Effect of Stress Annealing on Domain Wall Dynamics in Nanocrystalline Hitperm-Type Microwires

A. Talaat[1,2,*], P. Klein[3], R. Varga[3,4], V. Zhukova[1,2], J.M. Blanco[2] M. Ipatov[1,2] and A. Zhukov[1,2,5]

[1]Dpto. Fisica de Materiales, UPV/EHU, 20018, San Sebastian, Spain

[2]Dpto. de Física Aplicada, EUPDS, UPV/EHU, 20018, San Sebastian, Spain

[3]RVmagnetics, a. s., Hodkovce 21, 04421 Košice, Slovakia

[4]Institute of Physics, Faculty of Science, University of Pavol Jozef Safarik, Park Angelinum 9, 041 54 Kosice, Slovakia

[5]IKERBASQUE, Basque Foundation for Science, 48011 Bilbao, Spain

Abstract: We have studied the effect of stress induced anisotropy on domain wall dynamics in as-cast and annealed nanocrystalline Hitperm-type microwires. Annealing without stress leads to stress relaxation of the strong stresses frozen-in post the production process. Stress annealing at 300°C under 222.7 and 270.9 MPa, respectively, has triggered most complex domain wall dynamics.

Observed results are discussed considering that annealing under higher stresses leads to an enhancement of longitudinal (axial) anisotropy due to positive magnetostriction of both bcc-(Fe, Co) grain as well as the residual amorphous matrix, and subsequently due to the decreasing of axial anisotropy due to back stresses arising from the glass-coating after removing the mechanical load.

Magnetic anisotropies (axial and radial ones) after stress annealing can be responsible for the considerable influence of the annealing conditions on domain wall dynamics observed in Hitperm-type microwires. As a result of decreasing both anisotropies by different ways, the domain wall velocity decreased.

Keywords: Magnetic wires, Hitperm, Nanocrystalline materials, Domain wall dynamics, Longitudinal anisotropy, Compressive stresses.

INTRODUCTION

Studies of the domain wall dynamics in magnetic nano- and microwires have attracted significant attention considering either their application possibilities in spintronics devices and various sensors as well as actuators, or from a theoretical point of view (supersonic boom, negative critical field for the domain wall propagation, estimation of the domain wall mass or etc) [1-6]. Fast velocity of the domain wall and temperature stable domain wall dynamics are needed for reliable work of such devices [6, 7]. In contrary, undesired effect as decreasing of the average domain wall velocity above Walker breakdown can be suppressed by various ways (application of transverse magnetic field, increasing of the temperature, small amplitude periodic structuring, various annealing in furnace below or above crystallization temperature in order to obtain stable nanocrystalline state) [8-11].

It has been considered elsewhere [6, 7, 9] that the domain structure of studied amorphous microwires is governed by two dominant anisotropies: magneto-elastic and shape anisotropy. As a result of magnetoelastic anisotropy, the domain structure of microwires with positive magnetostriction consists of a single axial monodomain in the center of the metallic core, which is surrounded by radial domain structure. Due to the shape anisotropy, small closure domains appear at both ends of the microwire in order to decrease the stray fields (see Figure **1a**). Such domain structure is characterized by bistable behaviour. Particularly, the magnetization in axial single domain can have only two states $+M_S$ or $-M_S$. Switching between these two stable configurations is driven by the domain wall propagation at the field called switching field.

Similarly, magnetocrystalline anisotropy in nanocrystalline microwires is averaged out over many grains with different orientation of its magnetic moments if size of grains is significant and distance between them is smaller than ferromagnetic exchange length. Thus, magnetocrystalline anisotropy is equal to zero and dominant anisotropy in nanocrystalline microwires is the magnetoelastic one [9, 13]. From above mentioned reasons bistable magnetic

*Address correspondence to this author at the Dpto. Fisica de Materiales, UPV/EHU, 20018, San Sebastian, Spain; Tel: +34-943-018611;
Fax: +34-943-017130; E-mail: ahmedtalaatfarag@gmail.com

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amorphous or nanocrystalline glass-coated microwires are ideal material to study single DW dynamics on macroscopic scale [9, 12].

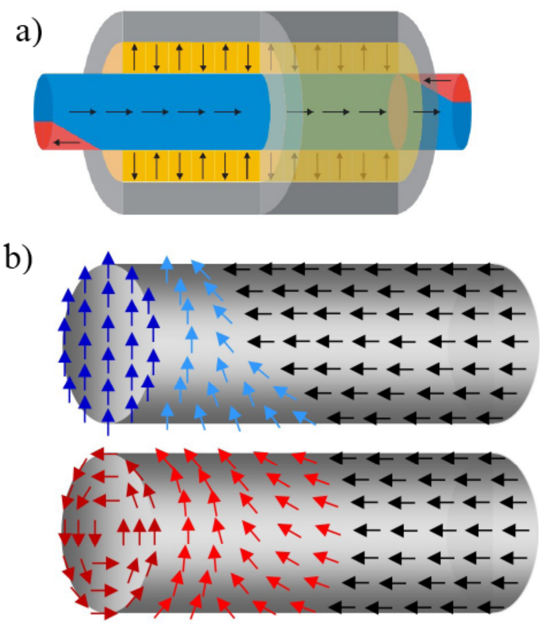

Figure 1: a) Schematic domain structure of amorphous and nanocrystalline microwire with positive magnetostriction, **b**) schematic picture of transverse (up) and vortex-type domain wall (down).

In fact the structure of propagating domain wall is quite complex and the length of propagating domain is much higher than the microwire diameter [11, 13, 14]. Therefore usually modeling of the propagating domain wall (DW) in amorphous microwires is very simplified. Generally experimental data can be described considering conical and elongated DW shape [11, 13, 14]. But in other papers in a very simplified approximation (considering huge dimensions of such DW and similarity to nanowires (where the length of propagating domain wall is few orders shorter) roughly two different domain wall structures can be assumed in glass-coated microwires. First, it is the transversal domain wall that has lower energy, lower domain-wall mobility, and appears in thinner wire or at low magnetic fields. On the other hand, a vortex domain wall needs more energy than the transversal one in order to be created. However, it has higher domain-wall mobility and appears in thicker wires or at higher magnetic fields [12, 13].

However, a considerable disadvantage of amorphous microwires is their unstability or change magnetic properties with time (ageing) and with temperature [15, 16]. One possible solution is to use nanocrystalline materials [17], which are prepared by controlled annealing from the amorphous precursors and they consist of small nanocrystalline grains embedded in the residual amorphous matrix.

The most common nanocrystalline materials known as Finemet (FeSiBNbCu) show excellent soft magnetic properties (high permeability, low coercivity, and high saturation magnetization) comparable to Co-rich amorphous materials. Second well known class called Nanoperm (Fe-*X*-B-Cu) where *X* is Zr, Nb, Hf, Mo or etc. is optimized for low magnetostriction and as well as for high saturation magnetization. However, disadvantage of Nanoperm is low Curie temperature. Therefore, third class of nanocrystalline materials was proposed and is known as Hitperm Fe-Co-*X*-B-(Cu) where *X* is Nb, Zr, Hf, Mo or etc. is characterized by high Curie temperature due to addition of Cobalt (over 1100^{o}C) and therefore it is suitable material for high temperature applications [18].

In given contribution, we present study of the DW dynamics in Hitperm-type glass-coated microwires, which metallic core is in stable partially nanocrystalline state already from production process. Particularly, the effect of annealing under stress on domain wall dynamics will be systematically studied. Moreover, we performed the stress annealing because induced anisotropy is two orders of magnitude higher in comparison to the annealing in high magnetic field as was found in nanocrystalline Finemet ribbons [19, 20].

MATERIALS AND METHODS

We have studied glass-coated microwires of nominal composition $Fe_{38.5}Co_{38.5}Mo_4B_{18}Cu_1$ with two different dimensions quoted as sample 1 (total diameter D= 22.5μm, metallic nucleus d= 9.4 μm, ρ= 0.41) and sample 2 (D= 16.6 μm, d= 10 μm, ρ=0.60) respectively prepared by a modified Taylor-Ulitovsky method. Essentially the fabrication process consists of the melting of the metallic alloys that fills the glass capillary and a microwire is thus formed where the metal core is completely coated by a glass shell. Then this composite microwire is drawn through the liquid jet that allows rapid quenching of the microwire.

Hysteresis loops have been determined by a fluxmetric method at frequency of applied magnetic field of 50 Hz.

Structure and phase composition have been checked using a BRUKER (D8 Advance) X-ray diffractometer with Cu K_α (λ=1.54 Å) radiation. The microwires were attached to the diffractometer sample holder at which each scan was made over the two theta angular range from 30 up to 90 degrees, step size of 0.05°and step time of 30 second for each step.

We have studied the domain wall dynamics by modified Sixtus-Tonks method described elsewhere [21-23] in which a system of three pick-up coils is used for estimation of the DW velocity. System of three pick-up coils is needed to ensure the single DW propagation regime, because at high enough magnetic field local DW nucleation on defects and inhomogeneities can take place [21, 22, 24]. To ensure the domain wall propagation from one sample end, we placed selected sample end outside the magnetization solenoid activating in this way the DW propagation always from the opposite wire end. The microwire (10 cm long) is placed coaxially inside of the magnetizing solenoid and three pick-up coils. We used 3 pick-up coils with distances d1-2 and d2-3 between coils of 27 mm mounted along the length of the wire. The propagation DW induces electromotive force (emf) in the coil which are picked up at an oscilloscope screen upon passing the propagating wall, as described elsewhere [25]. Then, the DW velocity can be estimated as:

$$v = \frac{l}{\Delta t} \quad (1)$$

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

Special stress annealing was carried out in a conventional furnace, which was turned into vertical position in order to apply constant tensile stress during thermal treatment using classical laboratory loads. Tensile stresses applied on metallic core and glass-coating of the microwire during heating, annealing and subsequent slow cooling were calculated using following equations [26]:

$$\sigma_m = \frac{kP}{kS_m + S_{gl}} \quad (2)$$

$$\sigma_{gl} = \frac{P}{kS_m + S_{gl}} \quad (3)$$

where $k=E_2/E_1$, E_i are the Young modulus of metal (E_2) and glass (E_1) at room temperature and P is the applied mechanical load.

For completeness, heating rate starting from room temperature up to the elevated temperature (300^{o}C in our case) was always 5^{o}C per minute.

RESULTS AND DISCUSSION

Both studied FeCoMoBCu-based microwires present nanocrystalline structure already from production process as was before confirmed by XRD-analyse, see Figure **2a** [27, 28]. The average grain size decreases due to nucleation and creation of a lot of new smaller grains of bcc-(Fe, Co) phase than already existing from production up to the annealing temperature of 450^{o}C. However, at higher temperatures the increasing of the average grain normally way is observed probably due to grain growth at temperatures above 450^{o}C (Figure **2b**).

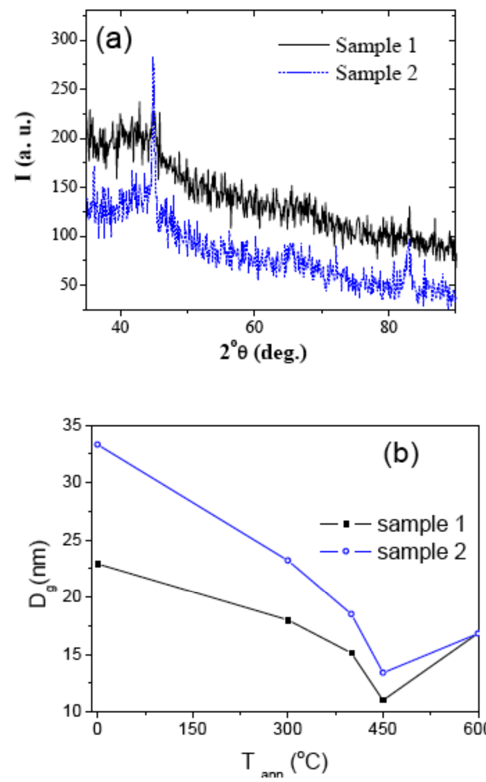

Figure 2: a) XRD patterns of as-prepared samples, **b**) dependence of average grain size on the annealing temperature.

Experimental data in FeCoMoBCu microwires are interpreted considering higher anisotropy in comparison to the classical FeSiB microwires which result in two dominant anisotropies (axial and radial ones) compensating each other. It is considered that due to this good compensation are achieved fast domain wall velocities [29].

Figure **3** shows the perfect rectangular hysteresis loops of the studied FeCoMoBCu microwires in the as-prepared state for sample 1 and for sample 2, respectively. Magnetic bistability is confirmed by the lack of experimental points between the two stable magnetic configurations.

Surprisingly, the sample with lower ρ-ratio (with higher internal stresses) presents lower coercivity. It is worth mentioning that the coercivity of both samples (about 500 A/m and 700 A/m for the samples 1 and 2 respectively) are rather higher than typical amorphous and nanocrystalline Fe-rich microwires.

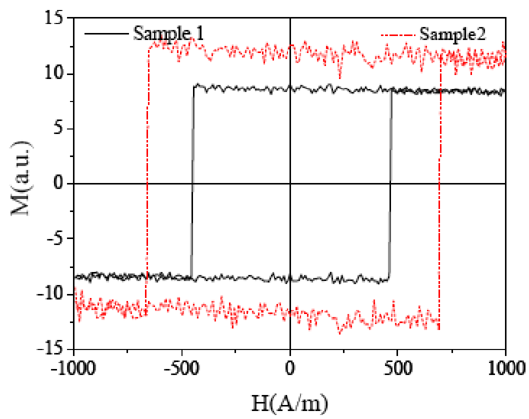

Figure 3: Hysteresis loops of as-prepared FeCoMoBCu microwires: sample 1 with ρ=0.41 and sample 2 with ρ=0.6 measured at f=50 Hz.

Dependencies of DW velocity, v, on magnetic field, *H,* measured in as-prepared samples are shown in Figure **4**.

The sample 2 presenting higher coercivity also presents higher DW velocity with DW mobility that at low magnetic field is S~0.49 m^2/A.s. while at H>1000 A/m the same sample presents higher mobility S~1.78 m^2/A.s. and with velocity up to 2600 m/s (see Figure **4**). Moreover above H=1000 A/m we can observe non-linearity that usually is explained as the nucleation of additional DWs in front of moving DW [21, 22].

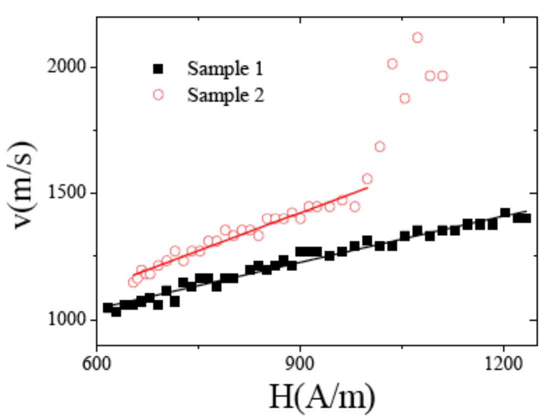

Figure 4: Domain wall velocity in as-prepared nanocrystalline Hitperm based microwires.

The domain wall dynamics is generally described by the linear dependence of the domain wall velocity *v* on applied magnetic field *H*:

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

where S is the domain wall mobility and H_0 is a critical field below which the domain wall propagation is not possible. Moreover, the domain wall mobility *S* is proportional to the domain wall width δ_S :

$$S \sim \delta_S \sim \sqrt{A/K} \quad (5)$$

where A is the exchange stiffness constant and K is the magnetic anisotropy constant. Hence the high domain wall velocities in any magnetic material are the result of two conditions: high domain wall mobility *S* and low (or high and negative) critical field H_0.

Although in present sample we did not measure the local nucleation field distribution that can provide the information on nucleation of DWs on local defects, we employed three pick-up coils. Therefore we can assume that the non-linearity of *v(H)* observed for the sample 2 can attributed to the change in the propagating DW features.

Thus, high DW velocities have been discussed considering compensation of both dominant anisotropies (axial one and radial one) [23]. In the case of nanowires higher DW velocity is usually attributed to the vortex domain wall regime. Consequently assuming that we always have single DW propagation we can explain the non-linearity as the transformation of the DW shape: at higher magnetic field DW presents higher mobility S~1.78 m^2/A.s and velocity up to about 2200 m/s (see Figure **4**).

DW dynamics have been remarkably affected after all kind of thermal treatments (stress annealing and conventional annealing) as can be observed for the sample 1 (Figure **5**).

To understand the effect of annealing we must assume that the annealing at 300 °C for 1 hour leads to the relaxation of strong stress introduced during production process. Moreover, annealing temperature of 300°C is optimum temperature for stress relaxation as was found before in ref. [30]. As a result of such annealing the lower field is needed for observation of the domain wall propagation (~ 450 A/m). This influence must be attributed to the magnetic softening earlier reported for annealed FeCoMoBCu-based microwires [27, 28]. But simultaneously considerable decreasing of the DW velocity takes place after such annealing: DW velocity decreases and lower DW mobility, S~ 0.52 m^2/As is observed (see Figure **5**).

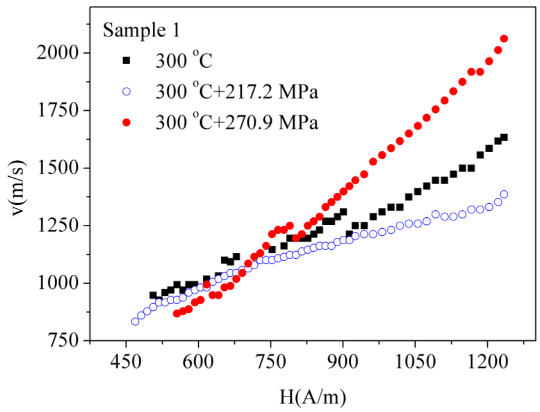

Figure 5: Domain wall velocity in annealed and stress annealed Hitperm-type microwires.

On the other hand, DW dynamics of stress annealed sample depends on the stress applied during the annealing. For certain applied tensile stresses the stress annealed sample present the opposite effect, *i.e.* we can observe increasing of the DW velocity and DW mobility after stress annealing as shown for sample 1 in Figure **5** and for sample 2 annealed at similar values of applied stresses and annealing conditions (see Figure **6**).

Roughly linear *v(H)* dependence is observed for the sample 1 (see Figure **5**). A change of the mobility at magnetic fields about 825 A/m (sample 1 annealed at 300 C under 270.9 MPa), can be ascribed to change the domain wall structure.

As can be appreciated in Figure **6**, the *v(H)* dependence for the sample 2 annealed at 300°C for 1 hour under stresses of 222.7 MPa presents essentially non-linear v(H) dependence.

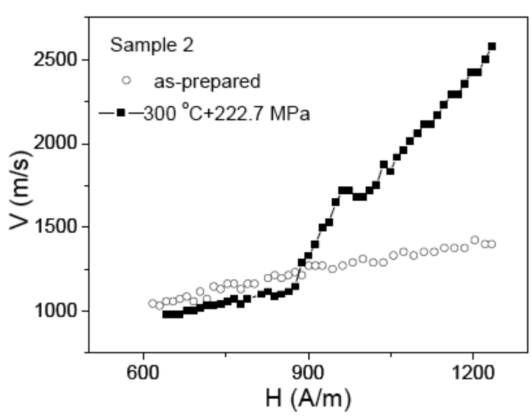

Figure 6: Domain wall dynamics in nanocrystalline microwire sample 2 annealed at 300°C for 1 hour under stress of 222.7 MPa.

The peculiarity of stress annealing is that the applied stress is maintained during all the time, *i.e.* during heating from room temperature up to elevated temperature, subsequent annealing at elevated temperature and consequent very slow cooling of the sample after annealing.

For explanation of observed data we must consider a few different effects occurring during stress annealing of glass-coated microwires, which will be detailed explained in the following.

Firstly, up to now, two concurrent explanations have been proposed for stress induced anisotropy: i) the tensile back-stress theory proposed by Herzer [31] and ii) Néel's model of atomic pair ordering adapted by Hofman and Kronmüller [32].

Herzer attributes it to the magnetoelastic effect. In amorphous alloys the sign of induced anisotropy correlates with the sign of magnetostriction coefficient:

$$K_u = -\frac{3}{2}\lambda_S\sigma \qquad (6)$$

In nanocrystalline alloys, problem becomes more complex since the external stress generated internal stresses, which are heterogeneously distributed in two-phase material, thus the induced magnetoelastic anisotropy is deduced as [30]:

$$K_u = -\frac{3}{2}\left(x\lambda_C\sigma_C + (1-x)\lambda_a\sigma_a\right) \qquad (7)$$

where x is crystalline volume fraction, σ_c and σ_a denotes the stresses located in the volume fraction of nanograins and amorphous phase, respectively.

Additionally, as can be observed from Figure **2**, the structural changes (*i.e*. average grain size) change after anneling.

In our case we have nanocrystalline metallic core already from production process. Therefore during annealing under stress is induced strong longitudinal anisotropy because K_u is negative in according to the Eq. (7) due to positive magnetostriction of both bcc-(Fe,Co) grains and as well as residual amorphous matrix [33, 34].

Hofmann and Kronmüller gave the other interpretation, which is well known as Néel's model of atomic pair ordering [35]. Particularly, atom pair ordering is a diffusional process involving the ordering of the atomic pairs. Annealing at a higher temperature causes larger atomic mobility, enabling faster diffusion. Alloys with two or more different magnetic elements show considerably stronger induced anisotropies than amorphous alloys with only one transition element. In our case, atom pair ordering related to the ordering of Fe and Co atoms. Maximum value of anisotropy in FeCo-based amorphous metals is induced when ratio between Fe and Co is 1:1, what is our case [36]. Moreover, annealing is performed well below the Curie temperature at high internal magnetic field because stable nanocrystalline Hitperm-type alloys are characterized by very high Curie temperature over 1600^{o}C.

The case of the nanocrystalline Hitperm ribbons with positive magnetostriction is rather different to the nanocrystalline Finemet ribbons with negative magnetostriction where atom pair axis of FeSi tend to rotate perpendicular to the direction of the applied tensile stress. Particularly, annealing of Finemet-type nanocrystalline ribbons under tensile stress leads to the flattening of the hysteresis loops increasing the applied stress while in Hitperm-type nanocrystalline ribbons the only squared hysteresis loops were observed. In other words, for amorphous and nanocrystalline alloys, it was found that the easy axis could be either parallel or perpendicular to the tensile stress applied during the annealing, depending on the sign of the magnetostriction and the heat treatment conditions [36, 37].

Secondly, in the case of glass-coated microwires during the annealing the metallic core is relaxed under stress induced by the glass-coating. As known from previous publication on calculation of the internals stresses induced during the rapid solidification of metallic nucleus surrounded by the glass coating the prevailing internal stresses components induced by the glass-coating are the tensile stresses being few time larger than the radial or azimutal [38, 39] But, annealing under high stresses usually leads to transformation of the squired hysteresis loops into linear due to back stresses as was showed in previous work [26, 40]. Therefore, the velocity of the domain wall can increase after annealing under stress as a result of decreasing of longitudinal stresses in the metallic core (see Figure **4**).

Therefore as a result of annealing under stress, the anisotropies distribution can change after annealing. Moreover, radial anisotropy can increase at the expense of axial anisotropy.

The other typical feature of the stress annealed samples is the negative mobility observed for some of annealed samples. The origin of negative mobility (at about 825 A/m for the sample 1 and at about 970 A/m for the sample 2) might be attributed to the observation of the Walker breakdown. So-called Walker breakdown is usually ascribed to the cyclic transformation of the domain wall structure accompanied by the oscillation of the local domain wall velocity [41].

In the case of glass-coated microwires the characteristic size (length) of DWs propagating along the microwire is quite large (about 40-60 microwire diameters) and essentially non-abrupt being [11, 12, 14]. Moreover, the shape of propagating DW in mircowire is quite complex and the micromagnetic origin of this remagnetization front is still unclear. For explain the non-linear DW dynamics and considered that the only one DW propagates along the samples we can assume that the structure of propagating DW change under application of magnetic field.

It is worth mentioning that previously non-linear DW dynamics in glass-coated microwires has been interpreted considering Walker breakdown [42, 43].

The situation is different at more elevated values of stresses applied during the annealing (σ: 334.1 MPa and 556.8 MPa, respectively): with increasing the applied stress the DW velocity decreases (see Figure **7**). We can assume that the axial magnetic anisotropy induced during the preparation process is affected by the stress annealing. As previously discussed [26, 40] during the stress annealing the axial stresses can be reduced and even compensated by the back stresses arising during the stress annealing while transversal anisotropy can increase at the expense of axial anisotropy. Consequently magnetic anisotropy distribution is now different and we can observe only linear regime with much lower velocities up to 1600 m/s.

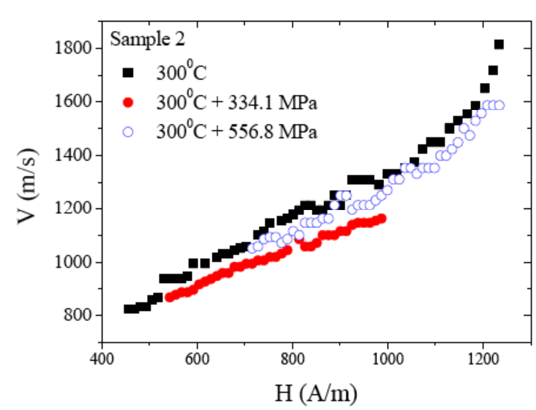

Figure 7: Domain wall dynamics in annealed and stress-annealed FeCoMoBCu microwires.

It is also worth mentioning that the minimum magnetic field for observation of the domain wall propagation after stress annealing considerably increases from ~ 450 A/m (conventional annealing) up to 720 A/m for the sample annealed under σ=556.8 MPa (Figure **7**).

CONCLUSIONS

It was found that optimum annealing conditions for achievement of fast domain wall is temperature of 300°C for 1 hour under tensile stresses of 270.9 and 222.7 MPa. Such annealed microwire is characterized also by non-linear and complex dependence of the DW velocity on applied magnetic field.

Annealing under higher stresses leads to the decreasing of axial anisotropy due to compressive stresses arising during the stress annealing. As a result of such annealing, the magnetic anisotropy distribution change and consequently DW dynamics is affected by the induced magnetic anisotropy

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