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Report:

The structural and magnetic properties of an Fe wedge sample exchange coupled to a CoO top-layer was traced with varying the Fe thickness.

Sample preparation was carried out in a multi-chambered ultra-high vacuum system with a base pressure better than 5 x 10^{-10} mbar. An 8 mm in diameter Ag(001) crystal was cleaned by sputtering with Ar^+ at 1 keV and annealing at 820 K for 30 minutes. An Fe wedge sample was thermally deposited while a shutter was gradually introduced causing a time gradient in the exposure of the substrate surface to the constant Fe flux. The thickness of the wedge varied gradually over 5 mm from 0 to 10 monolayers (MLs) and then a 2 mm-flat region of 15 ML was grown to demarcate the end of the slope. The Fe wedge sample was covered with 1 ML of metallic Co then transferred under UHV to another chamber for reactive growth. The sample was heated to 340 K and Co was evaporated under an O_2 pressure of 5 x 10^{-7} mbar to grow about 7 nm (33 ML) of CoO in 25 minutes. Such a CoO thickness is larger than the reported thickness above which exchange bias ceases to vary with the AFM thickness. The CoO surface seen in the Low Energy Electron Diffraction (LEED) pattern showed well-defined spots with an ordered cubic (1 x 1) surface. *Ex situ* Grazing incidence X-ray diffraction showed that CoO was grown epitaxially with (001) surface planes and the CoO[110] axes parallel to the Fe[100] and Ag[110] axes. The CoO film was almost totally relaxed with lattice parameters a = 4.251Å and c = 4.242 Å, very close to the bulk CoO value of 4.260 Å. The Fe layer was checked to be fully pseudomorphic above 6 MLs. The deposition of 1 Co ML previously the reactive growth intended to protect the Fe wedge from oxidation, nevertheless partial oxidation of the Fe did occur, as seen by X-ray absorption spectroscopy measurements showed in this report.

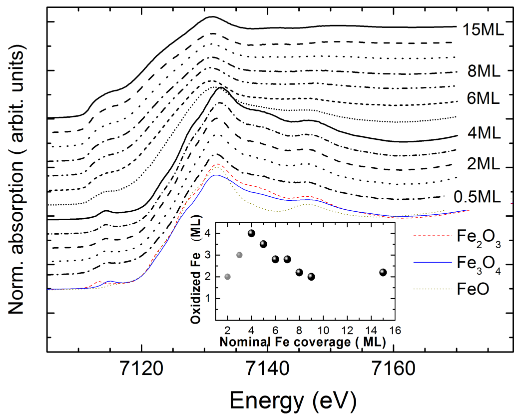
Experimental procedure :,

X-ray Absorption Near Edge Spectroscopy (XANES) at the Fe K edge was performed to investigate the oxidation state of Fe due to its proximity to the CoO and exposure to O_2 during initial CoO growth. XAS data were collected in fluorescence mode at room temperature. The samples were mounted in a goniometer and aligned at different orientations related to the X-ray beam. In-plane and out-of-plane data were collected at a grazing angle of about 5 degrees. The collected data turn out to be of very good quality, with the noise limited by the photon counting statistical. Simultaneously, the Co K edge was investigated.

Preliminary results :

Figure 1 shows the spectra at the Fe *K*-edge for various t_{Fe}. From 15 ML to the lowest Fe coverage, the spectra changes from basically that of pure Fe metal to that of an Fe oxide. By comparing with reference spectra of oxidized states of Fe viz., FeO, Fe_2O_3 and Fe_3O_4, it was seen that an inhomogeneous oxidation of the Fe has occurred. Furthermore, a qualitative comparison excludes the presence of FeO, and points to a linear combination of Fe_2O_3 and Fe_3O_4. The striking outcome here is that from the lowest coverage up to 4 ML, the Fe layer is fully oxidized and the spectra are essentially identical. It is clear that the oxygen has penetrated to 3-4 ML within the Fe. This is consistent with MOKE measurements where no open loop was detected in this region. Above 5 ML, a clear mixture of Fe metal and oxides shows up. A linear combination of the XANES data revealed that the thickness of the $Fe_{1-x}O_x$ phase (t_{ox}) varied with t_{Fe}. The oxide contribution (inset figure 1) reaches a maximum at 4 ML, then decrease monotonically down to only 2 ML for $t_{Fe} = 15$ ML. This suggests that probably up to the first 4 ML of Fe the growth is not smooth and there is a larger surface area exposed to the oxygen. Regan *et al.* [Regan *Phys. Rev. B* **64**, 214422 (2001)] have found for a 7 ML Fe layer in contact with CoO that only 2 ML were oxidized.

These XANES result combined to those previously obtained by MOKE allow to identify the Fe thickness regimes for induction of exchange bias by coupling with a CoO top layer: 1) the oxidized region up to 4 ML below the interface which does not contribute to the magnetic-optic signal, 2) between 4 and 6 ML where the spin-reorientation transition occurs from out-of-plane moments to the in-plane, 3) between 6 and 8 ML where the reoriented moments gradually become coherent in the film plane leading to the increase in coercivity and exchange bias with increasing Fe thickness. For Fe thickness larger than 8 ML the moments are well aligned and magnetic field cooling does not increase the exchange bias significantly more than is induced by zero field cooling.



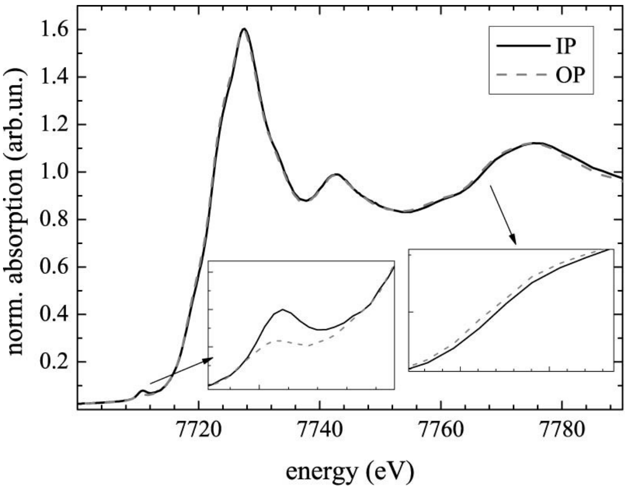


FIG. 1: XANES spectra at the Fe *K*-edge for various thicknesses of Fe. Also shown are the reference spectra for the FeO, Fe_2O_3 and Fe_3O_4 phases for comparison and, (inset) the estimated thickness of the oxidized Fe plotted against the nominal Fe thickness.

FIG. 2: XANES spectra at the Co *K*-edge for In-Plane and Out-of-Plane geometries. The insets show the difference.

Figure 2 shows the spectra at the Co *K*-edge for two different sample orientation related to the incoming beam. The shift of the first EXAFS oscillation agrees with a small shortening of the bond length in the film plane, due to epitaxial constrain. However the most striking feature is an unexpected anisotropy in the pre-edge range. From preliminary *ab initio* spectroscopic simulations, this large anisotropy seems to be dependent on the spin orientation of the cobalt atoms, and so related to the antiferromagnetic spin structure of the oxide. The study of the origin of this anisotropy and its relationship with crystallographic and spin orientation in the CoO film will be the object of a future proposal.