

# Hard x-ray resonant scattering study of $\text{Ni}_{81}\text{Fe}_{19}(111)/\text{CoO}(111)$ exchange biased bilayer

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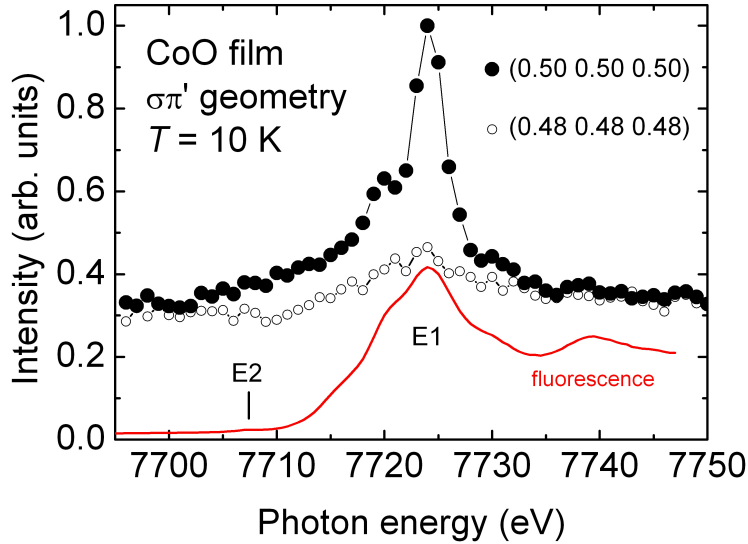
**Abstract.** We have employed hard x-ray resonant magnetic scattering to study the bulk antiferromagnetic domain state of the archetypal epitaxial  $\text{Ni}_{81}\text{Fe}_{19}(111)/\text{CoO}(111)$  exchange biased bilayer ( $\text{Ni}_{81}\text{Fe}_{19}$  = Permalloy = Py). Tuning the x-ray energy to the Co K-edge, we were able to detect the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  antiferromagnetic magnetic Bragg reflection from a 2000 Å thick CoO film covered by a 120 Å Py layer and capped by 50 Å gold. The temperature dependence of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg intensity and the rocking-width agrees well with previous neutron diffraction data. The average lateral size of antiferromagnetic domains is about 40 nm close to  $T_N = 291$  K.

## 1. Introduction

The exchange bias effect is of great importance for basic research and numerous device applications. It manifests itself in a shift of the hysteresis loop in the negative or the positive direction with respect to the applied field. Its origin is related to the magnetic coupling across the common interface shared by a ferromagnetic (FM) and an antiferromagnetic (AF) layer. The particular domain state of the AF layer has a significant effect on the magnitude of the exchange bias, which usually is much smaller than predicted by idealized models. Recently, the origin of the reduced exchange bias in epitaxial  $\text{Ni}_{81}\text{Fe}_{19}(111)/\text{CoO}(111)$  bilayer has been investigated by combined soft and hard x-ray resonant magnetic scattering (XRMS) and polarised neutron diffraction [1]. Here, we provide a detailed description of the XRMS results, including new data.

## 2. Experimental

A 2000 Å thick CoO film was grown on an  $\text{Al}_2\text{O}_3(11\bar{2}0)$  substrate, covered by a 120 Å Py layer and capped by 50 Å gold at the Helmholtz-Zentrum Berlin (HZB) by dc-magnetron sputtering as reported in [1]. The excellent smoothness of the Py/CoO interface has been proven by x-ray diffraction, showing the presence of several Laue oscillations around the Py(111) Bragg reflection. Polarized neutron reflectivity data result in a roughness parameter of  $\leq 7$  Å for the interfaces. The AF CoO layer was studied using XRMS close to the Co K-edge (7.71 keV). The measurements were performed in zero magnetic field at the beamline MAGS, operated by the HZB at the synchrotron source BESSY [2]. The sample was mounted on a closed-cycle cryostat equipped with Be domes. Cooling the sample from 300 K to 10 K in a 800 Oe magnetic field applied parallel to the sample surface, i.e., saturating the ferromagnetic Py layer, showed no significant effect on the widths or intensities of magnetic superlattice reflections compared to zero-field cooling data. In order to separate magnetic from structural contributions, linear polarisation analysis of the scattered beam was carried out using the (006) reflection of a highly



**Figure 1.** X-ray energy dependence of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection intensity through the Co K-edge measured for the 2000 Å thick CoO film with linear polarization analysis ( $\sigma\pi'$  channel). For comparison, the energy dependence at an off-peak reciprocal space position has also been measured, reflecting the background from fluorescence and ( $\pi\pi'$  cross-talk of) diffuse scattering. In resonance, the absolute peak intensity of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection was about 40 cts./sec above background at full ring current. The fluorescence signal is also plotted which gives a measure of the energy dependent absorption cross section.

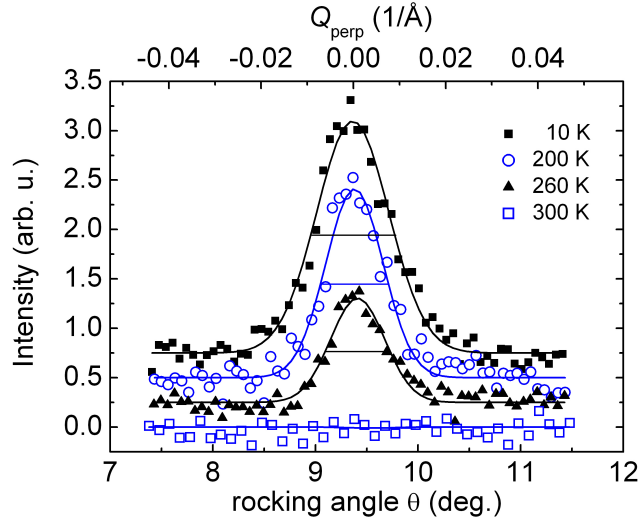
oriented pyrolytic graphite (HOPG) crystal, which gives an analyser Bragg angle of  $91.6^\circ$ , close to the ideal value of  $90^\circ$ . The degree of linear polarization of the primary beam was about 90%.

### 3. Results and Discussion

The energy dependence of the intensity at the CoO  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  position (Figure 1) shows a significant resonance enhancement at the Co K edge, indicating that the corresponding Bragg reflection is of magnetic origin. The observed resonance is dominated by the dipolar transition  $1s \rightarrow 4p$  (E1) with a maximum intensity at 7724 eV. The quadrupolar transition  $1s \rightarrow 3d$  (E2) at 7707 eV causes a weak anomaly in the fluorescence spectrum but is invisible in the Bragg intensity data. This contrasts the behavior observed for a bulk sample, where the E2 transition is prominent [3].

This discrepancy is easily understood taking into account the limited sample thickness and the variation of the x-ray penetration depth  $\mu$  across the Co K edge. In CoO, for energies closely below the edge we get approximately  $\mu_{\downarrow} \approx 29 \mu\text{m}$ , while  $\mu_{\uparrow} \approx 4 \mu\text{m}$  above. The  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  reflection appears at a Bragg angle  $\theta = 9.4^\circ$  and the effective sample thickness  $\tau$  is  $\tau = 2000 \text{ \AA} / \sin \theta = 1.2 \mu\text{m}$ , which in any case is smaller than the penetration depth. Therefore, the scattered intensity is limited by the sample thickness and scales with the ratio  $\tau/\mu$ . With respect to the scattering from a bulk sample this leads to a modification of the energy dependence of the scattered intensity. For the following experiments the x-ray energy was set to 7724 eV.

Longitudinal ( $\theta-2\theta$ ) scans of the (111) and  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  reflections exhibit significant line broadening related to the formation of small structural grains and AF domains [1]. The size  $L$  of these domains can be estimated using  $L = 2\pi/\text{FWHM}$ , where FWHM is the full width at half



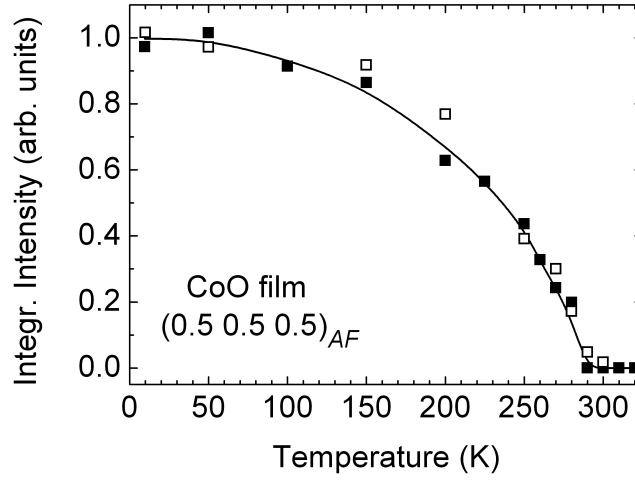
**Figure 2.** Transverse ( $\theta$  rocking) scans of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection for the 2000 Å CoO film. A diffuse background was determined from data taken at 300 K and subtracted. The transverse instrumental resolution was  $0.02^\circ$  and thus negligible. For clarity, the data are offset along the vertical axis.

maximum in reciprocal space. From the FWHM values of  $0.011 \text{ \AA}^{-1}$  and  $0.013 \text{ \AA}^{-1}$ , respectively, we infer an average grain size of 57 nm and an average AF domain size of 48 nm for the out-of-plane direction [4]. This suggests the formation of AF domains within structural grains.

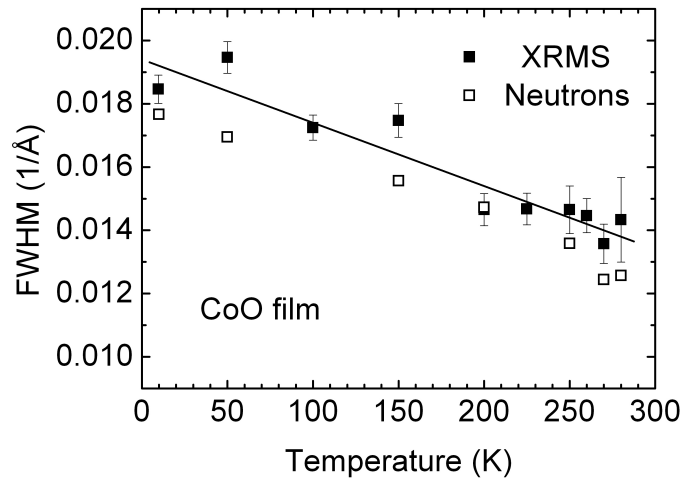
In order to get information about the in-plane AF domain size, the AF  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection was measured by transverse ( $\theta$  rocking) scans for various temperatures (Figure 2). The temperature dependence of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg intensity (Figure 3) reflects the square of the magnetic order parameter. The overall  $T$ -dependence and the observed transition temperature  $T_N = 291 \text{ K}$  are in excellent agreement with previous neutron scattering data.

The temperature dependence of the FWHM of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  reflection is shown in Figure 4. Again, the XRMS data agree well with the results from neutron diffraction experiments. The quantitative analysis yields a 40 nm average lateral AF domain size close to  $T_N = 291 \text{ K}$ . Interestingly, the line width is temperature dependent, suggesting a significant decrease of the AF correlation length on cooling. In the previous report we argued that the averaged sizes of AF domains and domain walls vary as function of temperature. This can be understood as an effect of the interplay between AF anisotropy and stiffness strength [1]. Quantitatively, however, an accurate estimate of the correlation length from the rocking width may be hampered by the structural changes of the CoO lattice below  $T_N$ . To this end we have measured rocking widths also of the (111) reflection (not shown). The preliminary data analysis shows that the temperature dependence of the (111) rocking width exhibits a similar temperature dependence as that of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  reflection, suggesting that the known monoclinic distortion in the magnetically ordered phase of CoO [5] may contribute to the observed temperature dependences by mosaic broadening. We have also measured the azimuth dependence of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  line width (not shown) and found no significant variation of the FWHM value as function of the azimuth angle, suggesting isotropic in-plane average domain sizes.

In conclusion, we have determined the temperature dependence of the intensity and the rocking-width of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection from a 2000 Å CoO film on sapphire by XRMS. The results correspond well with previous neutron diffraction data.



**Figure 3.** Temperature dependence of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  integrated Bragg intensity for the 2000 Å CoO film measured by XRMS (solid symbols). Corresponding neutron diffraction data (open symbols), taken from [1], are shown for comparison. The line is a guide to the eye.



**Figure 4.** Temperature dependence of the FWHM line width of the  $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$  Bragg reflection from the XRMS data (closed symbols). The corresponding neutron diffraction data (open squares), taken from [1], are shown for comparison. The line is a guide to the eye.

## References

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