Depth profiling of magnetic and atomic structures of ultrathin films by depth-resolved XMCD and XAFS techniques with a sub-nm depth resolution

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Abstract. Recent progresses in the depth-resolved X-ray magnetic circular dichroism (XMCD) and X-ray absorption fine structure (XAFS) techniques are presented. The depth-resolved atomic and magnetic structures of Co films grown on Ru(0001) was investigated by using the depth-resolved XMCD and extended X-ray absorption fine structure EXAFS. The three-dimensional magnetic structure of a micro-patterned Fe/Ni/Cu(100) film was studied by combining the depth-resolved XMCD with the X-ray microbeam. The technique was also applied to Co thin films covered with a relatively thick Mo overlayer, in order to demonstrate the possibility to investigate ex-situ samples with a protection layer.

1. Introduction
The depth profile of magnetic ultrathin films has attracted much interest because the surface and interface play important roles for the magnetic property of the whole film. In order to investigate the depth profile of ultrathin films, we have developed a depth-resolved technique based on X-ray magnetic circular dichroism (XMCD) and X-ray absorption fine structure (XAFS) spectroscopies [1-9]. We have studied the depth profile of magnetization in 3d transition metal films grown in-situ on Cu(100) [1-3,5,6], as well as the effects of gas adsorption on the films [4,7,9].

Recently, we have made some progresses on this technique. First, we tried to apply this technique to the extended X-ray absorption fine structure (EXAFS), in order to investigate depth-resolved atomic structure. Second, we have developed a three-dimensional XMCD/XAFS technique by combining the depth-resolved technique with the X-ray microbeam. Third, magnetic thin films...
covered with a protection layer such as Au was investigated, to which the in-situ measurements are not necessary [8]. In this contribution, the recent progresses in the depth profiling of the magnetic and atomic structures of ultrathin films are presented after a brief introduction to the depth-resolved technique.

2. Experiments

2.1. Depth-resolved XMCD/XAFS measurements

2.1.1. Principle

In the depth-resolved XMCD/XAFS technique, the probing depth of the spectrum is determined by the effective escape depth, $\lambda$, of the electrons, which depends on the electron emission angle. The experimental layout of the depth-resolved XMCD/XAFS measurement is schematically illustrated in figure 1. The electrons emitted after X-ray absorption were separately collected at different detection angles, $\theta_d$, by using an imaging type detector, which consists of a microchannel plate, a phosphor screen, and a CCD camera. A partial electron yield mode with a retarding voltage of 500 V was adopted, so that the Auger electrons were mainly collected. The effective electron escape depth, $\lambda$, was experimentally determined at each $\theta_d$ from the thickness dependence of the edge-jump intensity of the films [1]. One can investigate the depth profile of the magnetic and atomic structures of ultrathin films with a sub-nm resolution, by analyzing a set of XMCD/XAFS data recorded at different probing depths.

The spectra were recorded at normal and grazing x-ray incidences (0° and 60° from normal) if necessary, e.g., to study magnetic anisotropy. In the XMCD experiments, the sample was magnetized with a yoke coil (~500 Oe) and then the coil was retracted out during the measurement. Accordingly, the remanent magnetization was examined. All the measurements with a standard beam size were performed at the bending magnet (BL-7A and 11A) and undulator (BL-16A) soft x-ray stations in the Photon Factory [10,11]. The three-dimensional XMCD measurements, in which the depth-resolved XMCD technique is combined with X-ray microbeam, were performed at BL23SU in SPring-8 [12]. A 3-5 μm X-ray beam was obtained by using a two-step Kirkpatrick-Baez focusing system, which has been recently developed [13].

![Figure 1. Schematic layout for the depth-resolved XMCD/XAFS measurements in the normal (a) and grazing (b) incidence configurations.](image)
2.1.2. Definition of the probing depth

Figure 2(a) illustrates the attenuation of the electrons emitted at depth $Z$ from the surface, which can be expressed as an exponential function, $\exp[-Z/\lambda]$. Owing to the element specificity of the XMCD/XAFS technique, we only observe the signals from the Co thin film, in this example, as shown in figure 2(a) by bars. It is clearly recognized that the depth dependence of the Co signal is more prominent at smaller $\lambda$. Therefore, the XMCD/XAFS spectrum obtained at smaller $\lambda$ contains more contribution from the top Co layer, and is more surface sensitive.

It should be noted here that one can normalize the XMCD/XAFS spectra using the edge-jump intensity, which is independent of the magnetic, electronic and atomic structures. Consequently, the X-ray absorption intensity per Co atom is obtained. Therefore, the relative ratio of the signal intensity from each Co layer is essential in the analyses of the depth-resolved XMCD/XAFS data. The contribution of the signal from $i$th Co layer to the normalized XMCD/XAFS spectrum is given by

$$\frac{\exp[-(i-1)d_{Co}/\lambda]}{\sum_{j=1}^{\lambda} \exp[-(j-1)d_{Co}/\lambda]},$$ \tag{1}

where $d_{Co}$ represent the layer spacing for the Co layers. The calculated signal ratio is depicted in figure 2(b). A set of the XMCD/XAFS spectra with different $\lambda$ are normalized and analyzed by assuming the contribution from each layer according to eq. (1).

Figure 2. Exponential decay function for the emitted electrons (solid curves) and relative intensity of the Co signal from depth $Z$ (bars) in Co(5 ML)/Au(5 ML) (a), and ratio for the observed Co signal originated at depth $Z$ (b). Effective electron escape depth, $\lambda$, is assumed to be 0.5, 1.0, and 1.5 nm.

Next, let us consider that the sample is covered with some overlayer, e.g. Au(5 ML)/Co(5 ML)/Au(5 ML). In this case, the Co signal is reduced by the electron attenuation by the overlayer as shown in figure 3(a). Again, the signal ratio for each Co layer is essential, because the obtained XMCD/XAFS spectrum is normalized in the “per Co atom” basis. Since the signal intensity emitted
from the Co atom at depth \( z_{\text{Co}} \) from the top Co layer is expressed by \( \exp[-(5d_{\text{Au}} + z_{\text{Co}})/\lambda] \), the contribution of the signal from \( i \) th Co layer to the normalized XMCD/XAFS spectrum is given by

\[
\frac{\exp[-(5d_{\text{Au}} + (i-1)d_{\text{Co}})/\lambda]}{\sum_{j=2}^{\infty} \exp[-(5d_{\text{Au}} + (j-1)d_{\text{Co}})/\lambda]},
\]

where \( d_{\text{Au}} \) represents the layer spacing for the Au layer. The calculated signal ratio is depicted in figure 3(b). Since eq. (2) can be rewritten as

\[
\frac{\exp[-5d_{\text{Au}}/\lambda]\exp[-(i-1)d_{\text{Co}}/\lambda]}{\sum_{j=1}^{\infty} \exp[-5d_{\text{Au}}/\lambda] \sum_{j=1}^{\infty} \exp[-(j-1)d_{\text{Co}}/\lambda]} = \frac{\exp[-(i-1)d_{\text{Co}}/\lambda]}{\sum_{j=1}^{\infty} \exp[-(j-1)d_{\text{Co}}/\lambda]},
\]

the intensity ratio for the Co layer is independent of the overlayer thickness, as can be recognized from eqs. (1) and (2) and figures 2(b) and 3(b), and determined solely by the layer number, \( i \), from the top Co layer and the effective electron escape depth, \( \lambda \). Therefore, we hereafter denote \( \lambda \) as the probing depth of the XMCD/XAFS spectrum, regardless of the existence of the overlayer.

**Figure 3.** Exponential decay function for the emitted electrons (solid curves) and relative intensity of the Co signal from depth \( Z \) (bars) in Au(5 ML)/Co(5 ML)/Au(5 ML) (a), and ratio for the observed Co signal originated at depth \( Z \) (b). Effective electron escape depth, \( \lambda \), is assumed to be 0.5, 1.0, and 1.5 nm.
2.2. Sample preparation
All the samples were prepared in an ultrahigh-vacuum chamber, whose base pressure was $3 \times 10^{-8}$ Pa, according to the following procedures.

2.2.1. Co/Ru(0001)
A Ru(0001) single crystal was cleaned by repeated cycles of Ar$^+$ sputtering (2 keV) and subsequent annealing at ~1470 K. Co films were deposited on Ru(0001) at the substrate temperature of 363 K with the electron bombardment evaporation of a Co rod. The thickness of the Co films was controlled by monitoring the oscillatory intensity of a reflection high-energy electron diffraction (RHEED) spot. The Ru-covered samples were also prepared by subsequently evaporating Ru at the substrate temperature of 363 K with the electron bombardment evaporation of a Ru rod. The Ru deposition rate was calibrated by a homoepitaxial growth on Ru(0001) prior to the sample preparation.

2.2.2. Fe/Ni/Cu(100)
A Cu(100) single crystal was cleaned by repeated cycles of Ar$^+$ sputtering (1.5 keV) and subsequent annealing at ~900 K. A 6 ML (monolayer)-thick Ni film was uniformly deposited on Cu(100) at room temperature with the electron bombardment evaporation of a Ni rod. The thickness of the Ni film was controlled by monitoring the oscillatory intensity of a RHEED spot. A micro-patterned Fe thin film was subsequently deposited by putting a 200×40 μm$^2$ slit in front of the sample. The Fe evaporation was carried out by the electron bombardment evaporation of a Fe rod, and the deposition rate was calibrated by a growth on Cu(100) prior to the sample preparation.

2.2.3. Mo/Co/Au/Mo/Al$_2$O$_3$(11-20)
A 20 nm-thick Mo buffer layer was deposited on an Al$_2$O$_3$(11-20) single crystal at the substrate temperature of ~1270 K, by the electron bombardment evaporation from a Mo rod. A 10 nm-thick Au(111) substrate was subsequently prepared at room temperature by resistively heating a W filament covered with Au wires, followed with substrate heating to ~470 K for 30 min. Then a wedge-shaped Co layer (~0.5 nm/mm) was deposited at room temperature by the electron bombardment evaporation of a Co rod. Finally, the sample was covered with a 1.3 nm-thick Mo overlayer at room temperature. The deposition rates for Mo, Au and Co were calibrated by using the Auger spectroscopy prior to the sample preparation.

3. Results and discussion

3.1. Magnetic and atomic structures of Co/Ru(0001)

3.1.1. Depth-resolved XMCD
It was reported that Co thin films grown on a Ru(0001) single crystal exhibit the spin reorientation transition (SRT) from in-plane to perpendicular magnetization by capping with Ru [14]. In the present contribution, the magnetic structure at the surface and interface has been investigated in order to clarify the origin of the SRT. Figure 4 shows Co L-edge XMCD spectra taken at ~120 K. The XMCD intensity for the 3 ML Co film is much smaller than that for the 12 ML film. Moreover, the XMCD signal is larger in the surface sensitive spectrum (λ ~ 0.6 nm) in the 3 ML data. These results directly indicate that the Co atoms at the interface to the Ru substrate have smaller magnetization compared to the above layers. On the other hand, no significant λ dependence can be seen in the 12 ML data, indicating that the surface has similar magnetization to that in the inner layers. By a close look in the
12 ML spectra, however, one can find that the XMCD intensity at $\lambda \sim 0.6$ nm is larger than that at $\lambda \sim 1.4$ nm at the L$_3$ peak, while they are almost the same to each other at the L$_2$ peak. According to the XMCD sum rule [15], a larger orbital magnetic moment is suggested at the surface, compared to the inner layers. The estimated effective spin and orbital moments are also given in figure 4.

Upon Ru capping, the SRT to perpendicular magnetization occurs for the 3 ML Co film as shown in figure 5, in which the XMCD intensity at the normal incidence is twice as large as that at the grazing incidence. On the other hand, in-plane magnetization is kept in the 12 ML Co film even after Ru capping.

Figure 4. Co L-edge XMCD spectra for 3 and 12 ML Co films on Ru(0001), taken at probing depths of ~0.6 and ~1.4 nm. All the spectra were taken at grazing X-ray incidence (60° from normal). Effective spin, $m_s^{\text{eff}}$, and orbital, $m_l$, moments are given, which are estimated by applying the sum rules.

Figure 5. Co L-edge XMCD spectra for Ru (2 ML)-covered 3 and 12 ML Co films on Ru(0001), taken in the total electron yield mode.

Figure 6 gives depth-resolved XMCD data for the Ru capped films. For the 3 ML Co film, the XMCD intensity is much smaller at the surface sensitive spectrum ($\lambda \sim 0.6$ nm), which is in a clear contrast to the bare film. This again indicates that the interface Co to the Ru has smaller magnetization.
compared to the inner layer. Note that the top Co layer changes from the surface to the interface upon Ru capping. Moreover, the orbital magnetic moment in the 3 ML Co film with perpendicular magnetization is larger than that in the 12 ML Co film with in-plane magnetization. Furthermore, the in-plane orbital magnetic moment in the 12 ML Co film significantly decreases upon Ru capping, especially at the surface sensitive data, which might be the origin of the Ru-induced SRT.

Figure 6. Co L-edge XMCD spectra for Ru (2 ML)-covered 3 and 12 ML Co films on Ru(0001), taken with probing depths of ~0.6 and ~1.4 nm, together with the estimated effective spin and orbital moments. The incidence angle was chosen according to the magnetic easy axis of the Co film.

3.1.2. Preliminary results for depth-resolved EXAFS
The atomic structure must be one of the most important factors to determine magnetic anisotropy. We have tried to apply the depth-resolved technique to the EXAFS spectroscopy, in which the X-ray absorption intensity is measured in the same manner to the depth-resolved XMCD, but in a relatively wide energy range using the linear polarization. Figure 7 shows layer-resolved EXAFS spectra for the bare 3 ML Co film, which were extracted from a set of the EXAFS spectra with different probing depths, \( \lambda \), according to the following procedure [1,2,4,5]. The observed EXAFS spectrum, \( Y(E) \), after the background subtraction and the normalization to the edge jump intensity is given by

\[
Y(E) = C \sum_{i=1}^{n} \mu_i(E) \exp \left[ -d_{\text{Co}} \left\{ \frac{i-1}{\lambda} + \frac{1}{\cos \theta} \sum_{j=1}^{i-1} \mu_j(E) \right\} \right],
\]

where \( C \) is the normalization factor, \( n \) the Co layer thickness in ML unit, \( \mu_i(E) \) the absorption coefficient at the \( i \) th layer from the top Co layer, \( E \) the photon energy and \( \theta \) the x-ray incidence angle from surface normal. Since \( C \) is independent of the photon energy, it can be determined at each \( \lambda \), by using the known absorption coefficients at the pre- and post-edge energies. Then \( \mu_i(E) \) is optimized so as to reproduce the observed set of \( Y(E) \) at different probing depths, \( \lambda \).

Although the detailed analyses for the spectra have not performed yet, it can be seen that the EXAFS oscillation period is shorter at the surface in the grazing incidence data, while it is almost the same or slightly longer at the surface in the normal incidence condition. These results suggest that the out-of-plane lattice constant is larger at the surface, while the in-plane one is slightly smaller at the
surface. This seems reasonable because the lattice constant for bulk Ru is larger than that of Co by ~15%. Moreover, this is consistent with the Co K-edge EXAFS results [16], in which a larger in-plane and smaller out-of-plane lattice constants compared to bulk Co were observed in the thin film regions. Although the quantitative analyses of the L-edge EXAFS data are difficult due to the superposition of three absorption edges, a new approach using the Bayes-Turchin method has been developed and applied to the Fe L-edge EXAFS data [17]. The detailed analyses of the present data are now underway by using the Bayes-Turchin approach.

Upon Ru capping, the EXAFS spectra changes as shown in figure 8. Although the data are quite noisy due to the signal attenuation by the Ru capping layer, it might be suggested that the Co film has almost uniform in-plane lattice constant regardless of the depth. Again this seems consistent with the Co K-edge EXAFS results [16], in which a relaxation to the Co bulk lattice constant was reported upon Ru capping.

**Figure 7.** Layer-resolved Co L-edge EXAFS spectra for a 3 ML Co film on Ru(0001). Anomalous structures around 850 eV is due to the Ni contamination on the beamline optics.

**Figure 8.** Layer-resolved Co L-edge EXAFS spectra for Ru(4 ML)/Co(3 ML)/Ru(0001).
3.2. Three-dimensional micro XMCD for Fe/Ni/Cu(100): Combination with X-ray microbeam

3.2.1. Two-dimensional magnetic structure
In this subsection, the first data for the depth-resolved XMCD combined with the X-ray microbeam are reported, in order to demonstrate the possibility of the three-dimensional magnetic analyses. The sample was magnetized in plane after the deposition of Ni, and the Fe micropattern was subsequently formed without the magnetic field. Since the Ni(6 ML)/Cu(100) film shows the SRT to perpendicular magnetization at <0.5 ML Fe coverage, and then to in-plane at 1-2 ML Fe [6], the present sample is expected to have in-plane magnetization. Some anomalous magnetic state might remain, however, because both of the Fe growth and the XMCD measurements were carried out without the magnetic field.

Figure 9 gives two-dimensional XMCD imaging together with the geometry of the measurements. Essentially no signal can be seen in the normal incidence data (θ = 0°), which indicates in-plane magnetization as expected. In the grazing incidence data (θ = 60°), clear XMCD signals are observed when the incident X-ray direction is in the azimuthal angle, φ = 90°, while little signal can be seen in the data for φ = 0°. Some different magnetic structures are suggested, however, around the both edges of the micropattern, \(X = 0-35\) and 140-200 \(\mu\)m. The magnetization direction at each position along the centerline of the micropattern was estimated from the XMCD intensities observed at three directions, as depicted in figure 10. One can recognize that the magnetization direction is always in plane, but the azimuthal direction changes according to the position. Since the magnetic easy axis lies in [011] and [01-1], corresponding to φ = 0° and 90° in the present setup [6], some anomalous magnetic states are suggested around the both edges of the micropattern.

![Figure 9](image.png)

**Figure 9.** Two-dimensional XMCD imaging (top) and line scan (bottom) of Fe(2 ML)/Ni(6 ML) micro pattern with a 200×40 \(\mu\)m² size grown on Cu(100) taken at the Ni L3 peak energy. The difference between the intensities for the positive and negative helicities is plotted.
3.2.2. Depth-resolved XMCD measurements

The depth-resolved XMCD measurements were carried out at three positions, A, B and C indicated in figures 9 and 10, and the obtained spectra are shown in figure 11. Although the data are noisy, typical XMCD spectra are obtained at positions A and B, regardless of the probing depth, $\lambda$. On the other hand, large anisotropy between the L$_3$ and L$_2$ edges can be seen at position C, especially at the surface sensitive spectrum ($\lambda \approx 0.6$ nm), which suggests a large orbital magnetic moment at the surface. Moreover, the branching ratio between the L$_3$ and L$_2$ edges seems different from that at position A and B. Although an anomalous magnetization direction with some electronic state is thus suggested around the edge of the micropattern sample, more precise experiments are necessary to confirm it.

3.3. Application to samples with thick overlayer: Mo/Co/Au/Al$_2$O$_3$(11-20)

In this subsection, the application of the depth-resolved XMCD technique to samples with a relatively thick overlayer is reported, in order to demonstrate the possibility to investigate ex-situ samples with protection layers. We chose the Mo/Co/Au/Al$_2$O$_3$(11-20) sample, since the SRT has been reported at $\approx 1.5$ nm Co thickness [18].
3.3.1. Depth-resolved XMCD

Figure 12 shows Co L-edge XMCD raw data from a Co thin film covered with a 1.3 nm Mo overlayer. It should be noted here that the signal to background (S/B) ratio reduces as the probing depth decreases, due to the attenuation of the Co signal and the increase of the background from the overlayer. In fact, the S/B ratio at $\lambda \sim 0.6$ nm is about a half of that at $\lambda \sim 1.4$ nm. It is important to make the protection layer as thin as possible in the case of ex-situ samples.

![Figure 12. Co L-edge XAFS raw data taken at $\lambda \sim 0.6$ and $\sim 1.4$ nm for Mo(1.3 nm)/Co(1.4 nm)/Au(10 nm)/Mo(20 nm)/Al2O3(11-20).](image)

Figure 13 shows the probing depth dependence of the Co L-edge XMCD spectra. It is clearly seen that the XMCD intensity is larger at the L_3 peak but smaller at the L_2 peak for the surface sensitive spectrum ($\lambda \sim 0.6$ nm) in the Co 1.4 nm sample with perpendicular magnetization, and vice versa in the Co 1.7 nm sample with in-plane magnetization. Although we could not measure both of the perpendicular and in-plane orbital moments for the same sample, these data suggest that the orbital moment in the perpendicular (in-plane) direction is larger (smaller) at the Mo-Co interface. Therefore large perpendicular magnetic anisotropy is suggested at the Mo-Co interface, which can be the origin of the reported SRT [18].

![Figure 13. Co L-edge XMCD spectra taken at $\lambda \sim 0.6$ and $\sim 1.4$ nm for 1.4 and 1.7 nm Co films, together with the estimated effective spin and orbital moments.](image)
4. Summary
The depth-resolved XMCD and EXAFS for Co films grown on Ru(0001) suggested Ru-induced large perpendicular magnetic anisotropy and the depth-dependent atomic structure changes. An anomalous magnetic state was suggested for a micro-patterned Fe/Ni/Cu(100) film by combining the depth-resolved XMCD with the X-ray microbeam. The depth-resolved technique was also applied to Co thin films covered with a relatively thick Mo overlayer, indicating that the Co-Mo interface has large perpendicular magnetic anisotropy.

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