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Enhanced blocking temperature and isothermal control of hysteresis loop shifts in Co/NiO/[Co/Pt] heterostructures with orthogonal easy axes

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Heterostructures of Co 4 nm/NiO 1.1 nm/[Co 0.4 nm/Pt 0.6 nm] with mutually orthogonal easy axes allow for isothermal tuning of the hysteresis loop shifts along the applied field axis at room temperature, as well as displaying a greatly enhanced blocking temperature. The loop shifts can be varied up to 35 Oe through the application of moderate dc magnetic fields of 3 kOe. The presence of the [Co/Pt] multilayer with perpendicular anisotropy is responsible for a significant enhancement of the blocking temperature. For this thickness of NiO, the blocking temperature is expected to be well below 50 K, in contrast to the observed blocking temperature of 225 K. These effects may be tailored by a judicious choice of materials. The dependence of the loop shift on applied field will vary depending on the coercivity and remanance of the ferromagnetic layers. The enhancement of the blocking temperature is highly dependent on the in-plane and out-of-plane anisotropy constants of the intervening antiferromagnet. Both effects are directly relevant to a variety of modern spintronic applications.

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I. INTRODUCTION

The exchange bias (EB) effect[1] is a fundamental aspect of most realized spintronic devices.[2,3] This effect takes place at the interfaces of magnetic heterostructures and has been shown in ferromagnetic/antiferromagnetic (FM/AFM) and FM/ferrimagnetic bilayers as well as soft/ hard FM bilayers. EB is evidenced, among other effects, by a hysteresis loop shift (LS) along the field axis and an enhancement of the coercivity (H_c). The anisotropy constants of the AFM layer play a pivotal role in determining the minimum thickness and maximum temperature range over which EB exists. The magnitude of the LS depends on several intrinsic parameters including the exchange interaction at the FM/AFM interface, interface roughness, micromagnetic structure, and thickness. However, it is also possible to tune the EB externally; for example, field cooling in a variety of magnetic states[4–6] or extremely large field excitations.[7] These approaches are often tedious and prove inconvenient in a practical setting and thus are unlikely to be suitable for real world application. Recent isothermal approaches attempting to manipulate the interfacial magnetic spins include a [Pt/Co]/NiFe system, where an in-plane surface magnetization is attributed to Néel-type flux closure caps at the interface between the two FM layers leading to a NiFe loop shift[8] and a $Fe/Cr_2O_3/Fe$ system displaying a tunable exchange bias with moderate set fields (<10 kOe).[9]

Engineered spin valve structures in magnetic memory devices require stable operation at temperatures well above room temperature (RT). For AFM materials with low anisotropy constants, this implies a rather large thickness in order to stabilize the LS at higher temperatures, an undesirable constraint given the requirements for high-density recording. Here we show that heterostructures comprised of $Co/NiO(11\ \text{Å})/[Co/Pt]_5$ with easy axes perpendicular [Co/Pt] and parallel to (Co) the plane of the film, display an isothermally tunable in-plane LS at room temperature (RT) and an unusually high blocking temperature (T_B). The small thickness and weak in-plane anisotropy of the AFM NiO layer would normally restrict the EB to temperatures below 30 K (Refs. 10 and 11); we will show that the presence of the [Co/Pt] layer plays a pivotal role in stabilizing NiO grains and dynamically manipulating them (via in-plane set fields), thereby tuning the observed LS.

II. EXPERIMENTAL TECHNIQUES

Samples were prepared by dc and rf magnetron sputtering from separate Pt, Co, NiO, and Cu targets on similarly sized $4\times4\ \text{mm}^2$ Si substrates deposited in 2 mTorr Ar pressure with a base pressure of $\sim3\times10^{-8}$ Torr and consisted of

Sample A: $Si/Pt(200\ \text{Å})/Co(40\ \text{Å})/NiO(11\ \text{Å})/[Pt(6\ \text{Å})/Co(4\ \text{Å})]_5/Cu(100\ \text{Å})$.

In order to understand the individual role of each magnetic layer we also grew the constituent parts

Sample B: $Si/Pt(200\ \text{Å})/Co(40\ \text{Å})/NiO(11\ \text{Å})/Cu(100\ \text{Å})$ and

Sample C: $Si/Pt(200\ \text{Å})/NiO(11\ \text{Å})/[Pt(6\ \text{Å})/Co(4\ \text{Å})]_5/Cu(100\ \text{Å})$.

The thickness calibrations for these structures were checked using an *in situ* quartz-crystal monitor. Crystal structure was measured by x-ray diffraction; the Pt layers are polycrystalline but are highly fcc (111) textured; the Co layers are highly hcp (100) textured, and the NiO is polycrystalline but is shown to be strongly fcc (111) textured perpendicular to the film plane. Bulk NiO crystallizes in the rock salt (NaCl) structure, undergoing a slight rhombohedral distortion on cooling through the Néel temperature.[12] The spins order in antiferromagnetically coupled ferromagnetic (111) sheets within which the spins point in the $(11\bar{2})$ directions. We assume the bulk ordering structure for this thin NiO film, with the antiferromagnetic order-parameter perpendicular to the sample surface. Due to the lack of in-plane order, all

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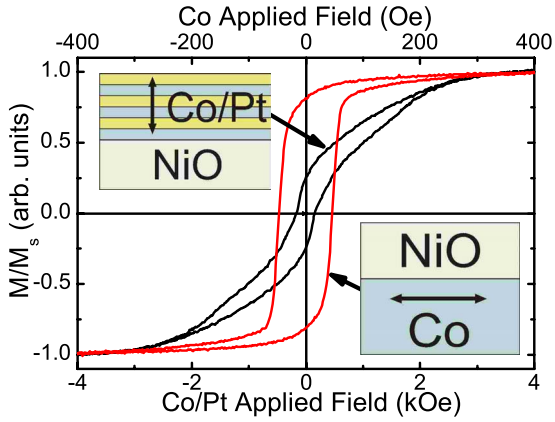

FIG. 1. (Color online) In-plane magnetization curves for samples B and C at room temperature. The thin films structures are also indicated. The Co/Pt layer has the expected hard axis S-shaped curve with a nonzero remanence value that is ~26% of saturation. The Co loop is square and is symmetric about the magnetization axis.

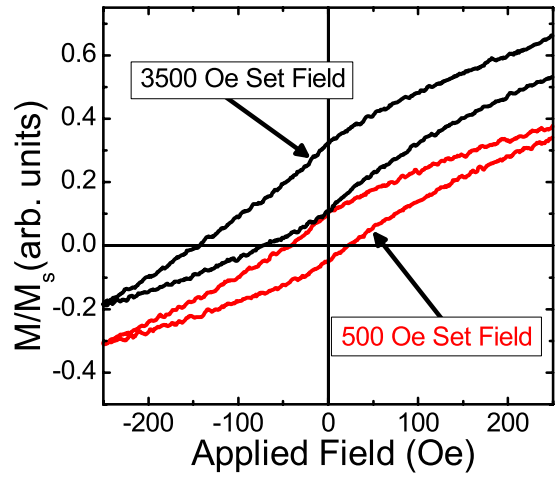

FIG. 2. (Color online) Two minor in-plane, [Co/Pt]/NiO magnetization loops, one from +3.5 kOe to −250 Oe and one from +500 to −250 Oe. The asymmetry in the in-plane magnetization increases with increasing set field.

possible spin orientations exist within the plane.

Extensive experiments on numerous previous samples with similar NiO thicknesses[13] grown under identical growth conditions in the same chamber indicate that above a thickness of 7 Å the NiO layer is pinhole free as evidenced by antiferromagnetic coupling at thicknesses of 7 Å and above. In addition, careful x-ray absorption spectroscopy (XAS) and x-ray reflectivity measurements on samples with similar thicknesses of NiO (Ref. 13) indicate that the Co/NiO interfaces are clean and abrupt.

Room temperature magnetic characterization of samples was done using alternating-gradient field magnetometry (AGFM), while temperature dependent characterization was done using the magneto-optic Kerr effect (MOKE) while in vacuum using a Janis cryostat with polarization preserving optical windows.

III. LOOP SHIFTS AT ROOM TEMPERATURE AND BELOW

The measurements described below consist of both major and minor hysteresis loops. To avoid confusion, we define the upper (lower) curve of the hysteresis loop as corresponding to the curve starting from positive (negative) field.

We first describe magnetization loops on the constituent samples B and C. The hysteresis loop for sample B is a typical square easy axis hysteresis loop, with an H_c of 46 Oe and a saturation field of ~250 Oe (Fig. 1). For sample C, all measurements reported here were made following perpendicular saturation to ensure that the [Co/Pt] layers were single domain. The major in-plane hysteresis loop of sample C (Fig. 1) exhibited the expected S-shaped hysteresis loop typical of a hard axis magnetization rotation mechanism, but with a nonzero remanent magnetization (M_R). The in-plane saturation field is ~3.5 kOe; however, the loop displays hysteresis between ±2.75 kOe leading to an M_R value of ~26% of the saturation magnetization. To elucidate the behavior of sample A, minor loops were measured on sample C between ±250 Oe, corresponding to the saturation value of the in-plane Co layer. These minor loops were measured after a variety of in-plane set fields, H_{set}, were applied to the sample. Since the field effectively cycles from our H_{set} value to −250 Oe and back to +250 Oe, the minor loops show an asymmetry, corresponding to differing values of M_R and H_c for the upper and lower curves of the hysteresis loop. This asymmetry increases with an increase in H_{set} up to a value of ~2.75 kOe, corresponding to the closing of the full in-plane hysteresis loop for the [Co/Pt] multilayer (Fig. 1). Two representative loops following an applied H_{set} of 2.75 and 0.5 kOe are shown in Fig. 2 displaying this change in asymmetry. Above H_{set}=2.75 kOe, further increases in magnetization with increasing field are solely due to reversible, rotational processes and the asymmetry of the minor loop is fixed. Based on this correlation between H_{set} and the [Co/Pt] in-plane magnetization asymmetry, for the remainder of the experiment the field H_{set} serves only as a measure of the asymmetry of the [Co/Pt] minor loop.

In-plane magnetization measurements of sample A (the entire heterostructure), taken between ±250 Oe at RT, are shown in the inset of Fig. 3. Similar to measurements on sample C, in order to control the in-plane component of magnetization in the [Co/Pt], the sample is magnetized perpendicular to the plane of the film to produce a single domain state, followed by the application of H_{set}, which induces the in-plane component of magnetization. The magnitude of H_{set} is varied from 250 to 3500 Oe (based on the magnetic properties of the [Co/Pt] multilayer, discussed above), which serves to increase the asymmetry of the in-plane component of magnetization in the [Co/Pt] multilayer. For each value of H_{set}, corresponding to a particular in-plane magnetization of the [Co/Pt] layer, in-plane magnetization measurements between ±250 Oe (sufficient to switch and saturate the in-plane Co layer) were taken. The result of these measurements is shown in Fig. 3. At H_{set} values between 250 Oe and 3 kOe, the upper curve shows a steady increase in H_c while the lower curve remains essentially unchanged. This leads to

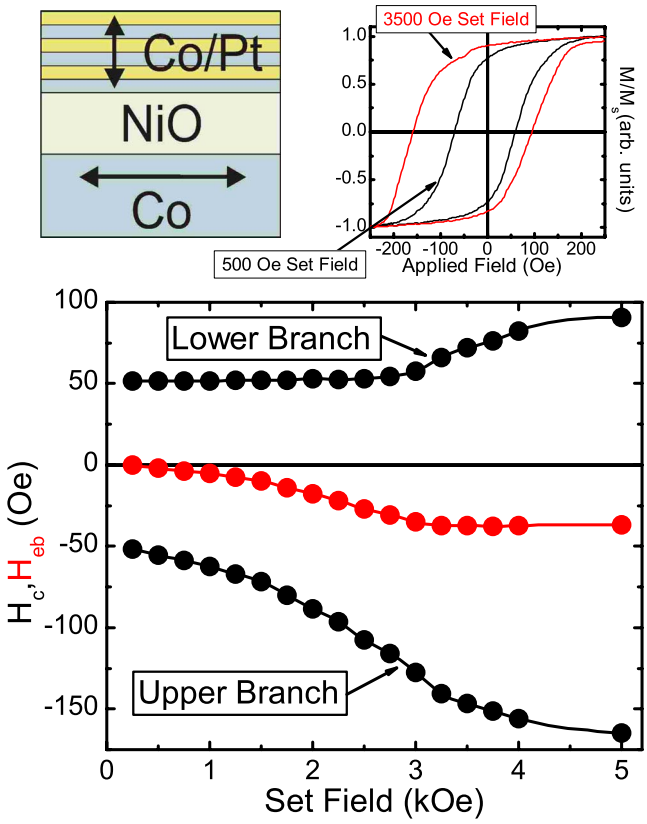

FIG. 3. (Color online) In-plane exchange bias of the Co/NiO/[Co/Pt] heterostructure as a function of H_{set}, where the structure is indicated in the upper left inset. The H_c for the upper and lower branches is also given; notice the lower branch shows no change until 2.75 kOe, above which the upper and lower branches show equal but opposite changes. Two representative loops taken at different H_{set} are shown in the upper right inset.

an increasing LS along the field axis for the Co magnetization with increasing H_{set}.[14] Increasing H_{set} above 3 kOe results in a symmetric increase in H_c for both the upper and lower curves of the loop such that the LS saturates at a H_{set} of 3 kOe. We have seen similar behavior for a wide variety of samples with differing NiO thicknesses; in all cases, the loop shifts and the dependence on the set field are qualitatively similar.

Temperature dependent measurements were taken on sample A after in-plane ac demagnetization from 3.5 kOe, which minimizes the [Co/Pt] in-plane magnetization. Prior to cooling, a small 250 Oe in-plane field is applied to saturate the in-plane Co layer; the application of this field may result in a very small in-plane remanence of the [Co/Pt] layer (<10% of the saturation magnetization). After cooling in zero field, the ±250 Oe in-plane magnetization measurements as a function of temperature are shown in Fig. 4. At each temperature, the sample underwent more than twenty ±250 Oe magnetic-field cycles, ensuring that any training effects (resulting in a decrease in LS with repeated magnetic-field cycles) for subsequent loops are negligible. Following these initial loops, ten subsequent loops were taken and averaged together; representative loops for three temperatures (150, 225, and 300 K) are displayed in the inset of Fig. 4. A

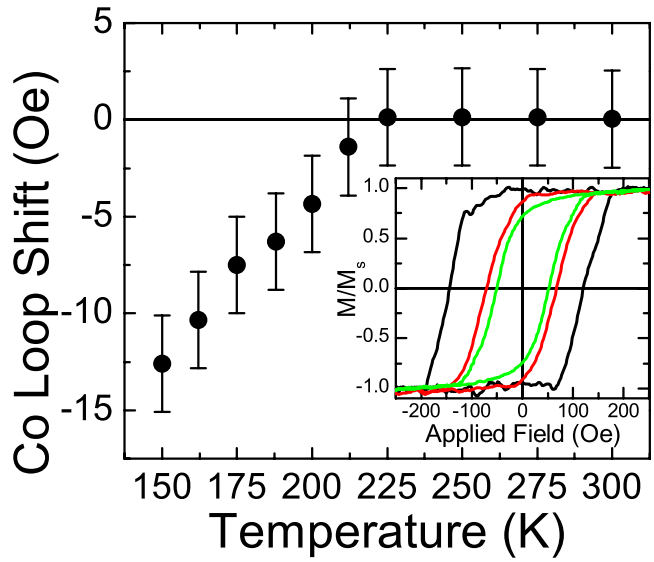

FIG. 4. (Color online) Co loop shift as a function of temperature. The loop shift decreases linearly with temperature with a T_B of 225 K. The inset shows the hysteresis loop at three representative temperatures (150, 225, and 300 K).

LS is apparent below a temperature of 225 K and the LS increases linearly with decreasing temperature. The result is consistent with the previously observed linear dependence of EB with temperature below T_B.[10,11,15–18] This value of T_B for NiO is vastly greater than other published values of less than 50 K (Refs. 10 and 11) for in plane FM/NiO heterostructures and very closely resembles the linear temperature dependence of LS expected for an AFM with cubic anisotropy and a $(1-T/T_N)^2$ temperature dependence of the anisotropy constant.[19–22] Note that the strong temperature dependence below T_B rules out magnetostatic coupling effects; due to the high Curie temperatures of the FM layers, the magnetization remains almost constant over the temperature range studied.

IV. DISCUSSION

The in-plane magnetization of the [Co/Pt] layer clearly has a large effect on the in-plane hysteresis loop of the Co layer in these heterostructures. For an 11 Å thickness of NiO, the Néel temperature is expected to be below room temperature.[23] However, neutron-scattering studies show that the AFM ordering can be stabilized by the presence of an adjacent FM or ferrimagnet.[18,24] Previous measurements on NiO films of similar thickness sandwiched between [Co/Pt] multilayers[13] indicate that the NiO is AFM ordered well above RT. Following the model of Stiles and McMichael,[25] we envisage the role of the [Co/Pt] as stabilizing the winding up of domain walls (DW) in the thin AFM NiO during the Co layer's magnetization reversal. In the absence of such stabilization, the partial domain wall in such a thin film will unwind from the back surface, destroying any possible LS. With only four monolayers of NiO, the concept of a DW may seem inadequate; however, the role of the [Co/Pt] magnetization is simply to pin the back surface of the NiO such that the unwinding process does not easily occur. Moreover, since

the [Co/Pt] magnetization direction varies with applied fields, the direction of pinning and subsequently the energy stored in the wound up DW can be externally controlled with modest applied fields. The data of Fig. 3 detailing the dependence of the EB field on the magnetic remanence of the out-of-plane [Co/Pt] layer and the temperature dependence of the EB presented in Fig. 4 both depend on the presence of the [Co/Pt] layer, albeit in differing ways. The role of NiO in this structure is fundamentally different above and below T_B leading to two distinct mechanisms.

Above T_B, but below T_N, the majority of NiO grains will rotate with the adjacent Co magnetization, contributing to an H_c enhancement. The dragging of AFM grains with the FM magnetization is responsible for the enhanced H_c in EB systems and has been shown to be ubiquitous in bilayer EB systems[26,27] and is present well above T_B.[28] We propose that the differing H_c for the upper and lower curves of the hysteresis loop (i.e., LS) occurs due to the asymmetry in the [Co/Pt] in-plane magnetization. Soft NiO grains with high coupling strengths will rotate with the respective magnetizations leading to small increases in H_c at low H_{set}. The asymmetry in H_c for the upper and lower curves is due to the NiO layer in contact with the [Co/Pt] layer rotating through a much larger angle on the upper branch than on the lower branch of the hysteresis loop. As the applied field changes from +250 to −250 Oe in the upper curve, the [Co/Pt] layer undergoes a highly asymmetric change in the magnetization angle (see Fig. 2). As H_{set} increases, this asymmetry increases. Coupling at the [Co/Pt]/NiO interface implies a similarly asymmetric change in the Ni spins at this interface which may be propagated into the rest of the NiO layer. In the lower curve, the very small changes in H_c with increasing H_{set} reflect the small change in angle in both the [Co/Pt] magnetization and hence the corresponding NiO orientation.

For set fields above 3 kOe there is no increase in the asymmetry of the [Co/Pt] loop, as the full [Co/Pt] loop is closed; this is responsible for the saturation of the Co LS seen in Fig. 3. Above this saturating H_{set} the symmetrical increase in H_c for both the upper and lower curves (Fig. 3) is attributed to the further rearrangement of the NiO grains. At these larger H_{set} values, the [Co/Pt] magnetization has a larger in-plane component leading to an increase in coupling energy with the NiO layer. This increased coupling at the [Co/Pt]/NiO interface will increase the alignment of NiO grains along the H_{set} direction, increasing the effective coupling at the Co/NiO interface. In this scenario, harder NiO grains will be dragged symmetrically during Co magnetization reversal leading to a symmetric increase in H_c. This symmetric H_c increase will continue until the [Co/Pt] layer saturates in plane.

Below the observed T_B of 225 K, the LS is no longer due to an asymmetry in the [Co/Pt] magnetization since the applied fields are now symmetric (±250 Oe) in this study. The perpendicular anisotropy of [Co/Pt] has been shown to remain constant with temperature below RT,[29] so in the ±250 Oe loops at lower temperatures there will be no asymmetry in the [Co/Pt] in-plane magnetization. Instead, the LS occurs due to a balance between the coupling at each interface and the AFM DW energy within a NiO grain. Each NiO grain differs in volume (and hence anisotropy energy), coupling strength (to both the in-plane Co and out-of-plane [Co/Pt]), and in-plane orientation. We have assumed (i) an even number of AFM layers and (ii) AFM coupling at the NiO/[Co/Pt] interface and FM coupling at the Co/NiO interface. Although we certainly have atomic level roughness, the average value of the thickness (as measured by an *in situ* quartz monitor) is 11 Å leading to a preponderance of grains with four monolayers of NiO. The assumed sign of coupling at either interface is based on previous x-ray magnetic circular dichroism (XMCD) studies, which indicate a Ni terminated Co/NiO interface and an oxygen terminated NiO/[Co/Pt] interface leading to opposite signs of coupling.[13] As in the Stiles and McMichael paper,[25] we define $\hat{\mathbf{M}}_{\mathbf{FM}}$, $\hat{\mathbf{u}}$ and $\hat{\mathbf{m}}(\mathbf{0})$ as the directions of the FM magnetization of the in-plane Co layer, the direction of the AFM NiO layer furthest from the FM layer and the direction of the AFM NiO layer closest to the FM Co, respectively. For simplicity, we assume that both the Co and [Co/Pt] layers are in a single domain state and can be described as a macrospin coupled to an ensemble of NiO grains.

The direction of $\hat{\mathbf{u}}$ in this heterostructure is controlled by the magnetization direction of the [Co/Pt], the strength of the coupling at the [Co/Pt]/NiO interface and the anisotropy constants of NiO (including the AFM ordering parameter). For NiO, the anisotropy constant for in-plane rotation [within the (111) plane] is suggested to be ~5% of the out-of-plane rotation [perpendicular to the (111) plane], $K_1 = 3.32 \times 10^6$ erg/cm^3.[10,12] These widely differing values of anisotropy lead to significantly different T_B for in-plane vs out-of-plane EB. Experiments on in-plane Ni/NiO(28 Å) (Ref. 11) indicate a T_B of 34 K whereas experiments on [Co/Pt]/NiO(11 Å) bilayers and trilayers with perpendicular anisotropy indicate a T_B above 200 K.[19,20] These T_B values correspond to the thermal energies needed to switch a particular AFM grain, corresponding to K_1V (K_2V) in the case of out-of-plane (in-plane) rotations, where V is the volume of a grain. Above the cited T_B, all AFM grains are thermally switched in a particular anisotropy direction.

The initial 250 Oe in-plane field at RT to saturate the Co layer will have the effect of inducing a small in-plane remanent magnetization in the [Co/Pt] layer resulting in a magnetization direction that is ~5° from the normal. This direction of magnetization together with the coupling will define the vector $\hat{\mathbf{u}}$, where $\hat{\mathbf{u}}$ may cant out of and rotate within the (111) plane. Rotation within the (111) plane is governed by the magnitude of K_2, whereas canting out of this plane is governed by the magnitude of K_1. Although $K_2 \ll K_1$, XMCD measurements[13] have shown that the NiO spins do cant out of plane at [Co/Pt]/NiO interfaces. Thus, we can expect $\hat{\mathbf{u}}$ to have an out-of-plane component during the cooling procedure and lie within the plane defined by $\hat{\mathbf{M}}_{\mathbf{FM}}$ and the [Co/Pt] magnetization. At the opposite interface, the direction of $\hat{\mathbf{m}}(\mathbf{0})$ is dictated by the direction of $\hat{\mathbf{M}}_{\mathbf{FM}}$, the interfacial coupling, the anisotropy constants of NiO (particularly K_2), and, most importantly, the strength of the AFM ordering.

Below 225 K the following constraints hold (i) energy minimization requires that all the NiO spins lie in the plane $\vec{\mathbf{A}}$ defined by the magnetization of the in-plane Co layer, $\hat{\mathbf{M}}_{\mathbf{FM}}$, and the pinned NiO layer, $\hat{\mathbf{u}}$ [25] (ii) the energy cost

associated with overcoming K_1 prevents the NiO spins from further canting out of the (111) plane; however, at these temperatures spin rotation within the plane is energetically allowed and (iii) the NiO layers adjacent to the Co and [Co/Pt] layers are coupled to their respective FM layers. Based on these three constraints on the NiO spins we envisage $\hat{\mathbf{u}}$ rotating only slightly within the NiO (111) plane during the magnetization reversal of both the Co and [Co/Pt]. It is energetically unfavorable for $\hat{\mathbf{u}}$ to completely track along with the [Co/Pt] magnetization; when the [Co/Pt] reverses, it does so by passing through a vector perpendicular to the NiO (111) plane. For $\hat{\mathbf{u}}$ to track the [Co/Pt] magnetization along this path, it must overcome the K_1 anisotropy. Instead $\hat{\mathbf{u}}$ will attempt to switch via rotation within the NiO (111) plane; however, this mode will only allow for relatively small rotations due to constraints (i) and (ii). Allowing $\hat{\mathbf{u}}$ to rotate in the NiO (111) plane, a small amount minimizes the energy cost associated with overcoming the AFM ordering but also satisfies our three constraints. In this scenario, $\hat{\mathbf{u}}$ is sufficiently pinned to produce a DW in the NiO layers resulting in slightly differing energies for positive and negative Co saturation, leading to very small LS values. Our measured LS is orders of magnitude smaller than those seen for perpendicular [Co/Pt]/NiO EB measurements. As the temperature decreases below 225 K, the anisotropy constants increase leading to the observed linear temperature dependence of LS.

There are numerous configurations of NiO spins that may occur within the constraints given to produce the observed LS. To experimentally observe the specific configuration will require depth profiling measurements of the local magnetization in the NiO layer, similar to the recent investigation of a [CoO/NiO] multilayer in contact with a Pt-Co layer.[30]

V. CONCLUSION

We have demonstrated an isothermally tunable LS in a Co/NiO/[Co/Pt] heterostructure with an 11 Å thick NiO interlayer. This heterostructure exhibits a LS along the field axis and H_c enhancement at RT. The addition of the [Co/Pt] multilayer allows dynamic control of the NiO AFM structure by way of stabilization due to the exchange interaction at the NiO/[Co/Pt] interface. Variation in the in-plane component of magnetization in the [Co/Pt] multilayer leads to changes in the NiO, which in turn lead to a change in the H_c of the Co layer. This H_c change is asymmetric for the upper and lower curves of the hysteresis loop due to an asymmetry in the [Co/Pt] magnetization leading to a tunable LS. The addition of the [Co/Pt] layer also greatly enhances T_B of this structure by adding an additional constraint to the NiO layer during Co magnetization reversal. Such isothermal control of the LS at RT and the greatly enhanced T_B is useful in a variety of modern approaches to spintronic applications.

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