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## X-ray magnetic circular dichroism in Fe/NiO thin films

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### Abstract

The interface of thin films of Fe/NiO (1 0 0)-oriented single crystals was studied by X-ray magnetic circular dichroism (XMCD). Thin layers of Fe were removed by “in situ” sputtering, followed by XMCD measurements, resulting in a depth profile. A decrease from the bulk value in the value of the Fe moment at the interface of Fe with the antiferromagnetic NiO(1 0 0) was observed. © 2001 Elsevier Science B.V. All rights reserved.

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Thin films consisting of different adjacent magnetic materials have attracted great technological and academic interest [1,2]. Thin films have been used, for instance, in spin-valves and magnetic recording heads, devices based on effects like giant magnetoresistance, and exchange biasing. In particular, the system Fe/NiO has been investigated under the scope of the exchange bias coupling [3–5]. Basically, this effect [1–3] is attributed to interactions of the spins at the interface. On modeling this effect, the magnetic ordering of interfacial spins must be accounted for, which still needs further experimental investigation [1,2 and references therein].

Soft-X-ray magnetic circular dichroism (XMCD) is nowadays a well-established technique

for investigation of the magnetism of surfaces and thin layers, since the probing depth is around 50 Å [6–8]. The XMCD is measured by the difference between the absorption cross-section of left and right circularly polarized X-rays at inner-shell absorption lines of magnetic systems. For the 3d transition metals, L<sub>2</sub> and L<sub>3</sub> are the absorption edges, corresponding respectively to transitions from the spin-orbit split 2p<sub>1/2</sub> and 2p<sub>3/2</sub> core levels to the 3d-valence band. Element-specific magnetic information is thus given by these dipole-allowed excitations [9–12].

In this work, we have studied the magnetic properties of Fe/NiO (1 0 0), a ferromagnetic (Fe) / antiferromagnetic (NiO) system. Thin films of Fe deposited over NiO [1 0 0] single crystals were investigated by X-ray magnetic circular dichroism (XMCD) performed at L<sub>2,3</sub> Fe and Ni edges as a function of Fe thickness. The XMCD was measured after partial sputtering of the

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Fe-layer from the surface down to the Fe/NiO interface.

Thin films of 200 Å Fe were deposited onto 1 mm-thick platelets of NiO single crystals, cleaved at the [100] direction, and cleaned with acetone ultrasound bath. The films were evaporated in a UHV chamber at a base pressure of  $10^{-9}$  Torr, using an Omicron Evaporator with integrated flux monitor. Samples were grown with the NiO substrate heated at 280°C, above its Néel temperature (250°C). A NdFeB magnet placed very close to the sample provided a 1 kG magnetic field parallel to the plane of the sample, in order to set the bias field direction of the ferromagnetic layer. Atomic force microscopy (AFM) was used to characterize the surface of the Fe film, attesting its very good quality.

The XMCD spectra were obtained at the SGM beam line at the Brazilian National Synchrotron Light Laboratory (LNLS). Thin layers of material were removed by means of ‘in situ’ sputtering, followed by room-temperature XMCD measurements, resulting in a depth profile. The synchrotron light passes through a slit that selects a beam with a circular polarization ratio of 85%, is monochromatized at the spherical grating monochromator, crosses an Au grid that measures the incoming flux, and finally impinges on the sample. Scanning the energies through the  $L_3$  and  $L_2$  absorption edges of Fe and Ni, the spectra were recorded monitoring the sample drain current. The sample stays at an angle of 45° with the X-ray beam propagation direction. A 2 kG magnetic field, parallel to the X-ray propagation direction, was applied with a permanent magnet, providing a larger projection of the sample magnetization  $M$  parallel to the beam direction. XMCD was measured as the field was reversed by rotating the magnet, and spectra collected with the field parallel and antiparallel to a fixed photon helicity. Several pairs of spectra were recorded at each sputtering session.

The spectra were flux normalized by the incoming flux, and the background was removed. The XMCD signal results from the difference of two spectra taken sequentially. The computed XMCD results are normalized [7] by the total absorption: the area under  $L_3$  XMCD is divided by the area of

the sum of the  $L_3$  absorption measured at both field directions. The normalized results are, therefore, proportional to the average magnetic moment per atom. The unknown Fe moments can be determined by normalizing, and then comparing their XMCD to that of a “standard” “in situ” sputter-cleaned bulk sample of Fe with a known moment of  $2.2 \mu_B$ . This standard was measured in each experiment, following the procedures of references [13–15].

The results of measurements with two samples of Fe (200 Å)/NiO grown without a capping layer are shown in the figures. Fig. 1 shows the X-ray absorption spectra of the Fe  $L_3$  and  $L_2$  edges for different Fe layer thickness: the full line (dotted line) is taken with field and helicity antiparallel (parallel). The XMCD spectrum is depicted below each pair, representative of the beginning, middle and thinnest Fe layer. Fig. 2 shows the spectra of

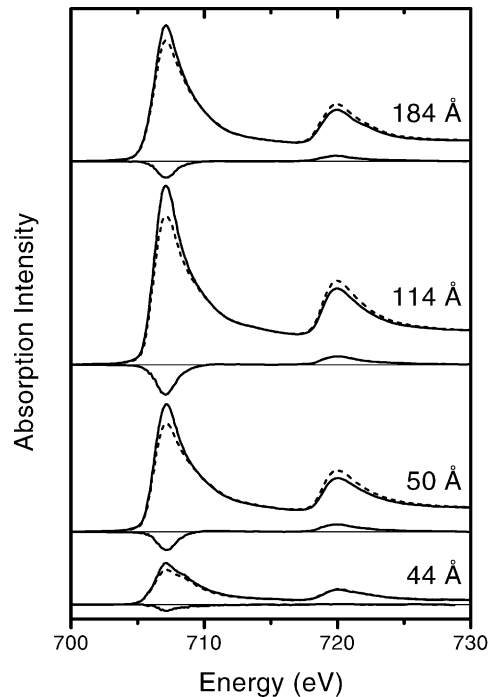


Fig. 1. X-ray absorption at the  $L_{2,3}$  edges of Fe, at different Fe layer thickness, taken with helicity of the photons and magnetization directions parallel (dotted line) and antiparallel (full line); the difference spectrum, below each pair, is the XMCD. The spectra are shifted for clarity.

the Ni  $L_{2,3}$  edges from NiO. Closest to the Fe/NiO interface, at 50 Å, there is some dichroism signal shown in the graph. As the Fe layer is totally removed, the signal from NiO does not present XMCD.

The intensity of the  $L_3$  absorption of Fe (circles) and Ni (triangles) as a function of Fe layer thickness is shown in Fig. 3a; the lines are just guides to the eyes. Considering the escape depth of the electrons around 50 Å, as the thin layers are removed, a depth of 50 Å is probed in this XMCD experiment. After cleaning the oxide layer, the Fe absorption signal remains constant, and the signal from the NiO substrate is barely seen. As the Fe layer becomes thinner, approaching the interface, the Ni in NiO absorption signal increases and the Fe signal decreases, consistent with the proportionality of the X-ray absorption with the amount of material present. The sputtering process continues until the Fe signal is not detected in the absorption spectra. At this point, the Ni signal has reached a maximum.

The resultant Fe  $L_3$  XMCD is plotted in Fig. 3b; notice that on the right axis the dichroic signal is expressed in units of magnetic moment. The thick horizontal line at  $2.2 \mu_B$  corresponds to the bulk Fe XMCD, from a reference sample measured at the same experiments. The statistical deviations from repeated measurements are just a few percent; the error bars at the points are the systematic errors as discussed in references [13] and evaluated as

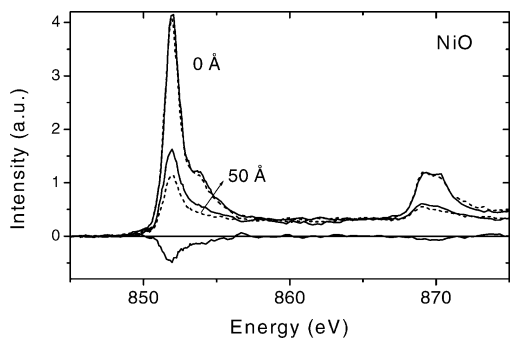


Fig. 2. X-ray absorption spectra of Ni  $L_{2,3}$  edges from NiO, as a function of the Fe layer thickness: close to the Fe/NiO interface (50 Å) there is a small dichroism (shown in the figure), absent from inside the NiO crystal (0 Å).

$\sim 10\%$ . Region (1) shows the increase of the XMCD signal as the iron oxide surface layers are removed. The X-ray absorption spectra present clear shoulders at higher energies on  $L_3$  and  $L_2$  edges when oxides are present [16,17]. As the layers are removed by sputtering, the spectra become “clean”, showing the absence of iron oxides. Region (2) exhibits an almost constant value corresponding to the middle of the Fe layer. As the Fe layer becomes thinner (region (3)), the interface is being probed, and the Fe XMCD decreases. The XMCD was measured until the Fe signal became too weak, making the difference spectra too noisy. It should be remembered that the use of the polarization slit cuts the intensity of the signal down to one third. In this way, the absorption without the slit can be measured further than the XMCD. Notice that the Fe XMCD decreases before the absorption signal starts to diminish. This fact confirms the probing

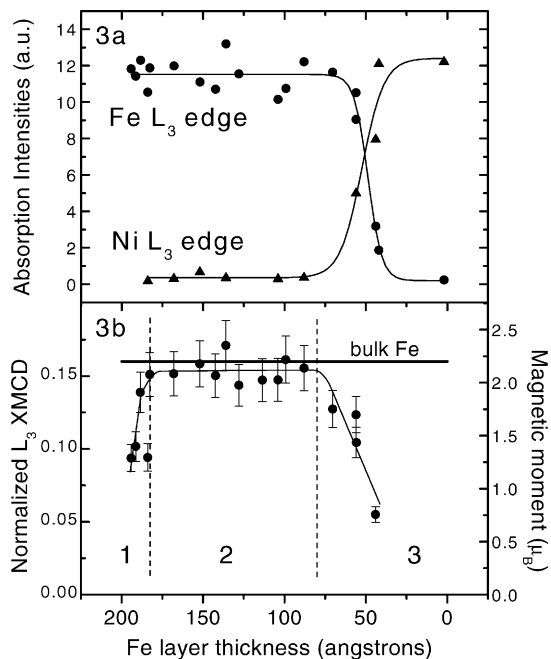


Fig. 3. (a) shows the intensity of the  $L_3$  absorption line of Fe (full circles) and Ni (triangles) as a function of Fe layer thickness. The Fe  $L_3$  XMCD is plotted in (b) the thick horizontal line at  $2.2 \mu_B$  corresponds to the bulk Fe XMCD, from a reference sample.

of Fe layers with reduced magnetic moment, close to the interface Fe/NiO.

One may formulate several hypotheses for the decrease observed in the Fe moments close to the interface. One can claim roughness at the atomic scale, due to some diffusion of Fe into the NiO crystal, or FeNi alloy formation during the process of Fe deposition, that could lead to a decrease in the measured average Fe moments.

The most probable explanation is the formation of paramagnetic FeO at the interface during the growing process, with oxygen released from the NiO. At the interface, this would lead to a line broadening at the absorption spectra of Fe, which was difficult to observe due to the poor signal to noise ratio, since there is only a small amount of Fe. A result that supports this idea is the observation of a small dichroic signal from Ni, which could be an indication of ferromagnetic order of Ni atoms.

In conclusion, the depth magnetic profile of the Fe/NiO (100) bilayer investigated by XMCD shows that, at the interface, the Fe moments decrease, possibly due to disorder at the interface (Fe–NiO interdiffusion or alloying) with the formation of FeO.

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