Quantification of Residual Stress Governing the Spin-Reorientation Transition (SRT) in Amorphous Magnetic Thin Films

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Abstract

Soft magnetic thin films with in-plane uniaxial magnetic anisotropy are of significant importance for a broad range of technological applications, including high-frequency power conversion. In-plane uniaxial anisotropy in amorphous films is of particular interest for ultra-low materials loss and GHz frequency operations. The present work is focused on one of the fundamental mechanisms, i.e., residual stress, that negate the uniaxial anisotropy in amorphous films by originating perpendicular magnetisation and hence, undermines the soft magnetic performance. It is quantified how the nature of residual stress, compressive or tensile, transforms the magnetisation from in-plane to out-of-plane configuration, also well-known as spin-reorientation transition (SRT). A correlation between engineered residual stress in multilayer stacks, induced by the uneven expansion of metallic/dielectric layers following a thermal-shock scheme, and SRT mechanism demonstrates tensile stress inside the films undermines the soft magnetic performance. We suggest the magnetic softness can be retained by eluding sources of tensile stress during fabrication or post-processing of the amorphous films.

Keywords: Spin-reorientation transition, residual stress, multilayer stacks, perpendicular magnetisation, nano-indentation, magnetoelastic anisotropy.

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1. INTRODUCTION

Soft magnetic amorphous thin films are the potential candidate for the inductor/transformer core applications, especially when high flux density (B_s > 1 T) materials are the focus of interest for the miniaturisation of magnetic components for power supplies on chip (PowSOC) concept(Hao Wu 2012, al. 2015, Masood, McCloskey et al. 2018, Ansar Masood 2019). Lamination of these films with thicknesses below the skin depth, with a suitable insulator, is a well-adopted approach for an optimal material performance(Mathuna, Wang et al. 2012, al. 2015). These materials need to retain in-plane uniaxial magnetic anisotropy for minimal energy losses, attributed to the magnetisation reversal through coherent magnetisation rotation instead of domain wall displacement, and high-frequency drive operations(Fergen, Seemann et al. 2002, Masood, McCloskey et al. 2017). Uniaxial anisotropy of thin films can suffer from many intrinsic and extrinsic factors, such as demagnetisation effects from material dimensions (shape anisotropy), magnetostriction constant of the alloys, and nature of deposition process/parameters(R. Harris 1977, Herzer 2005). Consequently, the uniaxial anisotropy is suppressed by the perpendicular magnetic anisotropy (PMA), which eventually redefines the magnetisation orientation in the perpendicular to the plane of films. Materials exhibiting PMA are of interest in the area of perpendicular recording media where a higher recording density can be achieved and novel spintronic applications such magnetic tunnel junctions(Joshi 2016, Dieny and Chshiev 2017). Nevertheless, this work highlights the applications where PMA contributions are undesired. In such a case, the PMA significantly undermines the functionality of thin-film materials (i.e., low permeability, high materials loss, multimodal ferromagnetic resonance behaviour) for high-frequency applications. Therefore, understanding the mechanisms contributing to the PMA to retain the uniaxial anisotropy in-plane is of significant importance for high-frequency applications, including electrodynamic energy conversion, magnetic sensing and magnetic shielding(Fergen, Seemann et al. 2002, Kisielewski, Maziewski et al. 2007, Masood, McCloskey et al. 2017).

Several mechanisms have been proposed as an origin of the perpendicular magnetisation in amorphous films, such as high degrees of atomic randomness, large magnetostriction constants of the alloy and residual stress produced due to the nature of the fabrication process, such as magnetron sputtering(P. Sharma 2006, Coisson, Celegato et al. 2008, Coïsson, Celegato et al. 2008). Furthermore, perpendicular magnetisation in amorphous films depends on film thickness(P. Sharma 2006, Coisson, Celegato et al. 2008), alloy composition(P. Sharma 2006), method of the fabrication process(P. Sharma 2006, Coisson, Celegato et al. 2008, Coïsson, Celegato et al. 2008), substrate temperature(P. Sharma 2006) and post-annealing conditions. Such an important example is magnetron sputtered Co-Fe-Zr-Ta amorphous system where a thickness-dependent change in the residual stress at micro-scale plays an important role and, eventually, transforms the magnetisation in the perpendicular direction, well-known as a spin-reorientation transition (SRT)(Kisielewski, Maziewski et al. 2007, Masood, McCloskey et al. 2017, Conca, Niesen et al. 2018). In addition to the micro-scale stress, the strain induced at the substrate-film interface contributes significantly to the perpendicular magnetisation, being another detrimental contribution to soft magnetic properties(Vonhorsten, Soffge et al. 1984, Dieny and Chshiev 2017). This was attributed to the interfacial anisotropy at small thickness with gradually increasing magnetoelastic anisotropy at higher thicknesses(Coisson, Celegato et al. 2008). Nevertheless, interfacial anisotropy is prominent mainly on ultra-thin (< 25 Å) layer system; hence it can be ignored for thick amorphous films. Sharma *et al.(P. Sharma 2006)* and Marco *et al.(Coisson, Celegato et al. 2008)*, separately, reported that the SRT in amorphous films may have origin in residual stress and if reduced by thermal annealing the magnetisation can transform back to the in-plane orientation. However, a comprehensive analysis of stress-induced SRT requires further investigations to understand how the residual stress (the magnitude as well as its nature) overcomes the in-plane uniaxial anisotropy and configures the magnetisation in perpendicular orientation.

Thermal shock scheme in multilayer laminations, and indeed in any alloy, can cause a significant expansion between the magnetic layers, dielectric and the substrate, especially when laminated stack contains several layers and there is a substantial difference in the thermal expansion coefficients of the materials. This can induce a large residual stress in the system that, eventually, can work as a system under “stress-induced magnetoelastic anisotropy” in the plane of the compression or tensile stress(Mandal, Vazquez et al. 2000, Herzer 2005). Hence, this type of system provides an opportunity to quantify how residual stress in amorphous thin films contributes to the SRT, and, consequently, undermines the material performance for high-frequency applications. Currently, soft magnetic Co-Zr-Ta-B/AlN multilayer system is considered as an ideal candidate for it its application as inductor/transformer cores for integrated power conversion applications. However, the how the residual stress induced by fabrication process or during device packaging can undermines the soft magnetic properties requires further investigations. In the present work, an engineered residual stress in an amorphous multilayer Co-Zr-Ta-B/AlN stack, induced by following a thermal shock scheme from various temperatures, was correlated to the SRT to quantify how its nature transforms the magnetisation from in-plane to out-of-plane orientation. This work demonstrates that tensile stress in thin films must be eluded to avoid its detremental effects on the soft magnetic performance for high-frequency applications.

2. Experimental Methods

Laminated magnetic stacks consisting of 2 µm (8 x 250 nm layers) of $Co_{84}Zr_4Ta_4B_8$ (atomic %) alloy were deposited by magnetron sputtering (Nordiko Advanced Energy NDX 2500-W) deposition technique. The sputtering chamber was pumped down to a base pressure of ~ 10^{-7} mbar. Prior to deposition, the substrates (100 mm diameter Si/SiO_2, 0.25 µm thermal oxide) were cleaned by generating an RF plasma in the sputtering chamber at 1 kW for 25 minutes using high purity argon gas. An adhesive layer of 20 nm thickness of Ti was deposited prior to the deposition of the first dielectric layer. An AlN dielectric layer of 20 nm was deposited by a DC sputtering of an aluminium target with reactive nitrogen gas. Further, DC sputtering was used on an 8″ single alloy target (Testbourne Ltd., 99.9% purity) with a throw distance of 5.5 cm. An aligning magnetic field was used during deposition of films. A DC power of 1 kW was applied to the magnetic alloy target with an argon sputter pressure of 1.5 mTorr. The sequential deposition of AlN and CZTB was repeated until 8 layers of magnetic films were achieved. The deposition was in a bottom-up configuration, and the wafers were sitting on a carousel. The carousel rotation throughout the deposition was kept constant at 10 revolutions per minute (RPM) to attain the uniform thickness of the multilayer stack. The static magnetic properties of the stacks were measured using M-H loop tracer (SHB Mesa 200) on 2 x 2 cm^2 diced samples. Magnetic force microscopy (MFM, Bruker Dimension Icon) measurements were obtained at the remnant state of magnetization of the multilayer stacks. The atomic structure of thin-film stacks was investigated using an X-ray diffraction technique (XRD, Philips Xpert diffractometer, Cu-K_α λ=1.54 Å) to investigate or confirm any phases present in the samples. The residual stress (σ_R) of the stacks was measured using a nanoindenter (Keysight G200) following a method described by Hardiman *et al.*(Hardiman, Vaughan et al. 2016) A total number of 25 indentations were carried out to a maximum depth of 1 μm on each sample. The continuous stiffness measurement technique was employed to measure the hardness continuously with indentation depth.

3. Results and Discussion

A well-defined uniaxial magnetic anisotropy in the as-deposited multilayer stacks, induced by the aligning magnetic field during the deposition process of films, was confirmed by easy- and hard-axis M-H loops, as presented in Fig. 1(inset). Further, the stacks were annealed in a temperature range T_a = 300-400 ^{o}C for 60 min in an argon atmosphere and, subsequently, exposed to room temperature to induce tensile effects in films, following a thermal-shock scheme. The M-H loops along the hard anisotropy axis of the post-annealed stacks (see Fig. 1) show a systematic evolution in the global magnetic behaviours, suggesting a coherent effect of thermal shocks (i.e., temperature-dependent) on the magnetic properties. A dramatic change in the shape of loops from a uniaxial sheered to an isotropic two-sloped loop, also known as a transcritical loop, at $T_a \geq 350$ ^{o}C suggests a transformation in the magnetization state from in-plane to out-of-plane configuration. The out-of-plane magnetization in thin-film stacks could be attributed to the dominant perpendicular magnetic anisotropy component emerged as a consequence of successive induced residual stress and, consequently, magnetoelastic anisotropy. It is clear that until treated above 325 ^{o}C temperatures, the multi-layered stack did not develop sufficient residual stress, as to induce large magnetoelastic anisotropy energy to overcome its in-plane uniaxial anisotropy, due to in-situ magnetic alignment and shape anisotropy. These increasingly larger perpendicular anisotropy components were believed to be as a result of residual stress occurring at the interface between the magnetic and non-magnetic multilayers.

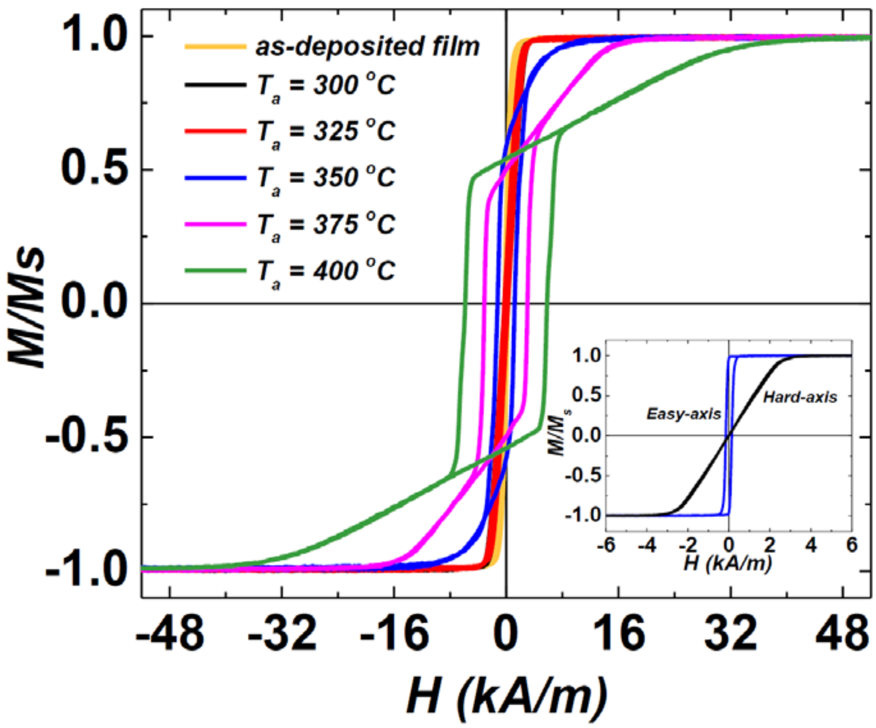

Fig. 1. M-H loops of the films annealed in the temperature range of 300-400 ^{o}C for 60 min. The inset represents easy- and hard-anisotropy axes of the as-deposited film.

MFM images were obtained at the remanent state of magnetization of the stacks, as presented in Fig. 2. A single magnetic domain state seen in the as-deposited, 300 ^{o}C, and 325 ^{o}C annealed samples could be attributed to the well-retained uniaxial anisotropy, as confirmed by the well-defined easy and hard axis M-H loops of the films. At 350 ^{o}C, the in-plane order is beginning to break down as seen with multiple contrasting regions indicating the formation of a multi-domain structure(Ambrose and Stamps 2013). Moreover, distinct striped domain patterns of the stack annealed at 375 ^{o}C and 400 ^{o}C are clearly formed, indicating a fully spin-reoriented perpendicular anisotropy. The evolution of magnetic domains is in good agreement with the M-H loops displayed in Fig. 1 with the perpendicular anisotropy completely outweighing any induced anisotropy contributions. The difference in contrast of striped domains is related to the orientation of the magnetic spins, which are pointing either upward or downward in relation to the film plane. The change in sign of the magnetization component between domains causes a reduction in the magnetoelastic anisotropy energy, as explained elsewhere(Coisson, Celegato et al. 2008). The combination of striped domains in conjunction with transcritical M-H loops, which are well known as a sign of an anisotropy transformation, confirm that multi-layer thin films have developed out-of-plane magnetisation."

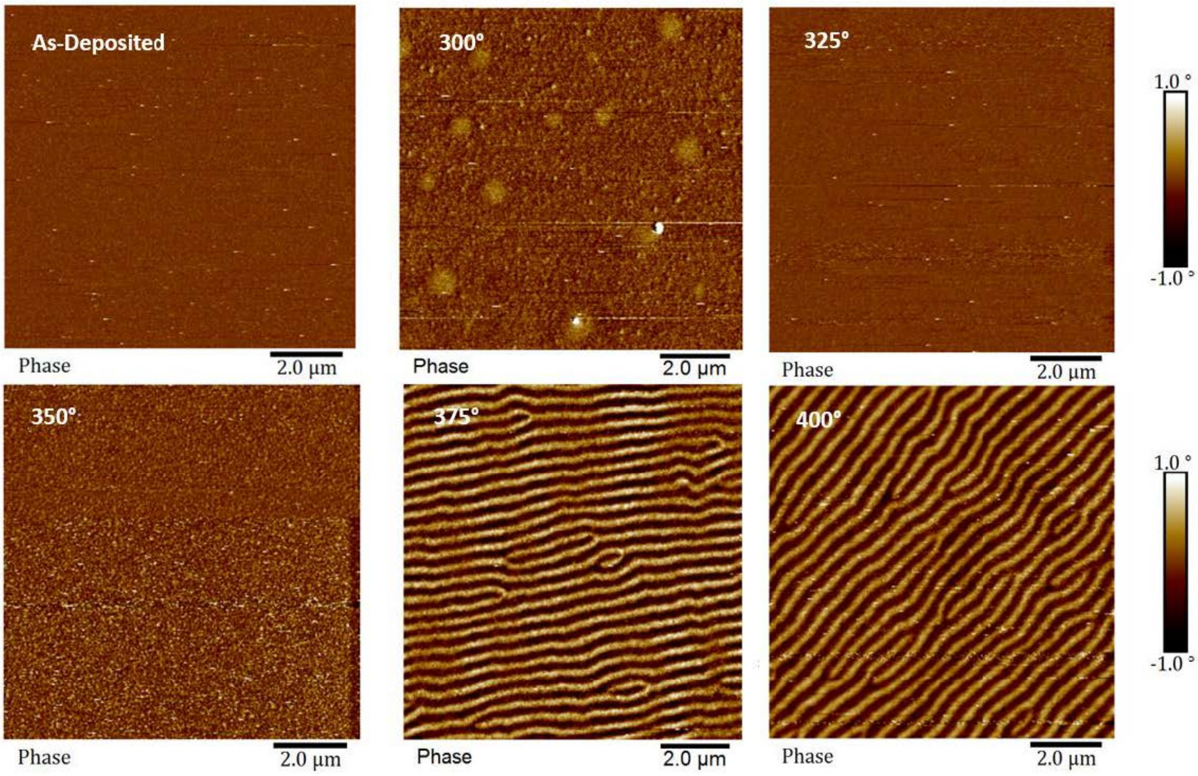

Fig. 2. Magnetic force microscopy (MFM) images of the magnetic domains carried out at the remanent state of magnetisation after subsequent thermal shock scheme from 300 ^{o}C – 400 ^{o}C (in addition to the as-deposited state).

The atomic structure of thin-film stacks was investigated using XRD to confirm if any phases were precipitated after heat treatment. Partial crystallisation may cause PMA contributions as a result of magnetic coupling in the out-of-plane direction. This has been seen with FeO thin films where the SRT was produced by various crystalline phases forming as a function of the oxidation temperature(Lin, Sivertsen et al. 1986). Metal nitrides have been known to cause compressive stress leading to out-of-plane anisotropy components reducing material performance for in-plane applications(Gupta, Dubey et al. 2008). In that particular instance, the stress dependence was deduced from the variation of the iron oxides' *d*-spacing as a result of annealing temperature(Gupta, Dubey et al. 2008). Similarly in cobalt nitride studies, the PMA was clearly as a result of γ-CoN phases, the return to in-plane characteristics was subsequently formed when the nitrogen was dissociated from the thin films(Matsuoka, Ono et al. 1987). The presence of amorphous structure is clear from a broad halo (2θ=40-50^{o}) in the XRD spectrum of the annealed stacks, as presented in Fig. 3. Further, it confirms no nitrides were formed during thermal shock events, hence eliminates the chances of any contribution of exchange coupling or internal CoN magnetocrystalline anisotropy from partial crystallisation. The AlN compound is the sole peak observed as the insulator, and its intensity has not increased or shifted furthermore. The boundaries of the 2θ axis have been chosen such that they may contain the phases of interest.

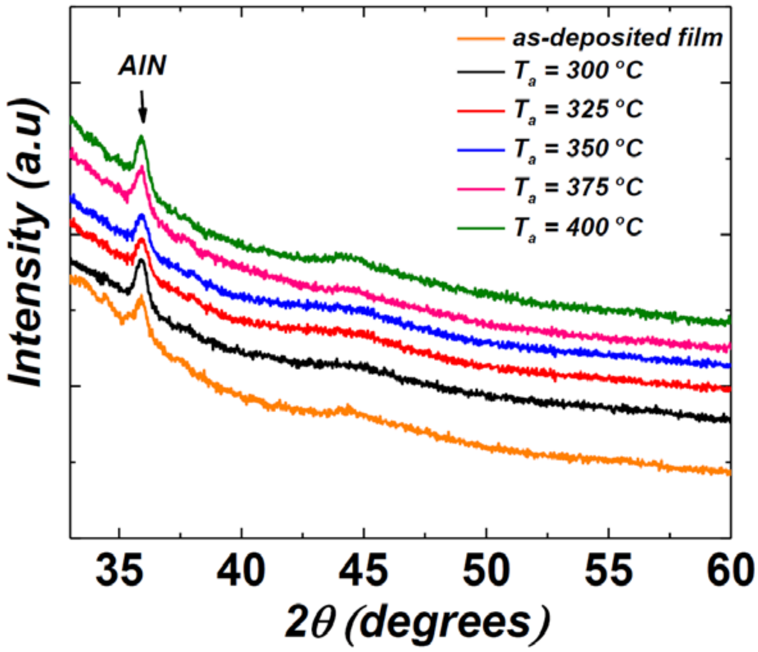

Fig. 3. X-ray diffraction (XRD) pattern of films undergone through thermal shock events at various temperatures (300-400 ^{o}C). The XRD pattern of as-deposited film is presented for comparison.

The hardness values were averaged from 100-200 nm to ensure there was no substrate influence on the measurements based on the film thickness of 2 µm (i.e., thickness/depth ratio ≤ 10 %). From the hardness, the residual stress of the samples was isolated and derived from the total stress using the Vickers Hardness method, $H = C\sigma$, where C is the constraint factor (approximately 2.8) dependant on probe dimensions and its method of procedure(Jang 2009). The residual stress value of the as-deposited stack was considered as a reference point and was subtracted to find each value of residual stress post-anneal (i.e., $\sigma_{R\ annealed} - \sigma_{R\ as\text{-}deposited}$). Fig. 4 shows the measured residual stress of each stack post heat treatment, it is clear how the initial compressive stress is increased towards tensile stress upon each increment of annealing temperature. Upon reaching 375^{o}C, its total residual stress is completely tensile. This would indicate that the tendency of SRT for multi-layered magnetic thin films is evidently related to tensile stress. One might argue that the magnetisation is fully out of the plane when the total compressive stress is removed as seen by the positive value of σ_R in Fig. 4 and its correlation to its M-H loops in Fig. 1. This tendency has also been seen in ultra-thin Ni/NiO layers where tensile stress, originated due to partial crystallisation of amorphous thin films, induced the PMA(Anyfantis, Sarigiannidou et al. 2019). For clear transcritical loops 375 – 400 ^{o}C, where the magnetisation is fully out of plane, $K_{\perp}$ values ranged between 7 and 28 kJ/m^3 through the expression $K_{\perp} = \mu_0 M_s H_k / 2$ ($\mu_0 M_s$ =1.2 T) consistent with existing studies(Umeda, Fujiwara et al. 1996, Coisson, Celegato et al. 2008).

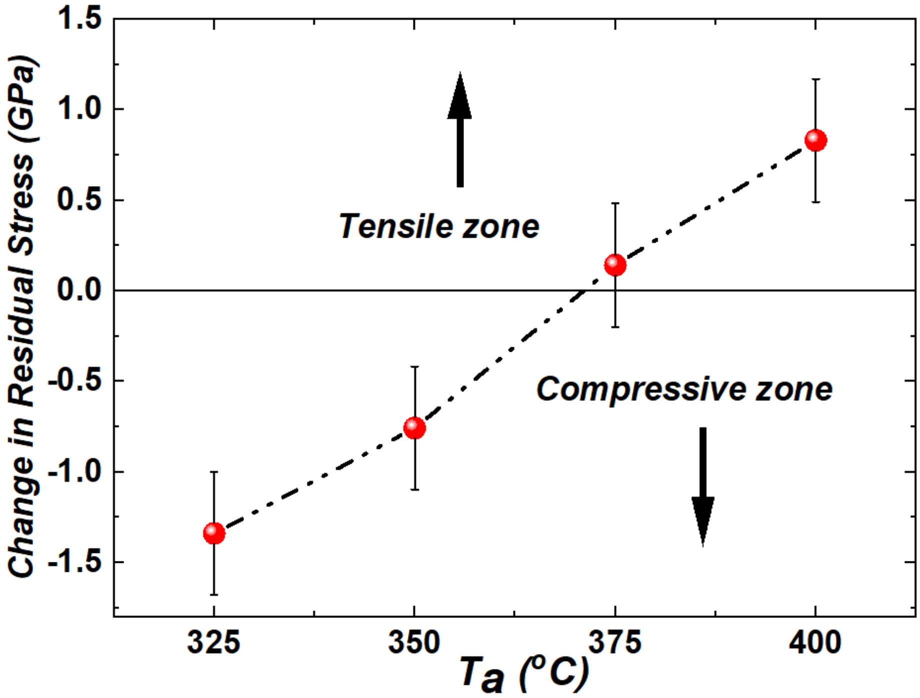

Fig. 4. The residual stress induced in thin-film stacks as a function of annealing temperature (325-400 ^{o}C) on rapid cooling measured by nanoindentation (including the range of uncertainty). The residual stress of as-deposited stack was considered a reference point.

The induced residual stress could be attributed to uneven contraction/expansion of multilayers as a consequence of thermal shock scheme. The uneven cooling rates between metallic Co-Zr-Ta-B and dielectric AlN causes a substantial tensile stress in the normal direction on immediate exposure of the samples from T_a to a room temperature, hence giving rise to an extensive magnetoelastic anisotropy field in multilayer stacks. This becomes a dominant anisotropy field, greatly outweighing any induced or uniaxial shape anisotropy factors that retained the magnetisation in-plane in the as-deposited stacks. Cobalt and aluminium nitride have the mean thermal expansion coefficients of ~13 and ~4.4 ppm/^{o}C, respectively(Paweł Rutkowski 2016). This has been justified using both the finite element analysis and experimental work in earlier studies, whereby the application of heat treatment increases residual tensile stress in between multi-layered thin films upon rapid cooling(H. J. Wang 2015). Conversely, this could also be taking place at the substrate-film interface upon rapid cooling. Studies on single layer amorphous films show SRT occurring due to large differences in thermal expansion coefficients between the magnetic and silicon layers(S.M. N 2002). It was unlikely that significant stress was caused by the 10 nm Ti adhesion layer due to its smaller volume than the CZTB/AlN in addition to the smaller difference in its coefficients of thermal expansion (α_{Ti} = 8.5 ppm/^{o}C). We conclude that the tensile stress was taking place between the layers as the nanoindentation characterised the tensile stress to a depth of 1 μm without reaching the substrate.

Furthermore, it's possible to employ the SRT phenomenological model to the current mixed anisotropy system in this experiment(Popov 2012). Namely, from a ground state in-plane single-domain structure to multi-domain perpendicular orientation via a transcritical or 'canted' state. The general phenomenological model for a multi-layer can be expressed as:

$$\Psi = -JM^2 \sum_{n=1}^{N-1} cos(\theta_n - \theta_{n+1}) + K_{1S}M^2 sin^2 \theta_1 + K_B M^2 \sum_{n=2}^{N-1} sin^2 \theta_2 + K_{NS}M^2 sin^2 \theta_N \tag{1}$$

where the exchange interaction is assumed to positive, isotropic and homogenous independent of respective magnetic layers. In this instance, K_{1S} and K_{NS} are the surface anisotropy at both sides of the film and K_B is the bulk anisotropy constant of the thin film from the dipole interaction(Popov 2012). θ_n is the orientation angle between the magnetisation *M* and the film plane. Theoretically, for an amorphous thin film stack, the anisotropy free energy contributions can be simplified to two terms assuming no sub-lattice magnetisation between layers(Delmoral 1992, Popov 2012). The individual surface and interface anisotropy terms in this system can be approximated as a bulk in-plane anisotropy with the inclusion of a second order perpendicular magnetoelastic anisotropy term(Timopheev, Sousa et al. 2016). Using this model, the simplified energy density can be reduced to

$$E/_V = K_B \, sin^2 \theta_1 + K_\sigma \, sin^4 \theta_2 \tag{2}$$

where K_B and K_σ are the bulk in-plane anisotropy and residual stress induced anisotropy terms respectively ($K_\sigma \propto \sigma_R$). Specifically, θ_1 is the angle between the multilayer's magnetisation *M*, to the film plane and θ_2 to the surface normal respectively. The second order term K_σ, is the sum of each stress contribution at every CZTB/AlN interface rather a large expansion as seen in equation (1). Analogous to works by Popov *et al(Popov 2012)* and Delmoral *et al(Delmoral 1992)'s* work, this may be a simplified approximation to the phenomenological model of the anisotropy energy density assuming the initial in-plane anisotropy is uniform and homogeneous. Subsequently, with increasing residual stress σ_R, K_σ will quickly increase, becoming the dominant higher order perpendicular term in the model as the bulk shape anisotropy become weaker with annealing temperatures(Timopheev, Sousa et al. 2016).

4. Conclusion

The present work quantifies how the nature of residual stress, compressive or tensile, contributes to the magnetoelastic anisotropy of the amorphous films and consequently, transforms the magnetisation from in-plane to the out-of-plane configuration. The residual stress in the multilayer stacks was induced following a thermal shock scheme from various temperatures and its effect on the global magnetic behaviours were investigated. The in-plane uniaxial anisotropy, induced by magnetic alignment during deposition of the films, remained well aligned in thin film system when the compressive stress remained dominant, while it evolved to perpendicular anisotropy when the nature of stress transformed to tensile. This transformation in the nature of stress redefined the spin orientation from in plane to out-of-plane. The present work highlights that sources of tensile stress must be taken into account for soft magnetic thin films that require prominent in-plane uniaxial anisotropy for inductor and transformers cores for high-frequency power transfer applications. Any magnetoelastic anisotropy formed inside the films by tensile nature of stress can undermine the film's efficiency.

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