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Publication Date

2006

Peer reviewed

Magnetization reversal of uncompensated Fe moments in exchange biased Ni/FeF$_2$ bilayers

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Abstract

The magnetization reversal of uncompensated Fe moments in exchange biased Ni/FeF$_2$ bilayers was determined using soft x-ray magnetic circular and linear dichroism. The hysteresis loops resulting from the Fe moments are almost identical to those of the ferromagnetic Ni layer. However, a vertical loop shift indicates that some Fe moments are pinned in the antiferromagnetically ordered FeF$_2$. The pinned moments are oriented antiparallel to small cooling fields leading to negative exchange bias, but parallel to large cooling fields resulting in positive exchange bias. No indication for the formation of a parallel antiferromagnetic domain wall in the FeF$_2$ layer upon magnetization reversal in the Ni layer was found.

PACS: 75.50.Ee, 75.60.Jk, 75.70.Ak, 75.25.+z

When Meiklejohn and Bean observed that hysteresis loops of Co/CoO particles shift away from zero field to negative values upon field cooling below the CoO Néel temperature [1], they readily recognized the relevance of uncompensated moments in the antiferromagnetic CoO for the understanding of this effect [2]. However, their assumption of a Heisenberg-like exchange across a fully uncompensated ferromagnet (FM)/antiferromagnet (AFM) interface results in loops shifts, that is exchange bias fields, that are almost two orders of magnitude larger than experimentally observed. An obvious way to resolve this discrepancy is to assume that not an entire atomic layer (AL) but only a very small fraction of it contributes to the exchange bias. Recent experimental efforts have focused on quantifying the amount of uncompensated moments near the FM/AFM interface in exchange biased bilayers. For example, by measuring the moments of field cooled CoO/MgO multilayers Takano *et al.* [3] concluded that about 1% of the interfacial AFM moments are uncompensated which is consistent with measured permalloy/CoO exchange bias fields that amount to 1% of the value derived using Meikeljohn and Bean's model. Studying exchange biased Co/FeMn bilayers with magneto-optic Kerr effect and x-ray magnetic dichroism, Antel *et al.* [4] showed that Fe forms an uncompensated surface with a net ferromagnetic moment of almost 1 AL at the interface. About half of these moments follow the magnetization of the Co layer whereas the other half is strongly coupled or pinned to the AFM layer. Ohldag *et al.* [5] imaged with x-ray absorption spectromicroscopy uncompensated Ni spins in the ultrathin $NiCoO_x$ layer forming at the surface of NiO upon Co deposition. Exchange bias fields for these samples are negligibly small indicating that the existence of uncompensated interfacial moments alone is insufficient for exchange bias. When these moments couple more strongly to the FM than the AFM, they reverse with the FM in an external field and yield no bias. Using x-ray magnetic circular dichroism (XMCD) measuring the total electron yield, Ohldag *et al.* also showed that in Co/NiO, Co/IrMn, and CoFe/PtMn bilayers approximately 0.5 AL of uncompensated moments are present near the interface but that only 0.04 AL pinned by the antiferromagnet cause the observed exchange bias [6]. Similarly, magnetic force microscopy measurements performed on exchange biased CoPt/CoO multilayer by P. Kappenberger *et al.* [7] revealed that in this system about 0.07 AL of uncompensated spins are pinned by the AFM layer up to external fields as high as 7 T. Nogués *et al*. [8] inferred antiferromagnetic coupling between the ferromagnetic layer and pinned uncompensated spins in the AFM from their observation of negative and positive exchange bias in Fe/$FeFe_2$ bilayers after field cooling in small and large external fields, H_{cf}, respectively. Roy *et al*. [9] concluded from soft x ray scattering measurements on positively biased Co/FeF_2 bilayers an antiferromagnetic coupling between Co and uncompensated, unpinned Fe moments at the interface.

Using XMCD and soft x-ray magnetic linear dichroism (XMLD), we have studied in detail uncompensated Fe moments in positively and negatively exchange biased Ni/FeF_2 bilayers. The hysteresis loops resulting from these Fe moments are almost identical to those of the ferromagnetic Ni layer. A nominal coverage of about 0.01 AL of the uncompensated Fe moments are pinned by the antiferromagnetic FeF_2. Most importantly the pinned moments are aligned antiparallel to small cooling fields leading to negative exchange bias but parallel to the large cooling fields resulting in positive exchange bias. Magnetic linear dichroism measurement do not provide any indications for the formation of a parallel antiferromagnetic domain wall in the FeF_2 layer upon magnetization reversal in the ferromagnetic Ni layer.

The Ni/FeF_2 bilayers were prepared by electron beam deposition of 50 nm FeF_2 onto a MgF_2(110) single crystal surface heated to $T_S = 575$ K and subsequent growth of 3 nm Ni at $T_S = 425$ K. The FeF_2 grew epitaxially along (110) and the Ni was polycrystalline [10]. The bilayer was capped with 3 nm Al to prevent oxidation. The samples were exchange biased by saturating the Ni magnetization at $T = 150$ K in external fields of 0.55 T applied along the FeF_2 easy axis, that is the [001] direction, and then cooling the sample in an external field, H_{cf}, below the FeF_2 Néel temperature of 78 K. All spectra and hysteresis loops were obtained with the eight pole magnet at ALS beamline 4.0.2 [11] at sample temperatures of 55 K to eliminate the influence of sample charging effects on the electron yield measurements.

Figure 1(a) shows x-ray absorption spectra of the Fe and Ni $L_{3,2}$ edges measured at $T = 55$ K with elliptically polarized x-rays (S_3=0.98) at 30^o grazing incidence. An external field of 0.65 T along the x-ray beam was reversed for each photon energy resulting in parallel and antiparallel alignment of sample magnetization and photon spin. The difference of the two spectra, the XMCD spectrum shown in Figure 1(b), is proportional to the average atomic magnetic moment $\langle\mu\rangle$ in the Ni and FeF_2 layer. The strong XMCD signal at the Ni $L_{3,2}$ edges verifies its expected ferromagnetic order. For an ideal antiferromagnet the XMCD signal is zero. However, uncompensated Fe moments in the FeF_2 layer reversing their orientation upon field reversal lead to a pronounced XMCD signal at the Fe $L_{3,2}$ edges. It exhibits the same sign as the Ni XMCD signal indicating parallel alignment of Ni and uncompensated, unpinned Fe moments. Note that these unpinned Fe moments do not contribute to magnetic exchange bias. From the magnitude of the XMCD signal we estimate using the approach discussed by Ohldag *et al.* [5, 6] that nominally 0.8±0.15 nm of uncompensated Fe moments are present near the Ni/FeF_2 interface [12]. Our measurements do not allow us to distinguish between a continuous Fe layer near the interface and a homogeneous distribution of uncompensated Fe moments throughout the antiferromagnet or any intermediate configuration between these two limiting cases. However, exchange bias persists up to (78±2) K in the bilayer indicating that the Néel temperature of the FeF_2 layer is consistent with that of bulk FeF_2 [8].

Element-specific hysteresis loops of the ferromagnetic Ni layer and uncompensated Fe moments in the FeF_2 were obtained by monitoring the sample current as function of external field with the photon energy tuned to the Ni and Fe *L* edges and calculating asymmetry ratios between loops obtained with left and right elliptically polarized x-rays [6]. Figure 2(a) shows Ni loops acquired after field cooling the sample in external fields of $H_{cf} = 0.02$ T and $H_{cf} = 0.55$ T. Field cooling in small external fields ($H_{cf} = 0.02$T) leads to negative exchange bias whereas a large cooling field ($H_{cf} = 0.55$ T) leads to positive exchange bias of the same magnitude. Note that inverting the loop showing negative exchange bias leads to a hysteresis loop identical to the one showing positive bias, i.e. $M_{\mathrm{positive\ bias}}(\mathrm{H}) = -M_{\mathrm{negative\ bias}}(-H)$. Figure 2(b) shows hysteresis loops resulting from uncompensated Fe moments in the bilayer sample obtained under the same field cooling conditions as the Ni hysteresis loops shown in Figure 2(a). The Fe hysteresis loops are almost identical to those obtained at the Ni edge and the two loops again overlap after an inversion and translation. Interestingly, we observe a vertical shift of $\delta = (0.06\pm0.01)\%$ between the positively and negatively biased sample [see loop details in Figure 2(c)]. A vertical loop shift of both positively and negatively biased samples could be due to imperfect normalization of loops acquired with left and right circularly polarized radiation. However, in our experiments, a relative shift is observed between positively and negatively biased samples acquired under otherwise identical conditions.

To interpret this vertical loop shift we distinguish between uncompensated, unpinned Fe moments that follow the external field and the Ni magnetization and uncompensated, pinned Fe moments that are uneffected by external fields in the hysteresis loop measurement. Uncompensated, unpinned moments do not cause magnetic exchange bias as demonstrated by Ohldag *et al.* [5]. The contribution of uncompensated, pinned moments to the XMCD signal is independent of the external field, that is, it is observed as a vertical loop shift. That the loop for the positively biased sample is shifted to positive XMCD values by $\delta = +(0.06\pm0.01)\%$ indicates that for this bias direction – compared to negative bias – *more* uncompensated Fe moments are pinned *parallel* to the positive external field. This means also that these spins are oriented parallel to the cooling field, $H_{\mathrm{cf}} = 0.55$ T, when the FeF_2 spin structure is frozen in below T_{N}. Analogously, for negatively biased samples the loops are shifted to negative XMCD values – compared to positively biased samples –indicating that for this bias direction *more* uncompensated Fe moments are pinned *antiparallel* to the small positive cooling field, $H_{\mathrm{cf}} = 0.02$ T, when the spin structure is fixed at T_{N}. Hence, our measurements directly confirm the assumption by Nogués *et al.* [8] that uncompensated Fe moments in the FeF_2 layer aligned parallel to large cooling fields, pinned by the FeF_2 lattice below T_{N}, and antiferromagnetically coupled to the Ni moments are the cause of positive exchange bias.

For intermediate cooling fields, $0.02\ \mathrm{T} \le H_{\mathrm{cf}} \le 0.55$ T, the hysteresis loops consist of two subloops shifted oppositely from zero field by the same exchange bias field, and represent a superposition of the two limiting cases shown in Figure 2(a). The contribution showing positive exchange bias increases with increasing cooling field. This loop bifurcation is due to a coexistence of domains with uncompensated Fe moments pinned parallel and antiparallel to the cooling field [10]. From the occurrence of loop bifurcation and the fact that $M_{\text{positive bias}}(H) = -M_{\text{negative bias}}(-H)$ for the Ni loops, we conclude that the same amount of moments pinned parallel to the cooling field for $H_{\text{cf}} = 0.55$ T is pinned antiparallel to the cooling field in the case of $H_{\text{cf}} = 0.02$ T. This leads us to a nominal coverage about 0.01 AL of pinned uncompensated moments in the Ni/FeF_2 bilayer sample assuming an escape depth of 1.7 nm [12].

We employed XMLD to characterize the impact of external magnetic fields and the magnetization reversal in the Ni layer on the spin structure of the antiferromagnetically ordered FeF_2 in an experiment completely analogue to that preformed by Scholl *et al.* on a NiO single crystal exchange coupled to a thin Co layer [14]. Scholl's observation of a strong XMLD effect at the Ni $L_{3,2}$ edges indicated the formation of a planar antiferromagnetic domain wall in NiO near the interface upon magnetization reversal in the Co layer as proposed by Mauri *et al.* [13] for exchange coupled FM/AFM bilayers exhibiting strong interface coupling but weak anisotropies in the antiferromagnetic layer. Figure 3(a) shows the Fe $L_{3,2}$ x-ray absorption spectrum obtained in normal incidence. The XMLD spectrum, that is the difference of spectra measured in normal incidence by switching the magnetic field in the sample plane between parallel and perpendicular to the polarization axis of the linearly polarized x rays, is shown in Figure 3(b). If the magnetization reversal of the Ni layer induced a domain-wall like rotation of the antiferromagnetically ordered Fe moments in the FeF_2 layer, the spectrum should show characteristics of FeF_2 XMLD [15] in the same ways as a Ni^{2+} XMLD spectrum was observed in NiO by Scholl *et al.* upon magnetization reversal in a Co layer exchange coupled to a NiO single crystal [14]. However, the Fe XMLD spectrum is almost identical to that obtained from ferromagnetic Fe [16] indicating that the XMLD almost entirely originates from uncompensated Fe moments in the FeF_2 layer, following the external field and the Ni magnetization. Due to the strong anisotropy fields (about 15 T [17]) in FeF_2 the antiferromagnetic domain structure is not appreciably influenced as predicted by Mauri *et al*. [13].

In summary, studying exchange biased Ni/FeF_2 bilayers we verified the assumption by Nogués *et al*. [8] that positive exchange bias is caused by an antiparallel coupling between the ferromagnet and uncompensated Fe moments pinned in the FeF_2 layer; the latter are forced to align parallel to large cooling fields. No indications for the formation of a parallel antiferromagnetic domain wall in the FeF_2 layer upon magnetization reversal in the ferromagnetic Ni layer were found.

The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231. Work at UCD was supported by ACS-PRF (#43637-AC10) and the Alfred P. Sloan Foundation. Work at UCSD was supported by DOE.

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Figure Captions

Figure 1

(a) Fe and Ni $L_{3,2}$ spectra of a 20 Å Ni / 500 Å FeF_2 bilayer measured at $T = 55$ K. Spectra obtained for parallel and antiparallel orientation of external field and photon helicity are indicated by a dotted and a solid line, respectively. (b) XMCD spectrum of the same sample, that is, the difference of the spectra shown in (a).

Figure 2

Hysteresis loops measured at the (a) Ni and (b) Fe *L* edges after cooling the sample in fields of $H_{cf} = 0.02$ T (solid line) and $H_{cf} = 0.55$ T (dotted line) to 55 K. A magnified view of (b) at high fields is shown in (c).

Figure 3

(a) Fe $L_{3,2}$ spectrum measured in normal incidence with linearly polarized x-rays at $T = 55$ K. The average of spectra obtained with the polarization axis parallel and perpendicular to the FeF_2(001) direction is shown. (b) XMLD spectrum, that is the difference of the spectra observed in normal incidence by switching a magnetic field of 0.65 T in the sample plane between parallel and perpendicular to the polarization axis of the linearly polarized x-rays.

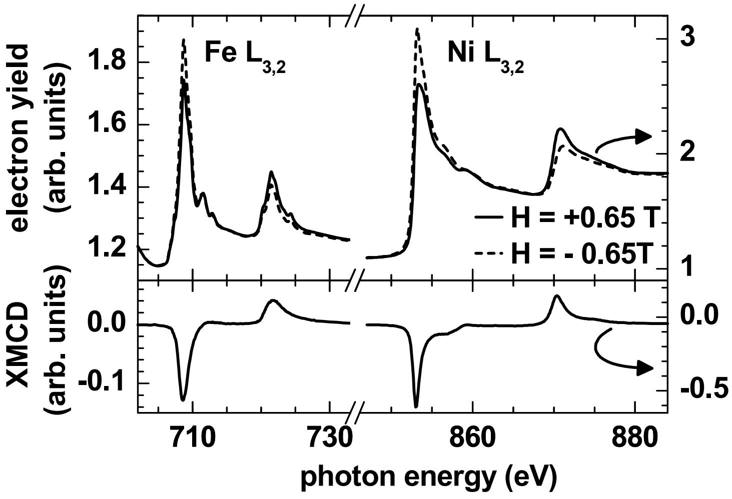
electron yield (arb. units)
1.8
1.6
1.4
1.2
Fe $L_{3,2}$
Ni $L_{3,2}$
3
2
1
H = +0.65 T
H = - 0.65T
XMCD (arb. units)
0.0
-0.1
0.0
-0.5
710
730
860
880
photon energy (eV)

Figure 1

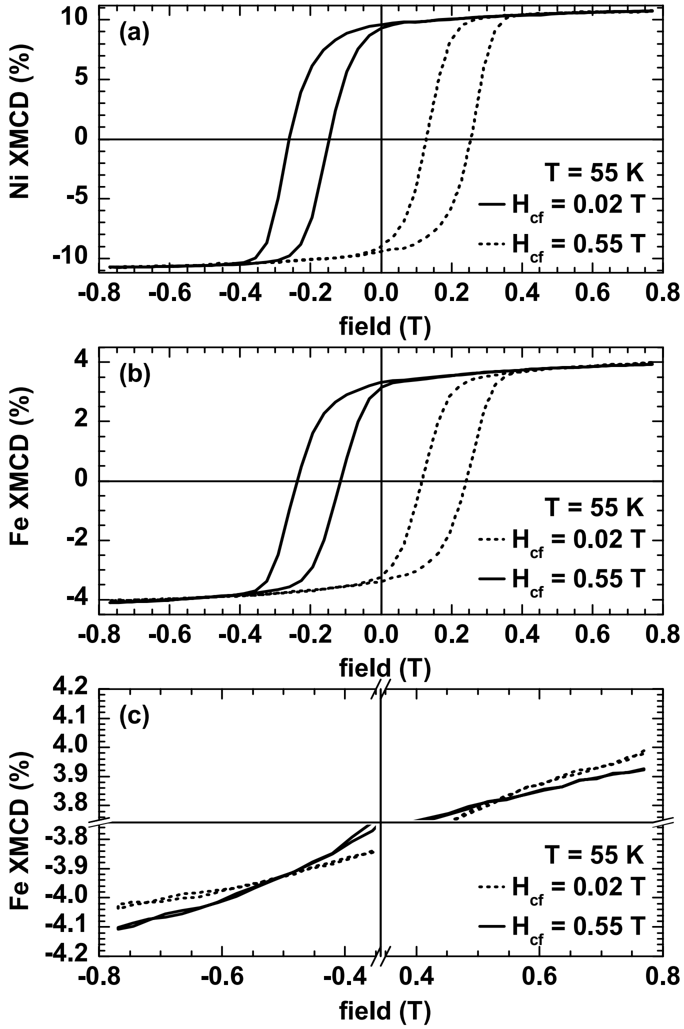
(a)
Ni XMCD (%)
10
5
0
-5
-10
T = 55 K
H_cf = 0.02 T
H_cf = 0.55 T
-0.8
-0.6
-0.4
-0.2
0.0
0.2
0.4
0.6
0.8
field (T)
(b)
Fe XMCD (%)
4
2
0
-2
-4
T = 55 K
H_cf = 0.02 T
H_cf = 0.55 T
-0.8
-0.6
-0.4
-0.2
0.0
0.2
0.4
0.6
0.8
field (T)
(c)
Fe XMCD (%)
4.2
4.1
4.0
3.9
3.8
-3.8
-3.9
-4.0
-4.1
-4.2
T = 55 K
H_cf = 0.02 T
H_cf = 0.55 T
-0.8
-0.6
-0.4
0.4
0.6
0.8
field (T)

Figure 2

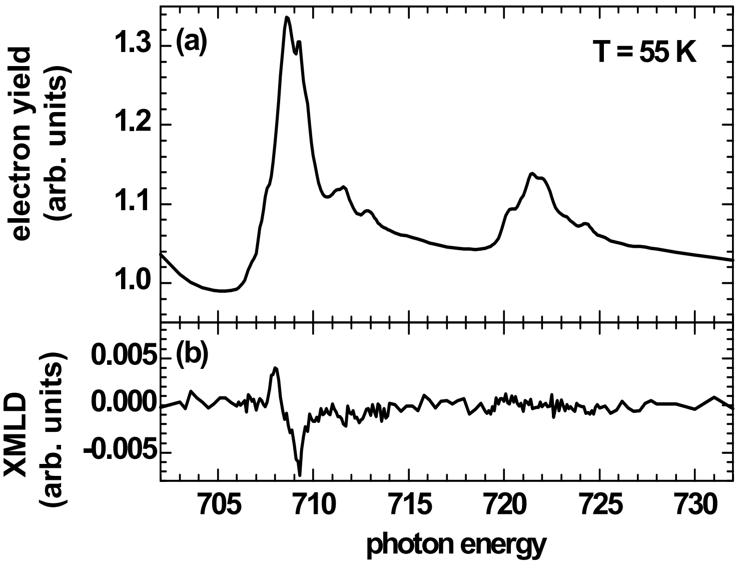
(a)
T = 55 K
electron yield (arb. units)
1.3
1.2
1.1
1.0
(b)
XMLD (arb. units)
0.005
0.000
-0.005
705
710
715
720
725
730
photon energy

Figure 3