Enhancement of the magnetic interfacial exchange energy at a specific interface in NiFe/CoO/Co trilayer thin films via ion-beam modification

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Keywords
modification, beam, ion, via, films, magnetic, thin, enhancement, trilayer, co, coo, nife, interface, specific, energy, exchange, interfacial

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Enhancement of the magnetic interfacial exchange energy at a specific interface in NiFe/CoO/Co trilayer thin films via ion-beam modification

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A series of ferromagnetic Ni₈₀Fe₂₀(55 nm)/antiferromagnetic CoO (25 to 200 nm)/ferromagnetic Co (55 nm)/SiO₂(substrate) trilayer thin films were fabricated by ion-beam assisted deposition in order to understand the role of ion beam modification on the interfacial and interlayer coupling. The microstructural study using transmission electron microscopy, X-ray reflectometry, and polarised neutron reflectometry showed that ion-beam modification during the deposition process led to an oxygen-rich Co/CoO nanocomposite interface region at the bottom layer. This interface caused a high exchange bias field for the ferromagnetic cobalt. However, the exchange bias for top permalloy ferromagnet remained low, in line with expectations from the literature for the typical interfacial energy. This suggest that the ion-beam enhancement of the magnetic exchange bias is localized to the Co/CoO interface where local microstructural effects provide the dominant mechanism. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4865569]

I. INTRODUCTION

The unusual magnetic anisotropy known as exchange bias was first discovered in Co/CoO nanoparticles, and results in the unidirectional shift of the magnetic hysteresis. More recently, the deliberate implantation of oxygen atoms using ion-beam techniques has been shown to lead to an anomalously high interfacial exchange energy in ion-beam modified Co. The origin of this exchange field enhancement in disordered Co/CoO nanocomposites remains unclear, but the relative strength compared to high quality Co/CoO thin films is both noteworthy and interesting. Moreover, our past work showed that the deposition order of the two layers, which may be either Co/CoO/Substrate or CoO/Co/Substrate, affected the penetration and diffusion of ions in the thin film interfaces. However, it is a topical question whether the bulk of the antiferromagnet, away from the ferromagnetic and interfacial regions, is an important component in this enhanced field. It is not clear, for instance, whether, as proposed for FeFₓ, a long-range spin structure in the (antiferromagnet) AF could lead to a different exchange bias field sensed at the opposite interface from the ion-beam modified interface. In previous work, we showed that exposure of the Co layer to moderate energy oxygen ion-beams during the deposition of the CoO led to the implantation and diffusion of oxygen into the underlying Co, leading to a pillar-like microstructure with an anomalously high exchange bias. A natural question is whether similar effects can be realized in a trilayer structure, and whether the third layer on the opposite side of the AF structure is affected by the modified exchange coupling at the nanocomposite interface. Previous work on (ferromagnet) FM/AF/FM layers in the 20–200 nm thickness regime reported a strong coupling between the two layers ascribed to the important role of bulk AF spins in such a structure. In this work, we fabricated Ni₈₀Fe₂₀/CoO/Co spins valves using ion-assisted deposition to test whether, upon modification of the cobalt layer, the permalloy layer also experienced an altered exchange field or whether the effect was localised to the bottom interface.

II. THEORY

By selecting two ferromagnetic materials with different coercive fields, it is possible to create a well-resolved double-step magnetic hysteresis loop that is the superposition of the two independent magnetic layers as shown in Figure 1. In this work, we chose Ni₈₀Fe₂₀ as the second ferromagnet, due to its soft magnetic character, and used CoO as the intermediary layer to form a Ni₈₀Fe₂₀/CoO/Co trilayer structure. This allows for the direct calculation of the exchange bias fields of each interface independently. For the scenario illustrated in Figure 1, one can then calculate the coercivity and exchange bias for each ferromagnetic layer labelled FM1 and FM2 using the formulae

\[ H^{FM1}_{EB} = \frac{(A + B) + (C + D)}{4}, \]  
\[ H^{FM2}_{EB} = \frac{(E + F) + (G + H)}{4}, \]  
\[ H^{FM1}_{F} = \frac{(C + D) - (A + B)}{4}, \]  
\[ H^{FM2}_{F} = \frac{(G + H) - (E + F)}{4}. \]  

If the ferromagnetic layers are truly independent, then this method may be used to reliably deconvolute the resulting

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double hysteresis to obtain the collinear configurations of each decoupled ferromagnet. A depth-resolved probe, however, such as polarized neutron reflectometry can provide a direct check of the magnetic depth profile. Once the behavior of such a trilayer is understood, the judicious selection of a specific field selected at points A-F can set the nanoscale spin alignment of each ferromagnetic layer independently during the field-cool process. For the typical case of negative exchange bias, this allows to one to safely field cool parallel or antiparallel alignments through the temperature range of the field cool process. Past work has shown that the formation of partial or anti-phase domains in the antiferromagnet can mediate magnetic frustration through the antiferromagnetic layer, with two possible mechanisms illustrated in Figure 2. In one case, it was shown that antiparallel alignment during the field cool process resulted in a lowering of exchange bias in one layer, whereas in other cases there is a change in the overall loop shape associated with a new anisotropy axis. Using similar techniques, we investigated whether ion-beam modification of the Co/CoO nanocomposite interface had a detectable effect on the bias at the Ni80Fe20/CoO interface across the CoO spacer layer in a trilayer structure.

III. EXPERIMENTAL

A dual ion-beam deposition technique was used to prepare the Ni80Fe20/CoO/Co trilayers on a Si(100) substrate that had previously been annealed to provide a thick SiO2 surface layer. A Kaufman ion source (800 V, 7.5 mA) was used to focus an argon ion-beam onto a commercial Co or Ni80Fe20 target surface. An End-Hall source (V_{EH} = 100 V, 500 mA) was used to in-situ bombard the growing film during CoO layer deposition with a mixture of 15% O2/(Ar + O2), which was previously found to form the rock-salt phase. Five samples were deposited with the layer thicknesses summarised in Table I.

The thicknesses were chosen in the same length scale regime as Ref. 5 and the two ferromagnet layers were deposited to give a constant thickness across the series, whereas the thickness of the oxide spacer layer (x) was systematically varied. The antiferromagnet CoO was selected because of its similarity to FeF2 in terms of antiferromagnetic domain state formation. The deposition was done at room temperature, and no external magnetic field was applied during deposition. Cross sectional transmission electron microscope (TEM) was performed on a JEOL [JEM-2010] TEM operating at 200 kV. X-ray reflectometry and diffraction was conducted on a Panalytical X’Pert Pro lab source using Cu-Kα radiation (λ = 0.154 nm). Polarised neutron reflectometry was conducted on the PLATYPUS reflectometer at the OPAL Research Reactor, Sydney. Variable temperature measurements were performed on a commercial Quantum Design Physical Property Measurement System vibrating sample magnetometer. Except where explicitly stated, the samples were field-cooled by applying a +10 kOe field at 350 K to saturate the sample, before applying a smaller field (0–1000 Oe) while cooling to 200 K where the exchange bias was measured.

IV. DATA AND ANALYSIS

A. Film structure

Figure 3 shows the X-ray diffraction patterns for each of five samples in the trilayer series collected using CuKα radiation, where the data for each sample has been offset for clarity. From the appearance of the broad diffraction peaks, the top two layers are polycrystalline, consisting of the face-centered cubic Ni80Fe20 (a = 3.55 Å) and the CoO (a = 4.25 Å) rock-salt phase. The cobalt layer is highly polycrystalline with nanoscale crystallites leading to the lack of clear X-ray diffraction features. Figure 4(a) is a high resolution cross-sectional TEM of sample 1. It shows a clear 3

FIG. 1. Schematic illustration of double hysteresis loop resulting from volume-average measurement of two uncoupled ferromagnetic phases. (a) Softer magnetic phase. (b) Harder magnetic phase. (c) Superposition of hard and soft magnetic phase.

FIG. 2. Two possible mechanisms for long-range frustration mediated through an antiferromagnetic spacer in a FM/AF/FM structure for the situation that the top ferromagnet is dominant, and both interfacial exchanges are positive (J_{int1} > J_{int2} > 0). (Left) Partial spiral domain wall parallel to the surface causing a non-collinear arrangement at the bottom interface. (Right) Different populations of antiferromagnetic phase domains with walls perpendicular to the surface. In both figures, only one antiferromagnetic sub-lattice has been drawn for clarity.
layer structure with a reasonably sharp interface between the Ni$_{80}$Fe$_{20}$ and the CoO layers, corresponding to low interface roughness ($\sigma_1 < 2$ nm). However, the boundary between the cobalt layer and the CoO layer is less distinct, with a diffuse interface showing evidence of layer-intermixing on a longer length scale ($> 5$ nm). Figure 4(b) is the selected area electron diffraction (SAED) pattern of the Co/CoO region. It confirms that three nanocrystalline phases co-exist in this vicinity: hcp metallic Co, rock-salt phase CoO, and a smaller component of the Co$_3$O$_4$ spinel phase. Figures 4(c) and 4(d) are the bright field and dark field image of the Co layer showing small grains with sizes in the range of 3–18 nm. Thick grain boundaries are evident separating grains of the same phase. The presence of some oxygen ($\approx 4\%–11\%$ per atom) within the cobalt layer was qualitatively confirmed using energy dispersive spectroscopy mapping. Figure 5 shows the X-ray reflectometry (XRR) pattern for each sample in the series, where the data and fit for each sample has been offset horizontally by 0.05 Å$^{-1}$ for clarity. The X-ray reflectometry was fitted with a 3-layer model using the genetic fitting and least-squares algorithm in the Motofit software package. Table I shows the fitting results for the thickness of each of the three layers for samples 1–5. The spacer-layer thickness ranged from 25 to 150 nm showing a linear dependence on the deposition time. For all samples, from the XRR fits, the roughness of the Ni$_{80}$Fe$_{20}$/CoO boundary was found to be significantly lower ($0.8–1.8$ nm) than the CoO/Co boundary ($6–23$ nm).

![FIG. 4. (a) TEM cross-section, (b) electron diffraction pattern, (c) bright field, and (d) darkfield images for Ni$_{80}$Fe$_{20}$/CoO/Co sample 1.](image)

![FIG. 5. X-ray reflectometry fits and data for Ni$_{80}$Fe$_{20}$/CoO(x)/Co (samples 1–5). Intensities have been systematically offset in Q$_x$ for samples 2–5 for the sake of clarity. Thick black lines are the experimental data and thin colored lines are the fit to the data. Solid lines are fits to the data for each sample. Table I summarises the fitted layer thicknesses for the three layers in each of the five samples.](image)
B. Magnetic properties

Figure 7 presents the room temperature magnetometry data for samples 1–5. The data have been over-plotted to emphasize the overall similarity in the magnetic properties of the films. A clear double step hysteresis is seen for all samples, reminiscent of the schematic diagram in Fig. 1. This is interpreted as the independent switching of each ferromagnetic layer separated by the paramagnetic CoO spacer layer. All samples were found to be saturated at fields above 2 kOe, as evident in the flat magnetic response for increasing fields up to 50 kOe. At room temperature, using Eqs. (3) and (4), the coercivity for the two phases is calculated as $H_{EB}^1 \approx 10$ Oe and $H_{EB}^2 \approx 250$ Oe. The inner loop coercivity is essentially identical for all samples, whereas small differences occur for the various films at the outer-loop switching point, which shows a distribution of coercive fields in a relatively narrow range (200–350 Oe). The exchange bias values for $H_{EB}^{FM1}$ and $H_{EB}^{FM2}$ are both zero within experimental uncertainty ($0 \pm 5$ Oe), which is to be expected since the Néel temperature of CoO is usually below room temperature (293 K for bulk).\(^\dagger\) Figure 8 summarises the angular dependent in-plane magnetic properties for sample 5 obtained at room temperature by taking multiple hysteresis loops at different in-plane angles. The lack of any strong angular dependence for the quantities $H_{FM1}^c$ and $H_{FM2}^c$ confirms the polycrystalline nature of the ferromagnetic grains, which, on average, lack a uniaxial or biaxial in-plane easy-axis. It should be noted that, from the room temperature magnetometry presented in Fig. 7, it is ambiguous which of the quantities ($H_{FM1}^c$ and $H_{FM2}^c$, or $H_{FM1}^2$ and $H_{FM2}^2$) belongs to the cobalt layer because neither of the dual loops gives the properties normally expected of cobalt. For instance, one could assume the typical case, which is that the cobalt layer has a higher coercivity (25–130 Oe)\(^\ddagger\) than the permalloy (2–10 Oe), in which case the outer loops belong to cobalt. However, in that scenario, the outer loop should have a step-size that is $\approx 2/3$ of the overall magnetic saturation, since bulk cobalt has 1400 emu/cm\(^3\)) versus bulk permalloy which has 780 emu/cm\(^3\),\(^\dagger\) and the layers are of almost the same thickness. Therefore, from the room-temperature magnetometry, there are two possibilities: either the cobalt has a reduced magnetic moment or the Ni\(_{80}\)Fe\(_{20}\) has an enhanced coercive field. To resolve this anomaly, and correctly understand the magnetic depth profile of the sample in the saturated state, we conducted polarised neutron reflectometry at 4000 Oe and room temperature (i.e., in the saturated state).

C. Magnetic depth profile

Figure 9 reveals the polarised neutron reflectometry pattern obtained for sample 1. No spin-flip scattering was observed in the saturated state, implying a collinear arrangement of moments with the field.\(^\ddagger\) Fitting of the two non-spin-flip (NSF) channels resulted in the magnetic depth profile illustrated on the right of Fig. 9. From the high-quality fit, it is obvious that there are missing magnetic moments located in the cobalt layer. The permalloy layer obtains a value close to its bulk magnetization (0.9 $\mu_B$ per formula unit Ni\(_{80}\)Fe\(_{20}\)) whereas the average magnetic moment in the cobalt layer is reduced to 0.4 $\mu_B$ per Co, which is only 25% of the bulk value (1.7 $\mu_B$ (Ref. 21)). To prove that this magnetic depth-profile is the more feasible fit between the two possibilities elicited from the magnetometry data, Fig. 10 shows a low-quality fit resulting from the magnetic depth model where the cobalt retains a higher average magnetic moment (0.9 $\mu_B$ per formula unit), but the...
permalloy is oxidized (0.4 \( \mu_B \) per formula unit). It is obvious that the model depicted in Fig. 9 is the only one of the two possibilities which accurately describes the data. Although in some magnetic multilayers, a non-collinear relationship of one ferromagnet with respect to the other has been found, leading to a similar reduction in the aligned magnetic moment, in that case, neutron spin-flip scattering would be expected.\(^{20,22} \) and it should not be possible to fit the NSF data with a collinear model.\(^{18} \) The best-fit nuclear scattering length density is also slightly higher for the cobalt layer, consistent with an increased oxygen-content. Recently, we studied CoO/Co bilayers fabricated in the same way and found that during the deposition of the CoO, oxygen ions penetrated into the underlying Co layer, forming pillar-like CoO features embedded in the Co layer.\(^{23} \) It is believed that a similar mechanism is at work in these trilayer films, explaining the diffuse interface and the nanocomposite diffraction peaks found in the cobalt region. Both the coercivity enhancement and the lower magnetic saturation in the film series are attributed to the penetration of oxygen into the cobalt, and the formation of CoO nanoscale structures embedded within the Co layer.\(^{23} \) The interpenetrating CoO and Co\(_3\)O\(_4\) regions are nominally antiferromagnetic and lower the average magnetization for the nanocomposite layer measured directly by polarized neutron reflectometry in the saturated state, along with thicknesses and exchange biases measured separately for the cobalt layers and permalloy layers, the interfacial energy \( E_{\text{int}} \) for the permalloy inner-loop is an order of magnitude smaller than that of Co at 200 K \( (H_{\text{EB}}^{\text{FM2}}) \). Moreover, the coercive field of the permalloy \( H_{\text{FM1}} \) is seen to be \(<10\text{Oe}\) for both the 300 K and 200 K measurements, but the cobalt layer shows a five-fold enhancement in coercive field at low temperature \( (H_{\text{FM2}}^{\text{FM1}} \approx 1000\text{Oe}) \). Taking account of the saturation magnetization for the nanocomposite layer measured directly by polarized neutron reflectometry in the saturated state, along with thicknesses and exchange biases measured separately for the cobalt layers and permalloy layers, the interfacial energy \( E_{\text{int}} = t_{\text{FM}}H_{\text{EB}}M_{\text{sat}} \) for the cobalt and permalloy layers can be calculated for each layer. The magnitude of the exchange bias for the cobalt layers in the five samples \((t_{\text{FM}} = 55\text{ nm}, M_{\text{sat}} = 400\text{ emu/cm}^2)\) is anomalously high, leading to an estimate of the interfacial energy \( E_{\text{int}} \) in the range of 0.45–0.6 ergs/cm\(^2\) at 200 K, which is higher than values of CoO. Figure 11 is the in-plane magnetic hysteresis measured for sample 1 under each of these two different field-cooling conditions, where only the first (untrained) loop is shown. In both cases, it is apparent that inner and outer loops shift either left or right, giving an exchange bias with the opposite sign to the cooling field. This is typical behaviour for most exchange bias systems. Table II summarises the quantities \( H_{\text{FM1}}^{\text{FM1}}, H_{\text{FM2}}^{\text{FM1}}, H_{\text{EB}}^{\text{FM1}} \) and \( H_{\text{EB}}^{\text{FM2}} \) for samples 1–5 under the +100 Oe cooling condition after first saturating at 10 kOe at 300 K.

The magnitude of the loop-shifts, \( H_{\text{FM1}}^{\text{FM1}} \), is found to be similar for all films within experimental uncertainty, whereas a maximum in \( H_{\text{FM2}}^{\text{FM1}} \) is apparent for sample 3, which had a 89 nm CoO layer. The exchange bias shift is direct evidence for magnetic coupling between ferromagnetic (Ni\(_{80}\)Fe\(_{20}\) or Co) and antiferromagnetic (CoO) spins. The lack of a strong spacer layer thickness implies that, in this case, the interfacial regions for the Ni\(_{80}\)Fe\(_{20}\)/CoO and CoO/Co regions dominate the magnetic effects. We note that the loop-shift \( (H_{\text{EB}}^{\text{FM1}}) \) for the permalloy inner-loop is an order of magnitude smaller than that of Co at 200 K \( (H_{\text{FM2}}^{\text{FM1}}) \). Moreover, the coercive field of the permalloy \( H_{\text{FM1}} \) is seen to be \(<10\text{Oe}\) for both the 300 K and 200 K measurements, but the cobalt layer shows a five-fold enhancement in coercive field at low temperature \( (H_{\text{FM2}}^{\text{FM1}} \approx 1000\text{Oe}) \). Taking account of the saturation magnetization for the nanocomposite layer measured directly by polarized neutron reflectometry in the saturated state, along with thicknesses and exchange biases measured separately for the cobalt layers and permalloy layers, the interfacial energy \( E_{\text{int}} = t_{\text{FM}}H_{\text{EB}}M_{\text{sat}} \) for the cobalt and permalloy layers can be calculated for each layer. The magnitude of the exchange bias for the cobalt layers in the five samples \((t_{\text{FM}} = 55\text{ nm}, M_{\text{sat}} = 400\text{ emu/cm}^2)\) is anomalously high, leading to an estimate of the interfacial energy \( E_{\text{int}} \) in the range of 0.45–0.6 ergs/cm\(^2\) at 200 K, which is higher than values

D. Exchange bias and layer-resolved interfacial energy

Sample 1 was field-cooled in one of two cooling fields \((\pm 1000)\) Oe to 200 K, which is below the blocking temperature of CoO. Figure 11 is the in-plane magnetic hysteresis measured for sample 1 under each of these two different field-cooling conditions, where only the first (untrained) loop is shown. In both cases, it is apparent that inner and outer loops shift either left or right, giving an exchange bias with the opposite sign to the cooling field. This is typical behaviour for most exchange bias systems. Table II summarises the quantities \( H_{\text{FM1}}^{\text{FM1}}, H_{\text{FM2}}^{\text{FM1}}, H_{\text{EB}}^{\text{FM1}} \) and \( H_{\text{EB}}^{\text{FM2}} \) for samples 1–5 under the +100 Oe cooling condition after first saturating at 10 kOe at 300 K.

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reported for typical thin films that use CoO (0.16–0.28 ergs/cm²) measured at >100 K with similar blocking temperatures. On the other hand, the interfacial energy for the permalloy layers (Mₘₐₓ = 790 emu/cm², tₚₘₐₓ = 55 nm, and Hₖₚₘₐₓ = 10 Oe) is 0.04–0.05 ergs/cm², which is a more typical value for polycrystalline CoO pinning layers in thin films (0.03–0.12 ergs/cm²). The vast difference in interfacial energies for the two ferromagnets coupled to nominally the same antiferromagnet implies that the modified nanocomposite interface structure at the Co/CoO has a strong, local effect on the exchange bias.

Along with the microstructural investigations, the increased low temperature exchange bias and coercivity in the Co/CoO layer provides indirect evidence for the implantation of oxygen into the cobalt layer, which alters the magnetic spin structure of the resulting nanocomposite, leading to stronger apparent coupling between the Co and CoO regions. One way to understand this is to remember that for two perfect thin film layers, there is only a single planar interface, whereas for a nanocomposite there can be numerous interfaces formed between Co/CoO, which may resemble an array of core-shell nanoparticles. In such a system, there is a natural tendency towards higher effective “surface” area for the interfacial magnetic coupling to occur. Indeed, the exchange bias effect was first discovered in core-shell Co/CoO nanoparticles, and the loop shift of disordered magnetic particles is often higher than in the film equivalents. In general, for films with high quality interfaces, the exchange bias loop-shift usually decrease with a 1/tFM relationship, where tFM is the thickness of the ferromagnet layer. The data, however, shows that the nanocomposite region breaks this trend allowing for a strong exchange bias to exist even for a comparatively thick ferromagnetic layer (55 nm). This agrees with the recent finding that strong exchange bias could be realized in 100 nm epitaxial Co thin films ion-implanted with oxygen, although in that work, a high energy implantation energy was used (60 keV), presumably resulting in a different microstructure. It should be noted, that while increased, the interfacial energy is somewhat lower than the previous report of interfacial energies up to 2.5–3.5 ergs/cm² in ion-beam modified Co/CoO bilayers, suggesting that the unique morphology in that case played an important role.

The vast difference in interfacial energies also implies a second important point: The two ferromagnets are probably decoupled across the spacer layer, since upon parallel and antiparallel field cooling; we find the exchange bias has the same magnitude for the individual ferromagnets, and the relative magnitude is unchanged. A previous study found that antiparallel field cooling led to a lower exchange bias in one of the two ferromagnets. It is also conceivable that, in the case of a negative interfacial exchange constant at one of the interfaces, one may expect the inverse result, therefore necessitating that all configurations are considered. To test this directly, we explored the different field-cooling configurations. To test this, sample 1 was saturated in +1000 Oe at room temperature. It was then field-cooled to 200 K in one of 4 fields (±25 Oe or ±1000 Oe). In principle, the positive applied fields should not alter the exchange bias magnitude since they preserve the parallel alignment during cooling. A negative −25 Oe field, however, should reverse the permalloy layer during cooling, but preserve the positive direction in the cobalt, while a −1000 Oe field should reverse both the cobalt and permalloy causing their alignment to cool in a collinear-negative direction. Figure 12 shows that only the inner hysteresis is affected by the change in field-cooling.

<p>| TABLE II. Exchange bias and coercivity at 200 K for Ni₈₀/Fe₂₀/CoO/Co samples 1–5 after field-cooling in +1000 Oe (after initial saturation). |</p>
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Modification during deposition. This resulted in a larger coercivity and exchange bias for the bottom cobalt layer. However, the top Ni$_{80}$Fe$_{20}$/CoO interface was nominally more ideal, and did not sense the enhanced exchange field.

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