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Closely linear temperature dependence of exchange bias and coercivity in out-of-plane exchange-biased \([\text{Pt/Co}\,\text{3/NiO}\,(11\,\text{Å})]\) multilayer

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Strong out-of-plane exchange biasing has been observed in out-of-plane exchange biased \([\text{Pt}\,(5\,\text{Å})/\text{Co}\,(4\,\text{Å})]/\text{NiO}\,(11\,\text{Å})]\) multilayer with perpendicular easy axis. Both the exchange field \(H_E\) and coercivity \(H_C\) display a closely linear temperature dependence except at very low temperatures. A thin NiO layer coated on the top of a Pt/Co multilayer has a great effect on the domain pattern of the Pt/Co multilayer, which is in a more irregular configuration with much smaller domain sizes than the uncoupled Pt/Co multilayer. A simulation according to Malozemoff’s random field model gives a good agreement to the experimental temperature dependence of \(H_E\) and \(H_C\), suggesting that the closely linear temperature dependences of \(H_E\) and \(H_C\) are strongly related to the behavior of the temperature of anisotropies and of the spin rotation inside the domain walls of a thin NiO layer. The blocking temperature of \(T_B = 220\,\text{K}\) is much higher than that observed in ferromagnetic/thin NiO systems with in-plane anisotropy. © 2003 American Institute of Physics. [DOI: 10.1063/1.1582378]

I. INTRODUCTION

When a ferromagnetic (FM) layer is in contact with an antiferromagnetic (AF) layer, exchange biasing at the interface causes the hysteresis loop of the FM layer to shift from its origin by an amount known as exchange field \(H_E\), and enhanced coercivity \(H_C\) is often observed. Since this effect was first discovered almost half century ago,\(^{1}\) it has been investigated extensively both experimentally and theoretically in an effort to understand the underlying microscopic mechanism.\(^{2,3}\) Most observations of exchange biasing have been reported for exchange biased FM/AF systems with in-plane anisotropy. Recently, out-of-plane exchange biasing has been observed in exchange biased FM/AF systems with perpendicular anisotropy.\(^{4-8}\)

A linear or almost linear temperature dependence of \(H_E\) has been observed in many exchange-biased FM/AF oxide systems with in-plane FM anisotropy.\(^{9-13}\) For FM/thin NiO bilayers with in-plane FM anisotropy, Takano\(^{9}\) and Gruyters\(^{10}\) have found a linear temperature dependence of \(H_E\) and a blocking temperature \(T_B\) much lower than the Néel temperature \(T_N\).\(^{13}\) The data from Refs. 9 and 10 are plotted in Fig. 1 and show the drop in \(T_B\) with a decrease in NiO thickness. Experimental studies\(^{9,14}\) have shown that the \(T_N\) of thin NiO film increases with an increase in the film thickness and is close to the bulk value of 523 K at 100 Å (see Fig. 1). Measurements of \(T_N\) for thin NiO films by Takano\(^{9}\) were performed using a microcrolimiter to directly measure the specific heat of magnetic NiO/MgO superlattices. Heat capacity measurements of microgram thin films up to 540 K are made possible by the dramatic reduction of the contributions of the substrate, thermometer, and heater to the total heat capacity. Néel temperature \(T_N\) can be obtained from the peak position in a specific heat curve, which is the sign of a magnetic phase transition. The accuracy for the measurement of \(T_N\) is within \(~3\%\) as estimated by Takano.\(^{9}\) Measurements of \(T_N\) for thin NiO films by x-ray absorption spectroscopy (XAS) and magnetic dichroism were also performed by Alders\(^{14}\). In ferromagnetically ordered materials, the presence of interatomic superexchange interactions causes long-range magnetic order and therefore influences the interatomic spin–spin correlation functions, resulting in a spontaneous atomic magnetic moment \(\langle M\rangle\). Using circularly polarized x rays, the temperature dependence of \(\langle M\rangle\) in ferromagnets can be measured via the magnetic circular dichroism (MCD) in the x-ray photoabsorption spectra, because MCD is sensitive to \(\langle M\rangle\). Even though the MCD effect disappears for antiferromagnets, both theoretical and experimental studies have shown that for x rays with linear polarization, magnetic linear dichroism (MLD) is proportional to \(\langle M^2\rangle\). Hence, this MLD effect in XAS can be used to measure the long-range magnetic ordering in antiferromagnets. The critical point at which the temperature dependence of the MLD effect disappears corresponds to Néel temperature \(T_N\). By performing Ni \(L_2\) XAS experiments on thin NiO films with the x-ray polarization vector parallel to the sample surface, Alders\(^{14}\) determined Neel temperatures for thin NiO films which are in good agreement with neutron diffraction results. The Neel temperatures of thin NiO films measured by both Takano\(^{9}\) and Alders\(^{14}\) are consistent.

The Neel temperatures determined by Takano\(^{9}\) and by Alders\(^{14}\) are for uncoupled thin NiO films. Recently, van der Zaaag\(^{13}\) have observed that the Neel temperature of a thin CoO film with thickness less than 50 Å is increased, higher than the bulk value, when it is in contact with a ferromagnetic \(\text{Fe}_3\text{O}_4\) layer. It appears to be a fact that the antiferromagnetic ordering in an AF layer can be stabilized if it is exchange coupled to a FM layer. However, the blocking temperature becomes much lower than the bulk Neel temperature,\(^{13}\) and its mechanism is not yet clear.
Above $T_N = 523$ K, bulk NiO has a face-centered-cubic (fcc) rocksalt structure. Below 523 K, magnetoelastic forces lead to slight contraction of the lattice along different (111) axes. The magnetoelastic anisotropy due to the small distortion causes the spins to lie in ferromagnetically ordered (111) planes with (111) planes stacked antiferromagnetically. Crystallographic twinning gives rise to four so-called $^2T$ domains that correspond to four possible (111) axes. Each $^2T$ domain can be further divided into three possible $^S$ domains with spins lying along three possible directions of $[11\bar{2}]$, $[1\bar{2}1]$, and $[\bar{2}11]$, respectively. There are two kinds of domain walls between $S$ domains: $S_1$, in which the wall is parallel to (111) planes, and $S_\perp$, in which the wall is perpendicular to (111) planes. In both $S_1$ and $S_\perp$ walls, spins rotate in the (111) plane. For very thin NiO films of 10 $- 20$ Å, it is believed to show no AF ordering due to discontinuity and/or interfacial diffusion.

In this article, we perform an investigation of out-of-plane exchange biasing in a glass Pt(100 Å)/[Pt(5 Å)/Co(4 Å)]$_3$/NiO(11 Å) multilayer with perpendicular easy axis. The observation of strong out-of-plane exchange biasing implies that even for a very thin NiO layer of 11 Å, it still presents AF ordering. The temperature dependences of exchange field $H_E$ and coercivity $H_C$ have been simulated according to Malozemoff’s random field model for FM/AF systems and extension of it by Zhang et al.

## II. EXPERIMENTS

The glass/ Pt(100 Å)/[Pt(5 Å)/Co(4 Å)]$_3$/NiO(11 Å) multilayer (sample I) was prepared by dc and rf magnetron sputtering from separate targets at deposition rates of 0.96, 0.199, and 0.188 Å/s for Pt, Co, and NiO, respectively. Ar pressure of 3 mTorr was used and the base pressure was $4 \times 10^{-7}$ Torr. An additional sample of glass/ Pt(100 Å)/[Pt(5 Å)/Co(4 Å)]$_3$/Pt(20 Å) (sample II) was grown simultaneously for comparison. No external field was applied during sample preparation. X-ray diffraction reveals that the Co layers are highly hexagonal close packed (hcp) textured, both Pt and NiO layers are highly fcc (111) textured. After growth, magnetic force microscopy (MFM) images at room temperature were obtained immediately for samples I and II in the as-grown state. All $M-H$ hysteresis loops were measured using a superconducting quantum interference device (SQUID) and AGFM magnetometers with the field applied perpendicular to the sample plane.

## III. RESULTS AND DISCUSSION

Figure 2(a) shows the $M-H$ loops at room temperature for samples I and II. Both loops are fairly square, implying out-of-plane easy axes. The loop for sample I does not show any shift, but does show greatly enhanced coercivity of $H_C = 887$ Oe in contrast to $H_C = 390$ Oe for sample II. Experimental studies have shown that five monolayer NiO ($\sim 10.5$ Å) has a Neel temperature of 295 K (Fig. 1). For 11 Å NiO film, the Neel temperature is estimated from Fig. 1 to be $\sim 310$ K. Thus, the enhanced coercivity of sample I is attributed to interfacial exchange coupling. The MFM images in Figs. 2(b) and 2(c) reveal clearly the difference in domain size for samples I and II due to the presence of the NiO layer. The presence of the NiO layer reduces the size of up and down domains by almost one order of magnitude, which can be seen clearly by comparing Figs. 2(b) and 2(d). This striking difference in domain size between samples I and II suggests that the formation of domains in the Pt/Co multilayer is strongly affected by the NiO layer through interfacial exchange coupling. A simultaneous effect of the Pt/Co multilayer on domain wall formation in the NiO layer also occurs (as will be discussed subsequently).

Sample I was cooled from room temperature to 30 K in 1 T magnetic field applied normal to the sample plane. After cooling, a series of $M-H$ loops were obtained at different temperatures, some of which are presented in Fig. 3. There is clear evidence of a two-step reversal process which may be related to the distribution of AF easy axes. The loop at 40 K displays a large exchange field of $H_E = -754$ Oe and large coercivity of $H_C = 5150$ Oe. The temperature dependences of...
$H_E$ and $H_C$ are shown in Fig. 4, and demonstrate that both $H_C$ and $H_E$ decrease almost linearly with an increase in temperature except in the very low temperature range, and that $H_E$ vanishes completely above the blocking temperature of $T_B = 220$ K. This blocking temperature shown in Fig. 1 ($\Delta$) is much higher than the previously observed $T_B = 52$ K, and ($\Delta$) 5 $234$, respectively. The ratio of $T_B/T_N = 0.71$ in the present case is much larger than $T_B/T_N = 0.33$ and 0.07 observed for permalloy/NiO(100 Å) (Ref. 9) and Ni/NiO(28 Å) (Ref. 10) systems, respectively.

Previous attempts to explain the linear temperature dependence of $H_E$ and the much lower blocking temperature assumed that the AF oxide layer was composed of an ensemble of exchange decoupled grains with size close to the superparamagnetic limit, and the blocking temperature was determined by the temperature at which the magnetocrystalline anisotropy energy is comparable to the thermal energy.\(^{10,20}\) Assuming that the NiO layer in the Ni/NiO bilayer is composed of grains with an average diameter of 100 Å, Gruyters\(^{10}\) calculated the anisotropy constant $K_{AF}$ in the NiO (111) plane as $K_{AF} = 25k_BT_B/V_{AF}$ with $k_B$ and $V_{AF}$ the Boltzmann constant and the average volume of AF grains, respectively. $K_{AF}$ was estimated to be $\sim 7.4 \times 10^5$ erg/cm\(^3\), giving the right order of magnitude in comparison to the theoretical values ($K_{AF}^{(1)} = 3 \times 10^6$ erg/cm\(^3\) and $K_{AF}^{(2)} = 1.5 \times 10^5$ erg/cm\(^3\) for the anisotropy constants out of and in the NiO (111) plane, respectively.\(^{21}\) In our sample, the NiO thickness is 11 Å. Assuming a grain diameter of 100 Å from previous work\(^{9,10}\) and using the measured $T_B = 220$ K we obtain a value of $K_{AF} = 8.79 \times 10^5$ erg/cm\(^3\), which is one order of magnitude higher than the theoretical value of $K_{AF}^{(2)}$, but close to that of $K_{AF}^{(1)}$. Thus, Gruyters’ proposal\(^{10}\) cannot explain the high blocking temperature of $T_N = 220$ K observed in sample I if all AF spins lie in (111) planes of the NiO layer with (111) texture.

Experimental investigations have demonstrated that the domains exist in NiO films, and that the spin ordering inside domains or domain walls can be strongly affected by FM magnetization through interfacial exchange coupling between FM/NiO bilayers.\(^{22,23}\) The dramatic difference in domain pattern between samples I and II, shown in Fig. 2, indicates that interfacial exchange coupling strongly affects domain formation in the Pt/Co multilayer, suggesting the existence of domains in the NiO layer. According to the random field model presented by Malozemoff\(^{18}\) and a recent sophisticated model reported by Stiles and McMichael(SM),\(^{24}\) the existence of domain walls in AF layers plays an important role in the exchange bias effect. $H_E$ is proportional to the energy $\sigma_{AF}$ stored in AF domain walls. In the SM model,\(^{25}\) the potential barrier to the reversal of AF spins is determined by $\sigma_{AF}$. The temperature dependence of $H_E$ and the blocking temperature would then be mainly controlled by $\sigma_{AF}$, the interfacial exchange coupling strength, and the Neel temperature. In sample I, the NiO layer is highly (111) textured. Due to the lower $T$ domain wall energy, we consider only $S_1$ and $S_\perp$ domain walls. For the very thin NiO layer of 11 Å, it is obviously impossible to form a $S_1$ domain wall parallel to the FM/AF interface. Thus, it is only necessary to consider $S_\perp$ walls. In bulk NiO, the spins in $S_\perp$ walls are confined by anisotropy energy and rotate only within the (111) easy planes. However, for a very thin NiO film in contact with a Pt/Co multilayer with a perpendicular easy axis, out-of-plane interfacial exchange coupling can make it energetically favorable to have out-of-plane spins. By considering rotation both out of and in (111) easy planes, the $S_\perp$ wall energy can be expressed by\(^{25}\)

$$\sigma_{AF} = 2 \sqrt{A_{AF}K_{AF}^{(1)}\cos \alpha + (2/3)\sqrt{2A_{AF}K_{AF}^{(2)}}\cos 6\beta}, \quad (1)$$

where $A_{AF}$ is the exchange stiffness, $\alpha$ is the rotation angle of the out of (111) easy plane, and $\beta$ is the rotation angle from the easy axis of [121] (or [211], [112]) within the (111) easy plane. The second term in Eq. (1) is determined by the weaker sixfold anisotropy in the (111) easy plane. $\sigma_{AF}$ will be determined by both $K_{AF}^{(1)}$ and $K_{AF}^{(2)}$, $K_{AF}^{(2)}$ is at least one order of magnitude less than $K_{AF}^{(1)}$, and experimental studies have shown that $K_{AF}^{(2)}$ for bulk NiO drops very sharply with an increase in temperature.\(^{26}\) For a very thin NiO film of 11 Å, its $K_{AF}^{(2)}$ is believed to be weaker than the
bulk value, and thus will then be more sensitive to the increase in temperature. The contribution of $K_{\text{AF}}^{(2)}$ to the exchange bias will be limited to a low temperature range. By assuming that $A_{\text{AF}}$ is constant with the temperature and $K_{\text{AF}} = K(0)(1 - T/T_N)^2$ for cubic anisotropy of AF layers, Malozemoff’s model successfully predicts the linear temperature dependence of $H_E$ with $\sqrt{A_{\text{AF}}K_{\text{AF}}(0)(1 - T/T_N)}$. Carey and Berkowitz argued that the temperature dependence of the anisotropy constant sets a scale for $T_B$, and that exchange bias will occur only when the anisotropy is above a certain threshold value. They substituted $T_B$ for $T_N$ and obtained $H_E \approx \sqrt{A_{\text{AF}}K_{\text{AF}}(0)(1 - T/T_B)}$. Using Eq. (1) and (2) and taking into consideration the fact that the anisotropy of very thin NiO film could deviate from cubic anisotropy, we express the temperature dependence of $H_E$ as

$$H_E = C^{(1)}(1 - T/T_B^{(1)})^{\gamma^{(1)}} + C^{(2)}(1 - T/T_B^{(2)})^{\gamma^{(2)}},$$

where $C^{(1)}$ and $C^{(2)}$ are two constants; $(T_B^{(1)}, \gamma^{(1)})$ and $(T_B^{(2)}, \gamma^{(2)})$ are determined by the temperature dependences of $K_{\text{AF}}^{(1)}$ and $K_{\text{AF}}^{(2)}$, respectively. Using two effective blocking temperatures determined by the two different anisotropy constants allows us to separate the contributions of $K_{\text{AF}}^{(1)}$ and $K_{\text{AF}}^{(2)}$ to exchange bias. As shown in Fig. 4, the temperature dependence of $H_E$ was fitted using Eq. (2). $(C^{(1)} = -943, T_B^{(1)} = 213 \text{ K}), (C^{(2)} = -234, T_B^{(2)} = 45 \text{ K})$, and $(\gamma^{(1)} = 1.28, \gamma^{(2)} = 0.98)$ were determined by the fit. The ratio of $C^{(1)}/C^{(2)} = 4.03$ is comparable to the theoretical value of $\sqrt{K_{\text{AF}}^{(1)}}/\sqrt{K_{\text{AF}}^{(2)}} = 4.47$, demonstrating the good simulation to the experimental temperature dependence of $H_E$. Both $\gamma^{(1)}$ and $\gamma^{(2)}$ are a little different from those for bulk NiO, suggesting that the anisotropy for very thin NiO film deviates from cubic anisotropy. The value of $T_B^{(2)}$ is much higher than that of $T_B^{(1)}$, but is close to the observed $T_B = 220 \text{ K}$, strongly suggesting that the main contribution to exchange bias comes from $K_{\text{AF}}^{(1)}$, and that the high $T_B$ observed is determined by the temperature dependence of $K_{\text{AF}}^{(1)}$. The contribution of $K_{\text{AF}}^{(2)}$ to exchange bias is limited to only a very low temperature range of $T < 45 \text{ K}$. The value of $T_B^{(1)} = 45 \text{ K}$ is close to the blocking temperature of $T_B = 34 \text{ K}$ observed by Gruyters in a Ni/(28 Å) NiO bilayer with in-plane anisotropy. For that system, the in-plane Ni magnetization will favor NiO spins lying in the (111) planes due to interfacial exchange coupling, and no rotation of spins inside the domain walls will occur out of (111) easy planes. With an increase in temperature, the weak $K_{\text{AF}}^{(2)}$ drops rapidly, and becomes too weak to pin the NiO domain walls in position and causes the exchange bias to vanish at very low temperature. This explains the observed small deviation of $H_E$ from the linear temperature behavior in the low temperature range shown in Fig. 4: both $K_{\text{AF}}^{(1)}$ and $K_{\text{AF}}^{(2)}$ are strongly related to the exchange bias at low temperature, and $K_{\text{AF}}^{(2)}$ drops much more rapidly with an increase in temperature. For our sample, the Co magnetization is perpendicular to the interface, i.e., the NiO (111) easy planes, so interfacial exchange coupling will favor rotation of AF spins out of the (111) easy plane, and then $K_{\text{AF}}^{(1)}$ will contribute to exchange bias in addition to the weak $K_{\text{AF}}^{(2)}$. Since $K_{\text{AF}}^{(1)}$ is at least one order of magnitude larger than $K_{\text{AF}}^{(2)}$, it is reasonable to assume that $K_{\text{AF}}^{(1)}$ drops more slowly than $K_{\text{AF}}^{(2)}$ with an increase in temperature, thereby giving an explanation for the higher $T_N = 220 \text{ K}$ than observed in FM/thin NiO bilayers with in-plane FM anisotropy.

By considering the effect of random fields on coercivity $H_C$ at the interface between FM/AF bilayers, Zhang et al. extended Malozemoff’s model and obtained as the expression of $H_C$

$$H_C = \frac{J_S}{M_s a_0 a_{\text{FM}}^{\frac{3}{2}}} \sqrt{a_0 a_{\text{FM}}},$$

where $J_S$ is the average exchange coupling constant of nearest-neighbor FM and AF spins at the interface, $a_0$ is the monolayer separation, $M_s$ is the saturation magnetization of the FM layer, $a_{\text{FM}}$ is the thickness of the FM layer, and $L$ is the size of the domain. In FM/AF systems, coercivity $H_C$ is related not only to random fields at the interface but also to the AF property. By assuming a square grid of domains with lateral dimensions in the AF layer, Malozemoff found $L$ to be $L = \pi \sqrt{A_{\text{AF}}/K_{\text{AF}}}$. Substituting this expression into Eq. (3) gives $H_C$ as

$$H_C \approx \frac{2 \sqrt{A_{\text{AF}} K_{\text{AF}}^{(1)} \cos \alpha}}{3} \left( \frac{2 A_{\text{AF}} K_{\text{AF}}^{(2)} \cos \beta}{\sqrt{3}} \right)^{1/2}.$$

Due to $K_{\text{AF}}^{(1)}$ being much stronger than $K_{\text{AF}}^{(2)}$, Eq. (5) can be rewritten to first-order approximation as

$$H_C \approx \left( 2 \sqrt{A_{\text{AF}} K_{\text{AF}}^{(1)} \cos \alpha} \right)^{1/2} + \frac{\sqrt{A_{\text{AF}} K_{\text{AF}}^{(2)} \cos \beta}}{3 \sqrt{\sqrt{A_{\text{AF}} K_{\text{AF}}^{(1)} \cos \alpha}}^{1/2}}.$$

Simulation to the temperature dependence of $H_E$ showed that the contribution of $K_{\text{AF}}^{(2)}$ to exchange bias is limited to only the low temperature range of $T < 45 \text{ K}$, and that the temperature dependence of both $K_{\text{AF}}^{(1)}$ and $K_{\text{AF}}^{(2)}$ deviates a little from the cubic anisotropy of bulk NiO. In general, enhanced coercivity $H_C$ still exists even if $H_E$ becomes zero above the blocking temperature. Taking these into consideration and omitting the effect of the temperature dependence of $K_{\text{AF}}$, the second term in Eq. (6), we rewrite for $H_C$ as approximately

$$H_C = D^{(0)} + D^{(1)} \left( 1 - \frac{T}{T_B^{(1)}} \right)^{\gamma^{(1)}} + D^{(2)} \left( 1 - \frac{T}{T_B^{(2)}} \right)^{\gamma^{(2)}},$$

where $D^{(0)}$, $D^{(1)}$, and $D^{(2)}$ are three constants. The inclusion of $D^{(0)}$ in Eq. (7) takes into consideration of the existence of $H_E$ above the blocking temperature. $\gamma^{(1)}$ and $\gamma^{(2)}$ take val-
ues of 1.28 and 0.98, respectively, determined by fitting to the temperature dependence of $H_E$. $T_B(1)$ and $T_B(2)$ are two parameters which will be determined by fitting to the temperature dependence of $H_C$. In general, $T_B(1)$ and $T_B(2)$ will be larger than $T_E(1)$ and $T_E(2)$, respectively, determined by fit to the temperature dependence of $H_E$, because enhanced coercivity still occurs above the blocking temperature. Fitting according to Eq. (7) has given a good agreement with the experimental temperature dependence of $H_C$ shown in Fig. 4. $T_B(1)=254$ K and $T_B(2)=70$ K from fit are higher than $T_B(1)=213$ K and $T_B(2)=45$ K, respectively, consistent with the experiments. The fit to the temperature dependence of $H_C$ shows clearly that the closely linear temperature behavior of $H_C$ at high temperature is determined by the behavior of the temperature of the stronger $K_{AF}^{(1)}$, and the deviation from the linear behavior at low temperature is due to contributions of both $K_{AF}^{(1)}$ and $K_{AF}^{(2)}$ and the more rapid drop of $K_{AF}^{(2)}$ with the temperature.

IV. SUMMARY

Strong out-of-plane exchange biasing was observed in a glass/ Pt (100 Å)/[Pt (5 Å)/Co (4 Å)]$_3$/NiO (11 Å) multilayer with perpendicular easy axis. Its domain pattern in the as-grown state is strongly affected by the NiO layer through interfacial exchange coupling, and it displays a more irregular domain configuration with much smaller domain sizes than sample II. The temperature dependences of both $H_E$ and $H_C$ are almost linear except in the very low temperature range of $T<45$ K, and the blocking temperature of $T_B=220$ K (above which $H_E$ vanishes) is much higher than that observed in FM/thin NiO bilayers with in-plane FM anisotropy. The simulations of the temperature dependence of $H_E$ and $H_C$ have shown that these phenomena are strongly related to the temperature dependence of anisotropies in thin NiO layers and the spin rotation behavior inside NiO domain walls induced by perpendicular Co magnetization through interfacial exchange coupling.

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