Weak magnetic moment on IrMn exchange bias pinning layers

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We present evidence from soft x-ray resonant magnetic scattering measurements at the Mn L_3 edge for the existence of a small magnetic moment on the antiferromagnetic IrMn pinning layer in a NiFe/Cu/Co/IrMn spin valve structure. The variation of the signal in an applied magnetic field shows that the moment lies antiparallel to the Co moment. Changes in the Mn L_3 edge signal as the Co moment is rotated into the hard direction are rapid and do not appear to be associated with thermal reordering of the antiferromagnetic domain structure. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1392304]

The shift in the hysteresis loop referred to as exchange biasing occurs when a unidirectional exchange anisotropy is created on cooling adjacent antiferromagnetic (AFM) and ferromagnetic (FM) films below the Néel temperature of the antiferromagnetic layer.[1] Despite its technical importance, the fundamental mechanism of exchange pinning in spin valve structures remains a major outstanding problem in thin film magnetism. A number of theories have been advanced to explain the phenomenon of exchange biasing, all based on forms of interface coupling. Most models assume that the AFM and FM anisotropy axes are collinear and that the AFM spins at the interface are uncompensated.[2,3] The simple models give rise to a bias field that is too high. Malozemoff[4] proposed a model with random exchange interactions that resulted in a lower bias field and Mauri *et al.*[5] suggested that formation of antiferromagnetic domains parallel to the interface would also reduce the bias field. More recently a micromagnetic calculation by Koon[6] suggested that the coupling between AFM and FM spins across a frustrated, but compensated, interface is perpendicular. There exists a canting of the AFM spins close to the interface that decays away within a few monolayers of the interface. For an antiferromagnetic exchange interaction, the canting is such as to create a small net moment on the AFM layer in the opposite sense to the FM magnetization. Kiwi *et al.*[7] have postulated a canted AFM interface structure coupled with an incomplete domain wall in the FM layer.

In recent experiments evidence in support of both parallel and perpendicular spin alignment was found. Takano *et al.*[3] found evidence of uncompensated spins in CoO/MgO and CoO/$Ni_{81}Fe_{19}$ and support for Malozemoff's model. Neutron scattering experiments of Ijiri *et al.*[8] on Fe_3O_4/CoO superlattices and magnetic measurements of Nogués *et al.*[9] on Fe/FeF_2 can be interpreted in terms of Koon's spin-canting model. On the other hand, direct observation by polarization-dependent x-ray magnetic dichroism spectromicroscopy of Co on $LaFeO_3$, showed that grain by grain the alignment of FM spins was parallel to the underlying AFM spin orientation.[10]

Like polarized neutron scattering, x-ray resonant magnetic scattering may be used to probe the magnetization of thin films. Although the x-ray technique cannot as yet determine the absolute value of the magnetization, it is element specific and Freeland *et al.*[11] have shown how this feature can be exploited to identify the order of layer switching in a NiFe/Cu/Co spin valve. Switching of the helicity of the incident x-ray beam results in a change in the intensity if a component of ferromagnetic moment lies in the incidence plane, the difference being a signal proportional to the magnitude of this magnetization component. For a perfectly compensated antiferromagnetic lattice, the difference signal will always be zero. However, we note that in experiments on $Fe_{25}Co_{75}$/Mn/$Fe_{25}Co_{75}$ trilayers, Chakarian *et al.*[12] did detect a net moment on the Mn layer, oriented 23° to the Co and Fe moments. Further, a weak moment was detected by resonant magnetic scattering on thin epitaxial $Fe_{75}Mn_{25}$ films grown on Ir(001) substrates.[13] However, in more recent experiments using magnetic circular dichroism on Co/FeMn bilayers it was found that the Mn spins are almost perfectly compensated for with no more than a few percent of one monolayer of residual ferromagnetic spins.[14] We report here resonant magnetic x-ray scattering experiments on NiFe/Cu/Co/IrMn which provide evidence for the existence of a small ferromagnetic moment on the antiferromagnetic layer in this system.

Soft x-ray resonant magnetic scattering experiments were performed at room temperature and in fields up to 70 mT on the Dragon beamline ID12B at the European Synchrotron Radiation Facility in Grenoble. The Helios-type helical undulator delivers two separate beams of x rays, each being between 80% and 90% circularly polarized, but in opposite senses. By displacement of a pinhole under the control of the station computer, the helicity of the x rays incident on the sample can be switched in a matter of seconds. Provided that a few minutes is allowed to elapse, enabling the thermal load to stabilize on the spherical grating monochromator, the normalized magnetization component in the incidence plane can be extracted reproducibly from the difference in the intensity for the two helicities. Measurements of the specular

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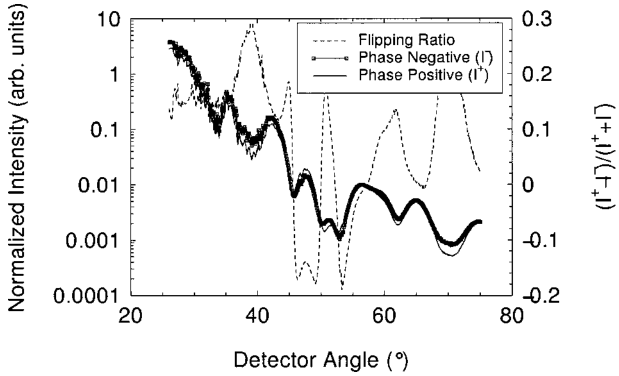

FIG. 1. Specular scatter for the sample with the thinner AFM layer, recorded at the Co L_3 edge, for positive and negative helicity.

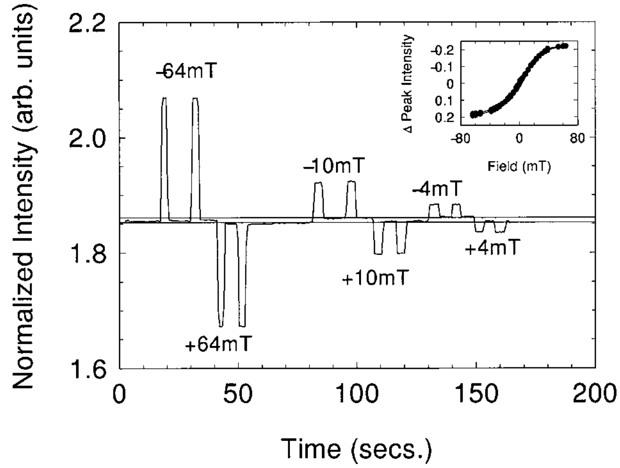

FIG. 3. Intensity at the Co L_3 edge (positive helicity) with the sample set on the specular ridge for the sample with the thick AFM layer. The field pulses applied are indicated. Inset: $M-H$ loop of the cobalt layer extracted from a similar data set with continually varying field applied along a direction close to the hard axis.

and diffuse scatter have been made at and away from the Co L_3 and Mn L_3 edges.

The samples were grown by magnetron sputtering on silicon (001) substrates and had nominal composition of Si/Ta (30 Å)$Ni_{80}Fe_{20}$ (40 Å)/Cu (30 Å)/Co (30 Å)/$Ir_{25}Mn_{75}$ (d Å)/Ta (15 Å) with $d=75$ and 270 Å. Vibrating magnetometer measurements on the samples showed classic spin-valve behavior with an exchange field of 40 and 50 (± 5) mT, respectively, for the two samples.

Although the penetration depth of these soft x rays is limited, we have shown previously[15] that it is possible to probe 1000 Å thick Cu/Co multilayers. Figure 1 shows the reflectivity from the sample with the thinner IrMn pinning layer recorded at the Co L_3 edge for both helicities. At high scattering angles the shape of the reflectivity curve is dominated by fringes originating from the tantalum buffer layer, showing a penetration depth greater than the total sample thickness. The net ferromagnetic moment of the cobalt layer is clear from the difference in intensity between the reflectivity curves recorded using the two opposite helicities.

At the Mn L_3 edge no obvious difference in the two full specular reflectivity curves can be detected upon reversal of the helicity. However, careful measurement across the specular ridge, made by scanning only the sample (Fig. 2), does reveal a small, reproducible, change in intensity ($\Delta I \sim 0.04$) upon reversal of the helicity that is substantially weaker, and only just above the noise level ($\Delta I \sim 0.001$) when the monochromator energy is tuned 20 eV below the Mn L_3 edge. The signal falls upon moving away from the

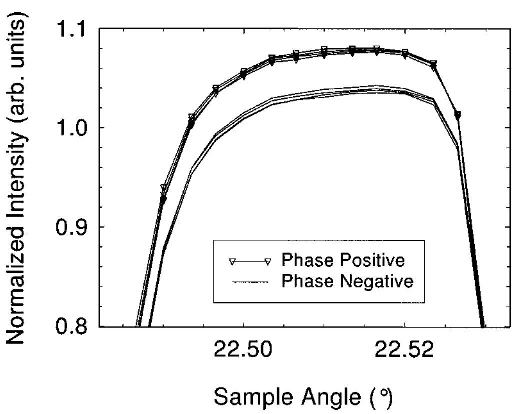

FIG. 2. Transverse scans across the specular ridge at the Mn L_3 edge. Four repeated scans are shown for each helicity.

edge to either higher or lower energies. As in previous soft x-ray resonant magnetic scattering experiments, this provides unequivocal evidence that a small net moment exists on the antiferromagnetic layer.

Measurements as a function of field were taken at a specific scattering vector, chosen such that the change in signal upon reversal of the helicity was large. By selecting a specific wave vector transfer the flipping ratio may be used to plot the magnetization curve as a function of the field. In this case the sample was oriented with the easy direction of magnetization, determined by the growth field direction, perpendicular to the incidence plane.

With positive helicity and with the sample set on the specular ridge at a wavevector transfer of 0.16 $Å^{-1}$, the application of a +64 mT field resulted in a 9.9% decrease in the signal intensity at the Co edge. Application of a −64 mT field resulted in an 11% increase at the Co edge, indicating that the easy direction was not oriented exactly perpendicular to the scattering plane (Fig. 3). This small net component of the magnetization in the scattering plane accounts for the two different "zero states" that are shown in Fig. 3. The curve corresponds to a hard axis magnetization loop of the Co layer and is in good agreement with the equivalent magnetization curve measured in a vibrating sample magnetometer when the component of magnetization associated with the very low field switching of the $Ni_{80}Fe_{20}$ layer is subtracted.

Upon application of the +64 mT field, no change in intensity was observed at the Mn edge, but in the −64 mT field, a 1.5% reduction was observed, revealing a change in the moment on the IrMn layer in the opposite sense to that in the Co layer. The reduction occurred each time that the −64 mT field was applied and the size of the signal change was independent of the field. Reversal of the phase and the direction of the trickle current in the electromagnet gave a decrease, after significant irreproducible "training,"[16] in the Mn edge signal in a +64 mT field as the in-plane AF domains reorder. In all cases, changes in the Mn edge signal were in the opposite sense to those at the Co edge (Fig. 4).

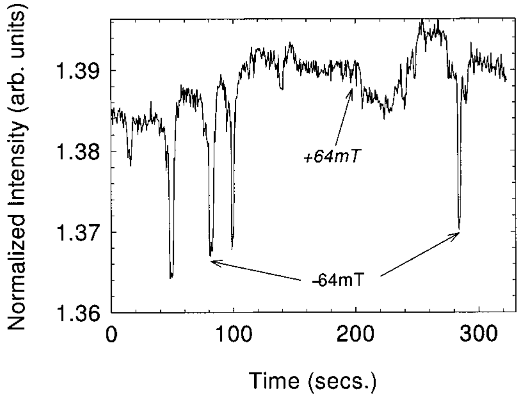

FIG. 4. Intensity from the same sample as in Fig. 3 recorded at the Mn L_3 edge under identical experimental conditions.

The observation of a net AFM moment oriented oppositely to the moment in the FM Co layer is in agreement with unpublished x-ray resonant scattering experiments at the Ni L_3 edge by Kao.[17] Measurement of the scattered intensity of circularly polarized radiation as a function of the field in the easy direction of a spin valve showed a small net moment on the NiO exchange pinning layer and a reversed, square loop associated with it. An induced moment on the AFM layer was observed in Fe–FeF_2 and Fe–MnF_2 bilayers by Nogués *et al.*[18] using high resolution superconducting quantum interference device (SQUID) magnetometry, the sense of the coupling depending on the microstructure and on the field in which the sample was cooled through the Néel temperature.

We can attempt to interpret the presence of the net magnetic moment on the AFM layer, either in terms of an uncompensated layer of spins at the interface[3] or a net moment due to canting of the AFM spins close to the interface.[4,7] Despite not being able to quantify the size of the moment due to lack of knowledge of the resonant magnetic cross section, the signal magnitude is not inconsistent with a single layer of spins. In the former case, we note that a small net moment arises only when there is an odd number of spins through the AFM layer. Roughness that is fully correlated through the AFM layer[19] does not affect the size of the moment, but uncorrelated roughness will average the effect to zero. Variation of the AFM layer thickness by more than one AFM unit cell will also average the net moment to zero. Roughness at the interface is of this order, and it is only in the case of totally correlated roughness that the effect can be observed. It is thus difficult to understand the origin of the observed moment in terms of an uncompensated surface layer. However, magnetometric and ferromagnetic resonance studies of exchange bias in $Ir_{20}Mn_{80}$ films suggested that a spin-flop model did not explain the data.[20] Our measurements as a function of the field applied in the hard direction indicate asymmetry in the rotation of such an interfacial moment. The moment changes seen in Figs. 3 and 4 occur over similar, rapid time scales. In both cases the changes in intensity occurred within the time resolution of the experiment (0.5 s). Unlike our previously studied Co/Cu multilayers, no magnetic viscosity effects were observed.[21] These changes cannot therefore be associated with the large scale AF domain reorientation that is postulated as being the origin of the time-dependent effects on the coercivity reported by Laidler and co-workers.[22] Nevertheless, the field dependence cannot be explained by a uniform rotation of the Mn moment. The present results provide further evidence for a net moment on the AFM layer but unfortunately do not provide a definitive test of the competing models.

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[1] J. Nogués and I. K. Schuller, J. Magn. Magn. Mater. **192**, 203 (1999).
[2] W. P. Meiklejohn and C. P. Bean, Phys. Rev. **105**, 904 (1957).
[3] K. Takano, R. H. Kodama, A. E. Berkowitz, W. Cao, and G. Thomas, Phys. Rev. Lett. **79**, 1130 (1997).
[4] A. P. Malozemoff, Phys. Rev. B **37**, 7673 (1988).
[5] D. Mauri, H. C. Siegmann, P. S. Bagus, and E. Kay, J. Appl. Phys. **62**, 3047 (1987).
[6] N. Koon, Phys. Rev. Lett. **78**, 4865 (1997).
[7] M. Kiwi, J. Majía-Lopéz, R. D. Portugal, and R. Ramírez, Appl. Phys. Lett. **75**, 3995 (1999).
[8] Y. Iriji, A. Borchers, R. W. Erwin, S. H. Lee, P. J. van der Zaag, and R. M. Wolf, Phys. Rev. Lett. **80**, 608 (1998).
[9] J. Nogués, D. Lederman, T. J. Moran, and I. K. Schuller, Phys. Rev. Lett. **76**, 4624 (1996).
[10] F. Notting, A. Scholl, J. Stöhr, J. W. Seo, J. Fompeyrine, H. Slegwart, J.-P. Locquet, S. Anders, J. Lüning, E. E. Fullerton, M. F. Toney, M. R. Scheifein, and H. A. Padmore, Nature (London) **405**, 767 (2000).
[11] J. W. Freeland, V. Chakarian, Y. U. Idzerda, S. Doherty, J. G. Zu, J.-H. Park, and C.-C. Kao, Appl. Phys. Lett. **71**, 276 (1997).
[12] V. Chakarian, Y. U. Idzerda, H.-J. Lin, C. Gutierrez, G. A. Prinz, G. Meigs, and C. T. Chen, Phys. Rev. B **53**, 11313 (1996).
[13] J. M. Tonnerre, L. Sève, A. Barbara-Dechelette, F. Bartolomé, D. Raoux, V. Chakarian, C.-C. Kao, H. Fischer, S. Andrieu, and O. Fruchart, J. Appl. Phys. **83**, 6293 (1998).
[14] W. J. Antel, Jr., F. Pejeru, and G. R. Harp, Phys. Rev. Lett. **83**, 1439 (1999).
[15] T. P. A. Hase, I. Pape, D. E. Read, B. K. Tanenr, H. Dürr, E. Dudzik, G. van der Laan, C. H. Marrows, and B. J. Hickey, Phys. Rev. B **61**, 15331 (2000).
[16] C. Schlenker and D. Paccard, J. Phys. (France) **28**, 611 (1967).
[17] C.-C. Kao, Polarisation Workshop, Daresbury Laboratory, UK, October 2000 (unpublished).
[18] J. Nogués, C. Leighton, and I. K. Schuller, Phys. Rev. B **61**, 1315 (2000).
[19] J. Clarke, C. H. Marrows, F. E. Stanley, R. J. T. Bunyan, B. K. Tanner, and B. J. Hickey, J. Phys. D **32**, 1169 (1999).
[20] S.-F. Cheung and P. Lubitz, J. Appl. Phys. **87**, 4927 (2000).
[21] T. P. A. Hase, B. D. Fulthorpe, S. B. Wilkins, B. K. Tanner, C. H. Marrows, and B. J. Hickey, J. Magn. Magn. Mater. **226–230**, 1748 (2001).
[22] X. Portier, A. K. Petford-Long, A. de Morais, N. W. Owen, H. Laidler, and K. O'Grady, J. Appl. Phys. **87**, 6412 (2000).