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Controlling exchange bias in FeMn with Cu

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To study the effect of non-magnetic layer (Cu) on magnetic properties of antiferromagnetic FeMn, multilayers of Ta(5 nm)/[FeMn(***t***)/Cu(5 nm)]$_{10}$/Ta(5 nm), where ***t*** is varied in the range of 5–15 nm, are fabricated by a combination of RF and DC magnetron sputter deposition. Magnetization curves for these samples exhibit magnetic hysteresis, and when the samples are cooled in an applied magnetic field, the hysteresis loops are shifted. This shift is attributed to an "intrinsic" exchange bias effect (i.e., it is observed without a separate ferromagnetic layer). Presented temperature and thickness dependences of the coercive field, magnetic moment, and exchange bias field provide insights into the origin and mechanism of the observed intrinsic exchange bias. © *2013 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4798310]

After more than 50 years of studies since the exchange bias discovery,[1,2] the microscopic mechanism of this proximity effect remains a puzzle. Exchange bias is typically observed in a ferromagnet-antiferromagnet (FM-AF) bilayer as a horizontal shift of the hysteresis field. While most models agree on the fact that some net uncompensated magnetic moment (magnetization) has to exist in the AF, figuring out the origin of that moment in all different exchange bias systems remains a key challenge on the way of creating a complete microscopic model of this effect. In addition to the fundamental interest, ability to control the uncompensated magnetization and hence control the exchange bias is important for various applications for magnetic recording and magnetic sensors.

In this work, we study properties of the antiferromagnetic FeMn in the absence of any ferromagnetic material and investigate how the presence of a non-magnetic Cu layer in contact with FeMn affects the properties of the uncompensated magnetization in FeMn. FeMn has a high T_N (490 K for bulk FeMn, smaller for thin films) which results in exchange bias for bilayer FeMn-FM systems (e.g., FM = NiFe) observed at and above room temperature.[3–6] It also had been shown that the presence of Cu next to FeMn modifies exchange bias in FeMn-FM systems.[7]

Two types of samples are fabricated for these studies: The first consists of the 10-repeats superlattice of FeMn/Cu: Ta(5 nm)/[FeMn(***t***)/Cu(5 nm)]$_{10}$/Ta(5 nm). The second type, control samples, consists of a single layer of FeMn of the same thickness as in the first family: Ta (5 nm)/FeMn(***t***)/Ta(5 nm), where the thickness ***t*** is varied in the range of 5—15 nm. For each of the sets, five 3 mm × 5 mm substrates are mounted equally spaced on a microscope glass slide, with the short side along the microscope slide for maximizing thickness uniformity within each samples. The FeMn layer wedge is grown using a confocal magnetron sputtering geometry with the substrates stationary during deposition; other layers (Ta and Cu) are deposited with the substrate rotating at 47 rpm, which ensures thickness uniformity. This procedure yields a set of samples of the same structure of the layers but a different thickness of FeMn layer. Based on the width of each sample, the spacing between the samples, and the assumption of uniform thickness gradient of the FeMn layer, the variation of the FeMn thickness within each sample is estimated to be within ±2% of the entire layer thickness range, i.e., ±0.2 nm for the 10 nm (=15 nm–5 nm) range.

The samples are prepared by a combination of RF (FeMn) and DC (Ta and Cu) magnetron sputter-deposition on top of Si/SiO_2 substrates. Prior to being loaded into the UHV chamber, the substrates are sonicated sequentially for 5 min in soap water, distilled water, acetone, and methanol. The system is pumped down to the base pressure of 1.8×10^{-8} Torr. The argon gas pressure during the deposition is set to 3 mTorr and substrate temperature is at 20 °C. The deposition rates for Ta, Cu, and FeMn are 0.76 Å/s, 2.1 Å/s, and 0.47 Å/s, respectively. The resulting samples are polycrystalline in the plane but (111) textured along the growth direction. FeMn is a metallic AF with the face-centered-cubic (fcc) structure.[8–12] X-ray diffraction measurements performed using Bruker-AXS Discover D8 diffractometer show overlap of (111) peak for all layers similarly to the results presented in Ref. 13.

The hysteresis loop measurements are performed using Quantum Design SQUID MPMS XL and SQUID-VSM. It is noteworthy that to avoid instrumental artifacts (e.g., such as those originating from the voltage variation over the scan time), we optimized the measurement procedure by choosing the mode and parameters of the measurements as listed here: Measurements with MPMS are performed in RSO (reciprocating sample option) mode with the scan range of 2 cm, center position scan at 1 Hz frequency with 10 cycles per scan and 5 scans per measurement. Measurements with SQUID VSM are performed using peak amplitude of 4 mm, field sweep rate of 100 Oe/s or smaller, averaging time of 1 s, and waiting time for each field step: 5 s.

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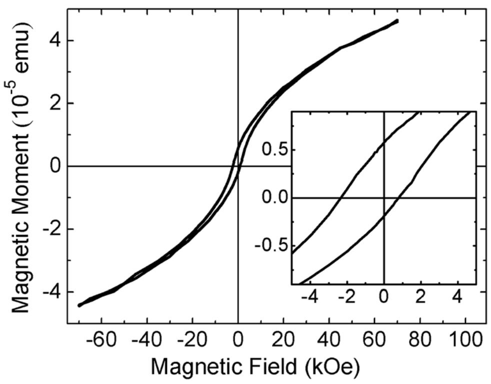

FIG. 1. Hysteresis loop measured at 10 K for the FeMn/Cu sample with 7.5-nm FeMn layers. $m_R = 0.38\ 10^{-5}$ emu, $M_{7T} = 4.6\ 10^{-5}$ emu, $H_E = -782$ Oe, $H_C = 1569$ Oe. Inset: the central portion of the hysteresis loop.

The magnetization curves measured at 10 K display finite remanences and coercive fields (Fig. 1). This is observed for both FeMn/Cu-superlattice samples and for single-layer-FeMn samples. However, for most FeMn/Cu samples, the coercive field is 1.7–2.4 times larger than that for the control samples with the same FeMn layer thickness.[14] The temperature dependence of the coercive field, H_C, for FeMn/Cu samples with a 7.5-nm-thick FeMn layer is presented in Fig. 2. The coercive field rapidly decreases from 1808 Oe at 10 K to 68 Oe at 50 K, and then it remains rather small at higher temperatures. Some studies of exchange bias systems with FeMn as an AF have shown a similar temperature dependence of the coercive field with a rapid increase of coercivity below $\sim$50 K.[15] Our observation might be an indication that it is intrinsic properties of FeMn that enhance coercive field below 50 K.

The hysteresis loops for the FeMn/Cu samples exhibit exchange bias when cooled from room temperature to 10 K in an applied field. The temperature dependence of the exchange bias field, H_E, presented in Fig. 2, also shows strong dependence at low temperatures. At 10 K, the absolute value of H_E is 897 Oe. Then, it decreases drastically, becoming about 180 Oe at 70 K and staying at that level until above 200 K. Finally, $|H_E|$ gradually decreases to zero between 250 and 300 K.

What is the source of this observed magnetization and what is responsible for the exchange bias? The magnetic moment of FeMn/Cu samples measured at 7 T, m_{7T} (after removal of the diamagnetic background due to the substrate) increases with the increasing thickness of the FeMn, but that dependence is almost linear (or increases slightly faster than linearly). This scaling suggests that the magnetic signal originates from the entire

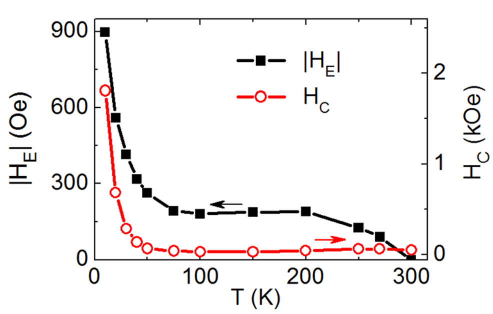

FIG. 2. Temperature dependence of the (a) coercive field, H_C, and (b) absolute value of the exchange bias field, H_E, for the 7.5-nm FeMn layers sample whose hysteresis curve is shown in Fig. 1. The line is a guide to the eye.

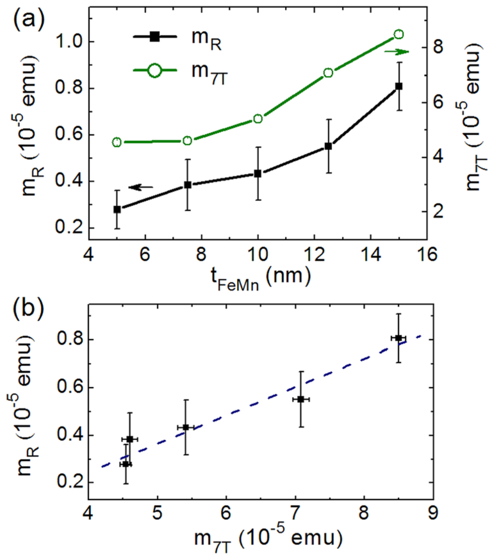

FIG. 3. (a) Magnetic moment of the samples at H = 7 T, m_{7T}, and the remanent magnetic moment, m_R, as a function of FeMn thickness; the line is a guide to the eye. The error bars for m_{7T} are smaller than the circles used for the data points. (b) Correlation between the two magnetic moments in (a); the dashed blue line is a linear fit.

volume of FeMn and not just from its surface. However, it is not clear what the source of the "excess" magnetic moment is. We also estimate the remanent magnetic moment, m_R, as the magnetic moment at H_E (and it is very close to the half-sum of the positive and negative magnetic moments at zero field which are not equal due to the loop shift from the exchange bias). While m_{7T} is the quantity that indicates the sum of the antiferromagnetic and ferromagnetic-like contribution to the total paramagnetic susceptibility, m_R is essentially a measure of the "ferromagnetic" component of the magnetic moment.[16] As it can be seen in Fig. 3(a), for the most part, m_R scales with the thickness of the FeMn. Moreover, m_R is proportional to the magnetic moment at 7 T as indicated in Fig. 3(b). This scaling indicates that the magnetization that is responsible for the hysteresis is distributed uniformly throughout FeMn.

We have eliminated contact with all metals during fabrication and measurement. In all steps of the substrate and sample handling, non-metallic, plastic, and ceramic tweezers are used in order to avoid transfer of magnetic contaminants to the sample.[17,18] During deposition, the samples are mounted onto a cleaned glass microscope slide and held in place with Kapton tape. With these precautions, we successfully avoid measurable magnetic contamination.

The observation of the exchange bias suggests that the entire magnetic moment has two components: pinned magnetic moment and unpinned (rotatable). The latter is responsible for the hysteresis, while the former provides the exchange bias. The thickness dependence of the coercive field does not demonstrate any interesting trend; further studies are needed to understand it. The dependence of the exchange bias field, H_E, on the thickness of FeMn layer is shown in Fig. 4 inset. This dependence can be described as the inverse proportionality between H_E and the thickness of FeMn (Fig. 4). The model of exchange bias for AF-FM bilayer by Malozemoff[19,20] predicts that H_E is inversely proportional to the thickness of the FM layer (i.e., rotatable

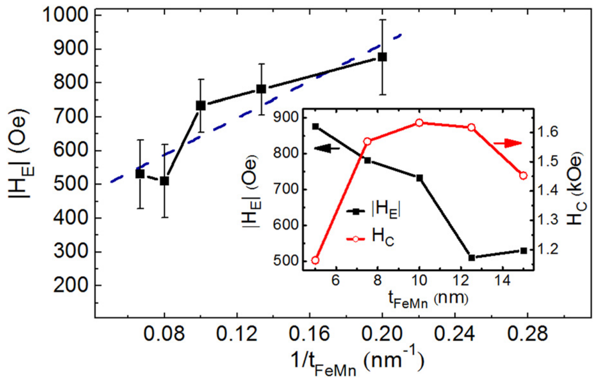

FIG. 4. H_E vs. inverse thickness, 1/t, of FeMn for the same samples as in the inset. The dashed blue line is a linear fit. Inset: Dependence of the coercive field, H_C, and absolute value of the exchange bias field, H_E, on the thickness of FeMn for FeMn/Cu multilayers. The line is a guide to the eye.

moments). Hence, our observation is consistent with the fact that the unpinned magnetization is uniformly distributed in the thickness of FeMn. Absence of measurable exchange bias without Cu layer suggests that the interface between FeMn and Cu is responsible for the pinned moments.

What could be the source of this magnetization? One possibility is that Cu diffuses into FeMn, which can result in the formation of CuMn and Fe. Both can provide a non-zero net magnetic moment which can also be exchange-coupled to the rest of FeMn system so that this coupling provides the pinning of this magnetization. To provide the amount of the total ferromagnetic-like magnetic moment present in our samples (of order of 10^{-5} emu), one needs equivalent of Fe from only seven to eight atomic layers of FeMn, which for 19 FeMn-Cu interfaces is about 40% of irons present at each interface. Additionally, CuMn is known to be a spin-glass material, which can provide the pinned magnetization as well. The spin-glass scenario is consistent with the observed training effect and excess noise in the hysteresis loops. Element-sensitive studies, such as x-ray magnetic dichroism (XMCD), are needed for verifying this hypothesis. While we suggest that this process may occur at the interface, it is possible that Cu diffuses into the grain boundaries of FeMn as it was observed for Cu diffusing in other similar materials.[21] Yet another possibility is that the strain that originates from the different lattice constants of Cu and FeMn creates some interfacial net magnetization in FeMn that is pinned due to its coupling to the AF-ordered spins in FeMn.

In either case, it is clear that Cu, when in contact with FeMn, affects the magnetic properties of FeMn and will likely affect the exchange bias in Cu/FeMn/FM FeMn/Cu/FM systems. This suggests that using Cu as a nonmagnetic spacer may have effect on exchange bias that is different than expected from just a nonmagnetic spacer.[22]

It is remarkable how similar the thickness and temperature dependences of H_E and H_C (Figs. 2 and 4) are to those measured for Cu/FeMn/FM systems (Figs. 4, 6, 7(a), and 7(c) in Ref. 6) The similarity suggests that these dependences might be primarily an intrinsic property of Cu/FeMn structures and might not be affected much by the presence of a FM. Moreover, the intrinsic exchange bias[23] (i.e., observed in an AF without a FM) might be determining the properties and the underlying mechanism of the exchange bias in AF-FM bilayers.[24] For instance, decrease in H_E with the FeMn grain size increasing observed by Bolon *et al.*[6] is consistent with the diffusion of Cu into the grain boundaries. The increasing grain size yields decrease in the amount of grain boundaries per FeMn volume, and, hence, smaller concentration of the pinned moment, leading to smaller H_E.

In summary, we observe magnetic hysteresis for FeMn samples. This hysteresis is especially strongly pronounced at low temperatures (10–50 K). When the samples are cooled in an external magnetic field, the hysteresis loop is shifted horizontally, exhibiting exchange bias. The value of the shift is rather large—close to 1.8 kOe at 10 K for 5 nm-thick FeMn layers. The value of the shift scales inversely with the thickness of FeMn layer, which fits Malozemoff's model, if we assume that the unpinned moments are uniformly distributed inside FeMn. Based on comparison of the properties of the Cu/FeMn superlattices with those of Cu/FeMn/FM, we propose that properties and the underlying mechanism of the exchange bias in AF-FM systems may be determined by the properties of the intrinsic exchange bias, i.e., exchange bias observed in the system without a FM.

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