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Exchange bias of mu-metal thin films

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ABSTRACT

The exchange bias of the soft ferromagnet mu-metal, $Ni_{77}Fe_{14}Cu_5Mo_4$, with the metallic antiferromagnet $Fe_{50}Mn_{50}$ has been studied as a function of ferromagnet thickness and buffer layer material. Mu-metal exhibits classic exchange bias behavior: the exchange bias (H_{EB}) and coercive fields scale inversely with the ferromagnet's thickness, with H_{EB} varying as the cosine of the in-plane applied field angle. Ta buffers, rather than Cu, allow the mu-metal to retain more of its soft magnetic character while exhibiting exchange bias. The ability to preserve soft ferromagnetic behavior in an exchange biased heterostructure may be useful for low field sensing and other device applications.

1. Introduction

Exchange bias is a phenomenon related to the interfacial exchange interaction between two ordered magnetic materials [1,2]. Observed primarily in structures composed of ferromagnet/antiferromagnet (FM/AF) interfaces (e.g., thin film heterostructures and nanoparticles), exchange bias manifests itself as a unidirectional magnetic anisotropy that shifts the hysteresis loop along the field axis by some amount known as the exchange bias, H_{EB}. The ferromagnet has a unique magnetization at zero field when H_{EB} exceeds the saturation field, which allows simple FM/AF bilayers to serve as a magnetic reference for spintronics devices [3]. In fact, there are many potential applications of exchange bias because H_{EB} is a function of many experimentally controllable parameters, including, but not limited to: ferromagnet and antiferromagnet thickness; temperature; interfacial structure and roughness; and grain size [4].

In this work, we focus on inducing exchange bias in $Ni_{77}Fe_{14}Cu_5Mo_4$, which is sometimes referred to as mu-metal or conetic. A member of the Permalloy family, this material has large permeability and saturation magnetization, and offers nearly zero magnetostriction and nearly zero magnetocrystalline anisotropy [5,6]. Introducing a unidirectional anisotropy via exchange bias in soft magnetic materials could be a useful for introducing additional control over phenomena and sensors such as giant magneto-impedance (GMI) [7]. Bulk mu-metal has been shown to have a large GMI ratio (300%) and a correspondingly high sensitivity (20%/Oe) [8,9], but its exchange bias properties have not been reported. Another potential area for impact is exchange spring system that combines material with perpendicular magnetic anisotropy with soft ferromagnet layers with in plane anisotropy. This leads to structures whose magnetization has an out of plane tilt angle that is tunable by the thickness of the soft ferromagnet [10]. Such structures are being explored for spin transfer torque devices [11].

2. Materials and methods

We investigated how the magnetic properties of several sets of $Ni_{77}Fe_{14}Cu_5Mo_4/Fe_{50}Mn_{50}$ (NiFeCuMo/FeMn) depend on NiFeCuMo thickness and substrate/buffer layer materials. We used Ta and Cu as buffer layers, with both grown on a 140 nm thick thermal oxide on Si (100) wafers (SiO*x*/Ta and SiO*x*/Cu). Some Cu-buffered samples were simultaneously grown on the native oxide of Si (100) wafers (Si/Cu); there was no significant difference in magnetic properties between these two substrate options, so we focus this report on samples grown onto the thermally oxidized Si. The Ta-buffered set had the structure Ta(50 Å)/NiFeCuMo(60–400 Å)/FeMn(150 Å), and were uncapped. The Cu-buffered samples had the structure Cu(300 or 800 Å)/NiFeCuMo (90–300 Å)/FeMn(150 Å)/Ta(50 Å). The FeMn thickness of 150 Å was chosen so that the Blocking temperature of $\sim$400 K was independent of the antiferromagnet's thickness [4]. The substrates were ultrasonically cleaned in acetone and methanol for 5 min each, blown dry with nitrogen gas, then inserting into the load lock. The samples were grown at ambient temperature in 3 mTorr of ultra high purity Ar in a magnetron sputtering system with a base pressure of 20 nTorr. The compositions noted were those of the sputtering targets. All targets were presputtered for 10 min prior to deposition. The sample holder was continually rotated during deposition, and the gun angle has been optimized to obtain

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deposition rates with variations less than 0.4% over the entire 75 mm substrate holding plate.

3. Results and discussion

X-ray diffraction results are shown in Fig. 1 for each sample type. The (111) orientation is specifically of interest because it is known to yield the largest exchange bias when using FeMn as the antiferromagnet [12]. Each sample shows shifted (111) peaks relative to the bulk (43.2°, 43.3°, and 44.2° for FeMn, Cu, and NiFeCuMo, respectively, for Cu k_α radiation). Of the materials used, the NiFeCuMo is the least strained along the growth direction, relative to the bulk. For SiO*x*/Ta(50 Å)/NiFeCuMo (400 Å)/FeMn(150 Å), the NiFeCuMo and FeMn peaks overlap, as observed via sequential XRD after deposition of the individual layers (such samples were used only for structural investigation to assist indexing of the peaks). The SiO*x*/Cu(800 Å)/ NiFeCu Mo(400 Å)/FeMn(150 Å) show distinct peaks for the Cu and NiFe CuMo/FeMn. The lattice mismatch at the Cu–NiFeCuMo interface may be related to the reduced level of (111) texturing in these samples. The 300 Å Cu-buffered samples have the weakest (111) texturing of all samples studied, indicating that this thickness buffer led to relatively low structural quality, as depicted in the inset of Fig. 1.

The (111) texture in the Ta-buffered samples is more coherent in the growth direction than that of the 800 Å Cu-buffered samples, as indicated by the relative intensities of the peak near 44° (Fig. 1). Although the Ta seems to be the more promising buffer from this standpoint, a simple Scherrer analysis of the (111) peak indicates that the coherence length is only about 80 Å. Thus, there may be further room for improvement of the structure. While annealing or higher temperature deposition may improve the structure, including possibly reducing the required Cu buffer thickness, this may come at the price of interdiffusion and subsequent loss of mu-metal's valuable soft magnetism. Note that NiFeCuMo films may be susceptible to deposition-induced structural perturbations: we find it necessary to rotate the samples during growth in order to obtain reproducible magnetic properties; growing with the sputtering flux at a fixed angle relative to a stationary substrate leads to unexpected (and difficult to control) magnetocrystalline anisotropy. It is possible that this structural sensitivity is playing a significant role in response to the differences in strain induced by the amorphous Ta and polycrystalline Cu buffers.

A custom substrate plate was used to deliver an in-plane field ~250 Oe in a local region of the plate, while the opposite side of the plate has a field below the detection level of a calibrated Lakeshore 421 Gaussmeter. This allows two control samples of Cu(300 Å)/NiFeCuMo(200 Å)/Cu(300 Å) to be produced simultaneously, one with and one without an applied growth field [13]. X-ray reflectivity was used to confirm that the deposition rate was independent of the magnetic field used during deposition. As shown in Fig. 2(a), the sample deposited in zero field shows quite isotropic magnetic behavior, with no significant difference in hysteresis loop shape for the magnetization measured along

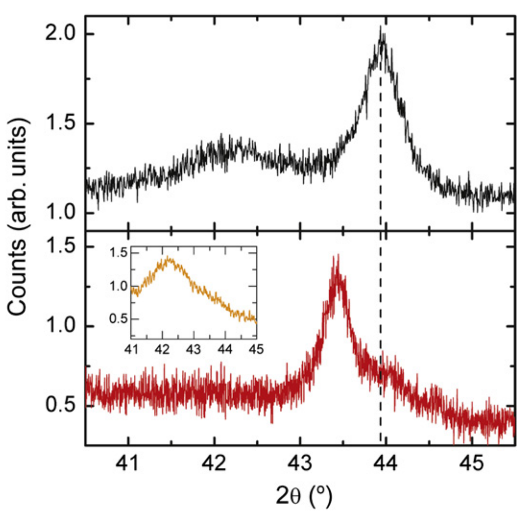

Fig. 1. X-ray diffraction results show that the Ta buffer (top) leads to more coherent (111) NiFeCuMo/FeMn texture with less strain than Cu buffers (bottom). The specific structures were SiO*x*/Ta(50 Å)/NiFeCuMo(400 Å)/FeMn(150 Å) and SiO*x*/Cu(800 Å)/NiFeCuMo(400 Å)/FeMn(150 Å). The NiFeCuMo and FeMn (111) diffraction peaks overlap near 44°; the Cu (111) peak is near 43.4°; the peak near 42° is related to the Ta. The inset shows that Cu(300 Å)/NiFeCuMo(300 Å)/ FeMn(150 Å) is insufficient to generate well defined (111) structure.

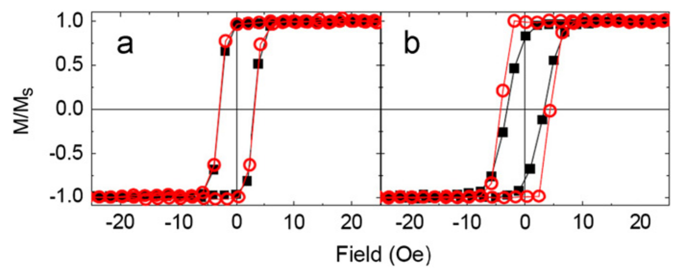

Fig. 2. Hysteresis loops of Cu(300 Å)/NiFeCuMo(200 Å)/Cu(300 Å) samples grown simultaneously in (a) zero field, and (b) 250 Oe, as measured by VSM. The measurement field was applied parallel (red, open symbols) and perpendicular (black, solid symbols) to the deposition field direction. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)

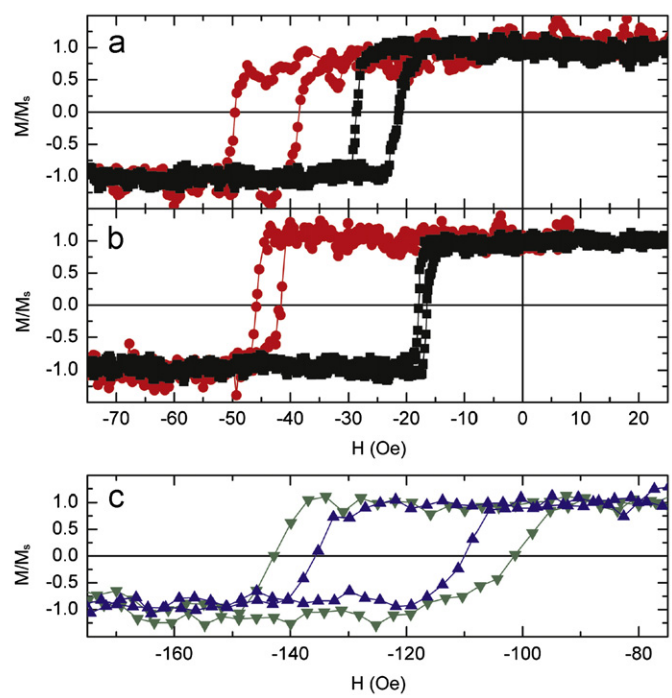

Fig. 3. MH loops measured along the easy axes of (a) NiFeCuMo(200 Å)/ FeMn(150 Å) and (b) NiFeCuMo(400 Å)/FeMn (150 Å), grown on substrate/buffer pairs of Si/Ta(50 Å) (black squares) and Si/Cu(800 Å) (red circles). (c) The low structural quality realized in Cu(300 Å)/NiFeCuMo(200 Å)/FeMn(150 Å) grown either on Si (blue up triangles) or SiO*x* (green down triangles) leads to significantly larger coercivity and exchange bias. Note that the magnitude of the field range is the same for all three panels. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)

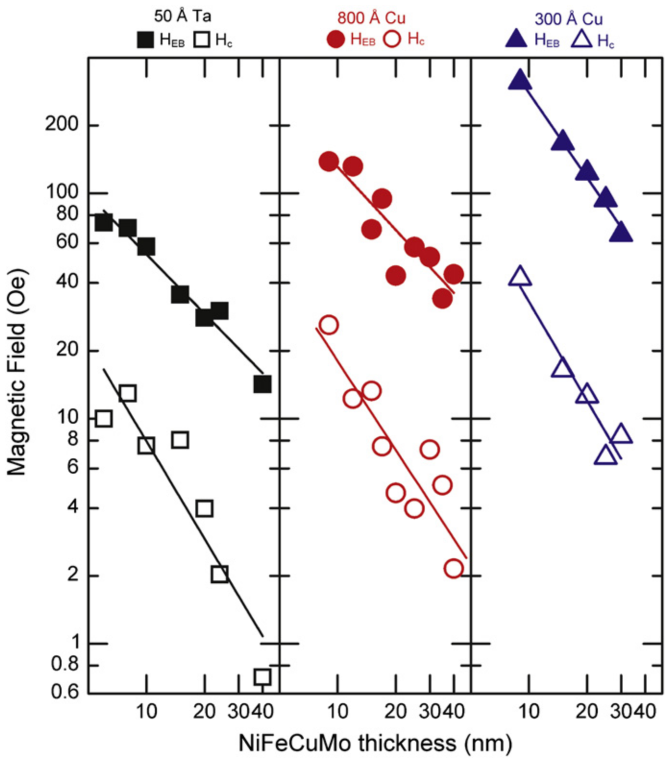

Fig. 4. Exchange bias and coercive fields are inversely proportional to the ferromagnet thickness; thin lines are linear fits.

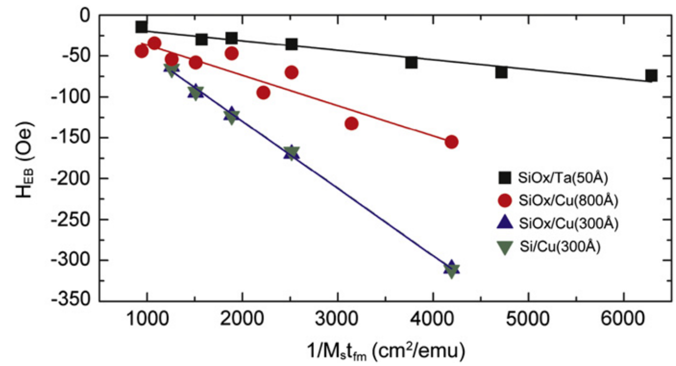

Fig. 5. The interfacial exchange energy per unit area J_{int} is the slope of the linear fits of the H_{EB} vs $1/M_s t_{FM}$, as described in the text.

two orthogonal directions; no measurable difference in M–H behavior was observed for any in-plane angle. In contrast, the field-grown sample has developed a uniaxial magnetic anisotropy, with the easy axis corresponding to the direction of the deposition field. The coercivity is slightly enhanced along the easy axis, while the hard axis coercivity is not measurably changed relative to the sample deposited in zero field. The saturation fields are in line with previous results on NiFeCuMo thin films [5,6]. These samples have no antiferromagnetic layer, and accordingly exhibit no exchange bias.

Relative to the control samples, a clear exchange bias develops when FeMn is deposited in a field onto the NiFeCuMo. Fig. 3(a) and (b) show room temperature hysteresis loops measured along the easy axes for 200 Å and 400 Å thick NiFeCuMo exchange biased with 150 Å FeMn. The H_{EB} found for 800 Å Cu-buffered samples is about double that of the Ta-buffered samples for each thickness. As for coercivity, the Ta and 800 Å Cu-buffered samples are comparable, but with the former having smaller H_c. In contrast, Fig. 3(c) shows that the 300 Å Cu-buffered samples have the most significant H_{EB} and H_c; these relatively anomalous values are likely due to the ill-defined structure shown in the inset of Fig. 1, which may result in more uncompensated moments per unit area at the NiFeCuMo–FeMn interface. The 300 Å Cu-buffered samples also have strongly enhanced switching field distributions, which indicate that the soft magnetic properties of the mu-metal have been essentially lost.

For a more global view, Fig. 4 shows the thickness dependence of H_{EB} and H_C for each sample set grown on thermally oxidized silicon. The dependence of the exchange bias on in plane field angle had $H_{EB}\cos\theta$ behavior for all measured samples.[1] Both H_{EB} and H_c are inversely proportional to the thickness, which is expected because exchange bias is an interface effect. While we forego a quantitative analysis here, it is apparent from the XRD and magnetic data that the exchange bias and coercivity are inversely related to structural quality. This is quite likely due to poor structure leading to greater uncompensated moments per unit area. Accordingly, the Ta/NiFeCuMo(400 Å)/FeMn sample, whose XRD suggests it has the most significant (111) texturing, shows that the soft magnetic properties of the mu-metal can be retained in exchanged biased structures: H_{EB}=14.1 Oe, H_c=0.7 Oe, and a saturation field on the order of 1 Oe.

Using values of H_{EB} measured along the easy direction for each sample, we can determine the interfacial energy per unit area according to $J_{int} = M_s t_{FM} H_{EB}$, where M_s and t_{FM} are the saturation magnetization and thickness of the NiFeCuMo, respectively. M_s was measured by vibrating sample magnetometry to be 265 emu/cm^3, independent of the thicknesses studied. Fig. 5 shows that linear fits of the exchange bias as a function of $1/M_s t_{FM}$ yield $J_{int} = -11.7 \pm 1.3$ merg/cm^2 for SiO*x*/Ta, and -37.1 ± 5.1 merg/cm^2 for SiO*x*/Cu(800 Å). To illustrate the previous claim that the SiO*x*/Cu(300 Å) and Si/Cu(300 Å) buffers lead to essentially the same magnetic behavior, we found J_{int} to be -82.2 ± 2.1 merg/cm^2 for the former, and -82.3 ± 2.0 merg/cm^2 for the latter. Despite the seemingly large spread, each of the J_{int} values are all in agreement with previous energy densities using FeMn (111) as the antiferromagnet [1]. This underscores the importance of structure in determining the ultimate magnetic properties of NiFeCuMo/FeMn heterostructures.

4. Conclusions

Together, these results show that mu-metal exhibits classic exchange bias behavior when grown in contact with FeMn. The differences in magnetic properties between the $Cu/Ni_{77}Fe_{14}Cu_5Mo_4/Fe_{50}Mn_{50}/Ta$ and $Ta/Ni_{77}Fe_{14}Cu_5Mo_4/Fe_{50}Mn_{50}$ samples are significant in respect to their applicability in low field sensing applications. The origin of the differences appears structural in nature. Although both Cu and Ta lend themselves to (111) texturing of the NiFeCuMo and FeMn, samples with Ta buffers preserved the soft magnetic properties of the mu-metal most effectively. 300 Å Cu buffer layers did not have as high quality (111) texturing as the other samples, which led to significantly enhanced exchange bias and coercivity, along with a broadened switching field distribution. One notable result here is the ability to preserve the soft features of the mu-metal while inducing the unidirectional anisotropy using Ta as a buffer layer. From a practical spintronics point, Ta may also be the most beneficial substrate because the relatively thin layer will lead to greater

[1] The uncapped Ta-buffered samples appear to have corroded over the course of two years with intermittent exposure to air, eliminating our ability to study their angular dependence; other measurements were performed within days of fabrication.

current density in the magnetic layers. This may thus impact devices and structures employing soft magnetic materials, such as giant magnetoimpedance and related sensors, and possibly exchange springs with tunable magnetization tilt angles.

Acknowledgments

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