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Sarbeswar Sahoo
University of Nebraska-Lincoln, sarbeswar@gmail.com

Christian Binek
University of Nebraska, Lincoln, cbinek@unl.edu

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Quenching of the Exchange Bias Training in $Fe/Cr_2O_3/Fe$ Trilayer

Sarbeswar Sahoo and Christian Binek

Department of Physics and Astronomy and the Nebraska Center for Materials and Nanoscience,
University of Nebraska, Lincoln, Nebraska 68588-0111, USA

Abstract. Exchange bias (EB) and its associated training effects are studied in an epitaxial Fe(10 nm)/Cr_2O_3(2.7 nm)/Fe(10 nm) trilayer heterostructure grown by molecular beam epitaxy. The EB decreases linearly with increasing temperature from T=5 K to T=50 K. It changes sign and becomes positive within 50 K < T < 200 K, finally changing back to regular EB for T > 200 K up to the highest measured temperature of T=395 K. Remarkably, the latter is far above the bulk Néel temperature T_N=307 K. EB training effects occur only at 5 K < T < 50 K. We show that this training can be quenched by subjecting the system to DC magnetic field, $\mu_0 H_{DC} \leq 7$ T. The applied field most likely induces a temperature dependent spin-flop transition. Upon its removal the antiferromagnetic Cr_2O_3 pinning layer evolves uniformly into its quasi-equilibrium spin configuration thus leading to quasi-equilibrium EB.

Keywords: Exchange bias, training effects, epitaxial thin films
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INTRODUCTION

The phenomenon of exchange bias (EB) was discovered by Meiklejohn and Bean in 1950 in exchange coupled ferromagnetic (FM)/antiferromagnetic (AF) Co/CoO granules [1]. It plays a major role in today's spintronic devices [2]. For instance, it constitutes an important component of the magnetic read head. The latter consists of a GMR or TMR type structure which includes a fixed layer and a free (sensor) layer. While the free layer is used to sense the flux emanating at the transition of bits in the media, the fixed layer is stabilized by combining a ferromagnetic film in proximity of an antiferromagnetic film (exchange bias). The discovery of GMR elements has revolutionized the present magnetic data storage industry for which this discovery was awarded the Nobel Prize in Physics 2007 [3].

EB is primarily established by three different experimental procedures: First, by applying a magnetic field during the fabrication process; second, by post-deposition annealing of the FM in presence of a magnetic field; third, by field-cooling (FC) the heterostructure below the AF Néel temperature T_N. While the first two procedures are industrially used, the last one is used for fundamental studies of EB and its related effects. EB manifests itself in the shift of the hysteresis loop along the magnetic field axis and a concomitant increase in the coercivity $\mu_0 H_c$. The EB magnitude $\mu_0 H_{eb}$ depends on intrinsic parameters such as the exchange coupling at the FM/AF interface, FM and AF film thicknesses, interface roughness and individual FM and AF micro(magnetic)- structure [4].

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Recently, tuning of the EB has been studied in a broad variety of systems with involved experimental protocols. They include cooling in different magnetic fields [5], cooling in zero field from different magnetization states [6], cooling in combinations of DC and AC fields [7], saturating the FM in large negative fields and then measuring the remaining loop with different wait times [8], high-temperature annealing [9], ion irradiation [10], and subjecting the system to pulsed fields [11].

In this paper, we investigate the exchange bias, its training and provide a mechanism to quench the EB training in an epitaxial Fe 10 nm/Cr_2O_3 2.7 nm/Fe 10 nm trilayer. Training refers to a gradual change of the bias field upon cycling the heterostructure through consecutive hysteresis loops. This structure with identical F thicknesses was chosen to take advantage of the induced moment at both Fe/Cr_2O_3 interfaces and the possibility of additional coupling between the FM layers across the AF buffer. In addition, the motivation of using Cr_2O_3 as the AF layer is based on our recent observation of significant magnetic moment in single Cr_2O_3 films [12]. There the direction of the induced moment could be tuned by applied DC magnetic fields. This has led the successful tuning of the EB by moderate DC fields [13]. Here, we explore the possibility to overcome the EB training owing to the non-equilibrium nature of AF by applying large DC fields, $\mu_0 H_{DC} \leq 7$ T. Interestingly, we find that quenching of EB training takes place isothermally without any specific field-cooling procedure.

EXPERIMENTAL DETAILS

The investigated trilayer structure, Fe 10 nm/Cr_2O_3 2.7 nm/Fe 10 nm, was prepared in a molecular beam epitaxy (MBE) chamber at a base pressure of 1×10^{-10} mbar. Since Cr exhibits multiple oxide states (+3, +4, +6), stringent control of the O_2 partial pressure and substrate temperature during the evaporation of Cr metal is crucial in order to optimize the growth of stoichiometric Cr_2O_3 films. In our case, (0001)-oriented c-Al_2O_3 substrate is heated to and maintained at 573 K during the deposition process. Thermal evaporation of Cr metal in an O_2 partial pressure of 2.2×10^{-6} mbar yields stoichiometric Cr_2O_3 films [12]. Prior to the growth of the top Fe film the base pressure was regained by evacuating O_2 from the chamber. The growth rates of Fe and Cr_2O_3 films were monitored by a calibrated quartz oscillator and were found to be 0.37 nm/min and 0.28 nm/min, respectively. We point out that no external magnetic field was present during the deposition of various films.

Detailed structural analyses of the trilayer structure were performed by large-angle X-ray diffraction (*Max-B, Rigaku D*) and small angle X-ray reflectivity as reported somewhere else. It is worth to mention that by using LEPTOS-2 program the interface roughness values were found to be 0.4±0.2 nm, 0.2±0.2 nm, 0.4±0.2 nm, and 0.1±0.05 nm for c-Al_2O_3/Fe, Fe/Cr_2O_3, Cr_2O_3/Fe, and Fe/air, respectively. This indicates highly epitaxial nature of the trilayer structure.

The magnetic measurements were carried out using the superconducting quantum interference (SQUID) magnetometer (*MPMS-XL, Quantum Design*) with magnetic fields applied in the sample plane.

RESULTS AND DISCUSSION

Figure 1(a) shows hysteresis loops measured at different temperatures after field cooling (FC) the sample in a magnetic field of $\mu_0 H$ = 50 mT from T = 395 K to the respective temperatures T = 5, 10, 20, 50, 100, and 150 K. The loops show typical features associated with EB effects. They are shifted along the $\mu_0 H$ axis by an amount $\mu_0 H_{eb}$ due to the exchange coupling at the interfaces in the trilayer Fe 10 nm/Cr_2O_3 2.7 nm/Fe 10 nm. Moreover, the exchange biased hysteresis loops have remanent to saturation magnetization ratio $M_r/M_S = 1$.

We use the conventional definitions of $\mu_0 H_{eb}$ and $\mu_0 H_c$, *i.e.*, $\mu_0 H_{eb} = \mu_0 (H_+ + H_-)/2$ and $\mu_0 H_c = \mu_0 (H_+ - H_-)/2$, where $\mu_0 H_+$ and $\mu_0 H_-$ are the field values at which the magnetization becomes zero on right and left branch of the hysteresis loop, respectively. Fig. 1(b) shows $\mu_0 H_{eb}$ and $\mu_0 H_c$ *vs* temperature. We identify three different temperature regimes of EB. First, between T = 5 K to T = 50 K the EB field, $\mu_0 H_{eb}$, decreases linearly with increasing temperature. Second, $\mu_0 H_{eb}$ *vs* T changes sign and becomes positive within 50 K < T < 200. Third, $\mu_0 H_{eb}$ *vs* T changes back to regular type for T > 200 K and remains non-zero up to the highest measured temperature of T = 395 K, which is larger than the Néel temperature T_N=307 K of bulk Cr_2O_3.

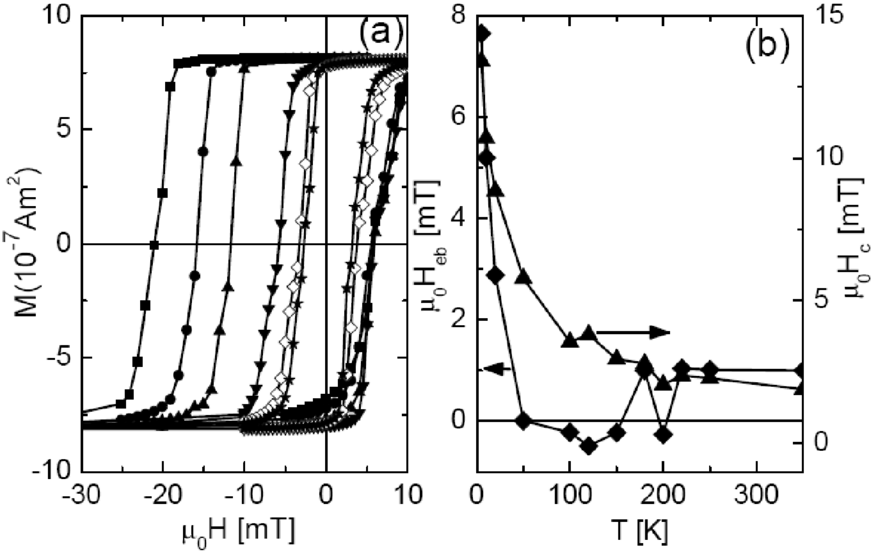

FIGURE 1. (a) Exchange biased hysteresis loops at different temperatures, T = 5 K (squares), 10 K (circles), 20 K (up triangles), 50 K (down triangles), 100 K (diamonds), and 150 K (stars). (b) $\mu_0 H_{eb}$ and $\mu_0 H_c$ *vs.* T. Lines are guide to the eye.

It should be pointed out that EB above T_N of the AF has also been observed in other FM/AF systems such as FeZnF2/Fe (Ref. 14) Co/NiF_2 (Ref. 15), Fe_3O_4/CoO (Ref. 16) and $Ni_{80}Fe_{20}$/Co_3O_4 (Ref. 17). There it is primarily attributed to either strain or proximity effects at the FM/AF interfaces. In our trilayer, the existence of the EB up to the highest measured temperature is tentatively attributed to the presence of magnetically hard interface regions or surface moments.

Training of the exchange bias offers a unique tool to test the deviation from the equilibrium of the AF layer [18]. Training refers to a gradual change of the bias field, which evolves upon cycling the trilayer through consecutive hysteresis loops and is a measure of the approach to equilibrium spin configuration of the AF. To this end, we measured the training effects at 5 and 20 K. Fig. 2(a) and 2(c) present selected hysteresis loops measured within each training sequence at T = 5 K and 20 K, respectively after field cooling from T = 395 K at a field of 50 mT. They reveal huge training effects associated with the non-equilibrium AF configuration of the Cr_2O_3 film below $T^* \approx 50$ K, which leaves its fingerprints in the $\mu_0 H_{eb}$ *vs.* T curve (Fig. 1b).

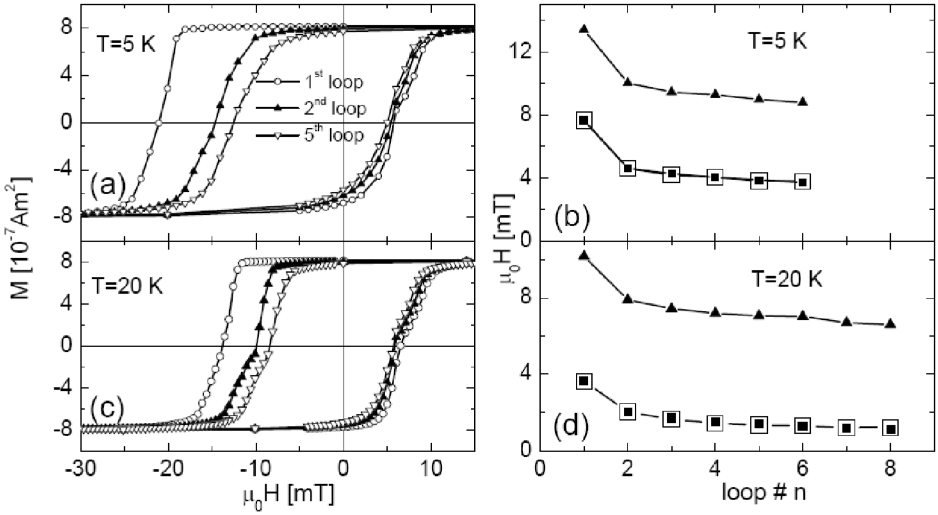

FIGURE 2. (a) Exchange bias training loops at T = 5 K. (b) Evolution of exchange bias (squares) and coercivity (triangles) with subsequently cycled loops, *i.e.*, $\mu_0 H_{eb}$ (squares) and $\mu_0 H_c$ (triangles) *vs.* loop number, n at T = 5 K. (c) Exchange bias training loops at T = 20 K. (b) $\mu_0 H_{eb}$ and $\mu_0 H_c$ *vs.* loop number, n at T = 20 K. The solid squares are the best fit of Eq. 1 to the training data. Lines are guide to the eye.

The gradual decrease of $\mu_0 H_{eb}$ (open squares) and $\mu_0 H_c$ (triangles) with the subsequent loop number, n, are plotted in Fig. 2(b) and 2(d), respectively. The huge training behavior indicates the spin configurational relaxation of the AF long range order. This aging process continues until the AF reaches its quasi-equilibrium state in the limit $n \to \infty$. The solid squares represent the best fit of $\mu_0 H_{eb}$ *vs* n data to a recently developed phenomenological theory [18,19,20,21],

$$\mu_0\left(H_{eb}(n+1)-H_{eb}(n)\right)=-\gamma\left(\mu_0\left(H_{eb}(n)-H_{eb}^{e}\right)\right)^3. \qquad (1)$$

The fits yield the values of the two fitting parameters: $\gamma = 1\times10^{-4}$ $(\mathrm{mT})^{-2}$ and $\mu_0 H_{eb}^{e}$=1 mT at T = 5 K while $\gamma = 3\times10^{-4}$ $(\mathrm{mT})^{-2}$ and $\mu_0 H_{eb}^{e}$=0.3 mT at T = 20 K. Note that γ contains the interface exchange coupling and the damping constant governing the relaxation dynamics of the AF spin configuration while $\mu_0 H^{\mathrm{e}}_{\mathrm{eb}}$ is the quasi-equilibrium exchange bias field [18].

Subsequently, we show that $\mu_0 H_{eb}$ *vs n* can be quenched exclusively by subjecting the system to large $\mu_0 H_{\mathrm{DC}}$ in our trilayer. Fig. 3 presents a comparison of the hysteresis loops at $T = 20$ K measured in an EB training cycle (first and eighth) to that obtained after subjecting the system to $\mu_0 H_{\mathrm{DC}} = 7$ T (circles). Note that the $\mu_0 H_{\mathrm{DC}} = 7$ T at $T = 20$ K has been applied for a duration of 300 s after FC the sample in precisely the same field of $\mu_0 H = 50$ mT. The comparison clearly reveals that the loop measured after subjecting the system to 7 T field approaches the trained 8th loop. Exposing the Fe(10 nm)/Cr_2O_3(2.7 nm)/Fe(10 nm) trilayer to large DC magnetic fields $\mu_0 H_{\mathrm{DC}} = 7$ T most likely induces a spin flop transition of the AF layer. Upon removal of the applied field a close to equilibrium AF order evolves where AF long range order establishes uniformly throughout the pinning layer leading to quasi-equilibrium EB and, hence, the absence of training.

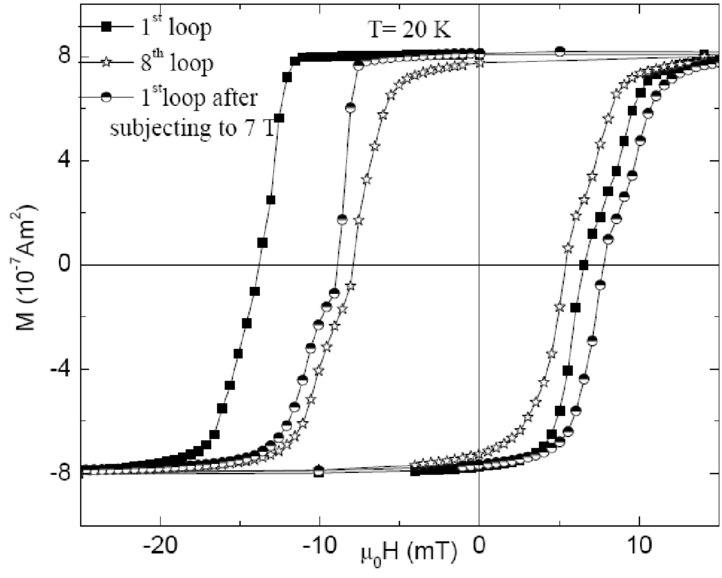

FIGURE 3. Quenching the exchange bias at $T = 20$ K by subjecting the system to large DC magnetic field after field cooled from $T = 395$K in $\mu_0 H$=50mT. The squares and stars represent the first and eighth loops in a training sequence while the circles represent the first loop measured immediately after subjecting the system to DC magnetic field of 7 T.

CONCLUSION

In conclusion, we studied the temperature dependence of the exchange bias and its training effects in an epitaxial Fe/Cr_2O_3/Fe trilayer. We, for the first time, found that in this heterostructure exchange bias training can be isothermally quenched by large DC magnetic fields. It indicates that the AF domain state of the pinning layer can be isothermally "annealed" by large DC fields such that AF quasi-equilibrium long range order establishes when the DC field is removed. The magnetically annealed EB heterostructure exhibits a stable EB field on subsequently cycled hysteresis loops. The latter corresponds to the quasi-equilibrium EB field which is asymptotically approached during EB bias training cycles of the AF pinning layer in a non quenched domain state.

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