Evidence of the Exchange coupling in the Co/α-Fe₂O₃ system

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Abstract. The magnetic exchange coupling in Co/α-Fe₂O₃(0001) interfaces has been studied. In this paper, we investigated magnetic properties at macroscopic (hysteresis loops) and microscopic (magnetic domain structure) scales. The system shows increased coercive fields and magnetic domain duplication likely mediated by the presence of a metallic interfacial Fe layer.

1. Introduction
Magnetically exchange coupled ferromagnetic (F) / antiferromagnetic (AF) thin films are intensively studied because of their important technological applications in spin-electronics, as for example in giant magnetoresistance based devices. An unidirectional magnetic exchange anisotropy is induced in a F material in two ways: by growing a F layer on AFs with high in-plane magnetic anisotropy under an external magnetic field or by cooling down the F/AF bilayer from above the Néel temperature (Tₙ) of the AF layer (and below the Curie temperature (Tₖ) of the F) in a saturating external magnetic field (field cooling process). It results in an exchange bias Hₑ corresponding to a hysteresis loop shift along the field axis and, eventually, in an enhancement of the coercive field, Hₑ [1,2]. This pinning phenomenon of the magnetization direction of a F layer by an adjacent AF layer was discovered almost 50 years ago [3]. Despite active research the exchange-bias effect is still subject to controversies. In particular, the conditions and materials parameters that favor the occurrence of Hₑ and Hₑ and determine their values, are not clear yet.

Recent works [4,5] evidenced the alignment of ferromagnetic spins along antiferromagnetic spin directions in exchange coupled bilayers. This phenomenon has been proposed to be a key feature in the understanding of the F/AF magnetic exchange coupling. Here, we present results for the Co/α-Fe₂O₃ system. The samples were grown without exposure to a magnetic field and were not field-cooled. Hematite (α-Fe₂O₃) is known to have limited in-plane magnetic anisotropy, large values of Hₑ are thus not expected. We will show that for the Co/α-Fe₂O₃ interface, magnetic coupling occurs through large Hₑ values and identical magnetic domain structures.
2. Experiment

All hematite layers, of thickness 20 nm, were grown in a dedicated setup by atomic oxygen assisted molecular beam epitaxy on α-Al₂O₃(0001) or Pt(111) substrates as described in references 6 and 7. The samples grown on Pt(111) substrates were dedicated to studies involving techniques hampered by charge build-up. Before growing Co layers of thickness $t_{Co}$, the air-exposed hematite layers were cleaned, by an annealing under an oxygen partial pressure of $10^{-5}$ mbar at 600K. For *ex situ* measurements, the samples were protected against oxidation by a thin (2 nm) Au capping layer.

The magnetic properties of the Co/α-Fe₂O₃ bilayers were studied by different techniques. Hysteresis loop measurements were recorded by *in situ* Magneto-optical Kerr effect (MOKE) and by *ex situ* Vibrating Sample Magnetometry (VSM). Chemical selective magnetic properties were obtained by *in situ* X-ray magnetic circular dichroism (XMCD), by *in situ* X-ray resonant magnetic scattering (XRMS) and by *in situ* X-ray photoemission electron spectro-microscopy (X-PEEM). The XRMS and X-PEEM experiments were performed at SLS (Villingen, Switzerland) on the SIM beamline. The X-ray absorption spectroscopy (XAS) and XMCD experiments were performed at the ID08 beamline at the ESRF (Grenoble, France). All measurements were carried out at room temperature.

3. Results and discussion

3.1. Macroscopic magnetic properties

3.1.1. Co-Fe ferromagnetic coupling

The growth mode and interface reactivity has been addressed in previous XMCD and XAS experiments [8]. The growth mode of Co is of a Stranski-Krastanov type and coalescence occurs at $t_{Co} \sim 2.5$ nm. The interface reactivity remains limited to about one atomic layer on either side of the interface which contains oxidized cobalt and reduced iron in the metallic form without any reduction sub-products of intermediate oxidation state. Importantly, the metallic iron layer only appears once Co is deposited. The magnetic coupling between the cobalt layer and the interfacial metallic iron was investigated by XRMS and XMCD. Figure 1 shows typical XAS spectra as well as the corresponding XMCD difference spectrum recorded using total electron yield (TEY) detection at the Fe and Co L₂,3 absorption edges for a 1.6 nm Co/20 nm α-Fe₂O₃/Pt(111) sample. The XMCD difference signals obtained for Fe and Co are of identical sign which reveals a parallel alignment of the magnetization between the Co and the interfacial metallic Fe.

![Figure 1. XAS (upper curves) and XMCD (lower curves) spectra of 1.6 nm Co/20 nm α-Fe₂O₃/Pt(111) recorded at the Fe (left) and Co (right) L₂,3 edges. (Inset) Element specific XRMS normalized hysteresis loops measured on Fe (□) and Co (■) in 1.6 nm Co/20 nm α-Fe₂O₃/Pt(111). The scattering angle was set to 2θ=26°](image-url)
normalized magnetization (M/Ms with Ms the saturation moment) as a function of the applied magnetic field. The hysteresis loops for the Co layer and for the metallic Fe layer are identical in shape and Hc values showing the coupling between the Co layer and the interfacial metallic Fe.

3.1.2. Coercive field Hc

Figure 2 shows the variation of Hc with respect to tCo for Co/α-Fe2O3/α-Al2O3(0001) and for Co/α-Fe2O3/Pt(111) samples measured by MOKE and VSM. From figure 2 it is clear that the choice of the substrate does not influence the coercive field value. In a previous work [10], we have shown that for identical preparation conditions and film thicknesses, hematite layers deposited on Pt(111) exhibit superior crystalline quality as compared to sapphire substrates. Although it is often claimed that the magnetic exchange coupling depends crucially on the F/Af interface quality, the crystalline quality seems not to be a determinant factor in terms of coercive field here. In all cases Hc is found to scale with 1/tCo which clearly demonstrates the interfacial nature of the effect [11].

The inset in figure 2 shows the hysteresis loops obtained for a Co layer with and without an adjacent AF layer. It reveals that the interface exchange coupling leads to an enhancement of Hc from ~5 Oe in 1.1 nm Co/α-Al2O3 (without AF layer) to ~137 Oe in a sample with the same thickness of cobalt evaporated on a hematite layer and, as expected, Hc = 0. Several possible explanations for the increased coercive field have been proposed as for example the anisotropic properties of the AF layers, the interfacial exchange coupling energy and the domain-wall energy density of the F layer [12]. The unpinned uncompensated AF spins were also suggested as responsible for the Hc enhancement in in-plane exchange F/Af systems [13]. In our case, the Hc enhancement may be understood by the following considerations. During the reversal of the F spins, the AF domains are dragged through the interfacial induced metallic iron spins mediating the exchange coupling. For the exchange coupled system the domain motion in the F layer requires thus more energy since it has to go beyond the interfacial exchange coupling energy and the domain-wall energy leading to enhanced Hc values.

Figure 2. Coercive field, Hc, versus cobalt thickness (tCo) for Co/α-Fe2O3/Pt (■) and Co/α-Fe2O3/α-Al2O3 (■). The values of the coercive field were obtained by Kerr and VSM. The continuous line corresponds to a 1/tCo dependence. (Inset) In plane Kerr-hysteresis loops of (1.1 nm) Co/α-Fe2O3/α-Al2O3 (…) and of (1.1 nm) Co/α-Al2O3 (—).

3.2. Local properties

The coercive field provides a macroscopic signature of the behavior of the F/Af interface when exposed to an external magnetic field. New techniques like X-PEEM allow to investigate directly the associated magnetic domain structure [4]. Fortunately, it has been shown that the L2 multiplet structure of α-Fe2O3 depends on the relative orientation between the spin orientation and the electric field direction for an incident linearly polarized beam [14]. This effect is exploited to obtain a contrast related to the magnetic domain structure in the AF hematite layer. For the ferromagnetic components (Co layer and interfacial metallic Fe) the magnetic domain contrast is obtained using left and right circularly polarized light at the L3 or L2 edge photon energy. The magnetic domain structure was investigated by X-PEEM measurements performed on a 1 nm Co/α-Fe2O3/Pt(111) sample. In figure 3, the α-Fe2O3 antiferromagnetic, the interfacial Fe ferromagnetic and the Co ferromagnetic domain structures, acquired at exactly the same sample position, are shown together. The magnetic domain structure is similar for the 3 components which imply a domain-by-domain coupling as already.
observed in other systems [4] and provides a direct evidence of the magnetic coupling at the local scale. Interestingly, the Co layer adopts a magnetic domain configuration properly dictated by the hematite layer although the demagnetizing field and magnetic anisotropy of Co would let expect larger domain sizes [15].

**Figure 3.** X-PEEM images. (Right) and (middle): XMLD and XMCD images at the Fe edge and (left) XMCD image at the Co edge revealing the AF and FM magnetic domain structures respectively. The red arrow gives the orientation of the x-ray incidence.

4. Conclusion
We have evidenced the presence of a magnetic exchange coupling in Co/α-Fe₂O₃ which consists of a strong increase of the Co coercive field at a macroscopic scale and magnetic domain duplication at a local scale. Both effects are likely mediated by the presence of a metallic interfacial Fe layer which shows a parallel ferromagnetic coupling with the Co layer. The onset of these properties is not linked to the presence of an exchange bias hysteresis loop shift.

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References

[1] Stiles M D and McMichael R D 2001 Phys. Rev. B 63 064405
[2] Mocuta C, Barbier A, Lafaye S, Bayle-Guillemaud P and Panabière M 2003 Phys. Rev. B 68 014416
[3] Meiklejohn W H and Bean C P 1956 Phys Rev 102 1413
[4] Nolting F et al Nature 405
[5] Ohldag H, Regan T J, Stöhr J, Scholl A, Nolting F, Lüning J, Stamm C, Anders S and White R L 2001 Phys Rev L 87 24
[6] Gota S and Gautier-Soyer M 2001 Phys Rev B 64 224407
[7] Barbier A, Belkhou R, Ohresser P, Gautier-Soyer M, Bezenčenot O, Mulazzi M, Guittet M J and Moussy J B 2005 Phys Rev B 72 245423
[8] Bezenčenot O, Barbier A, Ohresser P, Belkhou R, Stanescu S, Owens J and Guittet M J 2007 Surf Sci 601 4321-4325
[9] Tonnerre J M, Sève L, Raoux D, Rodmacq B, De Santis M, Troussel P, Brot J M, Chakarian V, Kao C C, Jonson E D and Chen C T 1995 Nucl Instrum Methods B 97 444
[10] Barbier A, Bezenčenot O, Mocuta C, Moussy J B, Magnan H, Jedrecy N, Guittet M J and Gautier-Soyer M 2007 J. Mat. Sci. Eng. B 144 19-22
[11] Shan R, Lin W W, Yin L F, Tian C S, Sang H, Sun L and Zhou S M 2005 Phys Rev B 71 064402
[12] Geshev J, Pereira L G and Schmidt J E 2002 Phys Rev B 66 134432
[13] Camarero J, Pennec Y, Vogel J, Pizzini S, Cartier M, Fettar F, Ehrnhöf F, Tagliaferri A, Brookes N B and Dieny B 2003 Phys Rev B 67 020413r
[14] Kuiper P, Searle B G, Rudolf P, Tjeng L H and Chen C T 1993 Phys Rev L 70 10
[15] Kittel C 1946 Phys Rev 70 965