



## Research articles

## Magnetic properties of the CoO/Fe(001) system with a bottom-up engineered interface



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## ABSTRACT

Ultrathin CoO films can be grown onto Fe(001) by exploiting Co buffer layers with nanometer thickness, with the result of avoiding the formation of Fe oxides at the interface. Such a system is characterized by a magnetic anisotropy that influences the magnetization reversal behavior, making the Fe easy magnetization axes inequivalent. Here, we exploit Magnetic Second Harmonic Generation to show that such an anisotropy is related to the buried interface and that it is not related to the magnetic properties of the antiferromagnetic CoO layer. In fact, CoO is magnetically ordered already at room temperature, even for low thicknesses and independently on the presence of Fe oxides. The magnetic domains configuration of CoO mimics those of both Fe and Co in all cases, as testified by magnetic PhotoElectron Emission Microscopy measurements.

## 1. Introduction

Intriguing magnetic properties can be obtained in systems containing antiferromagnetic (AF) transition metal (TM) oxides (O) both by low-dimensionality and by proximity to ferromagnetic (FM) layers [1]. So far, we have been investigating both chemical [2] and magnetic [3] properties of a variety of Fe/AF (NiO, CoO) layered structures. One of the most critical issues concerning reactive TMO/TM interfaces is the high degree of chemical mixing. In the case of the CoO/Fe system, for instance, the formation of a mixed oxide layer (i.e., containing different Fe oxides, such as Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>) commonly occurs at the interface, with thicknesses that, in the ultrathin range (a few layers) is comparable to that of the growing Cobalt oxide [4,5].

Recently we have been able, by exploiting a metastable Co buffer layer with the tetragonally distorted body centered cubic structure typical of ultrathin Co layers on Fe [6,7], to obtain a CoO/Fe interface free of any Fe oxide. The latter, on account of a misfit dislocation network developing at the interface, was also, interestingly, characterized by a dislocation-driven nanostructuring of CoO [8]. From the point of view of the magnetic properties, such an interface engineering resulted in the development of a significant uniaxial magnetic

anisotropy, induced by tiny amounts (monolayer range) of deposited CoO [9]. The occurrence of such an anisotropy was testified, in particular, by the observation of a multijump reversal behavior in the magnetization hysteresis loops as measured by the Magneto-Optical Kerr Effect (MOKE), in accordance with a reversal model in which the anisotropy energy was required to be larger than that needed to unpin 90° domain walls [10]. Thin Fe films are usually characterized by two easy magnetization axes directed along the two equivalent in-plane [100] directions. The presence of the mentioned anisotropy leads to a differentiation of the reversal behavior along the two axes, so that the formation of 90° domains is, in one of the two cases, more likely to occur. Further element-sensitive hysteresis loops, based on X-ray Magnetic Circular Dichroism (XMCD), confirmed such observations and revealed that an extremely thin metallic Co layer remains confined at the interface and has a magnetic behavior analogous to that of the underlying Fe substrate [9].

In this work, we aim to unveil further details related to the origin of the interface magnetic anisotropy, in particular by focusing on the magnetic properties of the interface itself and on the magnetic nature of the CoO overlayer. The first aspect was investigated by measuring magnetic hysteresis loops through Magnetic Second Harmonic (MSH)

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generation [11–13]. The latter technique is known to be particularly sensitive to magnetic effects associated with surfaces and interfaces, with several examples related in particular to AF/FM interfaces [14,15]. Furthermore, MSH generation was also shown to be capable of probing the magnetic arrangement at the surface of AF materials, even in magnetically compensated cases [16,17]. The use of MSH will allow us to disentangle the interface contribution to the magnetization reversal behavior previously measured with MOKE and XMCD, which is related to a portion of the substrate about 10 nm thick.

For what instead concerns the magnetic nature of the CoO overlayer, it was not directly measured in previous works. This kind of information would be useful to possibly confirm what already proposed as a tentative scenario in already published discussions. We performed XMCD and X-ray Magnetic Linear Dichroism (XMLD) in combination with Photoelectron Emission Microscopy (PEEM) to obtain the magnetic domains configuration with element sensitivity. In fact, XMCD-PEEM can be performed at either the Fe or Co absorption edges to observe the FM domains in the corresponding layers; XMLD-PEEM performed at a specific Co edge is instead capable of imaging the AF domains in the CoO layer. Spectroscopic data can be obtained by integrating over specific areas in the microscopy images, as described below.

The combination of these experimental approaches allows us to conclude that the source of the mentioned magnetic anisotropy resides indeed at the interface of the system and that it does not seem to relate to the magnetic order of the CoO layers, which is AF already at room temperature (RT) for all cases considered. In particular, MSH measurements suggest that the engineered CoO/Fe interface is characterized by the presence of pinned magnetic moments whose orientation is not dictated by the magnetic properties of the adjacent magnetic layers.

## 2. Experimental methods

Thin CoO films were grown onto the Co(001)- $p(1 \times 1)$  O surface of a ultrathin Co film (5 monolayers, ML) grown on a 500 nm-thick Fe(001) film, as described in detail in previous publications [4,18,6–9]. The oxygen-saturated Co surface is obtained by annealing the Co/Fe sample at 470 K for a few minutes; this also induces the formation of atomically flat terraces on the surface [19]. The CoO deposition was performed with the sample kept at 470 K by evaporating metallic Co in a pure O<sub>2</sub> atmosphere with partial pressure  $p_{O_2} = 1 \times 10^{-4}$  Pa [8], for an oxide thickness  $t_{CoO}$  up to 25 ML.

The MSH measurements were performed using a Ti:Sapphire regenerative amplifier operating at 800 nm wavelength with a pulse duration of 60 fs, a repetition rate of 1 kHz and a typical time-averaged beam power of 40 mW. Both *s* and *p* light polarizations were exploited. The laser beam was focused on the magnetic surface impinging at an angle of about 45°. An external magnetic field generated by Helmholtz coils was applied perpendicular to the light incidence plane (the so-called transverse geometry). The sample could be rotated around its surface normal in order to vary the orientation of the easy axes with respect to the applied field. The infrared component of the reflected laser beam was attenuated with band-pass filters and separated from its MSH signal using a prism. Then, the ultraviolet beam was detected by a photomultiplier connected to a lock-in amplifier. The MSH hysteresis loop was recorded by slowly and periodically (at a frequency of about 1 Hz) reversing the external magnetic field. Each reported loop is the result of about 50–100 averages.

The magnetic domains images were acquired at the PEEM end-station of the CIRCE beamline at the ALBA synchrotron light facility [20]. PEEM images were acquired using low energy secondary electrons (1–3 eV), with magnetic contrast provided by either the XMCD effect at the Co and Fe L<sub>3</sub>absorption edges, combining images with opposite circular photon polarization, or by the XMLD effect at the Co L<sub>3</sub>absorption edge, combining images with orthogonal linear photon polarizations. In the XMCD-PEEM images, the intensity (gray scale) is

proportional to the local magnetization, projected onto the incoming photon beam direction (which is at 16° degrees grazing angle from the surface). Similarly, the intensity in XMLD-PEEM images is proportional to the square of the local magnetization, thus allowing for the observation of AF domains. Magnetic fields along different directions were applied in situ using sample holders with in-built quadrupole electromagnets [21]. The images reported in this work were taken under very low magnetic fields (of the order of 1 Oe) applied along one of the sample easy axes, after having applied large enough fields to saturate the magnetization.

Local X-ray Absorption Spectra (XAS) were recorded by collecting a PEEM image series while varying the incoming photon energy (at fixed polarization) and extracting the average detected secondary electron intensity within defined areas in the image. Rather than subtracting spectra with different photon polarization, XMLD spectra were calculated from the difference (divided by the sum) of a single XAS from two different magnetic domains, using a linear photon polarization in the surface plane of the sample. The two domains had been previously determined by XMCD images to be aligned or perpendicular with the projection of the incoming beam. Such an approach allows to reduce the background in the spectra, thus obtaining better quality ones.

No capping layer was employed for ex-situ measurements. The chemical stability of the buried interface was later confirmed by either X-ray photoemission or XAS experiments on ex-situ measured samples (not discussed here).

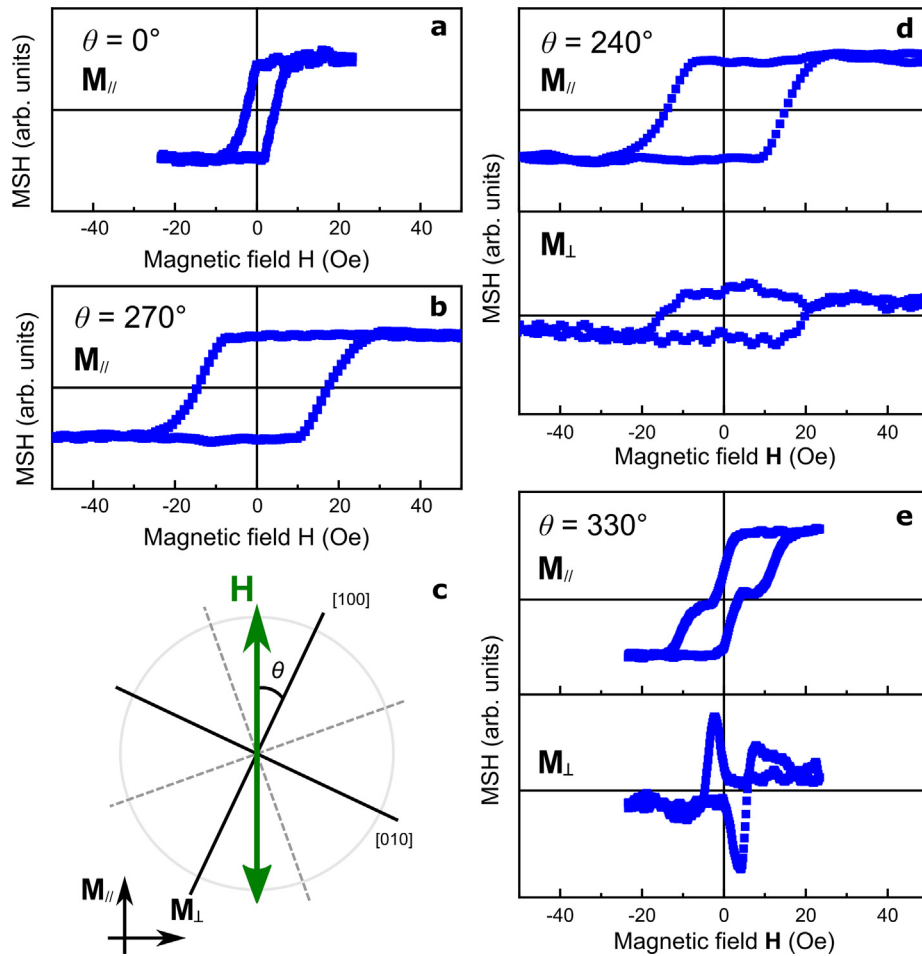
## 3. Results and discussion

MSH hysteresis loops were acquired at RT on the same samples, with  $t_{CoO} = 25$  ML, already discussed in Ref. [9]. A selection of the MSH measurements is reported in Fig. 1. The relative axes and field directions are sketched in panel c. In particular, **H** is the in-plane applied magnetic field and  $\theta$  the angle between **H** and the [100] direction of the Fe(001) substrate. During measurements, the direction of **H** was kept constant, while the sample was rotated in-plane by  $\theta$ . The hysteresis loops shown in Fig. 1 are related to the two components  $M_{\perp}$  and  $M_{\parallel}$  of the total in-plane magnetization **M** of the sample, which are either parallel or perpendicular, respectively, to the direction of **H**.

The hysteresis loops for the angles  $\theta = 0^\circ$  and  $\theta = 270^\circ$  are referred to the easy magnetization axes of the Fe film. A first interesting observation that can be made is that these loops differ in terms of the definitely larger coercive fields of the latter, despite they are expected to be totally equivalent in Fe films. This observation supports the conclusion, already discussed in Ref. [9], that the two axes are not fully equivalent, i.e. one is harder than the other (the lower coercivity here suggests that the easier one is that at  $\theta = 0^\circ$ ). This was attributed to the onset of a magnetic anisotropy, as discussed in the Introduction. The corresponding axes can thus be referred to as easy-easy axis and easy-hard axis, respectively.

On account of the mentioned anisotropy, the magnetization reversal along the easy-hard axis was accompanied by the formation of 90° magnetic domains, as shown in Ref. [9]. The two jumps behavior that would come along with the formation of 90° domains (and the corresponding  $M_{\perp}$  component) was not observed for the case reported in Fig. 1b (easy-hard axis), at variance with previously reported MOKE and XMCD loops. On the other hand, a multijump reversal behavior is found in the MSH loops for  $\theta = 330^\circ$ , as shown in Fig. 1e. Conversely, the MSH loops for  $\theta = 240^\circ$ , reported in Fig 1d, do not show a multijump behavior.

Overall, such magnetization hysteresis loops are similar to what already obtained by MOKE in Ref. [9], where multijump loops were observed when the field was applied in directions close to the hard axes. Apart from the absence of a  $M_{\perp}$  component in the easy-hard axis loops, another intriguing difference here is that the multijump behavior is seen when **H** is directed closer to the easy-easy axis ( $\theta = 0^\circ$ ), while in MOKE it occurs closer to the easy-hard one ( $\theta = 270^\circ$ ). This is confirmed



**Fig. 1.** Panels a, b, d, e: magnetic second harmonic hysteresis loops on a 25 ML CoO film on Co/Fe(001). Panel c: sketch of the experimental layout. The parallel and perpendicular components of the in-plane magnetization  $\mathbf{M}$  are referred to  $\mathbf{H}$ .

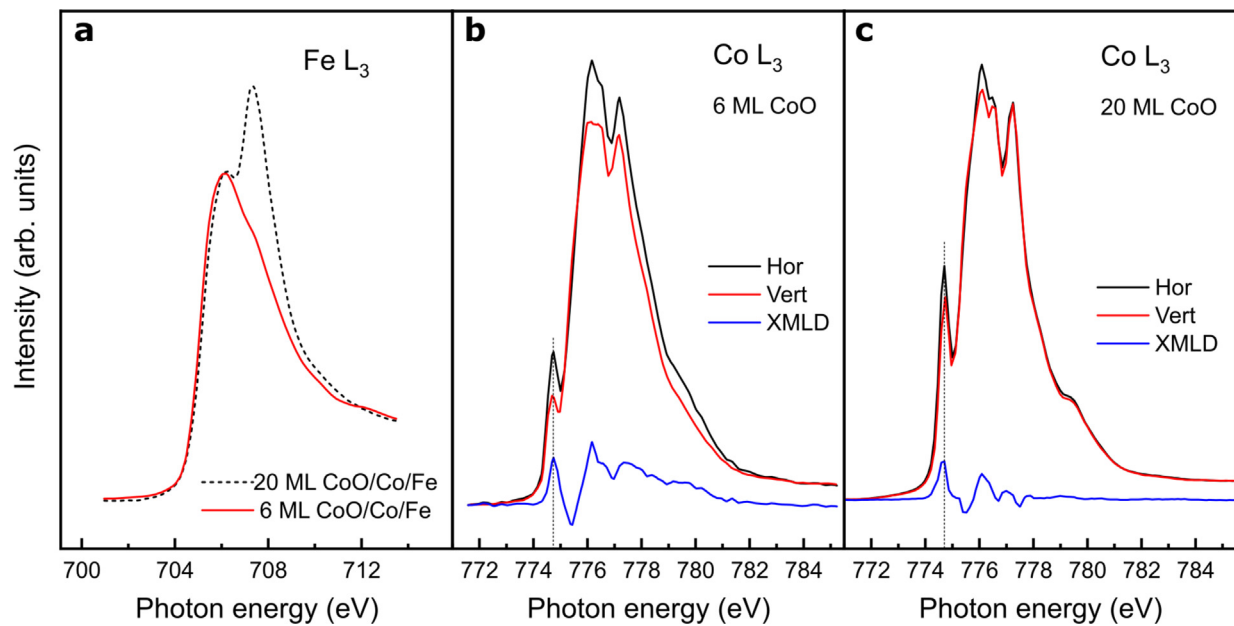
also by measurements at other angles, not shown here.

A possible explanation that would be in agreement with the above observations is the presence of pinned magnetic moments at the interface, which would be tentatively directed as a magnetic field applied close to the hard axis at  $\theta = 315^\circ$ . In the presence of such pinned moments, one would possibly expect the onset of an exchange bias at the interface, which would lead to a shift of the hysteresis loops. Such a shift is anyway not observed in any of the measured loops. On the other hand, it has been shown that pinned uncompensated spins can be found at an AF/FM interface also when the sample is above the so-called blocking temperature, which is the upper temperature at which the exchange bias effect is observed [14]. In this respect, we observe that the blocking temperature for CoO ultrathin films can be very low, even if the AF layer is coupled to a FM substrate [22]. Furthermore, the presence of pinned moments at the interface might be the reason for the fact that  $90^\circ$  domains that forms in the FM layer when the field is applied along the easy-hard axis do not stabilize at the interface and are thus not observed in the MSH loops. Finally, also effects related to the MSH response with respect to the alignment of the magnetic moments in the AF might have an influence on the observed reversal behavior. On the other hand, it has been shown that the polarization dependence of the MSH generation on the surface of a CoO (111) crystal is substantially isotropic [16], thus excluding anisotropy effects related to the experimental technique also in the present case.

In order to gain a further insight into the nature of the magnetic anisotropy that develops at the interface, we have investigated its possible relation to the magnetic order of the different layers. Fig. 2a shows XAS spectra taken at the Fe  $L_3$  absorption edge on two different

samples, characterized by different growth conditions and also by different CoO thicknesses. In particular, the sample with  $t_{\text{CoO}} = 6$  ML (continuous red curve) was successfully grown without forming Fe oxides, differently from that with  $t_{\text{CoO}} = 20$  ML (dashed black curve), which was prepared, on purpose, at a slightly higher temperature, sufficient to oxidize all of the buffer layer and in part the Fe substrate. In fact, while the former has the typical lineshape of oxygen-free Fe [23], the latter is characterized by a further peak at higher photon energy, which indicates the presence of Fe oxides at the interface [24].

Panels b and c of Fig. 2 report XAS spectra taken at the Co  $L_3$  absorption edge on the same samples of panel a and the corresponding XMLD spectra. In both cases, an XMLD signal is clearly present, which compares well with the known XMLD lineshape for CoO (despite some larger background in the 6 ML case) [25]. These measurements testify that the CoO film has a long range AF order in both cases, independently on the presence of Fe oxides at the interface. The XMLD signal of the thinner CoO film is slightly larger than that of the thicker one. This is most probably due to the fact that the 20 ML-thick sample features the presence of Fe oxides and, correspondingly, the CoO is not well ordered as for the case of the oxygen-free interface [26]. We believe that the increased film roughness is likely responsible for a decrease in the magnetic order of the film (e.g., by formation of multiple domains). It is worth to notice that also natural dichroism (i.e., related to the crystal field) partly contributes to the XMLD signal. The fact that the observed lineshape is for a large part due to magnetic order is anyway further confirmed by magnetic imaging (see below), so that we can conclude that the non-magnetic contribution to the spectra does not significantly alter the magnetic one.

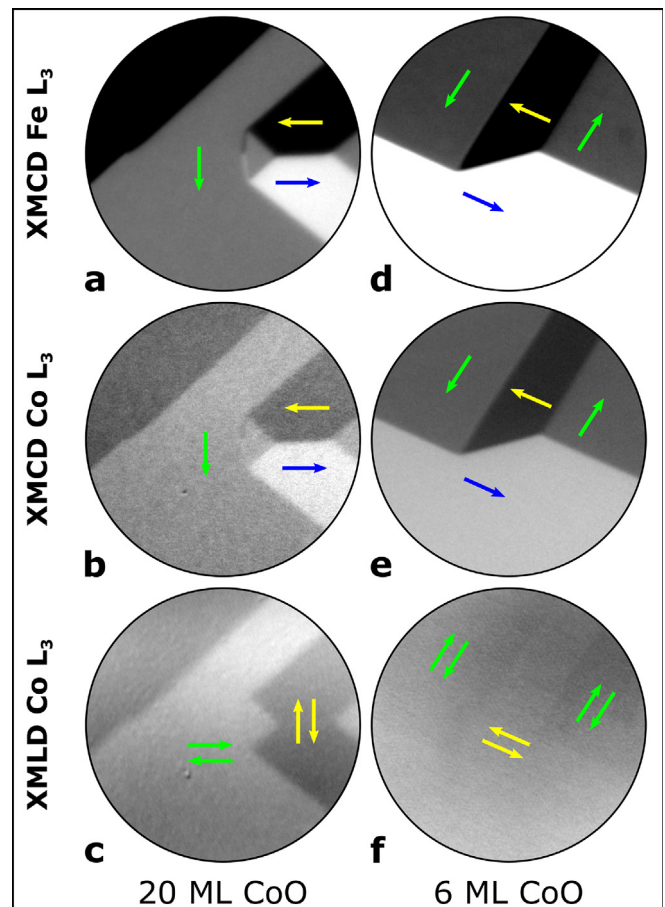


**Fig. 2.** Panel a: XAS spectra at the Fe  $L_3$  absorption edge on CoO/Co/Fe samples with different CoO thickness  $t_{\text{CoO}}$  (dashed black curve:  $t_{\text{CoO}} = 20$  ML; continuous red curve  $t_{\text{CoO}} = 6$  ML). Panels b and c: XAS spectra at the Co  $L_3$  absorption edge for the same samples, taken with orthogonal linear polarizations (Hor, black line, in-plane polarization; Vert, red line, out-of-plane polarization) and XMLD spectra (blue curves) calculated from the corresponding XAS spectra. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

We remark that, even if the Néel temperature ( $T_N$ ) of CoO is very close to RT for bulk samples ( $T_N = 291$  K), it can be enhanced for very thin films in proximity to FM layers [22]. It is therefore not surprising to observe an AF order even in very thin CoO films. These observations suggest, on one side, that the presence of a long range magnetic order in the CoO films can be due to the magnetic coupling with the adjacent FM layers and, on the other side, that such an order is not related to the onset of the interface magnetic anisotropy. In fact, we observe an AF order even in samples characterized by the presence of Fe oxides that, correspondingly, do not show any magnetic anisotropy.

The relationship among the magnetic properties of the substrate, the CoO film and the residual Co at the interface can be investigated by imaging the magnetic domains configuration through XMCD-PEEM and XMLD-PEEM. Representative magnetic domains images related to the same samples discussed in Fig. 2 are now reported in Fig. 3. Both XMCD-PEEM at the Fe  $L_3$  (panels a and d) and Co  $L_3$  (panels b and e) absorption edges are shown, as well as XMLD-PEEM at the Co  $L_3$  absorption edge (panels c and f). It is worth to notice that an XMLD signal could be due also to the FM Co ultrathin layer located at the interface. In order to be sure of retrieving pieces of information related to CoO alone, the XMLD-PEEM imaging was performed with a photon energy of 774.7 eV. Such an energy corresponds to the small peak indicated by vertical dashed lines in Fig. 2b and c. That peak is part of the multiplet splitting related to CoO [27] and is not present in XAS spectra of metallic Co [23]. XMCD-PEEM at the Co  $L_3$  edge was instead performed at a photon energy of 776.0 eV.

For both cases considered in Fig. 3, the magnetic domains were seen to extend over hundreds of  $\mu\text{m}$  and formed smaller structures close to domain walls. Some of such structures are seen in the reported images, which are characterized by a field of view of  $20\mu\text{m}$ . The X-ray beam impinges from the right, horizontally. The arrows in Fig. 3 give the interpretation of the magnetization direction in the domains. The different mounting directions of the samples are responsible of the direction of the domains magnetization with respect to the X-ray incidence direction, as seen in the images. The direction of  $\mathbf{M}$  in each domain is derived by comparing the domains gray scale. Brighter domains correspond, in particular, to  $\mathbf{M}$  parallel to the wave vector, while darker ones correspond to antiparallel domains with respect to the



**Fig. 3.** XMCD-PEEM and XMLD-PEEM images taken at the reported absorption edges for CoO/Co/Fe samples with CoO thickness  $t_{\text{CoO}} = 20$  ML (panels a–c) and  $t_{\text{CoO}} = 6$  ML (panels d–f). The field of view is  $20\mu\text{m}$  in all cases.



former ones. For what concerns intermediate gray scales, it is only possible to infer that they are oriented at  $90^\circ$  with respect to the other ones. The proposed directions were chosen to be consistent with those of the adjacent domains, i.e. by orienting the magnetic moments in order to avoid unfavorable head-to-head domains.

A direct comparison between magnetization reversal behavior and magnetic domain configuration is not possible due to the very different nature of the experiments. In the former case, the hysteresis loops are the average of tens of measurements and are not sensitive, therefore, to the particular reversal process occurring in each portion of a single loop. In the latter case, instead, the XMCD/XMLD-PEEM measurements are performed under a static magnetic field. It is therefore very difficult to distinguish between easy-easy and easy-hard axis in those experiments. In fact,  $90^\circ$  domains were seen to occur when applying  $H$  along different easy axes, even if they were apparently (i.e., without a statistically relevant certainty) easier to nucleate when the field was applied along one easy axis rather than along the other one.

The interesting evidences provided by the XMCD/XMLD-PEEM experiments are instead related to the relationships between the domains configuration for each component. It is in fact clear that, in both samples, the shape of the domains and the associated direction of  $\mathbf{M}$  mimic each other for the different layers. The comparison between the gray scales in the XMLD images and those in the corresponding XMCD ones allows us to conclude that the domains in the thicker CoO film are oriented at  $90^\circ$  in plane with respect to those of the underlying FM layers. The other way around occurs for the thinner CoO film, where the directions of  $\mathbf{M}$  in the AF are the same as those in corresponding domains of the FM substrate. These results are in very good agreement with previous ones, obtained for samples grown without the Co buffer layer [18], and seem thus to be independent of the presence of Fe oxides at the interface.

Therefore, for both thicknesses and independently of the presence of Fe oxides at the interface, the CoO films show a long range AF order, with domains clearly driven by those of the FM layers to which they are coupled (this is seen less clearly in Fig. 3f, on account of the low thickness of the CoO layer: the comparison with the corresponding XMCD-PEEM images help in observing the AF domains). The same observations were confirmed also for other samples in similar conditions. Therefore, we conclude that the ultrathin Co layer present at the CoO/Fe interface is FM and that its magnetization vector is always parallel to that of the Fe substrate. Also, the AF domains in CoO have magnetic moments directed either along or perpendicular to those of the FM counterparts.

#### 4. Conclusions

By combining the experimental observations described in this work, we can conclude that the magnetic anisotropy observed in CoO/Fe(001) layered structures prepared by exploiting a Co buffer layer is indeed an interface-related effect. We observe the occurrence of an AF order even in very thin CoO layers, with a magnetic domains configuration that mimics that of the FM components, which are in turn equal to each other. Although we can not, from our measurements, exclude a possible tilting of the AF moments with respect to the FM ones, such a mimicking testifies that the AF moments always rotate together with the FM ones upon field reversal and that the relative direction of the magnetic moments in corresponding FM and AF domains is the same in all cases, without any anisotropic behavior.

The MSH measurements suggest that the origin of the magnetic anisotropy resides at the AF/FM interface, likely on account of the presence of pinned magnetic moments. Given the behavior of the AF domains, in particular the fact that both the occurrence of the magnetic order and the correspondence of the domains shape between AF and FM layers does not seem to depend on the presence of Fe oxides, the observations lead to the conclusion that such pinned moments might not have a magnetic origin but could more probably be related to a

magnetic frustration induced by the dislocations developed at the interface [28]. The latter might develop in an anisotropic way on account of possible anisotropies related to the preparation procedure (e.g., on account of the sputtering direction), which remain hidden by stress accumulation in the substrate and are released upon CoO deposition. The misfit dislocations formerly observed by scanning tunneling microscopy appear anyway to be evenly distributed in the sample [8]. On the other hand, that kind of measurements are only related to the surface and are focused on regions of extremely small spatial extent; further measurements will then be needed in order to investigate the possible correlation between the structural and magnetic properties of these layered magnetic structures.

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