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Temperature and set field dependence of exchange bias training effects in Co/NiO/[Co/Pt] heterostructures with orthogonal easy axes

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ABSTRACT

Training effects in a new class of exchange biased ferromagnet/antiferromagnet/ferromagnet trilayers ($Co/NiO/[Co/Pt]_3$) with mutually orthogonal easy axes have been measured and successfully modeled. Previous experiments have demonstrated an enhanced blocking temperature as well as the ability to isothermally field tune the magnitude of the room temperature in-plane exchange bias. These effects have been attributed to the presence of the [Co/Pt] multilayer with perpendicular magnetic anisotropy, which variably pins the backside NiO domains. Here we show that the tuning of the exchange bias and the blocking temperature enhancement are highly dependent on both the temperature and the in-plane remanence of the normally out-of-plane [Co/Pt] multilayer, achieved using modest in-plane set fields. Training effects and their dependence on temperature and in-plane remanence are modeled using a thermodynamic approach. The in-plane remanence of the [Co/Pt] acts only to set the equilibrium exchange bias value and sets the scale for the blocking temperature; it has no effect on the training. We conclude that training effects occur only at the Co/NiO interface and that the relaxation towards equilibrium is confined to this interface. The field enhanced blocking temperature and isothermal tuning of exchange bias in these magnetic heterostructures with mutually orthogonal easy axes could play a role in the enhancement of exchange bias effects in future spin-valve devices. A thorough knowledge of the training effects is essential to account for the fundamental relaxation mechanisms that occur with repeated field cycling.

1. Introduction

Interfacial coupling at the interface between a ferromagnet (FM)/antiferromagnet (AFM) leads to a symmetry breaking and a subsequent shift of the hysteresis loop (the exchange bias), among other phenomena. Although discovered over 50 years ago [1], exchange bias continues to pose intriguing questions [2, and references therein], one of which is the training effect, in which the exchange bias field is progressively reduced on repeated magnetic field cycling [2,3]. Exchange bias has important technological applications in magnetic memory devices [4–6] and a clear understanding of the training effect could lead to technological advances by increasing the magnitude of the exchange bias. Exchange bias has been modeled by allowing for the formation of multiple domains, usually within the antiferromagnet. A net interfacial magnetization within the antiferromagnet, S_{AFM}, exchange couples to the FM. The training effect is commonly ascribed to the rearrangement of these domains towards equilibrium on repeated field cycling, thereby altering S_{AFM}. Numerous models based on experimental observations of the training effect have been proposed [7–13]. Much of the data on the training effect fit a $1/\sqrt{n}$ dependence [2,14–17], although an understanding of this dependence has been lacking. The addition of non-magnetic impurities to the AFM [18] leads to an increase in the exchange bias, and has been ascribed to the lower energy cost associated with the formation of a domain wall that passes through a non-magnetic impurity. Monte Carlo modeling of the interface magnetization of these diluted AFM films displays hysteretic behavior where the hysteresis loop does not close at positive saturation [18], implying a decrease in interfacial magnetization with subsequent loops leading to a decrease in the exchange bias. This effect has also been studied extensively by C. Binek in the framework of non-equilibrium thermodynamics, in which consecutive magnetization cycles rearrange the interface spins of the AFM towards equilibrium [11]. This more contemporary approach provides better insight and predictive power into the temperature dependence [19] of the training effect as well as a physical basis for the phenomenological $1/\sqrt{n}$ dependence, and was recently used to successfully model tunable training effects in a Ni/NiO bilayer structure [20].

In this paper, we measure and model the training effect in a new class of exchange biased magnetic heterostructures, consisting of

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two AFM/FM interfaces in a Co/NiO(11 Å)/[Co/Pt]$_3$ stack. Previous experiments [21] have demonstrated an enhanced in-plane blocking temperature as well as the ability to isothermally field tune the magnitude of the in-plane exchange bias, at temperatures well below the Néel temperature of the AFM. Both effects are large; the observed blocking temperature of 225 K is well above the 40 K or less expected for in-plane exchange bias with a similar thickness of NiO [22,23] and the isothermal field tuning shows room temperature changes of 35 Oe in the exchange bias on application of a 3 kOe in-plane set field. Both effects have been attributed to the [Co/Pt]$_3$ layer with perpendicular anisotropy, as discussed thoroughly in Ref. [21] The exchange interaction at the NiO/[Co/Pt]$_3$ interface pins the NiO domains, thereby increasing the energy cost associated with reversal of the AFM domains [21]. The effects seen in this trilayer sample are quite distinct from those seen in a Co/FeMn/CuNi stack [24], in which the presence of the Co underlayer effectively eliminates exchange bias effects at the CuNi/FeMn interface, although the approach to probing the AFM layer with a low T_c ferromagnet gives insight into the formation of exchange bias in a variety of systems, including our trilayer structure.

2. Experimental techniques

Three samples were prepared by dc and rf magnetron sputtering from separate Pt, Co, NiO and Cu targets on naturally oxidized Si substrates deposited in 2 mTorr Ar pressure with a base pressure of $\sim 3\times10^{-8}$ Torr. Samples A and B were made with a fixed thickness of NiO:

Sample A: Si⟨1 1 1⟩/Pt(200 Å)/NiO(11 Å)/[Co(4 Å)/Pt(6 Å)]$_3$/Cu(100 Å)

Sample B: Si⟨1 1 1⟩/Pt(200 Å)/Co(40 Å)/NiO(11 Å)/[Co(4 Å)/Pt(6 Å)]$_3$/Cu(100 Å) [25].

A third sample was made with a NiO thickness gradient:

Sample C: Si⟨1 1 1⟩/Pt(200 Å)/Co(40 Å)/NiO(6-20 Å)/[Co(4 Å)/Pt(6 Å)]$_3$/Cu(100 Å).

The 40 Å Co layer and the [Co/Pt]$_3$ multilayer stack display the expected in-plane and out-of-plane magnetic easy axes, respectively. The NiO is polycrystalline but strongly (1 1 1) textured. The thickness calibration and structural characterization are discussed elsewhere for samples A and B [21]. For sample C, the NiO wedge was characterized using X-ray reflectivity on a thicker wedge and scaled with time. The absolute thickness for the center of this NiO layer was checked with an *in-situ* crystal thickness monitor. Room temperature magnetic characterization of samples was done using alternating gradient field magnetometry (AGFM), while temperature dependent measurements were made using the magneto-optic Kerr effect (MOKE) in a Janis cryostat with polarization preserving optical windows.

The isothermally field tunable exchange bias previously measured on sample B [21] was confined to the first loop performed after application of an in-plane set field. Subsequent magnetization loops displayed a progressive reduction in the exchange bias, the subject of this paper. The blocking temperature of 225 K was measured at equilibrium after repeated ($n>20$) field cycling. This blocking temperature is similar to the reported value of $\sim$250 K [26] for out-of-plane exchange bias in [Co/Pt]/NiO bilayers with similar thicknesses. Above this blocking temperature, in-plane Co layer exchange bias was observed *only* when the [Co/Pt] multilayer acquired a non-zero in-plane magnetization and the magnitude of the Co exchange bias was directly proportional to this in-plane remanence. We have argued previously that the effect of the [Co/Pt] layer is to alter the configuration of domains in the NiO layer. Hence these heterostructures are ideal for investigations of the training effect, providing a precise method by which to control the configuration of AFM domains with application of fairly modest fields on a single sample. The role of the [Co/Pt] layer is to variably pin AFM domains, similar to previous measurements of the effects of dilution in the AFM layer [27], albeit on a single sample, allowing us to discount variations in interface roughness, crystallinity and coupling constants, all of which have an effect on the magnitude of both the training effect and the exchange bias field. Also in contrast to the dilution experiments, the magnetization direction of the [Co/Pt]$_3$ stack will control only the interfacial NiO layer, rather than altering the volume of the AFM.

In order to rule out dipolar coupling between the two FM layers, the results of loop shift measurements on sample C, with increasing NiO thickness, are shown in Fig. 1. Dipolar coupling would result in a monotonic decrease in coupling strength with increasing thickness of the spacer layer with the exact dependence a function of the domain structure and roughness of the two FM layers [28,29]. Below 8.5 Å (indicated by the gray bar), an exponential decay in the loop shift occurs. In this region, direct coupling dominates, presumably due to pinholes. Above 8.5 Å the loop shift deviates from this exponential decay showing a slight overall increase with thickness. Since dipolar coupling is expected to decrease with increasing thickness of the intervening layer, we assume that at a thickness of 11 Å, the thickness of NiO in samples A and B, the coupling effects seen are quite distinct from those due to dipolar coupling. Additional confirmation of this assumption comes from calculations of the magnitude of the expected magnetostatic energy density due to the presence of a domain wall. For a sample of [Co/Pt]/NiO/[Co/Pt], in which the FM layers are aligned, the magnetostatic coupling energy is $E_M^{(1)} \approx -0.02\times10^6\,\mathrm{erg/cm^3}$ for an 11 Å thickness of NiO [30]. This is the maximum possible energy; in our samples, the proportion of parallel aligned domains will be small, leading to a magnetostatic energy which is a small fraction of $E_M^{(1)}$. In contrast, for an exchange bias field (H_{eb}) of 35 Oe, the energy density is $E_{eb}=M_S H_{eb} = 1426\,\mathrm{emu/cm^3}\times 35\,\mathrm{Oe} = 0.05\times10^6\,\mathrm{erg/cm^3}$, where M_s is the saturation magnetization of the Co layer.

In order to understand the *quantitative* effects of the [Co/Pt]$_3$/NiO interface on the entire heterostructure, room temperature,

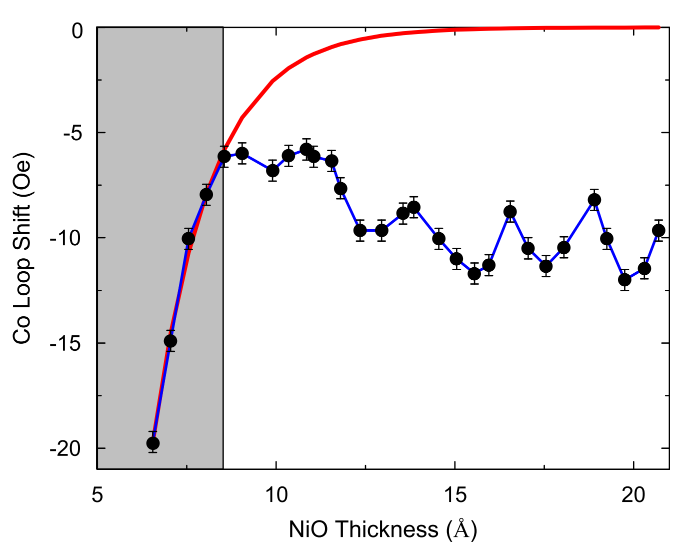

Fig. 1. Co layer loop shift (Sample C) as a function of NiO thickness. Below 8.5 Å (gray region), direct coupling between the Co and [Co/Pt] layers dominates and a monotonic, exponential decay is observed. At thicknesses greater than 8.5 Å, the Co loop shift deviates from this monotonic behavior, showing an overall increase.