



	Experiment title: Spin-glass investigations by Mössbauer spectroscopy with Synchrotron Mössbauer Source using ellipsometric approach	Experiment number: MA-3217
Beamline: ID 18	Date of experiment: from: 25 Oct 2016 to: 01 Nov 2016	Date of report: <i>Received at ESRF:</i>
Shifts: 18	Local contact(s): Dr. Rudolf Ruffer	

Names and affiliations of applicants (* indicates experimentalists):

Dr. ANDREEVA Marina¹, **BAULIN Roman**¹, **Dr. KISELEVA Tatiana**¹, **Dr. CHUMAKOV Alexandr**², **Dr. SMIRNOV Gennadij**³

¹M V Lomonosov Moscow State University Solid State Chair Faculty of Physics Leninskie Gory RU - 119991 MOSCOW,

²ESRF 71 avenue des Martyrs CS 40220 FR - 38043 GRENOBLE Cedex 9

³NRC Kurchatov Institute Pl. Kurchatova, 1 RU - 123182 MOSCOW

Report:

The main goal of our work was to apply the polarization analysis to the reflected from a Mössbauer sample radiation in order to clarify the interpretation of the complicated Mössbauer spectra typical for nanostructured objects (like that was observed for the cluster-layered $\text{Al}_2\text{O}_3/\text{Cr}(7 \text{ nm})/[^{57}\text{Fe}(0.12 \text{ nm})/\text{Cr}(1.05 \text{ nm})]_{30}/\text{Cr}(1.2 \text{ nm})$ sample [1]). Such samples have splitted magnetic spectrum only at low temperatures, so we put several multilayers with different thicknesses of layers to the cryo-magnet and cooled down all our samples to 4K.

The registration of the “rotated” polarization in Mössbauer scattering has been done for the first time and we have faced with certain difficulties during the experiment.

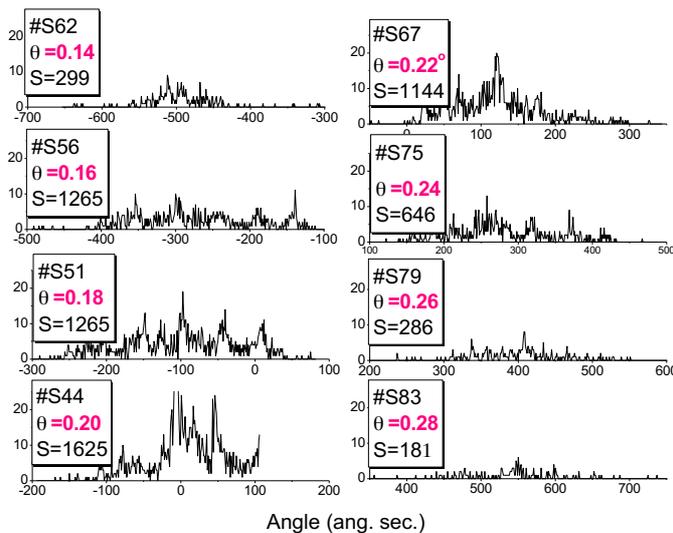


Fig. 1. Angular scans of the “rotated” polarization ($\pi \rightarrow \sigma$) reflected from $[^{57}\text{Fe}(0.8 \text{ nm})/\text{Cr}(1.05 \text{ nm})]_{30}$ sample at different grazing angle. During these scans the line width of the SMS was deliberately enlarged up to 5 mm/s to increase the intensity of the reflected radiation

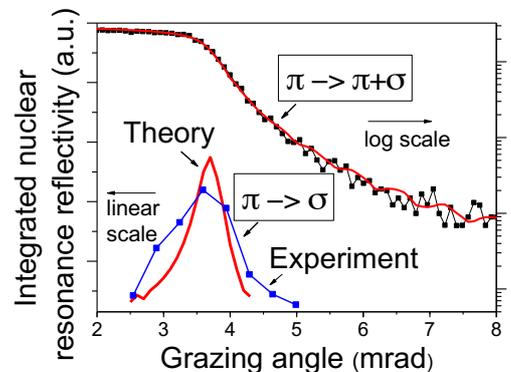


Fig. 2. The integral over scans presented in Fig. 1 revealed the peak for the “rotated” polarization predicted by theory.

We used Si channel-cut crystal (two (840) reflection with $\theta_B=46.9^\circ$ for 14.4 keV radiation) in order to select “rotated” σ -polarization in the reflected beam. The efficiency of the channel-cut crystal was only 4% and we had a great loss of the intensity. Moreover the perfect Si crystal has the very small angular acceptance but our samples had no perfect surface. Some of them were curved and others had mosaic-like surface. The angular divergence of the reflected radiation for them (as it was revealed by the angular scan with analyzer) was $\sim 200''$ (Fig.1), so we lost additionally 95% of the reflected intensity due to the divergence of the reflected beam. These circumstances make impossible the measurements from the sample with the thinnest ^{57}Fe layers (0.08 nm and 0.12 nm). The measurements at the “magnetic” maximum for $[\text{}^{57}\text{Fe}(0.8\text{ nm})/\text{Cr}(1.05\text{ nm})]_{30}$ sample characterised by the antiferromagnetic interlayer coupling [2] also occurred impossible due to the very low counting rate (~ 1 cps). The too low signal of the “rotated” polarization can be suppressed by the “background”, originated from a small contribution of the s-polarized radiation in the incident beam, which adds the wrong angular and energy dependences to the specific “rotated” polarization dependences. Note, that during our experiment the regular adjustment of the HRM to the resonant energy and the SMS was very important.

The nuclear resonance reflectivity (NRR) curve for the “rotated” polarization was obtained as an integral over angular scans at each grazing angle. The shape of the obtained NNR curve with σ -polarization selection shows a expected maximum near the critical angle (instead of plateau on the NNR reflectivity curve measured without polarization selection) (Fig. 2).

The Mössbauer spectrum of reflectivity for “rotated” polarization was measured at the peak for the “rotated” polarization on the NRR curve (Fig. 3). An acquisition of the spectrum was 8 hours. In spite of the bad statistics we see that resonant lines are presented in that spectrum as peaks and can be more or less reproduced by the fit. This spectrum essentially differs from the spectrum measured without polarization analysis. Additional spectrum measured at slightly lower angle show the unexpected asymmetry which we explain by the refraction effect.

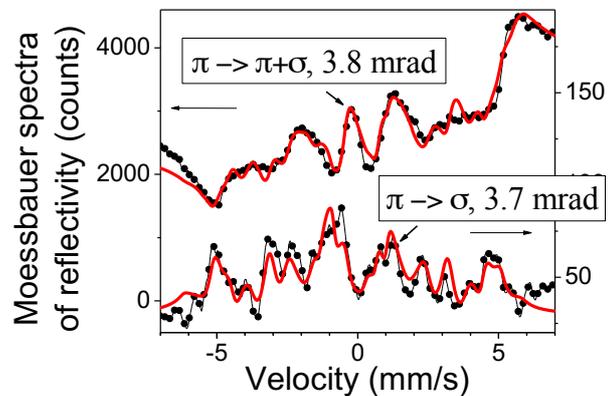


Fig. 3. Mössbauer spectra of reflectivity measured at the critical angle with analyzer ($\pi \rightarrow \sigma$) and without analyzer ($\pi \rightarrow \pi + \sigma$)

In summery:

During our experiment we have confirmed the basic features of the angular and energy dependencies of the “dichroic” signal in NRR reflectivity from magnetized sample. We understood that for the future applications of the polarization analysis to the practical tasks in Mössbauer spectroscopy we need the essential modification of the analyzer used in the present experiment in order to avoid the drastical loss of the measured intensity (use single reflection from analyzer, the crystal with the broad band angular acceptance, perform the measurements on the samples with better surfaces, etc.)

References:

- [1] Andreeva, M.A., Baulin, R.A., Chumakov, A.I., Rüffer, R., et al (2017). Field-temperature evolution of the magnetic state of $[\text{Fe}(1.2\text{ \AA})/\text{Cr}(10.5\text{ \AA})]_{30}$ sample by Mössbauer reflectometry with synchrotron radiation, *JMMM*, **440**, 225-229.
- [2] Andreeva, M. A., Chumakov, A. I., Smirnov, G. V., Babanov, Yu. A., et al (2016) Striking anomalies in shape of the Mössbauer spectra measured near “magnetic” Bragg reflection from $[\text{Fe}/\text{Cr}]$ multilayer, *Hyperfine Interactions* **237**(1), 1-9.