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On the interface magnetism of thin oxidized Co films: orbital and spin moments

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Abstract
An x-ray magnetic circular dichroism study of a polycrystalline Co/CoO bilayer is presented. Using both the chemical specificity and surface sensitivity in the core level techniques, we find that uncompensated Co^{2+} spin moments participate in the remanent ferromagnetic response of the bilayer that has oxygen nearest neighbors. These are likely located at the Co/CoO interface. As intermixing of magnetic species is not present in Co/CoO, it is concluded that the observed interface moments are due to interface roughness. Given their direction, these moments appear to not directly correlate to the exchange bias in these bilayers.

1. Introduction

Exchange bias is a well-studied phenomenon that has attracted scientific attention since it was discovered in 1956 by Meiklejohn and Bean (1956). Since then it has been observed in many different systems containing ferromagnetic (FM)–antiferromagnetic (AFM) interfaces. Thin FM/AFM bilayers, especially Co/CoO, have been the most widely studied type of system (Nogués and Schuller 1999).

Due to coupling between AFM and FM, the hysteresis loop after field cooling is shifted along the field axis, typically in the opposite direction of the cooling field. This loop shift is commonly referred to as the exchange bias field. Exchange bias occurs below the blocking temperature T_B, which is lower than or equal to the Neél temperature T_N of the AFM. Other effects, such as increased coercivity and asymmetric magnetization reversal, have been observed together with the loop shift.

It has been shown that the exchange bias increases significantly when non-magnetic impurities are introduced into the AFM. The domain state model (Miltény *et al* 2000) accounts for this by emphasizing the importance of AF domain formation which is promoted by the holes in the magnetic lattice. Such a diluted AFM is believed to be the experimental realization of the random field Ising model as first realized by (Fischman and Aharony 1979). Field cooling introduces random fields and generates a metastable ordered state below T_N, composed of micro-domains that enhance the exchange bias effect. It is clear that the magnetic morphology of the AFM at the interface is essential, yet the exact role of domain walls and uncompensated spins remains unclear.

Uncompensated spins have been observed at interfaces in CoO/SiO_2 (Ambrose and Chien 1996) and CoO/MgO (Takano *et al* 1997) from magnetization, and in metallic bilayers as Co/FeMn (Antel *et al* 1999, Offi *et al* 2003) and Co/IrMn (Hase *et al* 2001) using XMCD. A significant part of the interface contributes, but only a small number of spins seem to be anchored in the antiferromagnet and do not follow the external field. These latter are believed to cause the exchange bias effect. Such pinned moments have been observed

recently in CoO/(CoPt) with MFM (Kappenberger *et al* 2003), and in Co/NiO, Co/IrMn and PtMn/$Co_{90}Fe_{10}$ with XMCD (Ohldag *et al* 2003). The latter study shows that uncompensated spins are observed in about 0.5 ML of the interface layer, yet 4% are pinned. In CoO/(CoPt) 7% of pinned interfacial spins have been observed. With neutron reflectivity pinned spins have been observed inside the antiferromagnet in Co/FeF_2 while uncompensated spins at the interface are not pinned (Roy *et al* 2008). The size of the exchange bias field can quantitatively be understood using a simple extension of the Meiklejohn and Bean model. The origin of the pinned spins is nevertheless not clear, but they are speculated to be located at the grain boundaries of the polycrystalline films. Remarkably, soft x-ray resonant magnetic reflectivity demonstrates a change in sign in the magnetization of the oxide in perpendicular exchange coupled (CoO/NiO)/Pt–Co (Tonnerre *et al* 2008).

Various mechanisms of exchange coupling between spins at opposite sides of the interface have been reported. Antiparallel coupling due to the super-exchange in AFM oxides has been observed for pinned spins in CoO/(PtCo) (Kappenberger *et al* 2003). Parallel coupling due to the direct exchange interaction between metallic layers has been observed in Co/IrMn and PtMn/$Co_{90}Fe_{10}$, and also in Co/NiO (Ohldag *et al* 2003). Finally, perpendicular coupling due to the spin-flopped state of the AFM was suggested (Koon 1997) and observed in Co/CoO (Borchers *et al* 1998).

Our intention is to study the interfacial spins in Co/CoO exchange-biased systems with x-ray magnetic circular dichroism (XMCD), exploiting its surface sensitivity to measure the number and orbital and spin moments of the interface Co atoms. This has been successfully exploited (Ohldag *et al* 2001, Camarero *et al* 2003) on NiO/Co bilayers. In this latter study a significant interdiffusion has been observed and an interface region of $CoNiO_x$ is formed. This intermixing is believed to be responsible for a large number of uncompensated spins, which enhance the coercivity of the bilayer. Intermixing has also been observed in FeMn/Co layers (Antel *et al* 1999) giving rise to Fe moments participating in the FM as well as the AFM layer. In Co/CoO, diffusion is limited to two atom species which simplifies the interpretation and results in relatively well-defined interfaces and is a promising approach to maximize exchange bias (Inderhees *et al* 2008). Yet, both the ferromagnetic and the antiferromagnetic layer are composed of Co atoms and contribute to the resonant absorption at the Co $L_{2,3}$-edges. Previously it has been shown that the absorption signal of Co and CoO can be separated on the basis of their specific signatures due to differences in the chemical surroundings (Regan *et al* 2001, Alvarenga *et al* 2002).

We show in this paper that the interface moments can be observed by evaluating different magnetic states of the sample and using enhanced surface sensitivity. It is observed that a ferromagnetically ordered interface appears when the sample is cooled under pulsed applied fields. These interfacial Co atoms have oxygen atoms as nearest neighbors and show enhanced orbital moments as compared to the rest of the Co film.

2. Experimental details

A Co/CoO film has been fabricated by sputtering Co on a 500 nm thick SiO_2 substrate on an Si wafer. Subsequently the polycrystalline Co film is oxidized in a controlled environment under an oxygen pressure of 10^{-4} Torr for 90 s. Within three days this film was mounted in the UHV chamber for the XMCD experiment. Afterward the bilayer was examined with low-angle x-ray reflectometry (XDR) and showed 2 nm-thick CoO with an interface roughness below 1 nm. The thickness of the Co film equals 17 nm. A typical grain size distribution equals 20 ± 7 nm (Loosvelt 2004). Progressive oxidation of the bilayer was observed with time and the thickness of the CoO layer increased between the XMCD and XDR experiments. This is attributed to the fact that the initial CoO thickness was thinner than the native oxide. The exchange bias depends on the thickness of the CoO layer; consequently the latter could not be determined on the same system as mounted in the XMCD chamber. The exchange bias shift appears typically below $T_B \sim 100$ K in these polycrystalline Co/CoO films. Its magnitude depends strongly on the applied magnetic field and temperature. For the field cooling applied during the XMCD experiment a small positive exchange bias is observed in these films (Radu *et al* 2003, Gredig *et al* 2002).

X-ray absorption experiments were performed using beamline D1011 using radiation from the MAX II storage ring at the MAX Laboratory in Lund, Sweden. This bending-magnet-based beamline is equipped with a modified SX700 monochromator. The photon energy resolution was set to 0.1 eV at the Co L_3-edge and spectra were recorded between 740 and 840 eV to obtain a good background definition. The characteristic absorption length of the incident x-rays, Λ, is considerably longer than the escape depth of the electrons, λ.

The data were acquired both in total electron yield (TEY) by measuring the sample drain current, and in partial electron yield (PEY) by means of a channel plate detector mounted below the sample. A retarding voltage of 400 V was applied to the front of the channel plate detector. This enhances the surface sensitivity as the characteristic escape length of the Auger electrons and their secondaries is reduced, detecting mostly elastically scattered electrons. In figure 1 the surface sensitivity of the two recording channels has been illustrated. λ_{TEY} is reported $\sim$20 Å (O'Brien and Tonner 1994) and $\sim$30 Å (Regan *et al* 2001) for Co and CoO, respectively.

The sample was magnetized *in situ* by means of magnetic field pulses of 54 mT parallel to the sample surface. The experimental geometry allows independent rotation of the coils and the sample with respect to the vertical axis. The x-ray angle of incidence was chosen as 45° with respect to the sample magnetization in order to minimize saturation effects (Nakajima 1999).

Absorption data were acquired with circular polarization. The degree of circular polarization at BL D1011 has been measured to be $\sim$80%. The dichroic difference was obtained by switching the direction of the remanent magnetization in the sample. Spectra were recorded at room temperature and at 90 K after field cooling (FC). In the latter case the magnetization direction has been reversed via heating and

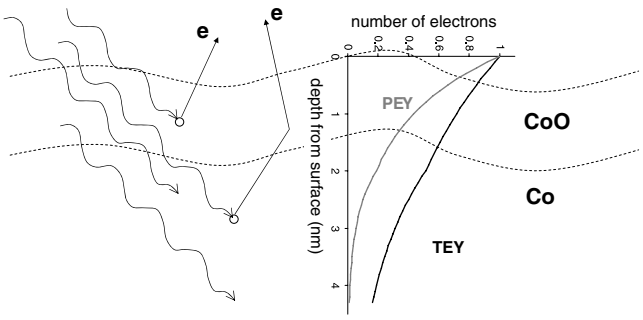

Figure 1. Origin of photoelectrons when measured in total electron yield (TEY) and partial electron yield (PEY). In PEY mainly electrons emerging from the top of the bilayer are detected and the escape depth λ is reduced significantly.

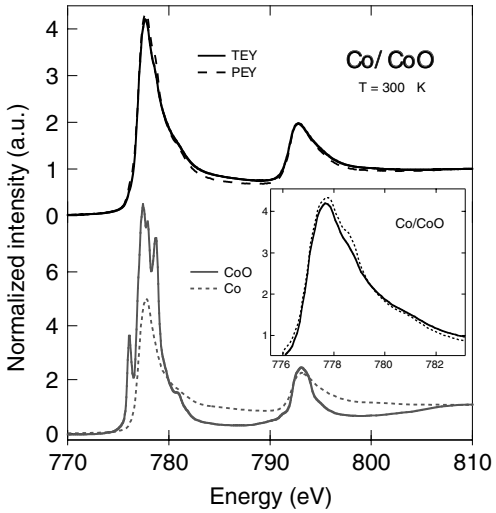

Figure 2. X-ray absorption spectra of Co/CoO at the Co $L_{2,3}$-edge obtained via TEY and PEY are shown at the top. The inset shows the detail of the L_3-edge. The bottom shows x-ray absorption spectra of metallic Co and CoO at the Co $L_{2,3}$-edge taken from (Regan *et al* 2001). The characteristic fine structure of the Co^{2+} ions in CoO is clear. The fine structure at the Co $L_{2,3}$-edge of the bilayer is enhanced in PEY confirming CoO is the top layer. All spectra are normalized on a per atom basis.

subsequent FC in the opposite direction. FC was obtained via cooling the sample from 300 to 90 K in 10 min, applying field pulses at intervals of 20 s.

All the data have been flux normalized by the photocurrent of a gold mesh reference monitor. Three spectra have been recorded for each setting and checked for reproducibility. They were averaged and normalized on a 'per atom' basis. The pre-edge and post-edge regions were used to correct differences in background between spectra recorded with opposite magnetizations.

3. XAS and XMCD

Figure 2 shows the Co $L_{2,3}$-edge of Co/CoO recorded with TEY and PEY at $T = 300$ K. The average of the spectra recorded with opposite magnetizations is used in order to

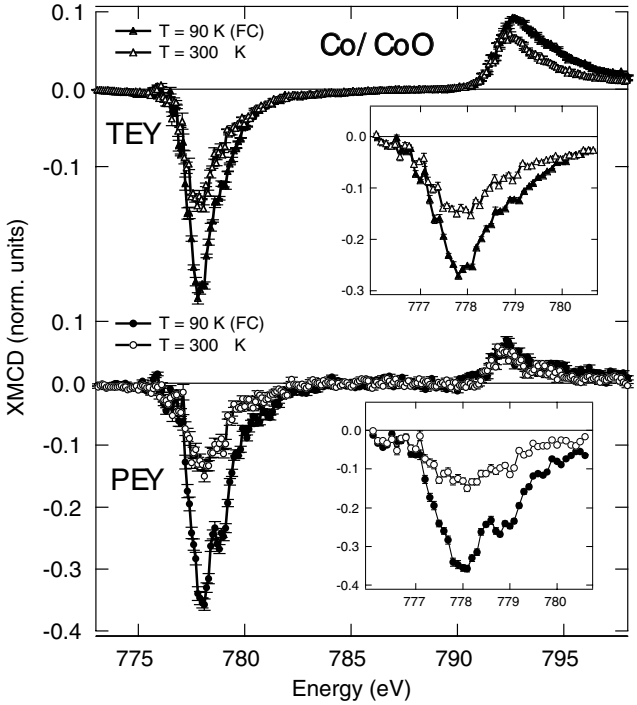

Figure 3. XMCD difference for Co/CoO bilayer at $T = 300$ K and after FC at 90 K, obtained in TEY (top) and PEY (bottom). The insets show the detail at the L_3-edge. The average orbital moment is enhanced near the surface, and at 90 K a fine structure emerges. This is most clear with PEY detection.

eliminate the magnetic part of the resonant absorption. The bottom of figure 2 shows the $L_{2,3}$ absorption edges of Co and CoO, taken from (Regan *et al* 2001) and normalized on a per atom basis. There is a clear difference between the multiplet-structured absorption spectrum of the CoO and the smooth profile of the Co spectrum. The oxidic contribution to the XAS spectrum is largest for PEY, as expected, as CoO is the top layer of the sample.

XMCD spectra obtained with TEY and PEY, after FC and at 300 K, are shown in figure 3. The orbital and spin moment, m_l and m_s, have been determined with the magneto-optical sum rules (Carra *et al* 1993, Thole *et al* 1992). To separate the transitions to unoccupied 3d states from the continuum states, a simple double step function was subtracted, whose step energies were set at the peaks of the L_3-and L_2-edges. The magnetic dipole term vanishes for polycrystalline samples. The electron occupation number was taken to be equal to 7.2 (Arvanitis *et al* 1996). For CoO a value of 7.1 has been calculated (Tanaka and Jo 1994, Ghiringhelli *et al* 2002). In practice there is some uncertainty in n_{3d} and m_l/m_s, making the relative changes in m_l and m_s the most reliable experimental quantities (Wu *et al* 1992). The obtained orbital and spin magnetic moments are corrected for the polarization of the light and for the angle of x-ray incidence. The results are listed in table 1.

$\langle m_s \rangle$ and $\langle m_l \rangle$ are the weighted spin and orbital moments of all Co^{2+} ions in the sample at depth t and lateral position

Table 1. Results of sum rule analysis for the total electron yield (TEY) and partial electron yield (PEY) XMCD spectra obtained at 300 K and at 90 K after pulsed field cooling. Also the deduced values for the interface region are given.

	TEY		PEY		PEY
	90 K	300 K	90 K	300 K	interface
$\langle m_s \rangle$ (μ_B/atom)	0.44(9)	0.30(6)	0.40(8)	0.21(5)	0.30(6)
$\langle m_l \rangle$ (μ_B/atom)	0.04(1)	0.02(1)	0.12(5)	0.05(2)	0.09(3)
m_l/m_s	0.08(2)	0.07(2)	0.29(6)	0.25(5)	0.31(6)

x, y:

$$\langle m_{l,s} \rangle = \frac{1}{\lambda S} \int_0^\infty \int_y \int_x m_{l,s}^{\parallel} e^{-t/\lambda} \, dx \, dy \, dt. \qquad (1)$$

$m_{l,s}^{\parallel}$ is the component of the Co^{2+} orbital or spin moment parallel to the photon wavevector. S is a surface large enough to yield a characteristic value of the surface topography. The addition of an integration along the surface plane (x and y directions) is necessary, given the existence of disorder, visible in the form of grains.

The remanence equals approximately 25% of the saturation magnetization in this Co/CoO bilayer at room temperature. Further, the sum of the spin moments in an antiferromagnet equals zero, while paramagnetic ions show no spontaneous magnetic moments. This leads to average Co spin moments well below those of a ferromagnetic Co layer.

The amount of multiplet structure observed in the x-ray absorption spectra at the L_3-edge is indicative of Co atoms probed within an oxidic environment and with a net ferromagnetic moment. It is deduced that the percentage of electrons originating from the CoO top layer is enhanced by a factor of two for PEY with respect to that of TEY. Consequently, TEY considers mainly Co ions from the ferromagnetic layer and $\langle m_s \rangle$ is observed to be 50% larger (table 1). The presence of grains as well as interface roughness does not allow us to perform a quantitative analysis with equation (1) and we present primarily an analysis of the relative variations of the moment values.

4. Interface region

Figure 3 shows an additional contribution to the XMCD contrast after FC. It is most apparent in PEY and consequently originates from the surface region of the bilayer. These additional moment components are oriented parallel to the magnetization of the Co layer.

As can be appreciated from the insets of figure 3, a multiplet-like structure emerges in the dichroic difference at the L_3-edge after FC. This feature relates to the chemical environment and indicates that some of the 3d electrons that give rise to the dichroic contrast are localized due to neighboring oxygen atoms. This additional XMCD contrast is denoted as $\Delta I_{PEY}^{interface}$ and equals

$$\Delta I_{PEY}^{interface} = R \, \Delta I_{PEY}^{300\ K} - \Delta I_{PEY}^{90\ K} \qquad (2)$$

where R equals the ratio of the remanent magnetization in the Co layer between 90 and 300 K. $R = 0.5$ has been determined from magnetization measurements after the field-cooling procedure used in the present study for similar Co/CoO films. This is due to a small positive exchange bias.

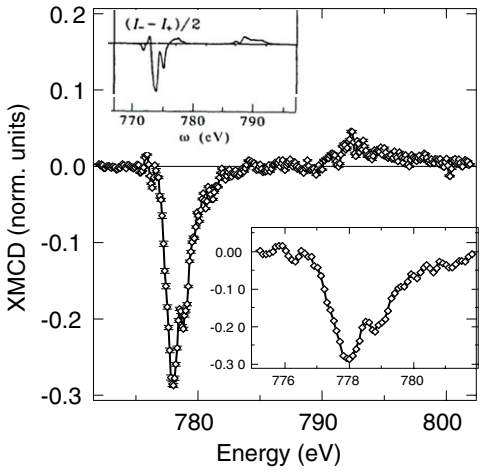

Figure 4. Signature of the Co^{2+} spins at the interface region of the Co and CoO layer. The bottom inset shows the detail at the L_3-edge. The top inset shows the calculated XMCD difference of magnetic Co^{2+} in oxidic environment, reprinted with permission of Imada and Jo (1992). Copyright Elsevier.

$\Delta I_{PEY}^{interface}$ is obtained from figure 3 and shown in figure 4. The relative contribution to the XMCD depends naturally on the depth of the particular Co^{2+} ion.

The multiplet structure is clearly visible in PEY and is very similar to that of magnetic Co^{2+}, as has been observed in $CoFe_2O_4$ (van der Laan *et al* 2008) and calculated by Imada and Jo (1992). The latter authors calculate the XMCD response of magnetic Co^{2+} in different crystal fields. Their prediction for octahedral symmetry with an additional trigonal distortion has been copied in figure 4 and gives $m_l/m_s = 0.27$, consistent with $m_l/m_s = 0.31(6)$ from $\Delta I_{PEY}^{interface}$.

The theory of (Solovyev *et al* 1998) gives 0.37 for Co^{2+} in CoO. Experimentally, $m_l/m_s = 0.48$ (Neubeck *et al* 2001) or higher (Jauch and Reehuis 2002) has been determined with x- and γ-ray diffraction while photo-emission gives 0.19 at 390 K (Ghiringhelli *et al* 2002). An XMCD study on CoO nanoparticles (Flipse *et al* 1999) reports $m_l/m_s = 0.31$ but the exact shape of the XMCD spectra is not reported. For metallic Co $m_l/m_s = 0.085$–0.091 has been determined with XMCD (Samant *et al* 1994, Alvarenga *et al* 2002). The latter study reports on a decrease in the orbital moment of Co near a CoO layer.

5. Discussion

Comparing the result from PEY with TEY indicates that the orbital moment is increased in the surface region of the bilayer. Such an increase of orbital moment near the interface is in contrast with the results from (Alvarenga *et al* 2002) where a constant m_l/m_s was reported. In the latter study depth profiling was obtained through sputtering of the Au/CoO/Co layers, suggesting that the ion bombardment modifies the structural and magnetic properties of the surface.

$\langle m_s^{\text{interface}} \rangle$ is substantial when compared to $\langle m_s \rangle$, indicating that a large part of the interface region participates. Moreover XMCD recorded with PEY indicates that the amount of ferromagnetic spins in an oxidic environment is considerable. These spins are either located at the surface, the interface or at grain boundaries.

The magnitude of the Co^{2+} spin moment calculated by (Imada and Jo 1992) equals 2.8 μ_B. This corresponds well with experimental estimates of CoO (Jauch *et al* 2001) and is taken as a reference. One ML of Co atoms at the interface is estimated to contribute 20% to the absorption, and with their spins fully aligned to the external field, gives $\langle m_s^{\text{interface}} \rangle = 0.56$. Yet, the preferential orientation of the Co^{2+} moments in CoO is randomly distributed due to the polycrystalline nature of the film. This is expected to result in a positive component along the field direction instead of a full alignment. Consequently, 0.65 ML of Co^{2+} spin moments aligned with their preferential orientation within the CoO matrix, yet closest to the applied field direction, accounts for our results. This number of spins corresponds well with that observed by Ohldag (Ohldag *et al* 2003).

This suggests that the interface is aligned more parallel to the applied field direction than the Co layer is. This is consistent with the large orbital moment at the interface, which enhances the coercivity at the interface. Possibly the remanence of the Co layer is larger near the interface because of coupling to the more anisotropic interface region. Figure 4 does not exclude that part of the interface signature originating from Co in a metallic environment.

The magnitude of interface moments is larger than expected for spin-flop coupling (Seehra and Silinsky 1979). Moreover, the exchange bias in this bilayer is positive and small, and the pinned spins are expected to be coupled antiparallel to the Co layer (Nogués *et al* 2000). Given that the dichroic difference of the interface has the same sign as that of the Co layer, we conclude that the interface signature of figure 4 is mainly due to uncompensated spins that do not contribute directly to the exchange bias. A similar conclusion has been reached for the interface moments observed in NiO/Co (Ohldag *et al* 2001). Uncompensated moments were attributed to the interdiffusion of the two magnetic species at the interface. Here we observe that a rough interface alone is enough to generate such moments.

It is remarkable that the uncompensated spins are aligned in opposite directions to the pinned spins that are believed to cause the exchange bias effect. This suggests that at least part of the mechanism generating these two groups of spins is different and exchange bias cannot be understood as uncompensated spins that happen to get pinned at grain boundaries.

6. Conclusion

It has been demonstrated that ferromagnetic or uncompensated spins at the Co/CoO interface can successfully be monitored by means of a partial electron yield mode in XMCD. Due to the chemical specificity of XAS the local environment of the Co atoms at the interface can be examined.

It appears that uncompensated Co^{2+} moments are present at the Co/CoO interface and that their magnetic moment components align more parallel to the applied field than the Co layer. These magnetic moments have oxygen atoms as nearest neighbors and their orbital moments are enhanced compared to the Co layer underneath. This introduces a larger magnetic anisotropy and enhanced coercivity at the interface compared to the rest of the Co film.

The magnetic morphology at the interface has changed after FC but, as the exchange bias is positive and small, it is concluded that the observed interface moments do not directly contribute to the exchange bias effect. Pinned spins are believed to align in the opposite direction to the observed interface moments, which indicates that the mechanism of exchange bias goes beyond the generation of uncompensated moments that are partially pinned.

Previous studies attributed uncompensated spins to the intermixing of different magnetic species at the interface. Here we conclude that interface roughness accounts for this effect.

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