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Magnetic anisotropy at the buried CoO/Fe interface

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(Dated: 18 October 2016)

Interfaces between antiferromagnetic CoO and ferromagnetic Fe are typically characterized by the development of Fe oxides. Recently, it was shown that the use of a proper ultra-thin Co buffer layer prevents the formation of Fe oxides [A. Brambilla, A. Picone, D. Giannotti, M. Riva, G. Bussetti, G. Berti, A. Calloni, M. Finazzi, F. Ciccacci, L. Duò, Appl. Surf. Sci. **362**, 374 (2016)]. In the present work we investigate the magnetic properties of such an interface and we find evidence for an in-plane uniaxial magnetic anisotropy, which is characterized by a multijump reversal behavior in the magnetization hysteresis loops. X-ray photoemission spectroscopy and element-sensitive hysteresis loops reveal that the occurrence of such an anisotropy is a phenomenon developing at the very interface.

Interfaces between ferromagnetic metals (FM) and oxide (O) films are ubiquitous in fields such as spintronics,[1,2] electronics,[3] and multiferroics.[4,5] The smaller the physical structures get, the more relevant interfaces become in determining their properties. This is true also for magnetic systems, where interface phenomena, such as exchange bias, have always been playing a major role,[6] and now are keys to obtaining an atomic-scale engineering of relevant magnetic features.[7,8] In the case of O/FM, several recent observations demonstrated how the interface chemical interactions directly influence the magnetic properties by, for instance, introducing uncompensated magnetic moments,[9] enhancing the coupling effects,[10] or even by creating such effects in unexpected ways, like the case of the nominally not exchange biased MgO/Fe interface.[11]

Among O/FM systems, those containing antiferromagnetic (AF) oxides have been the subject of a great number of both experimental and theoretical investigations.[12–16] The preparation conditions, as well as the growth order, are of great importance to understand and control their magnetic properties.[17] In particular, the CoO/Fe(001) interface has been a workbench for many investigations related to AF/FM systems with reactive interfaces, ranging from the exchange bias mechanism,[14,18] to the influence of structure and stoichiometry on the magnetic properties,[19] to the realization of peculiar magnetic configurations, like vortices.[20]

Very recently, we have demonstrated that it is possible to prepare a CoO/Fe(001) interface characterized by the absence of any Fe oxide,[21,22] which is a quite unique feature with respect to the common experimental situations.[10,19,23] This result has been accomplished by exploiting an ultra-thin Co buffer layer with a bct cubic structure.[21,24] In that case, the CoO films were also characterized by a well-ordered mesa mound morphology for CoO thicknesses above a few monolayers (ML), occurring on account of stress relaxation through the formation of a network of misfit dislocations.[25,26] In this Letter, we explore the magnetic properties of such an interface and we report on the occurrence of a uniaxial magnetic anisotropy that gives rise to multijump magnetic hysteresis loops. By exploiting an existing reversal model, we estimate the value of such an anisotropy and discuss about its possible physical origin. While the latter appears difficult to address, it is noticeable that, by following the correct preparation procedure, one can reproducibly obtain a magnetic anisotropy that may be used for settling a reference direction in the magnetic system, without exploiting exchange bias.

Ultra-thin films of CoO were grown on Fe(001) after deposition of a 5 ML of bct Co (1 bct Co ML = 0.140 nm)[27] onto the oxygen-saturated Fe(001)-$p(1 \times 1)$O surface of a 500 nm-thick Fe film, as described in Ref. 21. Subsequent annealing at 470 K for 5 minutes produces the Co(001)-$p(1 \times 1)$O surface,[24] characterized by a single atomic layer of chemisorbed oxygen, while iron at the interface remains free of oxygen. Reactive deposition of CoO, with a thickness t_{CoO} up to about 25 ML (1 CoO ML = 0.213 nm), was performed with the sample kept at 470 K by evaporating metallic Co in a pure O_2 atmosphere with partial pressure $p_{O_2} = 1 \cdot 10^{-4}$ Pa.[21]

Magnetic hysteresis loops were acquired at room temperature on samples characterized by $t_{CoO} = 25$ ML with the Magneto-Optical Kerr Effect (MOKE) apparatus described in Ref. 28 and are reported in Fig. 1, together with a sketch of the experimental layout. Making reference to the latter, **H** was the applied magnetic field and ϕ was 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 ϕ. The hysteresis loops shown in Fig. 1 are related to the $M_{\parallel}$ and $M_{\perp}$ components of the total in-plane magnetization **M**, which are either parallel or perpendicular, respectively, to the direction of **H**.

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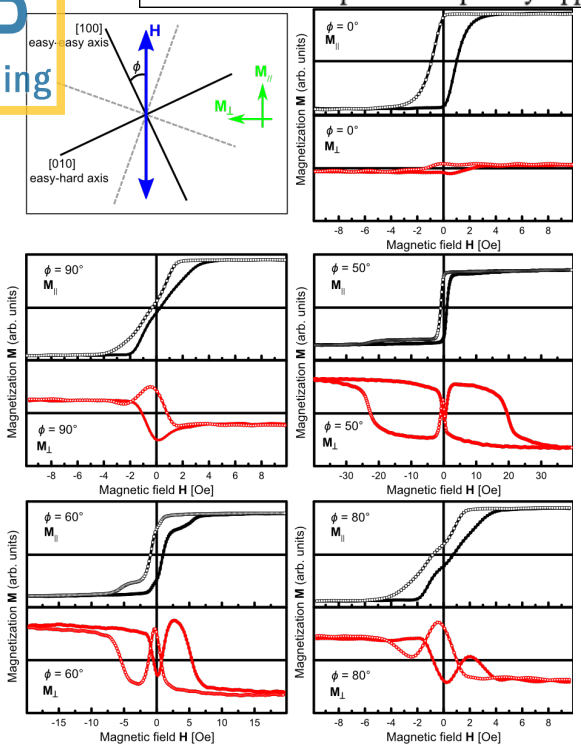

FIG. 1. MOKE hysteresis loops of the in-plane magnetization components either parallel (∥) or perpendicular (⊥) to the applied field **H**, taken for different azimuthal angles ϕ. Hollow and full symbols correspond to either decreasing or increasing loop branches. The sketch describes the experimental layout, where the continuous lines represent the sample easy axes and the dashed lines the hard axes. The measurements are taken on a sample with $t_{CoO} = 25$ ML.

Bare Fe(001) films are characterized by two equivalent easy magnetization axes, each coincident with the two in-plane equivalent [100] and [010] directions. Typical hysteresis loops acquired on Fe(001)-$p(1\times1)$O substrates (not shown) are like, e.g., those shown for $\phi = 0°$ ([100] // **H**) in Fig. 1. We underline that the small $M_\perp$ component here observed is due to a small misalignment (less than 5°) between **H** and the [100] direction of Fe.

In presence of the CoO film with $t_{CoO} = 25$ ML, the two easy axes turned out to be no longer equivalent. In particular, the hysteresis loops reported for $\phi = 90°$ ([010] // **H**) in Fig. 1 feature a 2-jump switching behavior of the magnetization at two different coercive fields, that are better visible separately in the $M_\parallel$ and $M_\perp$ components, suggesting the formation of 90° domains during the reversal. Such domains are parallel to the Fe [100] axis, meaning that such an axis is magnetically easier than the [010] axis. Further rotations of the ϕ angle reveal a change in the reversal behavior when crossing the hard axes. In particular, for angles $0° < \phi < 45°$ and $135° < \phi < 180°$ the magnetization reversal follows a 1-jump 180° switching (not shown). For angles $45° < \phi < 135°$ (excluding $\phi = 90°$), the reversal is instead characterized by a 3-jump switching route, better visible in the $M_\perp$ loops, as shown for some cases ($\phi = 50°, 60°$, and 80°) in Fig. 1. This reversal behavior is therefore symmetric with respect to the harder easy axis. It is interesting to notice that, in the 3-jump loops, the transition occurring first (i.e. the one for which the lower energy barrier has to be overcome) is always seen before the field switches sign. This can be observed in Fig. 1 by considering separately the decreasing and increasing loop branches, which are represented by either hollow or full symbols, respectively. In all measured 3-jump loops, such a transition is better evidenced in the $M_\perp$ component and it occurs at substantially the same field, independently of ϕ, of about 0.8 Oe (average absolute value).

Such a multijump magnetic switching behavior was formerly observed by Cowburn et al.[29] in Ag/Fe/Ag(001) films with ultra-thin Fe layers (0 to 13 ML range) and described, by the same authors, on the basis of a model that considers well-defined domain wall (DW) pinning energies separating energy minima for the magnetization configuration. The latter were calculated in a coherent rotation approach by superimposing a weak uniaxial anisotropy term K_u to the fourfold in-plane cubic magnetic anisotropy term K_1.[30] The reversal then occurs by DW displacement, in which the DW are allowed to propagate freely once the corresponding pinning energy (ϵ_{90} for a 90° DW, ϵ_{180} for a 180° DW) is overcome. In particular, the condition $K_u > \epsilon_{90}$ has to be fulfilled in order to observe 3-jump loops. In the present case, a value of $\epsilon_{180}/M = 2$ Oe, where M is the saturation magnetization, can be retrieved by measuring the coercive field H_c in the standard loops with either $\phi = 0°$ or $\phi = 180°$. From such a value one obtains $\epsilon_{90}/M = 1$ Oe, following the relation $\epsilon_{180} = 2\epsilon_{90}$ discussed in Ref. 29. Finally, these values can be used, together with the values of the lowest coercive field measured in each 3-jump loop, to obtain an estimation of the uniaxial anisotropy, yielding $K_u/\epsilon_{90} = 1.7 \pm 0.6$. This result, which describes a very small anisotropy, compares well with that obtained by Cowburn et al. ($K_u/\epsilon_{90} = 1.3 \pm 0.1$), even if our Fe films are two orders of magnitude thicker than those considered in the reference work, thus showing with even greater emphasis how big the influence of such a small anisotropy can be on the reversal behavior of a FM film.

Multijump magnetization reversal behaviors were only seldom observed, mainly on systems prepared on purpose by exploiting, e.g., vicinal surfaces,[31] ion bombardment,[32,33] oblique evaporation.[34] On the other hand, it was recognized that the development of a uniaxial anisotropy in Fe films and the mechanism of DW formation and propagation are effects related to the interface.[35,36] We underline that the magnetization loops reported in Fig. 1 are strongly dependent on the sample preparation recipe. Preparation approaches different from that described in Ref. 21 (in particular different growth temperatures), produced samples that did not show multijump magnetic hysteresis loops.

To go into further details about the role of the interface chemistry, we report in Fig. 2 X-ray Photoemission Spec-

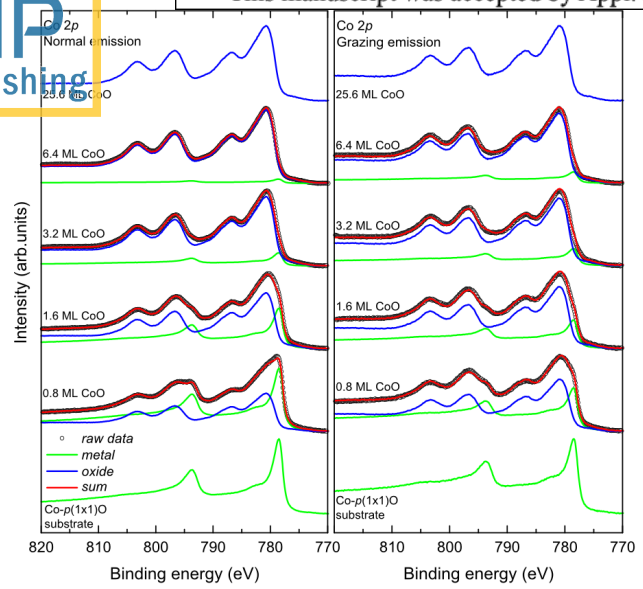

FIG. 2. XPS spectra taken at either normal (left panel) or grazing (60° off-normal, right panel) emission. From bottom to top the spectra correspond to increasing CoO thickness, from Co-$p(1\times1)$O (no CoO, metal lineshape reference) to t_{CoO} = 25.6 ML (oxide reference).

troscopy (XPS)[37] measurements, taken at either normal or grazing (60° off-normal) emission, on the Co 2p core level region, as a function of t_{CoO}. The reference experimental spectra are taken on the Co(001)-$p(1\times1)$O substrate (bottom row, green line) and on a film with t_{CoO} = 25.6 ML (upper row, in blue), for which the signal from the substrate can not be detected anymore by XPS and that perfectly agrees with literature.[25] The spectra with intermediate t_{CoO} values were then deconvoluted as a combination of metallic and oxide components. Confirming what previously discussed,[21] the Co buffer layer oxidizes during CoO growth and, at a thickness t_{CoO} = 6.4 ML, only a tiny metallic contribution is still visible in the spectra. Surprisingly, the latter appears to be greater for the grazing emission spectra (right panel) than for the normal emission ones. Such an observation might be explained by considering the presence of under-coordinated Co atoms, whose component in the XPS lineshape is very similar to that of metallic Co.[38] This may be understood when considering the peculiar three-dimensional morphology of the topmost layers of our CoO/Fe samples.[21]

In order to determine if the residual metallic Co has an influence on the magnetic anisotropy landscape of our samples, we performed element-dependent hysteresis loops by using X-ray Magnetic Circular Dichroism (XMCD) at the APE beamline of the Elettra synchrotron.[39] In particular, x-ray absorption spectra were acquired at both Fe and Co $L_{2,3}$ absorption edges. The energy of the photon beam was fixed, first at the maximum value of the respective XMCD spectrum of either Fe or Co, then at the corresponding pre-edges (for the sake of signal normalization), and the absorption signal was collected, for both photon helicities, while varying the external applied field **H**. Fig. 3 reports the element-dependent hysteresis loops obtained at different t_{CoO} values and also for the Co(001)-$p(1\times1)$O substrate. Here, **H** was oriented along one of the easy magnetization axes of the sample and both field and sample were jointly rotated to be either in the incidence plane of the x-ray beam, or perpendicular to it. The obtained components of the magnetization vector can thus be labeled as $M_{\parallel}$ (parallel to **H**) and $M_{\perp}$ (perpendicular to **H**) with the same labeling adopted in Fig. 1.

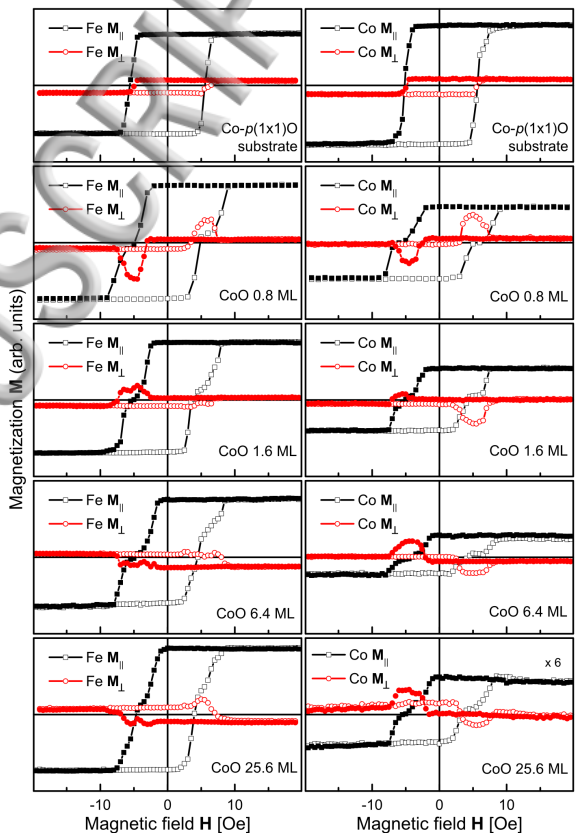

FIG. 3. XMCD hysteresis loops acquired at either Fe or Co absorption edges for different CoO thicknesses. The parallel and perpendicular components of the in-plane magnetization **M** are referred to the external field **H**.

In the Co(001)-$p(1\times1)$O case, we observe that the reversal behavior of Co spins is totally equivalent to that of Fe spins, suggesting a strong parallel coupling between the two layers. A substantially equivalent reversal behavior of Fe and Co is retrieved also for t_{CoO} = 0.8 ML. Moreover, a FM behavior of Co is observed also at larger coverages, even for t_{CoO} as high as 25.6 ML. The latter observation also demonstrates that a small fraction of metallic Co remains at the interface upon growth of a thick CoO layer, indicating that the process of the buffer layer oxidation stops at some coverage, before being complete. As reported in the bottom of Fig. 3, the XMCD spectrum at the Co edge for t_{CoO} = 25.6 ML had to be

multiplied by 6 in order to be comparable to the other spectra. This suggests that most of the magnetically active unreacted Co is located close to the interface.

The XMCD loops of CoO/Fe samples clearly show the reversal through formation of 90° domains, as evidenced by the $M_\perp$ component in the loops and by the clear 2-jump behavior visible in both magnetization components. This means that the configuration reported in Fig. 3 is analogous to the case $\phi = 90°$ of Fig. 1. Most unexpectedly, the same 2-jump behavior is observed also for t_{CoO} as low as 0.8 ML. It is worth noticing that XMCD loops had to be taken at different times for Fe and Co, which explains the differences in the shape of corresponding loops.

The above observations demonstrates that the uniaxial anisotropy is present since the early stages of CoO growth, fostering the conclusion that neither the initial metastable Co layer nor the residual Co species observed at higher t_{CoO} are at its source. Moreover, the observation of reversal through formation of 90° domains even before a complete CoO layer is formed suggests that the onset of an AF order is also not at the origin of K_u.

Further considerations can be made about a possible role of the misfit dislocation network that rapidly forms upon CoO deposition, as shown by scanning tunneling microscopy (STM).[21] Even in this case, STM demonstrated that the dislocations develop only after the first CoO layer is formed, while they were not observed for sub-monolayer coverages, thus pointing towards excluding them as the origin of the anisotropic magnetic behavior. Similarly, other topographic features, and in particular the terrace-steps distribution of the surface, described in Ref. 40, were observed to be substantially equivalent when the samples were prepared either with or without the Co buffer layer, implying that the substrate morphology is also not responsible for the magnetic anisotropy.

In conclusion, we have shown that a CoO/Fe(001) interface characterized by the absence of any Fe oxide develops a weak uniaxial magnetic anisotropy that is anyway sufficient to break the in-plane fourfold magnetic symmetry of a thick Fe layer, even in absence of an exchange bias effect. The presence of residual metallic Co species in the CoO film was shown to have no influence on such an anisotropy, which appear to be a clear interface phenomenon. This work demonstrates that a fine control of the oxidation/reduction processes occurring at a reactive AF/FM interface may be the key for the control of its large scale magnetic properties. We believe therefore that our system may be exploited in properly designed nanostructures with very small dimensions.

ACKNOWLEDGMENTS

Fondazione Cariplo is gratefully acknowledged for financial support through grants 2011-0389 and 2013-0623. Measurements at the Elettra synchrotron of Trieste (Italy) were allowed by Proposal No. 20140509.

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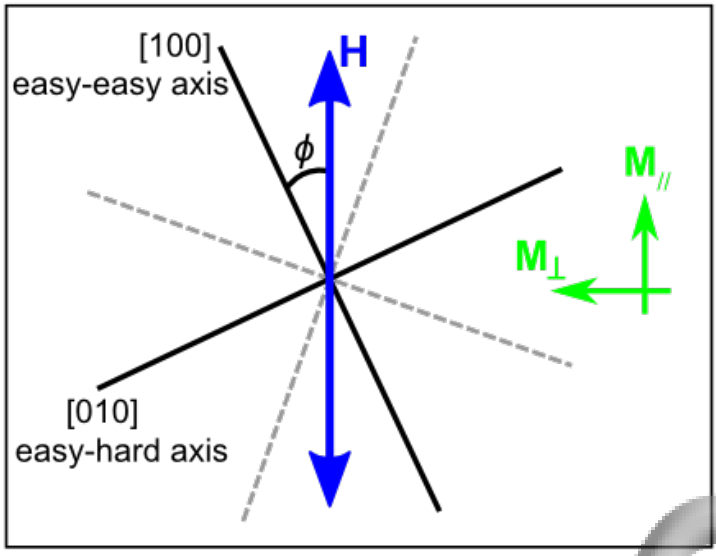
[100]
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H
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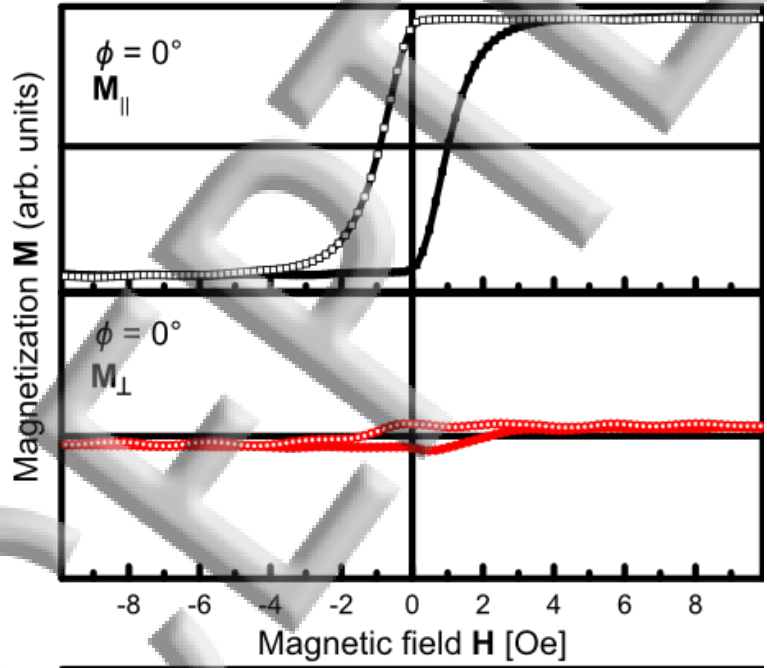
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M||
ϕ = 0°
M⊥
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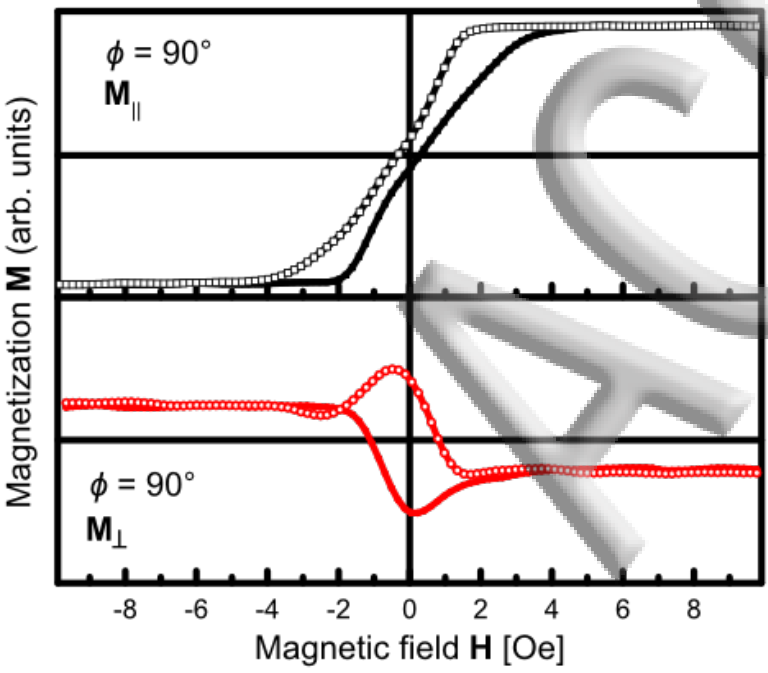
Magnetic field H [Oe]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M||
ϕ = 90°
M⊥
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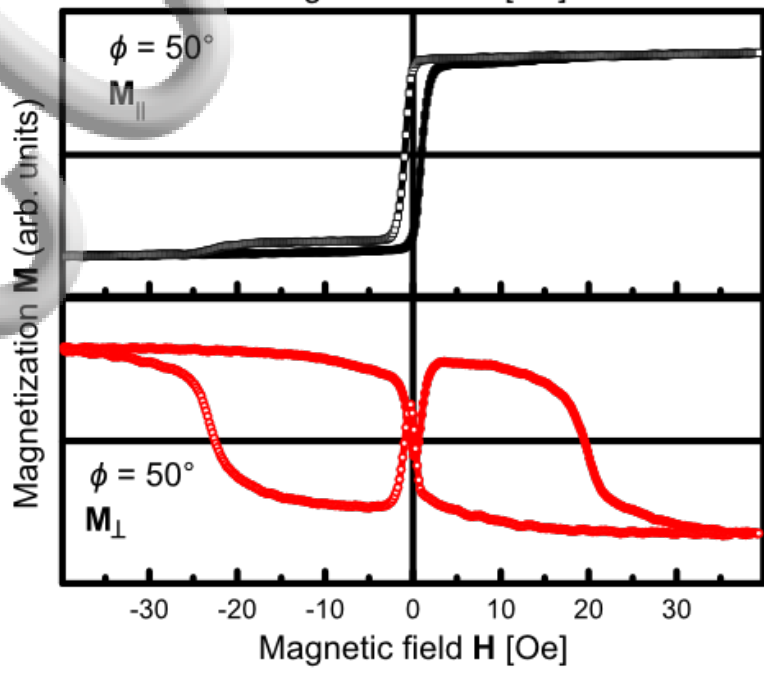
Magnetic field H [Oe]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M||
ϕ = 50°
M⊥
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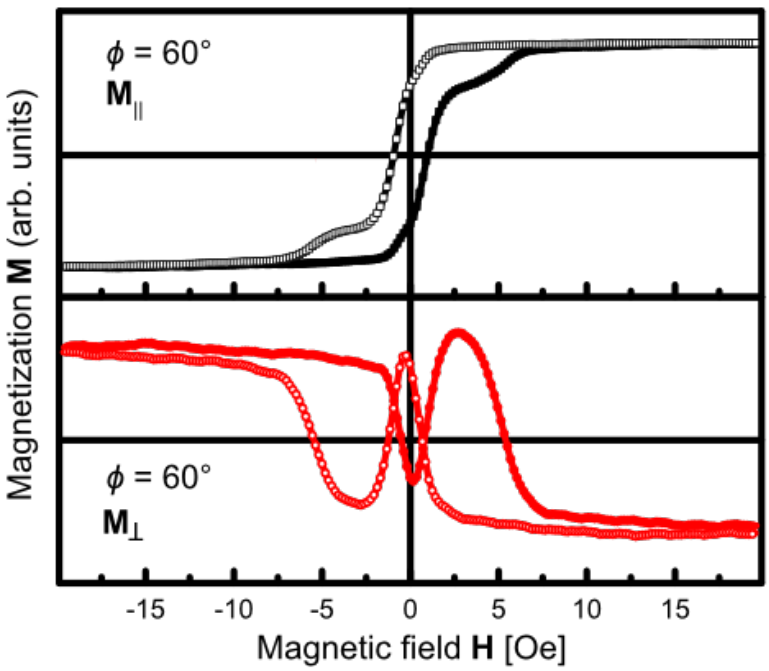
Magnetic field H [Oe]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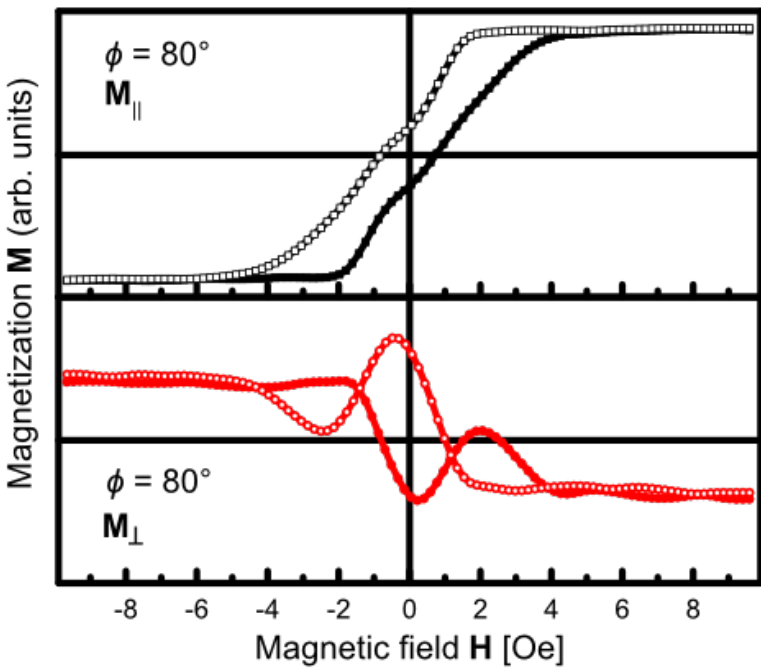
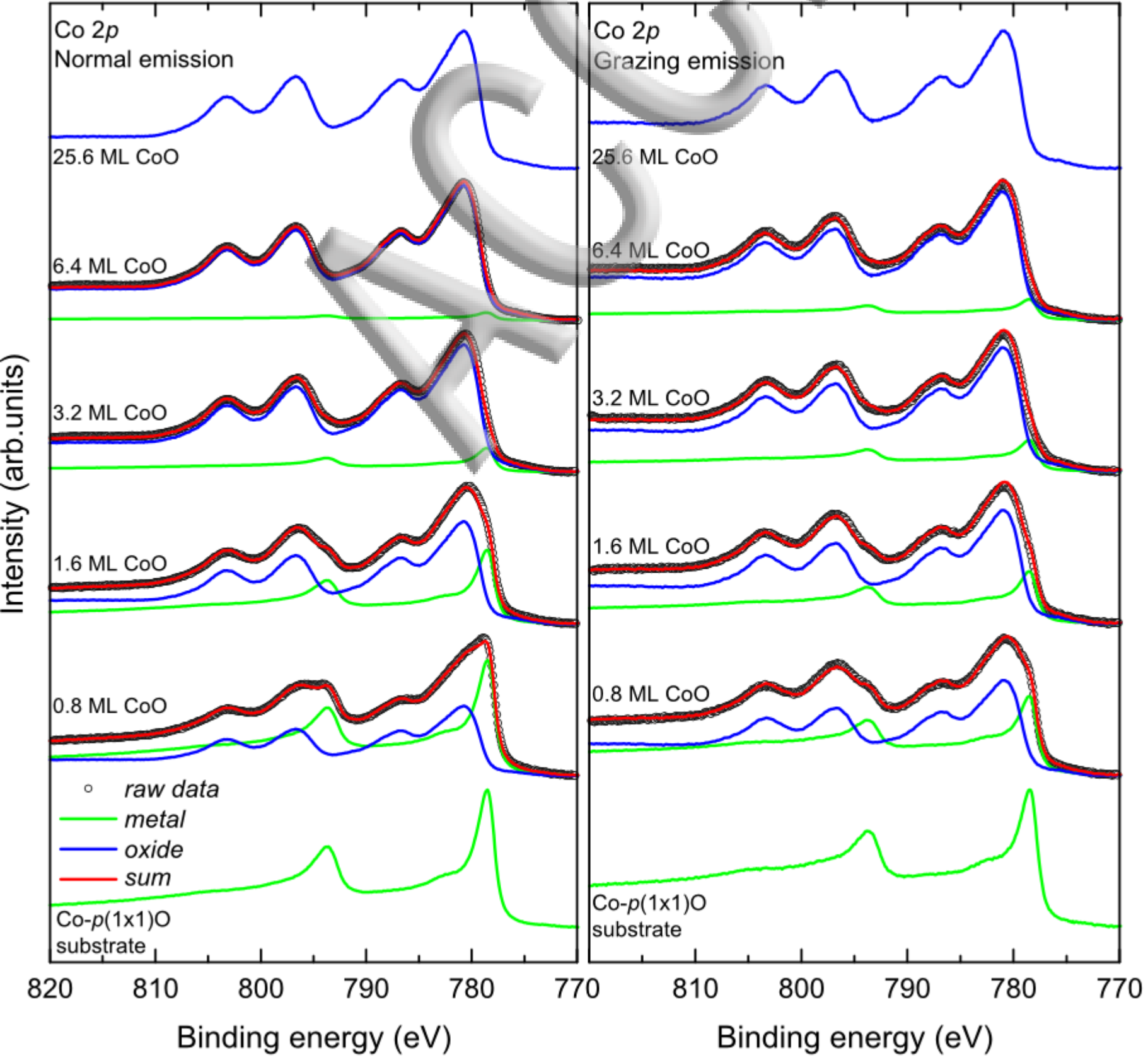
-15 -10 -5 0 5 10 15
Magnetic field H [Oe]


ϕ = 80°
M||
ϕ = 80°
M⊥
Magnetization M (arb. units)
-8 -6 -4 -2 0 2 4 6 8
Magnetic field H [Oe]


Co 2p
Normal emission
25.6 ML CoO
6.4 ML CoO
3.2 ML CoO
1.6 ML CoO
0.8 ML CoO
raw data
metal
oxide
sum
Co-p(1x1)O
substrate
Co 2p
Grazing emission
25.6 ML CoO
6.4 ML CoO
3.2 ML CoO
1.6 ML CoO
0.8 ML CoO
Co-p(1x1)O
substrate
Intensity (arb.units)
820
810
800
790
780
770
Binding energy (eV)
810
800
790
780
770
Binding energy (eV)