previous annealing: while current alone produces a slight increase of DW mobility of “Thick” microwires, “Thin” ones experience an observable reduction. However, current annealing after conventional annealing seems to have opposite effects, as “Thick” samples seem unaffected while the boost on mobility of “Thin” ones grows even bigger.

C. Giant Magneto-Impedance

The GMI ratio of all samples was measured for frequencies up to 1 GHz. A short, representative example is shown in Figure 5. This figure carries some interesting information points about the magnetic domain structure of our microwires, as well as about their behavior under the action of annealing and their contribution to the GMI effect efficiency. First, both type of microwires exhibit a double

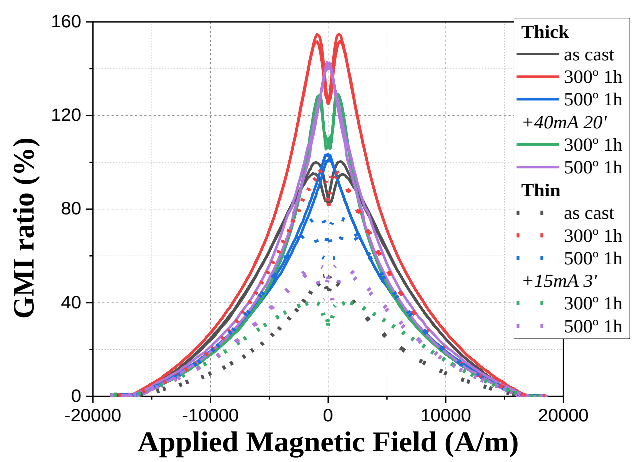

Figure 5. GMI ratios of microwire samples at 200 MHz.

peak structure in their GMI graphs, a phenomenon usually related to the magnetic domain structure of Co-rich microwires. This is congruent with previous results obtained by our own research [7]. However, when “Thick” samples are annealed at high enough temperatures, this double peak is lost at lower frequencies, appearing as the single peak expected in Fe-rich microwires. The most probable cause for this is the relaxation of internal stresses in the microwire, causing a reduction in size of the outer domain and, therefore, its contribution to the GMI effect.

Secondly, while as-cast “Thick” samples show a GMI max ratio (100%) quite higher than that of “Thin” ones (50%), and annealing of both microwires results in an enhancement of their ratios, the growth of “Thin” GMI (from 50% to 100%) is proportionally bigger than that observed in “Thick” ones (from 100% to less than 160%).

To better understand how annealing affects each microwire, Figure 6 and Figure 7 represent the max values of GMI ratios of annealed “Thick” and “Thins” samples respectively. When comparing both graphs, it becomes obvious that thicker microwires yield a higher GMI performance, as the highest ratios produced by annealed “Thin” samples does not surpass those of the “Thick” as cast sample. While it can be seen that annealing has indeed provided an enhancement on GMI ratios of both microwires, a closer look on both graphs reveals behaviors that are more complex. In Figure 6, conventional annealing at 300ºC results in the highest GMI values of the sample at both 1 and 3 hours, while annealing at 500ºC lowers said values back to a level similar to that of the as cast state. The addition of current annealing after these processes yields a negative effect in most cases, as the GMI ratios values become lower, with the exception of the annealing with 500ºC for 1 hour, which results in an enhancement comparable to those obtained at 300ºC.

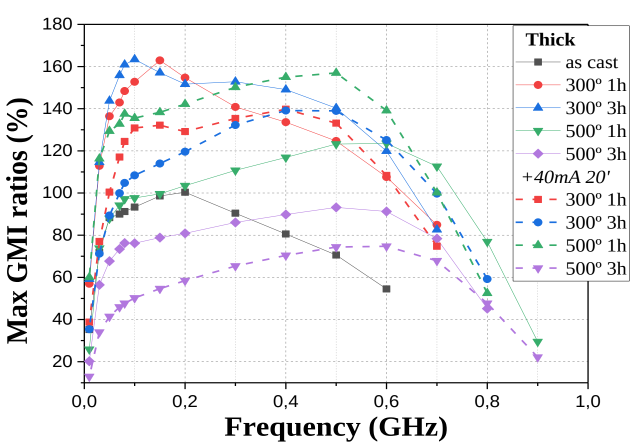

Figure 6. Max values of GMI ratios of annealed “Thick” samples.

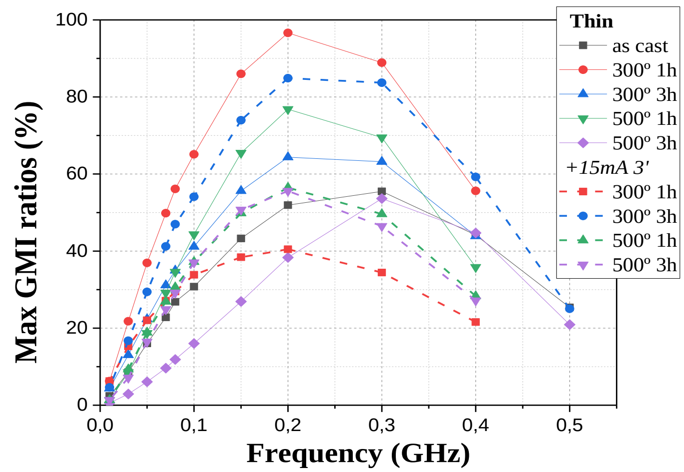

Figure 7. Max values of GMI ratios of annealed “Thin” samples.

IV. CONCLUSIONS

While thinner microwires possess better magnetic softness due to lower HC values, they also show a lower resistance to heat as, under the same annealing conditions, there are hints of alloy crystallization, not present on thicker ones. Current annealing may prove to be a decent alternative to heat annealing as, with only some minutes of treatment, the reduction of HC is bigger than annealing under 400ºC for 3h (Figure 2). Combination of both annealings has yielded a further reduction of these values, especially in thicker microwires. Opposite to these trends, DW dynamics show that annealing barely affects thick microwires, while thin ones can be subjected to either a boost or reduction of their DW velocities.

Conventional annealing of both types of microwires at 300ºC for 1 hour has shown to yield the highest values of GMI ratios, while de addition of current annealing after this process is mostly detrimental, except for specific cases in which said values are once again enhanced after their lowering due to excessive annealing temperatures or times. This might prove to be an interesting option to “repair” the sensitivity of microwires used in magnetic sensing technologies at high temperatures.

ACKNOWLEDGMENTS

This work was supported by EU under “INFINITE” (HORIZON-CL5-2021-D5-01-06) project, by the Spanish MICIN, under PID2022-141373NB-I00 project and by the Government of the Basque Country under PUE_2021_1_0009, Elkartek (MINERVA, MAGAF and ZE-KONP) projects and under the scheme of “Ayuda a Grupos Consolidados” (ref. IT1670-22). The authors thank for tech-nical and human support provided by SGIker of UPV/EHU (Medidas Magneticas Gipuzkoa) and European funding (ERDF and ESF).

We also wish to thank the administration of the University of the Basque Country.

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