Annealing Temperature and Thickness Dependencies of Perpendicular Magnetic Anisotropy and Dzyaloshinskii-Moriya Interaction of Pt/Co/MgO thin films

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Effects of the ferromagnetic layer thickness and annealing temperature were studied in Pt/Co/MgO-based systems grown on thermally oxidized Si substrates using magnetron sputtering system. The Co thicknesses were varied in the range 1-6 nm and the samples were annealed *ex situ* at 200, 300 and 400°C. Vibrating sample magnetometer was used to determine the magnetization at saturation and the magnetic dead layer thickness that were found to be insignificantly affected by the annealing. Brillouin light scattering, in Damon-Eshbach configuration, was used to measure the thermally excited spin wave frequencies versus the in-plane applied magnetic field and the spin wave vector which were used to investigate the perpendicular magnetic anisotropy (PMA) and interfacial Dzyaloshinskii-Moriya interaction (iDMI), respectively. The Co effective thickness dependence of the effective magnetization (M_{eff}), which provides information on the PMA constants, shows the existence of two linear regimes with different slopes due to misfit strain induced magnetoelastic anisotropy contributions to volume and surface terms. The volume magnetocrystalline anisotropy constant increases monotonously with annealing temperature whereas the pure surface (Néel type) constant increases with annealing up to 300°C and degrades at 400°C. The surface iDMI constant decreases with increasing annealing temperature and no obvious correlation with interface PMA constant has been found, suggesting different interfaces contribution to PMA and iDMI.

***Index Terms*—Interfacial Dzyaloshinskii-Moriya interaction, interface phenomena, perpendicular magnetic anisotropy, spin waves**

I. Introduction

INTERFACIAL DZYALOSHINSKII-MORIYA interaction (iDMI) [1], which is an antisymmetric exchange interaction between neighboring spins, is an emerging magnetic interface-related phenomenon that affects both static and dynamic magnetic properties of ultrathin film-based systems. Indeed, in such structures, if the layers on bottom and on top of the ferromagnetic (FM) ultrathin film are not similar, then the symmetry is broken in the direction perpendicular to the sample plane. Furthermore if (at least) one of the bottom or the top layers with FM has strong spin-orbit coupling, iDMI arises and manifests itself by an internal field that would act like the external in-plane field favoring a chiral arrangement of the magnetization. For instance, if iDMI is large enough, it changes the domain wall ground state from Bloch wall to homochiral Néel domain wall in PMA systems [2]. Moreover, skyrmions [3] are stabilized by interplay between iDMI and other interactions like symmetric exchange, dipolar and magnetic anisotropy. Therefore, a sizeable iDMI is an essential component in generating magnetic skyrmions, which can be used in high-density digital technologies for racetrack memory [4] and logic devices [5].

Due to the needed broken symmetry to induce iDMI and owing to its interfacial nature, any parameter that can make a change in interfaces or in symmetry properties of the heterostructure may affect iDMI. This includes asymmetric metal composition, asymmetric crystal structure [3], asymmetric induced magnetic moment [6], or asymmetric interface properties like roughness [7], intermixing and density of stacking faults [8]. Therefore, iDMI engineering aiming enlarging its strength could be achieved by optimizing the interface conditions [8], changing the materials adjacent to the ferromagnetic layer, and inserting thin layers at interfaces. Moreover, in such ultrathin film-based systems, the broken symmetry at interfaces induces surface anisotropy effects, which can generate perpendicular magnetic anisotropy (PMA). Although PMA has been already employed in magnetic recording media since many years, it is currently undergoing a revived interest due to its application in a variety of spintronic heterostructures exploiting effects such as spin torque.

Due to its interfacial nature, iDMI arising at the interfaces is distributed to the entire magnetic layer and its strength over the ferromagnetic layer is inversely proportional to the layer thickness. Therefore, changing the thickness of the ferromagnetic layer offers the possibility to tune the iDMI strength. Furthermore, annealing is well-used process in material science to change thin films physical properties after deposition. Indeed, thermal treatment can cause changes in structural properties, in atom arrangement and in the magnetic behavior of ultrathin films. Besides that it may induce alloy formation, intermixing and de-mixing, crystallization and change in Curie temperature, it can also lead to phase transition, elimination of residual stresses and changes in the interface roughness, modifying thus the demagnetizing factor. Therefore, annealing could be used to engine desirable iDMI and PMA values needed for chiral magnetic textures stability

Manuscript received April 1, 2015; revised May 15, 2015 and June 1, 2015; accepted July 1, 2015. Date of publication July 10, 2015; (Dates will be inserted by IEEE; "published" is the date the accepted preprint is posted on IEEE Xplore®; "current version" is the date the typeset version is posted on Xplore®). Corresponding author: M. Belmeguenai (e-mail: belmeguenai.mohamed@univ-paris13.fr). IEEE TRANSACTIONS ON MAGNETICS discourages courtesy authorship;

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such as skyrmions [9]. Therefore, the effect of the annealing temperature on the thickness dependence of both iDMI and PMA in Pt/Co/MgO systems is investigated in this paper. For this, Brillouin light scattering (BLS), ferromagnetic resonance with microstrip line (MS-FMR) coupled to vibrating sample magnetometery (VSM) techniques were used.

II. Samples and Experimental Techniques

Pt(3nm)/Co(t_{Co})/MgO(1nm) trilayers were grown at room temperature on thermally oxidized silicon substrates in a magnetron sputtering system having a base pressure lower than 2×10^{-8} Torr, using 3 nm thick Ta buffer and capping layers. The Co thickness (t_{Co}) has been varied in the range 1-6 nm to experimentally investigate the thickness dependence of the iDMI strength. The metallic layers were grown by dc sputtering under an argon pressure of 1 mTorr, whereas the MgO layer was grown by rf sputtering under an argon pressure of 10 mTorr. The samples were *ex-situ* annealed at variable temperatures (T_a= RT, 200°C, 300°C and 400°C) for 60 min in vacuum with a pressure lower than 2×10^{-6} Torr. The 3 nm thick Ta buffer and capping layers were used to improve the roughness, facilitating the (111) texturing of the upper Pt layer and to protect the samples from oxidation due to air exposure, respectively. Pt is employed as the heavy metal inducing significant iDMI since the intensity of this latter is related to the spin-orbit coupling strength.

For all the samples, the magnetic moment at saturation was determined from the hysteresis curves obtained by VSM, under in-plane applied magnetic field. BLS was used to investigate PMA and iDMI. For this, Stokes (S) and anti-Stokes (aS) spin wave frequencies were measured as a function of the in-plane applied magnetic field and the wave vector (k_{SW}). For PMA investigation, the field dependence of the mean frequency [$(F_S+F_{aS})/2$], at fixed k_{SW} of 4.1 µm^{-1} (incident angle of 10°) was used, whereas, the variation of the frequency mismatch between S and aS lines ($\Delta F=F_S-F_{aS}$) versus k_{SW} allowed to characterize the iDMI strength.

III. Results and Discussion

For precise characterization of the iDMI and the PMA, the magnetization at saturation (M_s) and the magnetic dead layer thickness (t_d) were deduced from the thickness dependence of the saturation magnetic moment per unit area measured by VSM, shown in figure 1a. Figure 1b, showing the annealing temperature dependences of M_s and t_d, reveals no significant variation. Indeed, the small variations of M_s and t_d are within the error bars and therefore M_s and t_d are T_a independent, in the limit of resolution of VSM, for the studied temperature range. Although this variation of M_s and t_d remains insignificant, one should mention the opposite trend of M_s and t_d with annealing, confirming the slight evolution of the sample quality with annealing. The presence of a magnetic dead layer in the as grown samples could be associated with a partial oxidation of Co when MgO is deposited on the top. When annealed at 200°C, diffusion of interpenetrating oxygen atoms in the Co layer toward the Co/MgO interface to form a sharp interface is expected as reported by Gweon et al. [10] and thus leads to sample quality improvement. Higher temperature annealing probably causes intermixing. Unless if it is explicitly mentioned, in the following, all parameters will be discussed as a function of the effective ($t_{eff}=t_{Co}-t_d$) thickness, where t_d is about 0.2 nm.

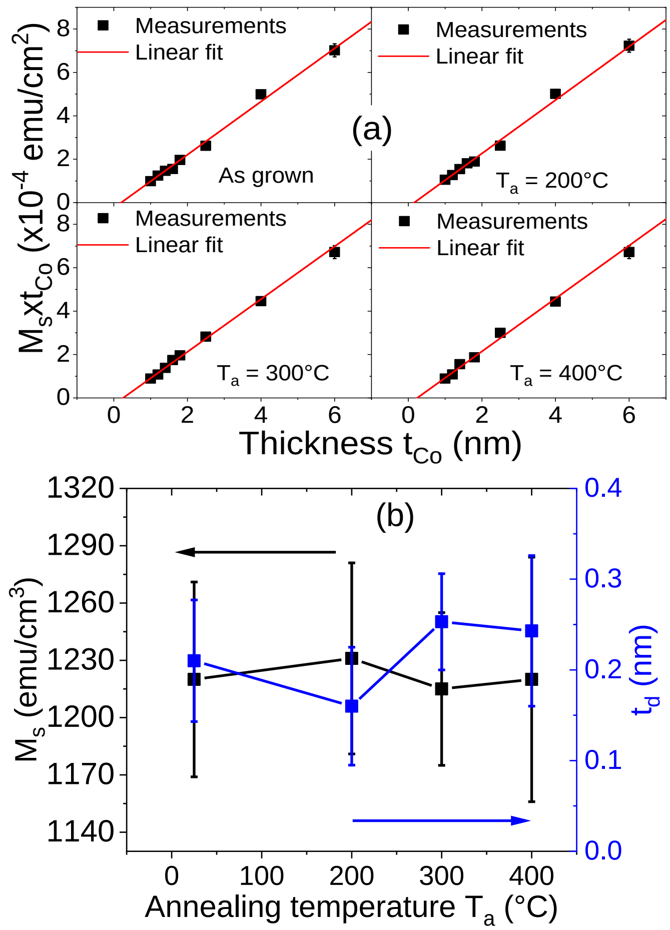

Fig. 1. (a) Saturation magnetic moment per unit area versus thickness of the as grown/annealed Co-based systems. (b) Annealing temperature dependence of the magnetic dead layer and the magnetization at saturation, deduced from the thickness dependence of the areal magnetic moment. Symbols refer to experimental data and straight solid lines are used for eye guides.

The effective magnetization, defined as $4\pi M_{eff}=4\pi M_s-2K_\perp/M_s$, where $K_\perp$ is the effective perpendicular uniaxial anisotropy constant, was deduced from the fit of the BLS data giving the field dependence of the mean frequency $(F_S+F_{aS})/2$,using equation (2) reported in [11] and are shown in figure 2a versus $1/t_{eff}$. The linear dependence of M_{eff}, suggests the existence of surface magnetic anisotropy and therefore, $K_\perp$ could be phenomenologically separated in a volume (K_v) and interfaces (K_s) contributions and approximately obeying the relation: $K_\perp=K_v+K_s/t_{eff}$. Note the existence of two regimes, separated at a given critical Co thickness (t_c) which is T_a dependent, characterized by different slopes of the linear dependence of M_{eff} versus $1/t_{eff}$ that can be attributed to misfit strain induced magnetoelastic anisotropy contribution. Indeed, strain in Pt/Co-based systems due to the difference in the (1 1 1) lattice planes spacing of Pt and Co was observed by x-ray diffraction [12] According to the variation of $4\pi M_{eff}$ with $1/t_{eff}$, the critical thickness decreases with the annealing temperature. For thicknesses over the

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critical thickness, the initial stress is relaxed by dislocations. Since annealing favors the relaxation, the critical thickness is reduced as T_a increases. Moreover, according to the model reported in [13], in region I (thinner Co films with a respect to t_c), the influence of the misfit strain appears as a volume contribution to the anisotropy (characterized by $K^{I}_{me,v}$ constant), whereas it leads to an apparent interface contribution (characterized by $K^{II}_{me,s}$ constant), in regime II (thicker Co films than t_c). K_v and K_s are given by $K_v=K_{mc}+K^{I}_{me,v}$ and $K_s=K_N$ in regime I and by $K_v=K_{mc}$ and $K_s=K_N+K^{II}_{me,s}$ in regime II.

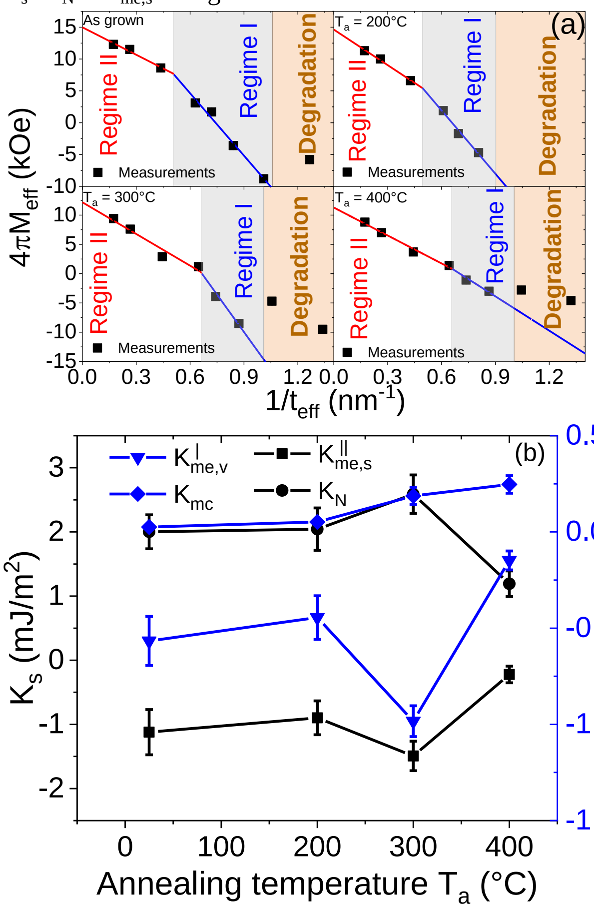

Fig. 2. (a) Thickness dependence of the effective magnetization ($4\pi M_{eff}$) extracted from the fit of BLS measurements of Pt/Co/MgO grown on Si substrate and annealed at various T_a. Symbols refer to measurements and solid lines are linear fits for regime I (blue lines) and II (red lines). (b) Variation of the different contributions of the magnetocrystalline (K_{mc}), Néel-type surface (K_N), the volume ($K^{I}_{me,v}$) and the surface ($K^{II}_{me,s}$) magnetoelastic anisotropy constants to the perpendicular anisotropy. Symbols refer to measurements and solid lines are used as guides for the eye.

The linear fits of the two regimes data in figure 2a combined to the above mentioned relations are used to determine K_{mc} (K_v), $K^{I}_{me,v}$, K_N(K_s) and $K^{II}_{me,s}$, which are shown in figure 2b versus T_a. Note the negative sign of the magnetoelastic anisotropy constants ($K^{I}_{me,v}$ and $K^{II}_{me,s}$) reinforcing the in-plane magnetic easy plane, suggesting a tensile out-of-plane strain owing to the negative magnetoelastic constant of Co. This is in line with the reported trends by Shepley et al. [14] who show that the tensile out-of-plane strain reduces the PMA of the Pt/Co/Pt. Note the similar trend of $K^{I}_{me,v}$ and $K^{II}_{me,s}$ versus T_a and their weak values at T_a=400°C, confirming the relaxation of strain at higher T_a as mentioned above.

Both the volume magnetocrystalline (K_{mc}) and the Néel type (K_N) anisotropy constants are positive and favor perpendicular easy axis (PMA). Figure 2b reveals that volume anisotropy constant K_{mc} increases monotonously with T_a. Its value is weak for low T_a (due to the defects of the film, like disorder) and approaches the magnetocrystalline anisotropy constant of hexagonal Co (K_v=0.51 MJ/m^3) [15] for T_a =400°C, probably due to lesser disorder. The interface anisotropy (Néel type: K_N) increases with increasing T_a up to 300°C, most likely due to the diffusion of the interpenetrating oxygen atoms in the Co layer toward the Co/MgO interface during annealing [10]. Further annealing (T_a=400°C) significantly degrades this interface anisotropy, probably due to the interdiffusion at interfaces. Note the deviation of M_{eff} from the linear dependence for further thinner Co films (Co thicknesses below regime I) most probably due to the degradation of interfaces of thinner Co layers.

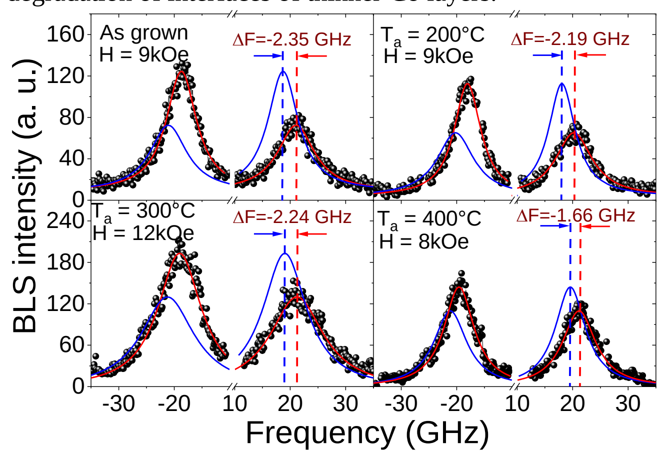

Fig. 3. BLS spectra measured for Pt/Co(1.4nm)/MgO annealed at various temperatures and measured at different in-plane positive applied magnetic field values and at a characteristic spin wave vector k_{SW} = 20.45 µm^{-1}. Symbols refer to experimental data and solid red lines are the Lorentzian fits. Fits corresponding to negative applied fields are presented in blue lines for clarity and direct comparison of the Stokes and anti-Stokes frequencies.

BLS, in Damon-Eshbach configuration, has been used to investigate the non-reciprocity of the spin waves (SW) propagation. For this, the thermally excited SW spectra have been recorded under saturating in-plane applied magnetic field, for each k_{SW} within the range 4.1-20.45 µm^{-1}. Figure 3 shows the typical experimental spectra for Pt/Co(1.4nm)/MgO annealed at various T_a compared to the corresponding Lorentzian fits. Although the fixed Co nominal thickness for the various T_a, the spectra show the positions of S and aS lines are different due to change of effective PMA field with T_a. Moreover, it is worth to note the frequency difference between the simultaneously detected Stokes and anti-Stokes lines ($\Delta F=F_S-F_{aS}$), reflecting the SW propagation non-reciprocity induced by iDMI.

The frequency mismatch varies linearly with k_{SW} for all samples as shown in figure 4a for Pt/Co(1.4nm)/MgO annealed at various temperatures. This linear dependence of ΔF is a signature of the iDMI contribution, especially for the ultrathin Co films investigated here and thus can be fitted by

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equation (1) reported in [16] to determine the iDMI strength, characterized by the effective constant D_{eff}.

$$\Delta F = F_S - F_{aS} = D_{eff}\frac{2\gamma}{\pi M_s}k_{SW} = \frac{D_s}{t_{eff}}\frac{2\gamma}{\pi M_s}k_{SW} \qquad (1)$$

where $\gamma/2\pi$=31 GHz/T is gyromagnetic ratio for Co [16] and $D_s=D_{eff}/t_{eff}$ is the iDMI surface constant.

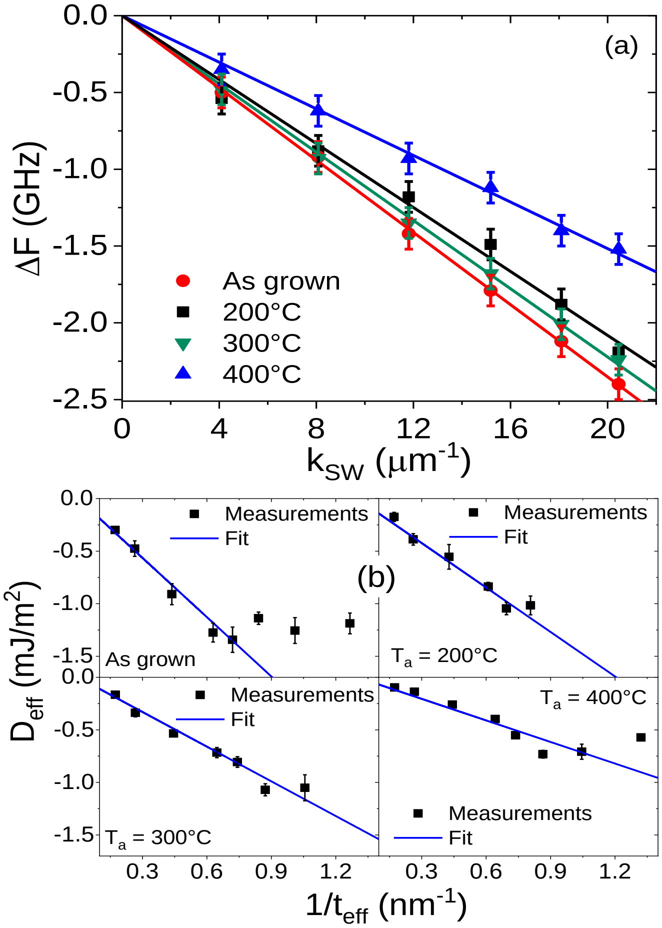

Fig. 4. (a) Wave vector (k_{SW}) dependence of the frequency difference ΔF of Pt/Co(1.4nm)/MgO-based samples. Symbols are experimental data and solid lines refer to linear fit using equation (1). (b) Variation of the effective iDMI constants of Pt/Co(1.4nm)/MgO systems versus $1/t_{eff}$. Symbols are experimental data and solid lines refer to the linear fit.

The variation of the deduced D_{eff} versus the reciprocal effective thickness of Co is shown in figure 4b for the various annealing temperatures. It is worth to note the linear dependence versus $1/t_{eff}$ with a deviation from this linearity for the thinner Co films, especially for Ta=RT and 200°C, most probably due to the degradation of the interface quality of the thinner ferromagnetic layer. In this Co thickness range where the linearity deviation is observed, D_{eff} remains constant or decreases slightly. It seems that the Co thickness below which this trend (linearity deviation) occurs decreases with annealing temperature most likely due to enhancement of the samples quality for higher annealing temperature. One should also mention the single linear regime for the thickness dependence of D_{eff} in contrast to the observed trend in M_{eff} behavior, suggesting the lesser sensitivity of iDMI to strain. Indeed, Shepley et al. [14] reported change of 12% of the effective PMA constant for 0.1% strain in Pt/Co(1nm)/Pt whereas Zhang et al. [17] observed the enhancement of D_{eff} up to 20% for the sample with 5.5% strain for Pt/Co(3nm)/MgO, which remains very large strain value compared to the misfit induced strain. Similarly for our samples, strain has a large effect on PMA revealed by the two regimes whereas it has probably no significant effect on iDMI.

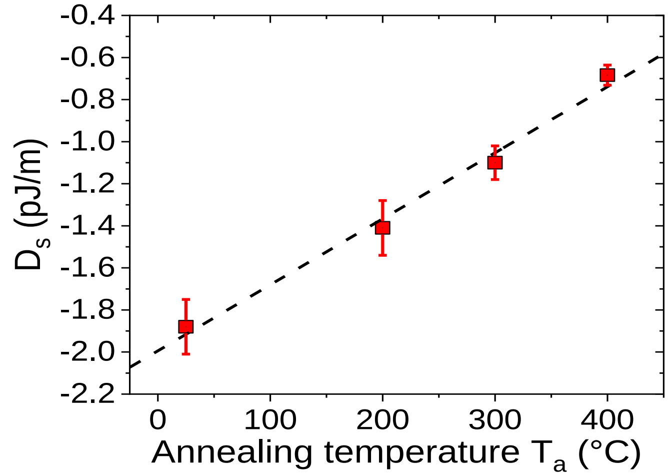

Fig. 5. Variation of the surface iDMI constant of Pt/Co/MgO-based systems versus the annealing temperature. Symbols are experimental data and dashed line is used as guide for the eye.

Figure 5 shows the annealing temperature dependence of the surface iDMI constants given by the slope of the linear fit of the D_{eff} versus $1/t_{eff}$, presented in figure 4b, where a significant decrease (in absolute value) is observed. We thus find that the surface and the magnetocrystalline volume anisotropy and iDMI constants follow opposite trends: the K_N and K_{mc} increase slightly with T_a, whereas D_s shows drastic decreases with increasing annealing. The increase of the anisotropy constants is coherent with the de-mixing of interpenetrating oxygen atoms from the Co layer and the formation of a sharp Co/O interface. However, since the iDMI is mainly generated by the Pt/Co interface, we conclude to the degradation of this interface due probably to interdiffusion. This is consistent with the increase of K_{mc} with T_a since CoPt alloys should have higher PMA [18]. The different behavior of PMA and iDMI versus T_a suggests a different origin in these samples. Indeed, PMA results from the two interfaces (Pt/Co and Co/MgO), whereas the iDMI effect is mainly due to the Pt/Co interface. Since both interfaces can be affected differently by T_a, it is not surprising to observe different trends for iDMI constant and PMA because the first involves only one interface while the second results from both of them.

To clarify the effect of T_a on PMA and iDMI, MS-FMR was used to measure the Gilbert damping (α) for the thicker Co films (6 nm) by investigating the frequency dependence of the half width at half maximum height (ΔH). The magnetic field was applied in the film plane direction, giving the minimal ΔH (determined from the variation of ΔH versus the in-plane magnetic applied field direction). α and the inhomogeneous broadening (ΔH_0) were then deduced from the linear fit of the frequency dependence of ΔH and are shown in figure 6. α decreases with T_a up to 300°C and then increases, whereas ΔH_0 which is related the sample quality, decreases monotonously with T_a indicating enhancement of the sample volume quality. The decrease of α could also be attributed to the reduced spin pumping efficiency at Pt/Co interface or to the decrease of the Co intrinsic damping due to enhancement of the sample volume quality. However, the increase of α for T_a= 400°C could result from the enhanced interdiffusion, introducing Pt impurity atoms in the Co layer and increasing its intrinsic damping.

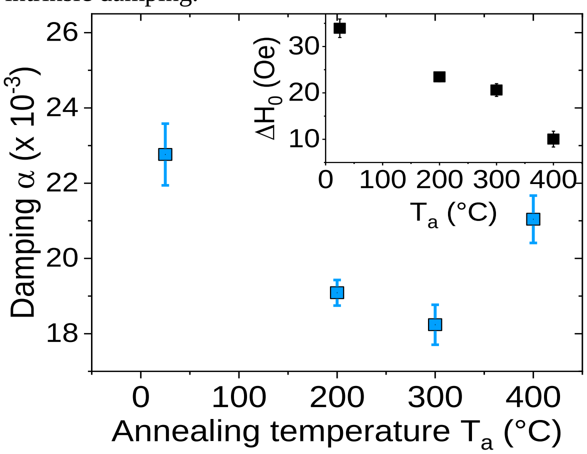

Fig. 6. Variation of the Gilbert damping coefficient of Pt/Co(6nm)/MgO versus the annealing temperature. The inset shows the inhomogeneous broadening of the linewidth.

IV. Conclusion

Pt/Co/MgO-based systems with variable Co thicknesses (in the range 1-6nm) have been grown by magnetron sputtering and then annealed at 200, 300 and 400°C. Their magnetic properties have been investigated by vibrating sample magnetometery and Brillouin light scattering (BLS). Field dependence of spin waves frequencies allowed determining the effective magnetization for which its linear dependence with the inverse of Co effective thickness follows two regimes of different slopes due misfit induced magnetoelastic anisotropy. The contributions of magnetoelastic terms to the interface and the volume anisotropy, depending on Co thickness, have been deduced and found to reinforce in-plane easy axis and decreases significantly for films annealed at 400°C. Moreover, BLS revealed frequency mismatch between Stokes and anti-Stokes lines, reflecting the spin waves non-reciprocity induced by interfacial Dzyaloshinskii-Moriya interaction (iDMI). The effective iDMI constant, characterizing iDMI strength, varies linearly with the inverse of the effective Co thickness and only single regime is observed probably due to the lesser sensitivity of the iDMI to strain. The deduced iDMI surface constant decreases significantly with increasing annealing temperature whereas, the interface PMA constant increases slightly with annealing temperature up to 300°C. We conclude to different behaviors of Co interfaces contributing to iDMI and PMA.

Acknowledgment

This research was supported by 'structure fédérative de recherche NAP MOSAIC' of the University Sorbonne Paris Nord and Conseil regional d'Île-de-France (convention 1763) through the DIM NanoK (BIDUL project). M.S.G, acknowledges the financial support for this work from MRI-CNCS/UEFISCDI through the PN-III-P4-ID-PCE-2020-1853-SPINSYNE grant number no. 182/2021.

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