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Striking anomalies in the shape of Mössbauer spectra measured near “magnetic” Bragg reflection from [Fe/Cr] multilayer

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Abstract The present work reports the observation of a peculiar asymmetry of the Mössbauer spectra of reflectivity from a periodic $[^{57}\mathrm{Fe}(8\text{Å})/\mathrm{Cr}(10.5\text{Å})]_{30}$ multilayer measured on both slopes of the half-order “magnetic” Bragg peak. The performed analysis and model calculations confirm the responsibility of the refraction effect for the observed features.

Keywords Mössbauer spectroscopy · Nuclear resonance scattering · Synchrotron Mössbauer Source · Magnetic multilayers

1 Introduction

The installation of the Synchrotron Mössbauer Source (SMS) [1] based on the (111) pure nuclear reflection from $^{57}\mathrm{FeBO}_3$ crystal recently launched at the ID18 of the European Synchrotron Radiation Facility (ESRF) [2] signifies the great achievement and substantial perspectives for applications of the nuclear resonance scattering at synchrotrons. In combination with the exclusively small angular divergence of synchrotron radiation this installation opens the perfect possibility to perform the Mössbauer diffraction and reflectivity

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measurements in the conventional energy domain. Nowadays it is well proved that the Mössbauer reflectivity method is very informative for the magnetic multilayer investigations.

For the first time the total reflection of the Mössbauer radiation by ^{57}Fe mirror was measured with the radioactive source in 1963 [3]. Difficulties of such experiments were related to the source intensity losses due to the necessity to cut out a narrow angular beam ($\sim$0.5 mrad) from radiation of a ^{57}Co source naturally emitted into 4π Sr. For this reason, such measurements were reported just in a few papers [4–9]. All these works demonstrated the excellent depth selectivity of this method: it has been shown that the depth profiles of the hyperfine fields were obtained with the resolution less than 1 nm. Another interesting experiments with Mössbauer specular reflection were devoted to the special features of the pure nuclear reflections from Grazing Incidence Anti-Reflection (GIAR-) films expected to be the pure nuclear monochromators [10]. Nowadays collective mode excitations in wave-guide structures showing up the energy spectrum distortions are thoroughly investigated [11, 12]. Synchrotron radiation opens an excellent possibility for the angular resolved measurements, however up to now the nuclear resonance experiments with synchrotron radiation have been performed in a so called time domain. Nuclear resonance reflectivity in the time-domain has been effectively tested in refs. [13, 14] and further it has been applied for numerous multilayer investigations (see e.g. refs. [15–20]). However, the interpretation of the time spectra of reflectivity was not as obvious as it could be done with spectra in the energy domain.

The spectrum of the radiation reflected by a Mössbauer mirror (e.g. ^{57}Fe film) essentially differs from the conventional Mössbauer absorption spectrum: at the grazing angles lower than the critical angle of the total external reflection θ_c the resonant lines appear as minima, near θ_c they takes the dispersive shape and at the angles larger than θ_c appear as peaks. For the explanation of this line shape variations in the first paper [3] the idea of interference of the nuclear resonance and nonresonant electronic scattering has been introduced. The idea has been based on the graphical illustration of the Fresnel intensity as a function of the real and imaginary part of the total refractive index, clearly showing the importance of the electronic scattering on the shape of the Mossbauer spectrum of reflectivity. Later the term of "interference" has been discussed as the very doubtful one due to the fact that the electronic scattering and the nuclear resonance decay take place at different time interval: the first one is prompt and the second one characterizes by some delay. This problem becomes more interesting when the nuclear resonance reflectivity has come under the measurements in the time domain with synchrotron radiation. Nevertheless, the peak observed near the critical angle on the delayed reflectivity curve has also been initially named as the interference peak [14, 21]. Later the correct explanation of the influence of the prompt electronic scattering on the delayed nuclear resonance signal has been presented [22]. It has been based on the concept of standing wave, created at the initial moment by prompt electronic scattering and influencing the nucleus excitation and following nuclear emission in the reflection direction. The interesting features of the spectrum of reflectivity from periodic multilayer in vicinity of the precise Bragg angle for the case of the single line resonance (the shift and peak doubling) has been theoretically analyzed in ref. [15]. The different shapes of the Mössbauer or time spectra of reflectivity measured at different orders of Bragg reflections from periodic structures, which stipulate the depth selectivity of the method, has been investigated in ref. [23].

In this article we report on the observation of the huge asymmetry of the Mössbauer sextet for a Al_2O_3/Cr(70Å)/[^{57}Fe(8Å)/Cr(10.5Å)]$_{30}$/Cr(12Å) multilayer in vicinity of the pure nuclear "magnetic" maximum [24]. In the absorption experiments the asymmetry of the magnetic sextet is explained by the existence of the specific helicoid or cycloid magnetic structures [25–27]. In our case the peculiar shape of the reflectivity spectra is explained by the refraction effects.

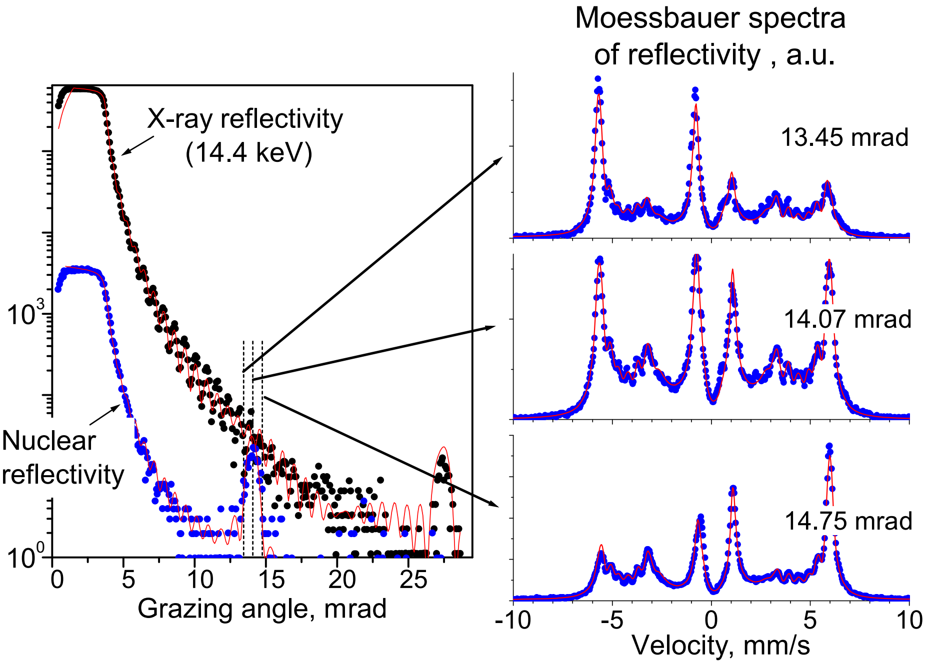


Fig. 1 X-ray and nuclear resonance reflectivity curves from the Al_2O_3/Cr(70Å)/[^{57}Fe(8Å)/Cr(10.5Å)]$_{30}$/Cr(12Å) sample - *left side*. On the *right side* the nuclear resonance reflectivity spectra at three angles in vicinity of the "magnetic" Bragg maximum, marked by the *dashed vertical lines* on the *left side*, are presented. The *dots* are the experimental data; *solid lines* (*red on line*) are the theoretical fit

2 Experiment

The Mössbauer spectra of reflectivity from a periodic [^{57}Fe/Cr]$_{30}$ multilayers have been measured [24] at the Nuclear Resonance Beamline [2] (ID18) of ESRF. The experiment was performed with the Synchrotron Mössbauer Source (SMS) opening the possibility to measure the reflectivity spectra in the conventional energy domain. The details of the experimental setup are described in [1, 2].

The results obtained for the Al_2O_3/Cr(70Å)/[^{57}Fe(8Å)/Cr(10.5Å)]$_{30}$/Cr(12Å) sample are presented in Fig. 1. The data were measured at 4 K and in the external filed of 0.025 T applied along the sample surface and perpendicular to the beam direction. The X-ray reflectivity curve was measured with the "Umweg" (Reninger) reflection from the $^{57}FeBO_3$ crystal after its azimuth rotation within the pure nuclear reflection position. The nuclear reflectivity angular curve was measured with the pure nuclear reflection from the $^{57}FeBO_3$ crystal (SMS setup) and obtained by integration over the reflectivity spectra in the range of ±10 mm/s. Mössbauer spectra of reflectivity at the specified angular point were measured by vibration of the $^{57}FeBO_3$ crystal providing the conventional Doppler change of the photon energy of the emitted radiation. In the nuclear resonance reflectivity angular curve (Fig. 1, left) a 1/2 order Bragg peak at ~14 mrad ("magnetic" maximum) appears revealing the doubling of the magnetic period due to a specific interlayer coupling in this sample (Fig. 1, left). The fit of the data was performed with the program package REFTIM [28,

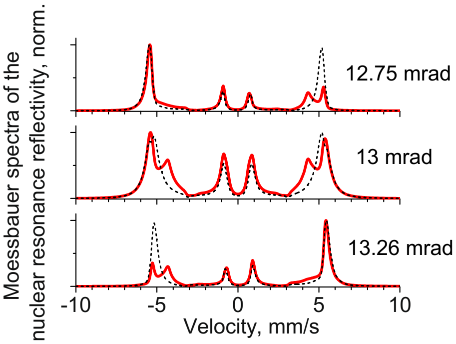


Fig. 2 Model calculations of the reflectivity spectra from the [^{57}Fe(7Å)/Cr(10.22Å)]$_{30}$ multilayer at three angles in vicinity of the exact 1/2 Bragg peak (13 mrad for the used model). The ratio of line intensities in the sextets (similar to the experimental one) corresponds to the +45°/-45° azimuth angles of B_{hf} orientations. The *dot lines* are the result for the model where only 1 sextet characterizes the hyperfine interactions in ^{57}Fe layers. The *solid lines* (*red on-line*) present the results for the model where an additional sextet with a smaller B_{hf} value is considered

29] adjusted for calculations of the reflectivity spectra in the energy domain (instead of the previous treated time domain version).

The X-ray reflectivity fit gives the multilayer period 1.587 nm (1.051 nm of Cr and 0.536 nm of ^{57}Fe) with a rather large interlayer mixing (~0.4 nm) of the Fe and Cr layers. Due to such intermixing the hyperfine parameters for ^{57}Fe layers, obtained by the joint fit of all the data presented in Fig. 1, includes 7 sextets corresponding to the magnetic hyperfine fields B_{hf} = 35.38, 32.50, 29.97, 27.00, 23.94, 20.80 and 17.43 T with relative weights of 24.33, 14.84, 13.80, 13.87, 14.02, 16.08 and 3.05 %, respectively. Notice that such regular set of the hyperfine field confirms the monocrystalline structure of our multilayer. Some distortion of the parameters in the top and bottom bilayers of the [^{57}Fe(8Å)/Cr(10.5Å)]$_{30}$ multilayer has been taken into account. The shape of spectra and the "magnetic" maximum intensity on the nuclear resonance reflectivity curve corresponds to the orientation of the hyperfine fields in the film plane with the effective azimuth angles (the explanation of the "effective" term is done in ref. [20]) in the adjacent ^{57}Fe layers ~ +70°/–70° relative the direction of the external field applied perpendicular to the beam direction. The details of the magnetic ordering at different values and directions of the external field will be discussed elsewhere. In this article we concentrate on the specific shape variation of the Mössbauer spectra of reflectivity.

The measured spectra of reflectivity were found to be quite surprising due to the strong opposite asymmetry of the 1st and 6th sextet lines at two sides of the "magnetic" peak on the nuclear reflectivity curve. The three spectra at the different angular settings of multilayer in the range of the magnetic Bragg peak are displayed in the Fig. 1, right side. A slight asymmetry and shifts of lines in the Mössbauer diffraction spectra were observed earlier in the angular resolved measurements in the vicinity of the pure nuclear (222) Bragg reflection from $FeB0_3$ crystal [30, 31]. But the primary goal of the earlier works was to find a resonance broadening at the Bragg angle and in this way to prove the existence of the coherent enhancement of the radiative channel. The observed slight asymmetry was only an accompanying result of that measurement. In our measurements we faced a strongly pronounced effect of asymmetry which should be correctly explained.

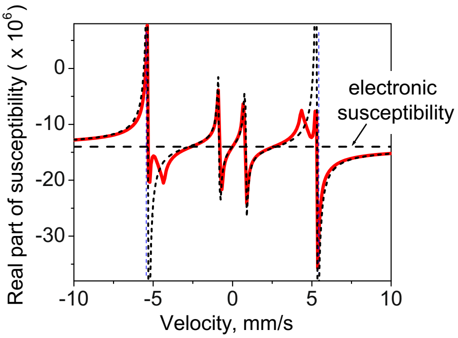


Fig. 3 The real part of the total susceptibility of the ^{57}Fe layer calculated for the model with one hyperfine field (*dot line*) and for the model with two hyperfine fields (*solid line, red on-line*). The real part of the electronic contribution is constant in this energy range and presented by the *dashed horizontal line*

3 Model calculations

For clear explanation of the observed asymmetry of the sextets in the reflectivity spectra we have performed the model calculations for a simpler model than the experimental one. At first we take just one sextet (33 T) for ^{57}Fe layer characterization.

With this simplest model we **did not get (!)** any asymmetry of the 1st and 6th sextet lines in the reflectivity spectra at the angles slightly deviated from the exact "magnetic" Bragg angle (see Fig. 2, dot lines). It became clear that one should suppose a strong perturbation of the resonance conditions at the energies corresponding to the 1st and 6th lines of our sextet. Having in mind that the refraction index has opposite signs at two sides of each resonance line (reaching the maximal magnitudes at the distances equal to the line width) it was natural to assume an existence of an additional magnetic sextet with a slightly smaller B_{hf}, which should create different refraction effects on the reflectivity at the energies corresponding to the 1st and 6th lines of the major sextet. (Note that the experimental spectra of our sample indeed reveal a whole set of sextets with decreasing B_{hf} values). The calculations with two sextets in the model (33 T and 30 T with broadening of 0.1 T and 3 T, respectively) remarkably confirm our expectations. (Fig. 2, the solid lines). The appeared asymmetry of the 1st and 6th lines of the larger sextet at two slopes of the Bragg peak occurs quite similar to that one observed in the experimental spectra.

Let us thoroughly explain the influence of the refraction of the waves in the sample on the intensity of the 1st and 6th lines of the major sextet in the spectrum of reflectivity. The condition for the Bragg peak appearance can be presented in the following way:

$$2k\sum_j \mathrm{Re}(\eta_j)d_j = 2\pi, \tag{1}$$

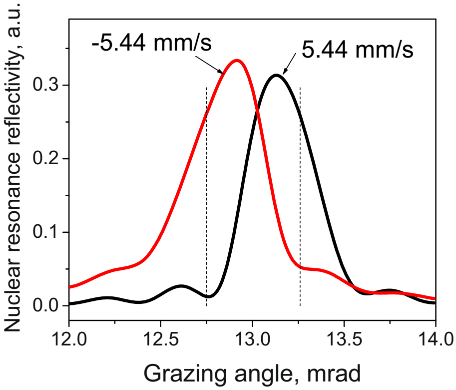
where $d_1 + d_2 + \ldots + d_n = D$ is the period of the multilayer, j is the number of sublayers in the period, $k = 2\pi/\lambda = \omega/c$ is the wave vector and

$$\eta_j = \sqrt{\sin^2\theta + \chi_j} \approx \sin\theta + \frac{\chi_j}{2\sin\theta} \tag{2}$$

is the normal component of the wave vector in j-th sublayer (in ω/c units), θ is the glancing angle for the incident radiation, χ_j is the total susceptibility of the j-th sublayer. Let us define θ in the following way $\theta = \theta_B + \Delta\theta$, where θ_B is the conventional Bragg angle $\sin\theta_B = \frac{\lambda}{2D}$ and $\Delta\theta$ is the small deviation of the Bragg peak position from θ_B due to the refraction. So

$$\sin\theta = \sin(\theta_B + \Delta\theta) \approx \frac{\lambda}{2D} + \Delta\theta\ \cos\theta_B \tag{3}$$

Fig. 4 The nuclear resonance reflectivity calculated in vicinity of the 1/2 order Bragg peak for the two photon energies corresponding ±5.44 mm/s in the Mössbauer spectrum at (the resonance energies of the 1st and 6th lines). The model includes two sextets described in the text



and from (1)–(2) we get

$$\frac{2}{\lambda}\left(\sin\theta\sum_j d_j+\sum_j \frac{\chi_j d_j}{2\sin\theta}\right)=1. \tag{4}$$

Using (3) we obtain

$$\sin\theta+\sum_j \frac{\mathrm{Re}(\chi_j)d_j}{2D\,\sin\theta}\approx\frac{\lambda}{2D}+\Delta\theta\;\cos\theta_B+\frac{\mathrm{Re}\overline{\chi}}{2\sin\theta}\approx\frac{\lambda}{2D}, \tag{5}$$

where we define the average susceptibility of the period as $\overline{\chi}=\sum_j \frac{\chi_j d_j}{D}$. So

$$\Delta\theta\;\cos\theta_B+\frac{\mathrm{Re}\overline{\chi}}{2\sin\theta}=0. \tag{6}$$

According to (6), the deviation of the Bragg peak position from θ_B can be determined by the simple formula:

$$\Delta\theta\approx\frac{-\mathrm{Re}\overline{\chi}}{\sin 2\theta_B} \tag{7}$$

We should suppose in addition that the Bragg peak shifts for the 1st and 6th sextet lines differ by the value comparable or larger than the Bragg peak width. In this case the condition for the enhancement of these lines will appear at different glancing angles. Neglecting absorption, the angular width of the Bragg peak W is determined by the simple expression

$$W=tg\theta_B/N, \tag{8}$$

where N is the number of periods in multilayer. In our case N=30, $tg\theta_B\approx 0.013$, so $W\approx$0.2 mrad.

The calculated real part of susceptibility in ^{57}Fe layer as a function of the energy shift across the resonant spectrum is presented in Fig. 3 for two models describing hyperfine interactions in ^{57}Fe layers: one sextet and two sextets.

It is clearly seen that the real part of susceptibility for the 1st and 6th lines is symmetrical in the case of one sextet in the model, but the additional sextet with smaller B_{hf} and consequently smaller line splitting drastically changes the refraction at the energies corresponding to the 1st and 6th lines of the major sextet: for 1st line the refraction is even positive and for 6th line it is strongly negative. The difference of the real part susceptibilities for the 1st and 6th lines is $\left|\mathrm{Re}\chi_{Fe1^{st}}-\mathrm{Re}\chi_{Fe6^{th}}\right|\approx 50\times 10^{-6}$ and the corresponding difference averaging over period in [Fe(0.7 nm)/Cr(1.02 nm)] multilayer is $Re\overline{\chi} \approx 0.021 \times 10^{-3}$. So the expected $\Delta\theta \approx 0.01$ mrad which is smaller than W but somehow comparable.

The direct calculation of the Bragg peak shifts for the photon energies in vicinity of the 1st and 6th lines of the larger sextet is presented in Fig. 4. The exact resonance positions for these lines correspond to the velocities of ±5.31 mm/s, but the real part of the resonant scattering amplitude has extremums at both sides of the exact resonance, so the angular curves in Fig. 4 are calculated for the velocities of ±5.44 mm/s, at these energies the peaks on the spectra in Fig. 2 take place. The widths of the calculated Bragg peaks and their shifts are slightly larger than obtained from the simplest expression (8) and (7), therefore the absorption should be taken into account for more accurate evaluation of these parameters. The maximal difference of the reflected intensity for the velocities of ±5.44 mm/s corresponds to the angles, marked by the vertical dashed lines, and exactly at these angles we have calculated the spectra presented in Fig. 2.

4 Conclusions

We have analyzed the origin of the essential anomalies in the Mössbauer spectra of reflectivity measured at both sides of the exact Bragg angle. Our theoretical estimations and model calculations confirm that the refraction effect, which shifts the Bragg peak position for different photon energies in the resonant spectrum, is responsible for the observed effect.

The influence of the refraction on the Bragg peak position is well known in the soft X-ray resonant magnetic reflectivity (XRMR) and has been used for the experimental determination of the spectral dependencies of the absolute values of the real part of susceptibility near the absorption edges [32, 33]. In the hard X-ray region this effect is too small and could be measured only in the extremely asymmetric diffraction geometry with the grazing incident angle near the critical angle θ_c [34]. In the Mössbauer case the radiation is rather hard ($\lambda = 0.086$ nm) but whereas the nuclear resonance interaction is very strong, the observable shift of the Bragg peaks on the reflectivity curve from periodic multilayers takes nevertheless place. In the most impressive form it reveals itself in the reflectivity spectrum shape. Hereby the investigation of the Mössbauer spectrum line shape opens the way to determine the absolute value of the refraction and consequently the direct measurement of some essential Mössbauer parameters like Mössbauer effect probability in multilayers.

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