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The use of quasi-Bragg diffuse scattering for express measurement of changes in multilayer d-spacing

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Abstract

The possibility of the use of quasi-Bragg diffuse scattering for express measurements of changes in multilayer d-spacing was studied. The error of this method was shown to be minimal, if the incident or scattered angles are sufficiently far from the critical total external reflection or Bragg angles. As an example, the measurements of the W/Si multilayer mirror with linearly varying d-spacing are presented. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Development of X-ray multilayer optics requires improvement of the methods to control the quality of multilayers. The usual control method is X-ray small-angle diffraction, which allows one to determine local values of multilayer d-spacing, thickness ratio of the layers of a bilayer, roughness dispersion, intermixed layer thickness and so on. Nevertheless, this method has two important disadvantages. Firstly, it is necessary to perform exact adjustment of the sample at every measurement point which sufficiently complicates the

experimental technique. Secondly, moderately large changes in the multilayer structure can cause significant changes in diffraction behavior due to strong dynamic effects. That is very difficult to take into account during experimental data treatment. This disadvantage is not important if the studied multilayer mirror structure is uniform over the sample square, and only very small changes in multilayer structure have to be controlled. If the studied multilayer mirror has a varying structure, the discussed effect can be sufficiently important.

From our point of view, the use of quasi-Bragg scattering [1–3] can be a more preferable method of control. This scattering is caused by the interfacial roughness imperfections, coherently repeating from one layer to another, and occurs at the total scattered angle equal to double Bragg angle ($2\theta_B$) independent of the incident angle.

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This technique has advantages over the conventional specular diffraction technique. Indeed, on the one hand, the diffuse scattering intensity dependence on the incident angle is very fine if its value differs remarkably from the Bragg angle or critical angle of total external reflection. Thus, no exact adjustment is necessary in this case. On the other hand, the kinematic approximation suits well for the theoretical description of this scattering, which greatly simplifies data treatment. Finally, it is very important that the quasi-Bragg scattering behavior depends on the multilayer structure in a very simple manner. The difficulties of the quasi-Bragg scattering modeling are caused by the nontrivial theoretical description of the interfacial roughness, which in many cases is completely determined by the substrate surface (for a moderately high lateral momentum transfer this fact is always true). Thus, if the substrate surface roughness is assumed to be uniform over the sample square, the changes in quasi-Bragg scattering are directly connected to the local changes in the multilayer structure.

This paper describes the measurements of the W/Si multilayer d-spacing with the use of quasi-Bragg scattering. As a test sample, a linearly varying d-spacing multilayer mirror was chosen.

2. Experimental

A multilayer mirror with a linearly varying d-spacing was deposited by magnetron sputtering in Ar^+ environment on a float glass substrate. In order to provide the desired gradient of multilayer d-spacing, the substrate was moved over the W and Si targets with variable velocity. The movement was performed by step motors under computer control. The sputtering regime was tuned so as to maintain the W-layer thickness to be constant and to ensure linear increase of the multilayer d-spacing due to the Si-layers thickness increase. The number of bilayers was 50.

The X-ray diffuse scattering measurements were performed using SR of the VEPP-3 storage ring of the Siberian SR Centre at Budker INP, which operates at 2 GeV, with a maximum stored current of 165 mA. The triple-axis diffractometer with a

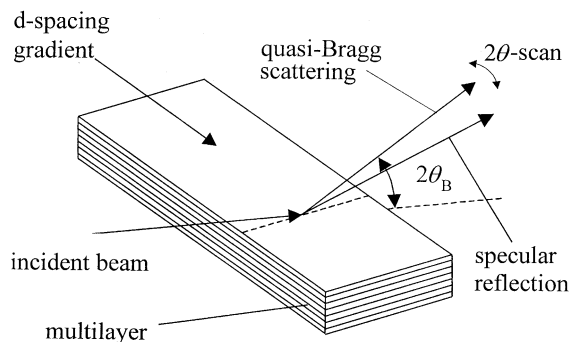


Fig. 1. The principal scheme of the experiment. Quasi-Bragg scattering occurs at the total scattered angle equal to double Bragg angle ($2\theta_B$), independent of the incident angle.

primary channel-cut single-crystal Si(1 1 1) monochromator and a Ge(1 1 1) crystal-collimator of the “anomalous scattering” station was used [4]. The measured angular broadening of the diffractometer had a full-width at half-maximum (FWHM) of $15''$. A scintillation detector based on an FEU-130 photomultiplier with a NaI(Tl) scintillator was used. The dynamic range of the detector system was about 5×10^4 . The photon energy was 8.048 keV.

The multilayer d-spacing values, which were obtained from quasi-Bragg diffuse scattering, were studied versus the incident angle in this work. The studied sample was placed so that the d-spacing gradient axis was perpendicular to the specular diffraction plane (Fig. 1). The incident beam was $100 \times 100 \mu\text{m}$ in size. For a given incident angle the 2θ -scan (total diffracted angle scan) (see Fig. 1) was performed. After that, the d-spacing value was calculated from this diffraction profile. In this manner, the d-spacing dependencies on the incident angle were obtained at several points of the sample. Finally, the d-spacing dependence along the gradient axis was obtained from the quasi-Bragg measurements.

3. Results

An example of d-spacing dependence on the off-specular angle, ω ($\omega = \theta_0 - \theta_1$, where θ_0 and θ_1 are the incident and diffracted angles, respectively),

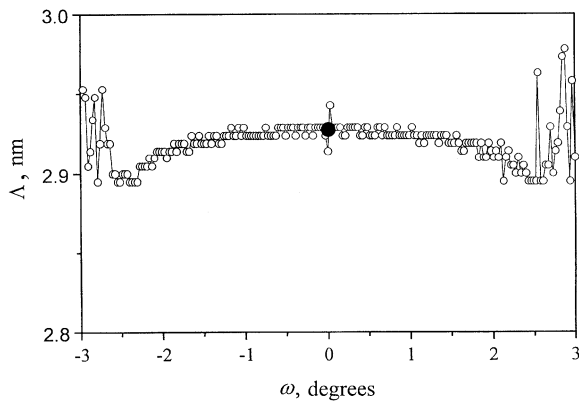


Fig. 2. The multilayer d-spacing value measured at the same sample point by quasi-Bragg scattering versus the off-specular angle, ω . The central black point is the d-spacing value measured by the conventional specular diffraction.

obtained from the quasi-Bragg scattering is shown in Fig. 2. The central black point ($\omega = 0$) in Fig. 2 corresponds to the d-spacing value measured by the conventional specular diffraction. The dependence behavior at the ends of the curve is evidently caused by the total external reflection effects.

The instability in the central narrow angle range is not so evident. Nevertheless, this behavior is not an artifact and is caused by the behavior of diffuse scattering near the Bragg point [3]. The latter effect demonstrates well the advantage of the quasi-Bragg scattering over conventional specular diffraction.

Values of multilayer d-spacing measured with the use of quasi-Bragg scattering are shown in Fig. 3. The smoothed changes in d-spacing are caused by the real deviations in d-spacing from linear behavior. As may be observed from Fig. 3, the technique errors do not exceed the errors of the conventional specular diffraction technique. It is necessary to mark the experimental simplicity of the discussed technique. So, only a few minutes were needed to measure the presented experimental data.

The discussed method can be used in the case of a multilayer with curved surface. It needs to be noted that the angle between the incident direction and that normal to the multilayer surface can be controlled by the measurement of the specular diffracted beam (Fig. 1) in this case.

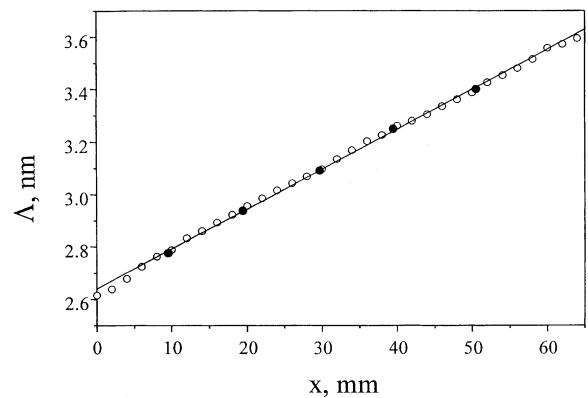


Fig. 3. The d-spacing values measured with the use of quasi-Bragg scattering along the gradient direction. The black points are values measured by the conventional specular diffraction.

4. Conclusion

The use of quasi-Bragg scattering in order to control the varying multilayer d-spacing was shown to have advantages over the conventional specular diffraction technique. One of those is the simplicity of the experimental conditions as well as of data treatment due to the kinematic nature of this scattering.

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