



Experiment title:

Polarimetry in Mössbauer reflectivity experiments for investigating noncollinear or spiral magnetic ordering in [Fe/Cr] multilayers

Experiment number:

MA-4040

Beamline:

ID18

Date of experiment:

from: 8 Jul 2018 to: 17 Jul 2018

Date of report:

Received at ESRF:

Shifts:

24

Local contact(s):

Dr. Aleksandr Chumakov

Names and affiliations of applicants (* indicates experimentalists):

Dr. Marina Andreeva (M.V. Lomonosov Moscow State University, Faculty of Physics)

Roman Baulin (M.V. Lomonosov Moscow State University, Faculty of Physics)

Boris Roshchin* (Institute of Crystallography (RAS))

Report:

The goal of our proposal was to improve a polarimetry technique in the Mössbauer reflectometry experiments and to show the advantages of this method in magnetic multilayer investigations. In the previous experiment (MA-3217) we were faced with some unexpected problems: the divergence of the reflected radiation from the samples was $\sim 200''$ and using Si-channel-cut crystal as an analyzer ($2\theta_{Br}=90.2^\circ$), having an acceptance of ~ 2 arcseconds, we lost $\sim 95\%$ of intensity.

To improve technique during the present session we tested several crystals as analyzer (Table 1), and we measured the divergence of reflectivity from different series of samples (prepared with different techniques and in different institutes). The smallest divergence was ~ 60 arcsec for [Fe/V] multilayers prepared in Uppsala University. The best choice of the analyzer for our measurements was LiF crystal.

We have measured the reflectivity from 3 samples: [Fe(30A)/Cr(12A)]₁₀ with antiferromagnetic interlayer coupling (AF), [Fe(15A)/V(15A)]₂₀ with ferromagnetic alignment (FM) and from cluster-layered [Fe(0.8A)/Cr(10A)]₃₀ sample, revealing Kondo-like behavior of resistivity. The samples were mounted in cryomagnet and cooled down to ~ 4 K.

Table 1

X-tal	(h k l)	$\theta_{Br} [^\circ]$	$\cos^2 2\theta_{Br}$	1/b	$\Delta\theta$	R
Si _{ch-c}	(840)	45.101	1.2×10^{-5}	29	2.3''	40%
Ge	(664)	45.506	3.1×10^{-4}	8.1	4.0''	15%
LiF	(622)	44.982	3.9×10^{-7}	2.8	100''	5%
Graphie	(0 0 10)	39.87	0.032	1	800''	1.4%
Graphie	(0 0 12)	50.29	0.034	1	800''	0.7%

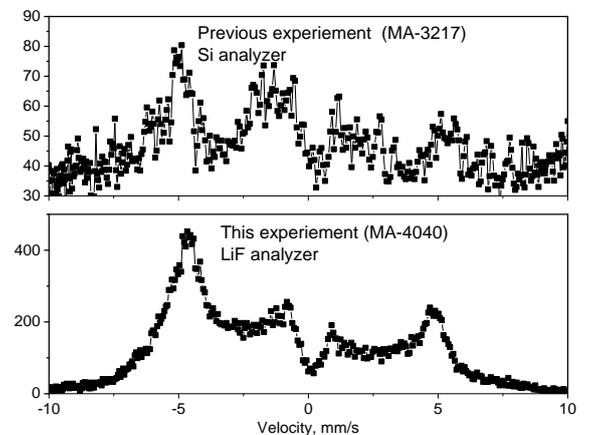


Fig. 1. Mössbauer $\pi \rightarrow \sigma'$ reflectivity spectra measured with analyzer at the critical angle for [⁵⁷Fe(8 Å)/Cr(10.5 Å)]₃₀ sample in the previous session (MA-3217) [1] and for [Fe(30 Å)/Cr(12 Å)]₁₀ sample in the present experiment.

We have significantly improved measurements of Mössbauer $\pi \rightarrow \sigma'$ reflectivity spectra of rotated polarization (Fig 1) comparing with previous experiment. The count rate was increased from ~ 0.2 cps to ~ 1.2 cps (while the intensity of incidence beam was the same). Moreover the LiF analyzer has better suppression of

the undesirable $\pi \rightarrow \pi'$ scattering than silicon. As a result the effect was increased significantly over the noise.

The improved analyzer allowed us to measure the $\pi \rightarrow \sigma'$ reflectivity spectrum from the AF $[\text{Fe}(30\text{\AA})/\text{Cr}(12\text{\AA})]_{10}$ multilayer even at the “magnetic” maximum. The presence of 2nd and 5th lines at $\sim\pm 3$ mm/s on the $\pi \rightarrow \pi' + \sigma'$ spectrum and their absence on the $\pi \rightarrow \sigma'$ spectrum (see Fig. 2) unambiguously indicates a canted magnetic ordering in the multilayer at the external field $H^{\text{ext}}=0.06$ T, applied perpendicular to beam. For the explanation see [2].

With the new analyzer the angular dependence of the $\pi \rightarrow \sigma'$ reflectivity was possible to measure at the broader angular range including $\frac{1}{2}$ Bragg and 1st Bragg maximum angles. Previously such measurements were done only near the critical angle and show the peak at the critical angle [2].

For the FM $[\text{Fe}(15\text{\AA})/\text{V}(31.5\text{\AA})]_{20}$ multilayer the spectra were measured at the critical angle and at the Bragg maximum of the 1st order with the external field (1 and 5 T) applied along the beam. The $\pi \rightarrow \pi' + \sigma'$ spectra have demonstrated the existence of the top oxide layer, magnetization of which does not allined by $H^{\text{ext}}=5$ T and so it does not contribute to the $\pi \rightarrow \sigma'$ spectra of reflectivity. In such a way we show the possibility to select the FM and AF fractions in the surface layers.

For the cluster-layered $[\text{Fe}(0.8\text{\AA})/\text{Cr}(10\text{\AA})]_{30}$ sample we measured the $\pi \rightarrow \sigma'$ spectrum only near the critical angle at $H^{\text{ext}}=4$ T applied along the beam. Due to the very small concentration of the ^{57}Fe nuclei the signal of the “rotated” polarization was very small (~ 0.2 cps). However, the $\pi \rightarrow \sigma'$ spectrum with “rotated” polarization demonstrates some separated lines contrary to the very smeared $\pi \rightarrow \pi' + \sigma'$ spectrum. We hope that from $\pi \rightarrow \sigma'$ spectrum it will be possible to restore the true distribution of the hyperfine magnetic fields in this cluster-layered sample.

At 16.07.2018 we were faced with the problem: some construction work close to ID18 (probably at BM18) caused vibration at ID18 during the whole day so the beam was very unstable and we had to spend a lot of time adjusting optics during that day.

Summary

We have tested several analyzers for the selection of the reflected radiation by polarization, the best choice was LiF. With LiF analyzer, we measured the nuclear resonant $\pi \rightarrow \sigma'$ reflectivity curves for the broader angular range including $\frac{1}{2}$ Bragg and 1st Bragg maximum angles. The quality of the measured Mössbauer $\pi \rightarrow \sigma'$ reflectivity spectra became acceptable for their quantitative interpretation. We have been convinced that the measured $\pi \rightarrow \sigma'$ reflectivity spectra provided us with an additional information about magnetic alignment, hyperfine field distribution, etc., not seen in the common Mössbauer $\pi \rightarrow \pi' + \sigma'$ reflectivity spectra.

References

1. M.A. Andreeva, R.A. Baulin, A.I. Chumakov, R. Ruffer, G.V. Smirnov, Yu.A. Babanov, D.I. Devyaterikov, M.A. Milyaev, D.A. Ponomarev, L.N. Romashev, V.V. Ustinov, Nuclear resonance reflectivity from $[\text{Fe}/\text{Cr}]_{30}$ multilayer with the Synchrotron Mössbauer Source, *J. Synchrotron Rad.* 25, 473–483 (2018).
2. M. Andreeva, R. Baulin, A. Chumakov, T. Kiseleva, R. Ruffer, Polarization analysis in Mössbauer reflectometry with Synchrotron Mössbauer Source, *Condensed Matter*. 4 (8), 1-17 (2019).

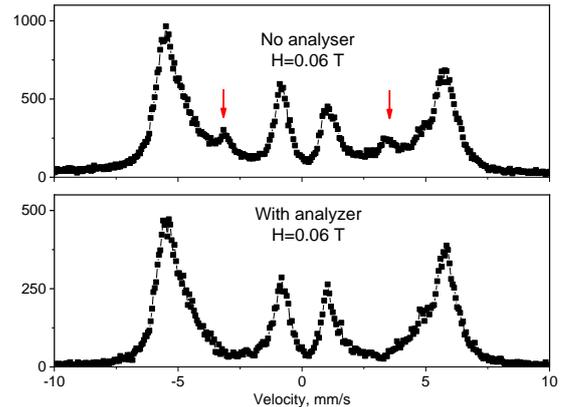


Fig. 2. Mössbauer reflectivity spectra for the FM $[\text{Fe}(30\text{\AA})/\text{Cr}(12\text{\AA})]_{10}$ sample measured at the “magnetic” maximum with and without analyzer.