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Study of the structural and reflective characteristics of short-period Mo/Be multilayer X-ray mirrors

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A series of short-period Mo/Be multilayer mirrors (MLs) have been studied using the methods of X-ray reflectometry, diffuse X-ray scattering and interferometry. It is shown that the structure of the MLs (the presence of diffraction orders in the angular dependence of the reflection coefficient) is observed at least up to a period of 1.29 nm. Also, when approaching extremely small period values, starting from 1.64 nm, a sharp increase in the width of the transition region between the mirror layers is observed.

Keywords: multilayer X-ray mirrors, synchrotron applications, X-ray monochromator, internal stresses.

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Introduction

The concept of short periodicity is largely conditional. Most often, in practice, values from 2–2.5 nm and less are considered to be short periods (d). These values correspond to normal incidence mirrors for operation in the wavelength range from 4–5 nm and below. Also, multilayer mirrors (MM) with similar periods are broadly used as grazing incidence optics for the hard X-ray range. In particular, short-period multilayer mirrors are used for monochromatization and focusing of hard X-ray radiation on synchrotrons.

Strong dependence of the reflection coefficients on the amount of interlayer roughness is an important feature of short-period multilayer mirrors. The effect of roughness on reflection becomes significant with such small d (< 2.5 nm). The reflection coefficient can decrease by several times even with extreme roughness.

The presence (or rather the absence) of absorption edges of mirror materials in the operating range of the monochromator is also important for monochromatization in the hard X-ray range. The presence of absorption edges does not allow the creation of a multilayer mirrors capable of providing the required high values of the reflection coefficient and spectral selectivity over the entire operating range, which ultimately results in the use of several mirrors in a monochromator instead of one, thereby complicating its use.

W/B_4C is the most commonly used pair of materials with periods less than 2 nm in the energy range of 2–20 keV [1–3]. However, insufficient selectivity is the disadvantage of this multilayer mirror for a number of tasks, which is associated, firstly, with high radiation absorption in W layers; secondly, with a large jump of electron density between W being the most absorbing material in the pair and B_4C being the least absorbing material. Also, the presence of a number of L-absorption edges of W in the area of 10–12 keV results in a decrease of the reflection coefficients of W-containing multilayer mirrors designed for operation in a wide spectral range of 10–18 keV, one of the most relevant areas in modern synchrotron studies, for example, the planned work on the 4th generation synchrotron SKIF [4].

Theoretically, Mo/B_4C and Mo/Be multilayer mirrors could replace W/B_4C in the range of 2–20 keV. Figure 1 shows the theoretical dependences of the peak reflection coefficient and spectral selectivity on energy. The calculation in the specified energy range was performed using such multilayer mirror parameters that ensured the reflection in the first order of diffraction in the range of grazing angles θ: 0.5–5° and the maximum reflection coefficient. The periods of the considered multilayer mirrors were in the range of 3.63–3.66 nm, the ratio of the thickness of the highly absorbent material in the period to the thickness of the period was approximately 0.32 ($\beta_{\mathrm{Mo,W}}$), the number of periods was $N = 150$. Studies of the structural and reflective characteristics of short-period multilayer mirrors based on Mo/B_4C were previously performed in other works [5–7]. Mo/B_4C multilayer mirrors with periods in the range of 0.8–3.5 nm were studied in one of the papers, which showed that these mirrors significantly exceed the W/B_4C multilayer mirrors in spectral selectivity having comparable reflection coefficients. It was also noted that there is no degradation of interfaces with the periods of up to 1 nm in case of Mo/B_4C, and the stability of reflective characteristics is observed during annealing at the temperature of up to 300°C. The prospect of usage of Mo/B_4C multilayer mirrors with periods of up to 1.8 nm for synchrotron applications is demonstrated.

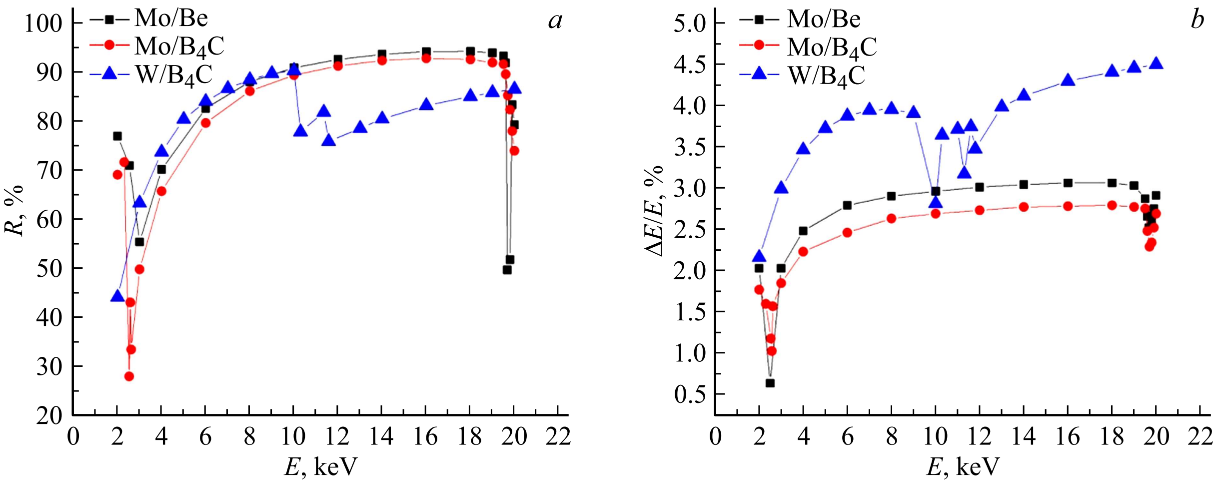

Figure 1. Theoretical dependences of peak reflection coefficient (*a*) and spectral selectivity (*b*) on energy. Calculation for the case of zero roughness at boundaries.

Mo/Be multilayer mirrors has been studied for use in the vicinity of the wavelength of 11.2 nm (0.11 keV). Their structural and X-ray optical characteristics were studied [8], as well as their thermal stability [9]. However, short-period Mo/Be mirrors have not been studied by anyone.

The problems of internal stresses and thermal stability of multilayer mirrors deserve special attention. The first problem results in an undesirable effect, namely, deformation of the substrate on which the multilayer mirror is applied, which can negatively affect, for example, the focusing properties of the multilayer mirror. The coating deposited on the substrate may peel off when the internal stresses exceed a certain threshold value. Therefore, the study of internal stresses in reflective coatings is of great importance for X-ray optics. The problem of heating of X-ray optical elements arises when they are exposed to high-energy radiation, for example, in monochromators of hard X-ray radiation on synchrotrons. Working with the hard X-ray range involves the use of short-period multilayer mirrors, the thermal properties of which may differ from thermal properties of multilayer mirrors with longer periods, and which are used to work in the adjacent soft X-ray radiation range.

1. Experimental method

In this work, Mo/Be multilayer mirrors with periods from 3.73 to 1.29 nm. A total of 17 samples were fabricated for the study. Three particular tasks can be distinguished in this study of the properties of short-period Mo/Be multilayer mirrors: 1) optimization of the thicknesses of materials in the Mo/Be multilayer mirrors with a period of ~ 3.6 nm was performed for maximizing the reflection coefficient; 2) the dependence of the reflection coefficient and the magnitude of the transition regions between layers on the magnitude of the period of multilayer structures was studied; 3) structural stresses and thermal stability of short-period Mo/Be multilayer mirrors were also studied.

The multilayer mirrors were fabricated by magnetron sputtering with a direct current in an facility with six planar-type magnetrons. The current on each magnetron was 600 mA. Pre-vacuum was 10^{-5} Pa. Sputtering was carried out in high-purity (99.998%) Ar at pressures of the order of 0.3 Pa. The film thicknesses were controlled based on the time the substrate was held above the magnetron. Layers of Mo and Be were sequentially applied (from the substrate) in each sputtering cycle with deposition rates of approximately 0.07 and 0.03 nm/s, respectively. Shaped precision diaphragms are provided above each magnetron to ensure uniformity of the coating over the substrate area. The distribution of the density of the flow of matter entering the substrate was controlled by changing the shape of the diaphragms. The accuracy of controlling the distribution of the period over the mirror area was less than 0.5% of the period value. The materials were deposited on ultra-smooth substrates of monocrystalline silicon with an orientation of (100) thickness of 0.47 mm. The study used substrates of different sizes depending on the type of study. Substrates of size 25×25 mm were used for the fabrication of multilayer mirrors for the purpose of optimizing the thicknesses of materials and searching for multilayer mirrors with a minimum period, the substrates with a size of 20×80 mm were used for studies of stresses in Mo/Be multilayer mirrors.

Multilayer mirrors were subject to qualification after the fabrication, which consisted in determining their main parameters, such as the period, thicknesses of individual layers, and widths of the transition regions. All these parameters were determined by fitting experimentally measured angular dependences of the reflection coefficient at wavelengths of 0.154, 0.989 and 1.759 nm, using a program for the reflectometric reconstruction of multilayer structures „Multifitting“ [10].

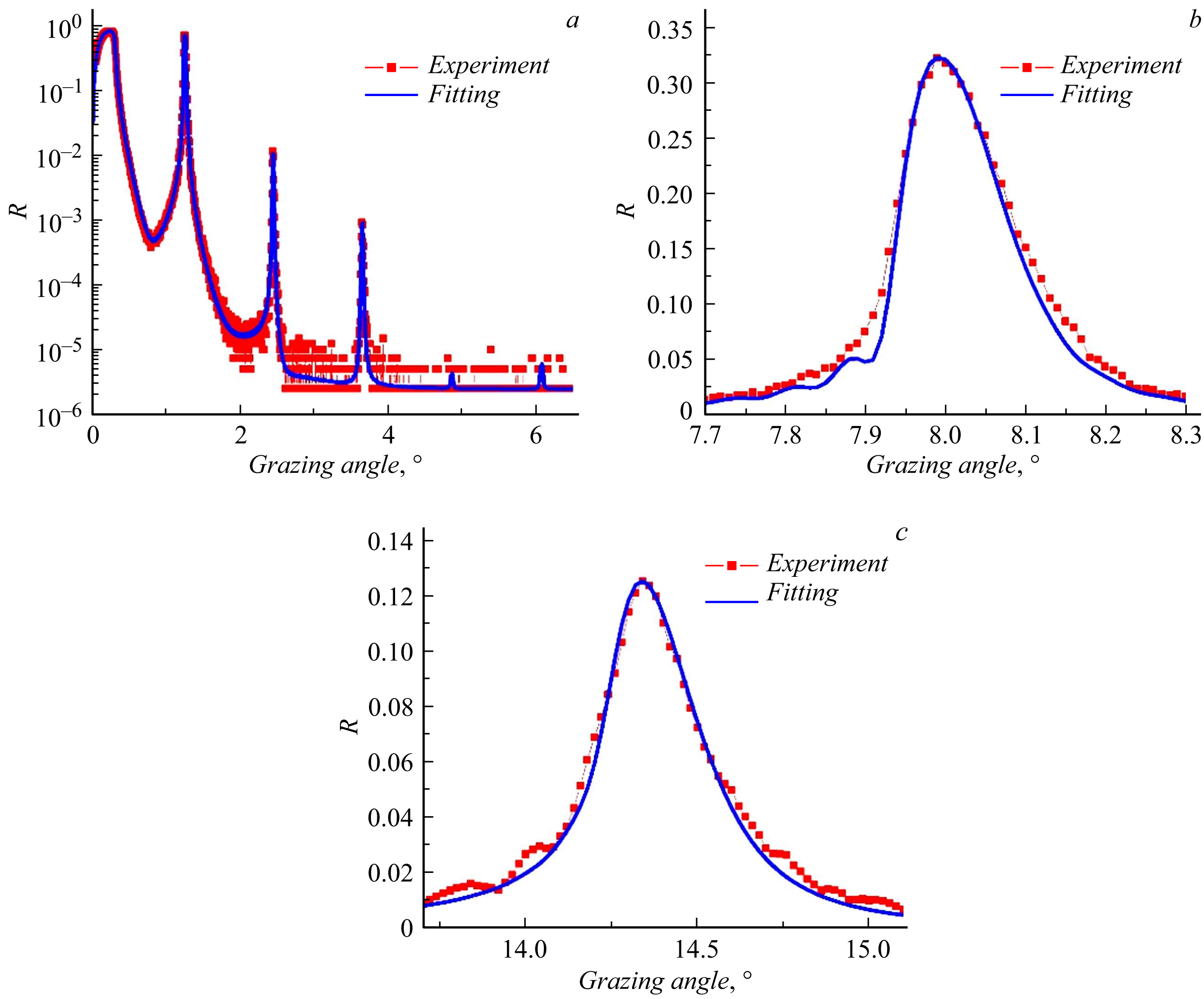

Figure 2. An example of simultaneous fitting of the angular dependences of the reflection coefficient measured at three wavelengths 0.154 (*a*), 0.989 (*b*) and 1.759 nm (*c*). Experimentally measured curves are shown by lines with dots, results of fitting are shown by solid lines.

Measurements at a wavelength of 0.154 nm were carried out using four-crystal high-resolution diffractometer PANalitycal X'Pert Pro. This device allows the study of crystalline materials and artificial multilayer systems by X-ray diffraction, including small-angle diffraction. The angular dependences of the reflection coefficient were recorded for all the samples studied in this work. The measurements were carried out in the angle range 0–6°. Diffuse scattering was measured for some samples in addition to angular dependencies. One of the types of diffuse scattering measurement was used in this study, in which the position of the goniometer of the sample (for example, $\theta = 1.51^\circ$) and the detector ($2\theta = 3.02^\circ$) corresponding to the first Bragg diffraction order was first selected, then the sample was scanned in this position (in the range of θ: $1.51-3.02^\circ$). The contribution of scattering roughness to the total transition region between layers for multilayer mirrors with periods 2.97 and 1.48 nm was separately defined using a combination of small-angle X-ray reflectometry and diffuse scattering methods, as well as numerical modeling.

Measurements in the field of soft X-ray radiation at wavelengths of 0.989 and 1.759 nm were performed using reflectometer with monochromator RSM-500 [11]. This device is designed in IPM RAS for the control of X-ray optical elements of arbitrary shape. The radiation source is a collapsible X-ray tube with replaceable target anodes, which allows for quick switching between wavelengths. Mg and Fe targets were used in the cases of wavelengths of 0.989 and 1.759 nm, respectively. Monochromatization of radiation is performed using lattice spectrometer-monochromator RSM-500, which ensures spectral resolution in the extreme ultraviolet (EUV) range of $\Delta\lambda/\lambda \approx 0.26\%$. An example of simultaneous fitting of the angular dependences of the reflection coefficient measured at three wavelengths for one of the samples is shown in Fig. 2.

Determination of the value of internal stresses is reduced to determining the radii of curvature of the substrate before and after deposition of a reflective coating on it. The radius of curvature of the substrate was determined using interferometer Zygo VeriFire 4 by comparing the profile of the substrate before and after application of the reflective coating with the reference profile. The interferometer measurement allows determining the deviation of the profile of the measured substrate from the profile of the reference. The reference plane is a transmission reference plane with size of 4 inches, reflection coefficient of 4%, shape accuracy better than $\lambda/20$. The claimed absolute error of determination of the profile using this device is 10 nm, the claimed maximum measured deviation from the plane is 15 μm. A detailed description of the measurements and calculation of the measurement error is given in Ref. [12]. The values of internal stresses in the multilayer mirrors were determined using the Stony formula [13]:

$$s = \frac{E}{6(1-\nu)}\frac{d_s^2}{d_f}\left(\frac{1}{r_2}-\frac{1}{r_1}\right), \quad (1)$$

where S — internal stresses, E — Young's modulus of the substrate material, ν — Poisson's ratio of the substrate material, d_s — substrate thickness, d_f — multilayer mirror total thickness, r_1 — initial radius of curvature of the substrate, r_2 — radius of curvature of the substrate after multilayer mirror deposition. The substrates had a size of 20×80 mm and a thickness of 0.47 mm. The error of determination of stress values, taking into account the thicknesses of reflective coatings of 364–235 nm (multilayer mirror periods of 3.6–1.5 nm), are of the order of $\pm 0.30 - \pm 0.46$ MPa, respectively. The substrates were cut out from plates of single-crystal silicon of orientation (100) with a diameter of 100 mm, used in the microelectronic production. Such plate had coefficient $E/[6(1-\nu)] = 30$ GPa.

The samples were annealed in a vacuum furnace at a pressure of $6 \cdot 10^{-5}$ Pa. The temperature was controlled by a chromel–alumel thermocouple with an accuracy of $\pm 5^\circ$C. One sample (with a period of 2.25 nm) was annealed at temperatures of 100, 200 and 300°C to trace the variation of the reflection coefficient of the multilayer mirror depending on the temperature. The annealing time was 1 h for each temperature (the time to maintain the annealing temperature without taking into account heating and cooling). The remaining samples were annealed at 300°C, the annealing time was also 1 h. Figure 3 shows the dependence of the sample heating temperature on time for the case of annealing at 300°C.

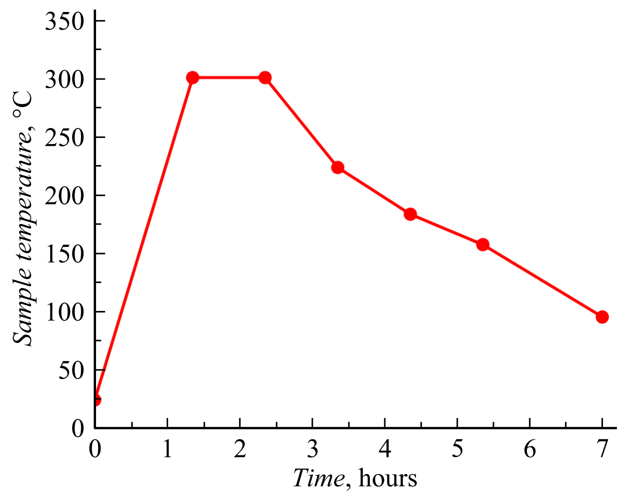

Figure 3. Dependence of the heating temperature of the sample on time for the case of annealing at 300°C.

2. Results and discussion

The thicknesses of the mirror materials were optimized at a constant period value at the first stage of the study for obtaining the maximum reflection coefficient. The optimization was performed by changing the parameter β_{Mo}, defined as the ratio of the molybdenum thickness in the period to the period value multilayer mirrors, and measurements of the angular dependence of the reflection coefficient in the first Bragg diffraction order at a wavelength of 0.154 nm. Spectral selectivity was also evaluated for each case ($\Delta\lambda/\lambda$). The estimation was carried based on the angular dependencies using the formula

$$\frac{\Delta\lambda}{\lambda} \approx \frac{\Delta\theta_{br}}{\mathrm{tg}(\theta_{br})}, \quad (2)$$

where λ — the wavelength of the radiation, $\Delta\lambda$ — the width of the spectral line at half height, θ_{br} — the angle corresponding to the Bragg peak, $\Delta\theta_{br}$ — the width of the Bragg peak at half maximum. A total of 5 samples were fabricated. The angular dependences of the reflection coefficient were measured for each sample and the main parameters of the studied samples were determined (Table 1). For clarity, the reflection coefficient and spectral selectivity data depending on the parameter β_{Mo} are shown in Fig. 4.

Fig. 4, *a* shows that a sample with the molybdenum proportion in the period equal to 0.33 has the maximum reflection coefficient in the studied series of five samples.

Table 1. The main parameters of the studied samples: β_{Mo} — molybdenum proportion in the period; d — mirror period; R — reflection coefficient; $\Delta\theta_{br}/\mathrm{tg}(\theta_{br})$ — characterizes the spectral selectivity of multilayer mirrors. The number of periods for each sample was $N = 100$. The measurements were performed at wavelength of $\lambda = 0.154$ nm

№of sample	β_{Mo}	d, nm	R, %	$\Delta\theta_{br}/\mathrm{tg}(\theta_{br})$, %
1	0.22	3.60	68.8	1.7
2	0.25	3.67	70.4	2.0
3	0.33	3.73	75.3	2.3
4	0.38	3.65	70.8	2.4
5	0.44	3.63	63.2	2.3

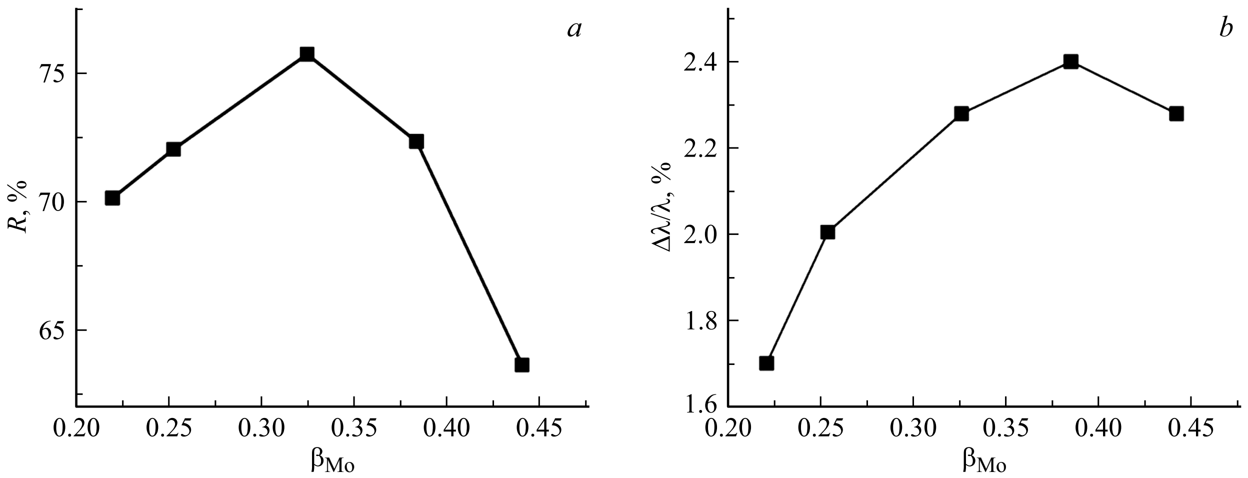

Figure 4. Experimental dependences of the peak reflection coefficient (a) and spectral selectivity (b) on the proportion of molybdenum in the Mo/Be multilayer mirrors period. The measurements were performed at the wavelength of $\lambda = 0.154$ nm.

The next step of the study was to determine the minimum possible value of the Mo/Be multilayer mirror period, at which diffraction orders are still observed based on the angular dependence of the reflection coefficient. A mirror with a high reflection coefficient and a ratio of material thicknesses in the period close to $\beta_{\text{Mo}} = 0.4$ was selected from the previous series of five samples for this purpose, so that it was possible to proportionally reduce the individual thicknesses of the mirror materials down to subnanometer values. Sample №4 from Table 1 was selected taking into account the noted conditions.

Next, a series of samples with periods in the range of 3.65−1.29 nm was fabricated. The ratio of material thicknesses in the period was maintained constant for all samples and was approximately 0.4. As the period decreased, the optimal value of the number of periods (N) was calculated for each case. The number of periods was selected by modeling in the „Multifitting“ program. At the same time, model data from the previous multilayer mirror sputtering were used for each subsequent multilayer mirror sputtering: a new multilayer mirror period was set in the new model, which was less than the previous one (while maintaining the condition $\beta_{\text{Mo}} = 0.4$), then the parameter N was increased in the model until the reflection coefficient (R) stopped significantly increasing (down to changes in tenths of a percent). The main parameters of the samples are listed in Table 2.

The multilayer mirror series was stopped at the period of 1.29 nm, since the reflection coefficient in the first Bragg order becomes lower than 1% with the lower values of d. Not only the reflection decreases when approaching the minimum multilayer mirror periods provided in Table 2, but also the shape of diffraction peaks (Fig. 5) on angular curves noticeably changes, starting from the period of $d = 1.64$ nm (Fig. 5, b). This indicates an increase of structural imperfection. There may be a partial loss of continuity of thin films of subnanometer thickness.

Table 2. The main parameters of the studied samples: β_{Mo} — molybdenum proportion in the period; d — mirror period; N — number of periods; M — layer material; $s(M)$ — width of the transition area (entry $s(M)$ corresponds to the area above M); R — reflection coefficient; $\Delta\theta_{\text{br}}/\,\text{tg}(\theta_{\text{br}})$ — characterizes the spectral selectivity of multilayer mirrors. The measurements were performed at the wavelength of $\lambda = 0.154$ nm

№ of sample	β_{Mo}	d, nm	N	s(Mo), nm	s(Be), nm	R, %	$\Delta\theta_{\text{br}}/\,\text{tg}(\theta_{\text{br}})$, %
4	0.38	3.65	100	0.35	0.52	70.8	2.4
6	0.40	2.97	120	0.31	0.51	59.0	1.7
7	0.40	2.50	120	0.32	0.54	41.9	1.3
8	0.39	2.25	120	0.33	0.53	35.0	1.1
9	0.40	1.99	130	0.31	0.55	19.2	1.0
10	0.39	1.69	140	0.31	0.57	10.3	0.7
11	0.42	1.64	140	0.30	0.74	6.4	0.7
12	0.41	1.48	140	0.31	0.78	2.6	0.7
13	0.41	1.29	160	0.33	0.78	1.4	0.6

However, a modulation of the electron density (dielectric constant) is observed despite such a change of the structure of the multilayer mirror at periods less than 1.69 nm. For comparison, Fig. 6 shows the electron density profiles for multilayer mirrors with the largest period in the series of $d = 3.65$ nm, with a period of $d = 1.69$ nm, after which the peak shape begins to change, and with one of the smallest periods of $d = 1.48$ nm.

Dependences of the reflection coefficient and spectral selectivity on the period of multilayer mirrors are plotted in Fig. 7 based on the data from Table 2. The depen-

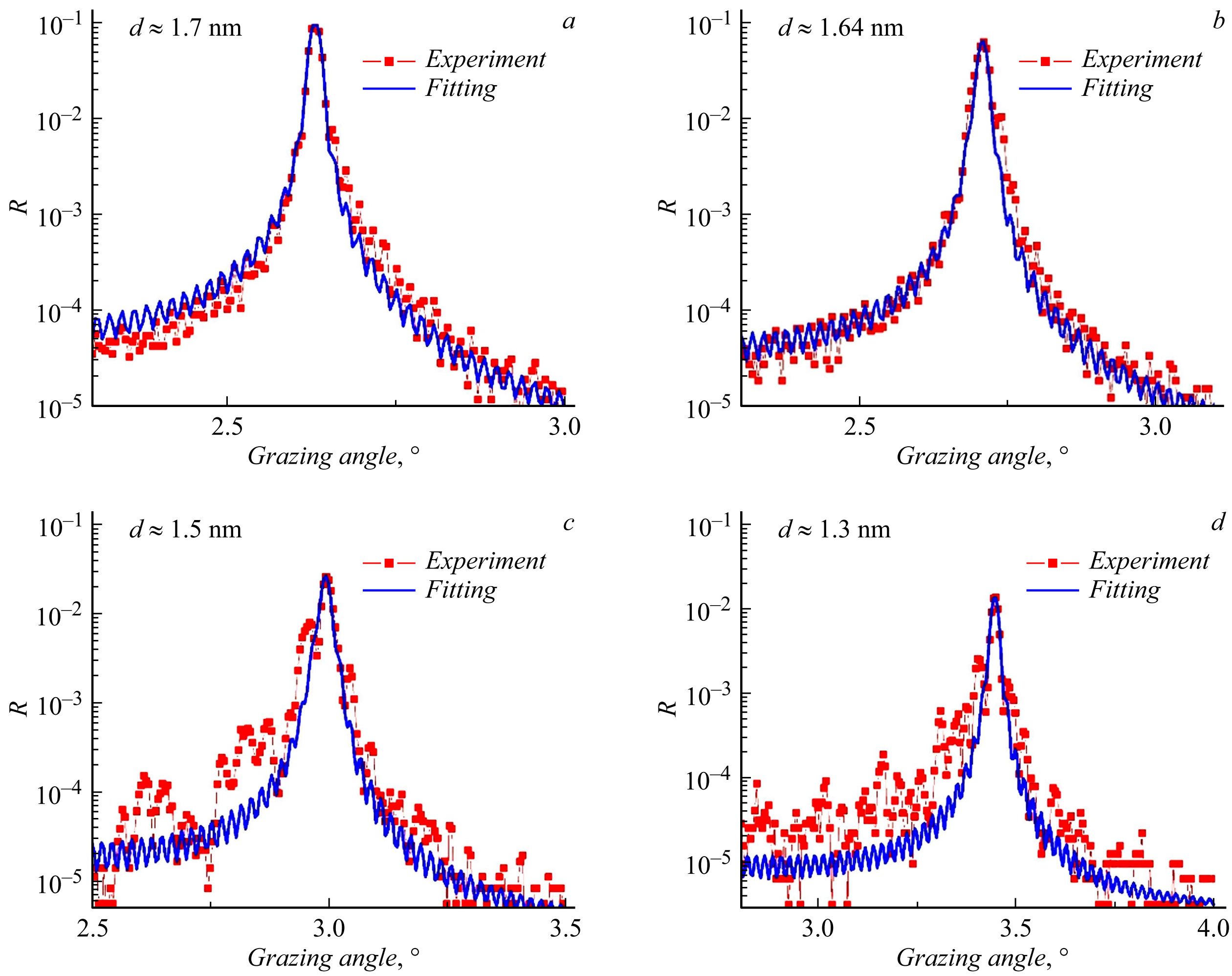

Figure 5. First orders of diffraction on experimentally measured angular dependencies of the peak reflection coefficient for Mo/Be multilayer mirrors with periods of $d = 1.69$ (a), 1.64 (b), 1.48 (c) and 1.29 nm (d). The measurements were performed at the wavelength of $\lambda = 0.154$ nm. Experimentally measured curves are shown by lines with dots, results of fitting are shown by solid lines.

dence $R(d)$ for each period value, but with zero transition region width ($s = 0$) is additionally shown on Fig. 7, a.

Fig. 7, a shows that the experimental reflection coefficient decreases more strongly compared to the theoretical one with a decrease of the multilayer mirror period (at ideal layer boundaries $s = 0$). The same cab be also noted by comparing this dependence $R(d)$ with a similar dependence for Mo/B_4C, given in Ref. [7], on which R does not fall so significantly with the decrease of the period. However, it is worth noting that a different approach was used in choosing N in the study of short-period Mo/B_4C multilayer mirrors. Perhaps the approach in choosing the number of periods for each subsequent multilayer mirror with a decrease of the period used in this paper is not entirely correct. When choosing a different approach, as in Ref. [7], where N was chosen from the condition of constancy of the total thickness of the mirror ($d_i N_i = \text{const}$), the experimental graph $R(d)$ in Fig. 7, a could look differently, on average the values of R would be larger. However, this would still not affect the type of dependency $s(d)$ shown in Fig. 8. It is possible to note the same decrease in the parameter $\Delta\theta_{\text{br}}/\,\text{tg}(\theta_{\text{br}})$ along with the decrease of the reflection coefficient, i.e. improvement of spectral selectivity (Fig. 7, b).

Changes of the Mo/Be multilayer mirror structure with a decrease of the period, as evidenced by changes of the reflection curves noted in Fig. 5, resulted in an increase of the width of the transition region between the layers, which ultimately led to such a sharp drop of the reflection coefficient. As can be seen in Fig. 8, a significant increase of the width of the transition region is observed with a decrease of the Mo/Be multilayer mirror period, starting from the value of $d = 1.69$ nm.

The transition regions between the layers in the multilayer mirror are formed due to several mechanisms, the main of which are the growth roughness and mutual mixing of materials, primarily due to the ballistic effect of the introduction of high-energy atoms (units-tens of eV) of the material entering the film, as well as diffusion and chemical interaction. It is possible to discriminate the

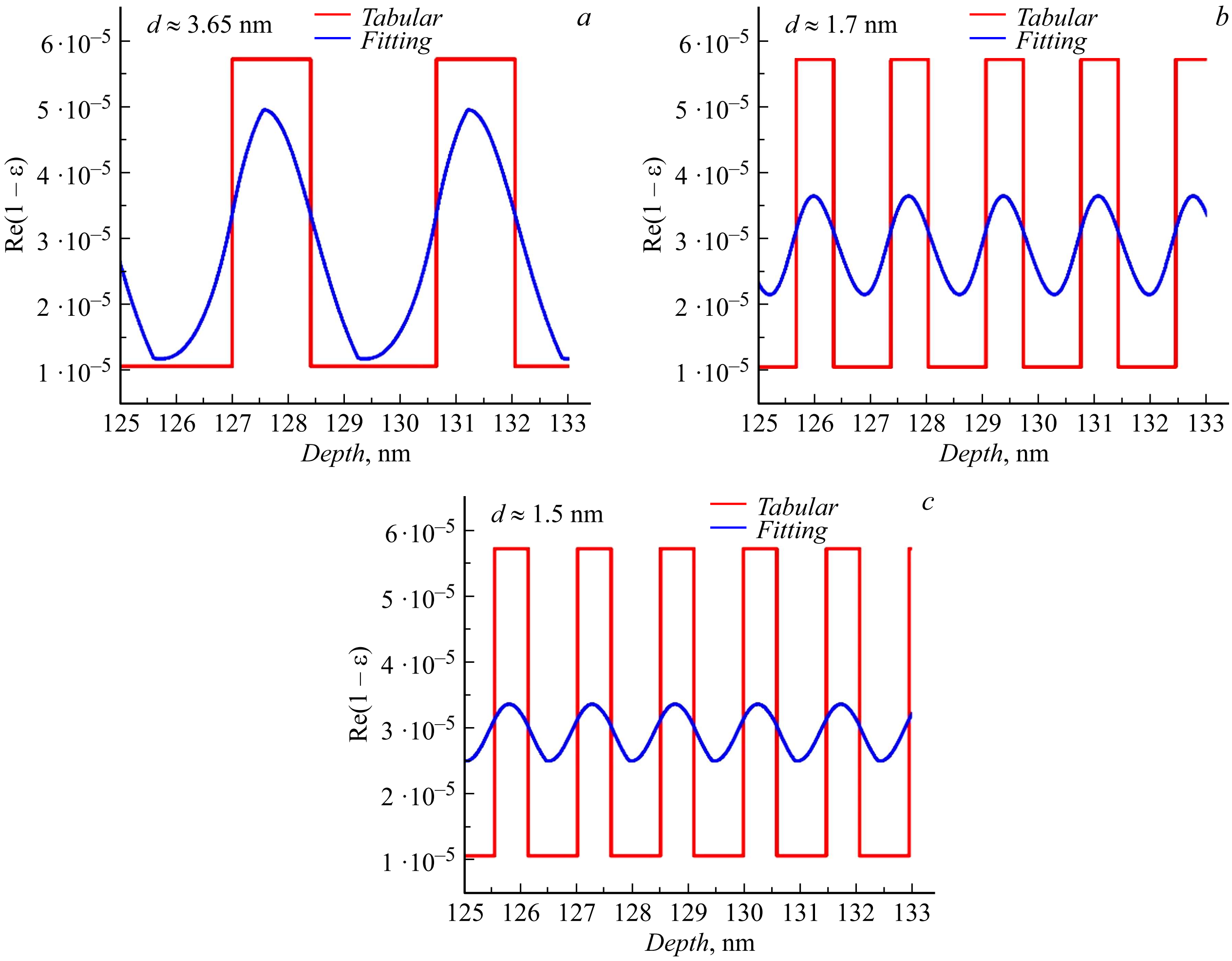

Figure 6. The electron density profiles of multilayer mirrors with periods of $d = 3.65$ (a), 1.69 (b), and 1.48 nm (c) reconstructed using X-ray reflectance data. The blue curve corresponds to the distribution of the electron density deep into the structure, taking into account the width of the transition region (s), the red curve corresponds to the case of an ideal structure ($s = 0$).

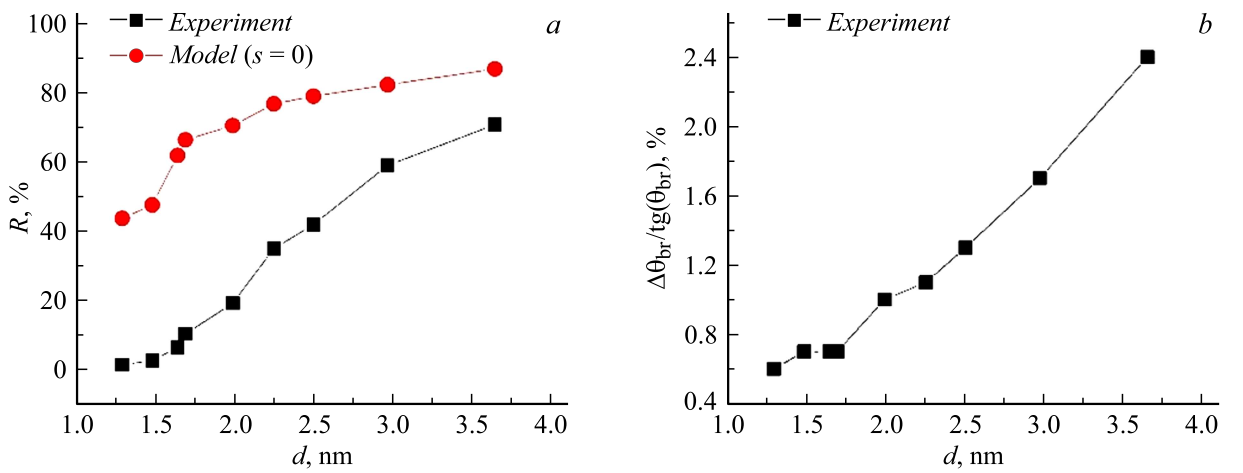

Figure 7. Experimental and theoretical dependences of the peak reflection coefficient (a) and spectral selectivity (b) on the Mo/Be multilayer mirror period. The measurements were carried out at a wavelength of $\lambda = 0.154$ nm.

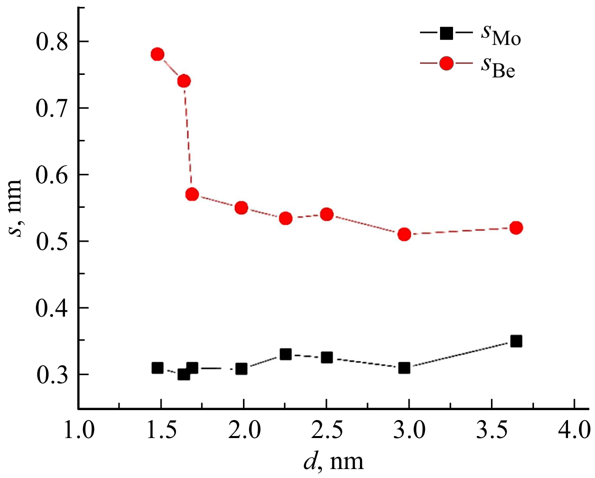

Figure 8. The dependences of the widths of the transition regions on the Mo/Be multilayer mirror period determined by the results of fitting.

Table 3. Parameters of the stress-tested samples: d — mirror period; N — number of periods; d_f — multilayer mirror total thickness; d_s — substrate thickness; S — internal stresses in multilayer mirrors

№of sample	d, nm	N	d_f, nm	d_s, nm	S, GPa
14	3.64	100	364.2	0.47	0.45
15	2.99	120	359.0	0.47	0.30
16	2.27	116	263.5	0.47	0.18
17	1.47	160	235.2	0.47	0.10
From [15]	5.80	70	403.0	0.50	0.33

contribution from the growth roughness to the total width of the transition region using the angular dependencies of the reflection coefficient and diffuse scattering curves at the same time with „Multifitting“ program. The contributions of growth roughness and mutual mixing for the Cr/Be multilayer mirror were discriminated in [14]. The diffuse X-ray scattering is used for this purpose because the interlayer roughness is responsible for scattering, while mixing leads only to a slight decrease of the intensity of scattered radiation due to a decrease of the electron density jump at the boundaries. Swing curves were measured based on this approach after measuring the mirror reflection curves at the wavelength of $\lambda = 0.154$ nm using the method described above for multilayer mirrors with periods of $d = 2.97$ and 1.48 nm (Fig. 9). The values of geometric roughness $\sigma \approx 0.11$ and ≈ 0.22 nm, respectively, were obtained as a result of the fitting. Thus, it can be concluded by comparing the obtained values of σ with the thicknesses of the transition regions in Table 2 that the contribution from geometric roughness to the transition region is not decisive.

Additional 4 multilayer mirrors were fabricated for the study of internal stresses with periods within the range of periods of the samples from Table. 2. The film parameters and the values of the internal stresses of the multilayer mirrors are listed in Table. 3. As can be seen from the above data, the stresses in the multilayer mirrors decrease with the reduction of the period. The sign of the parameter S indicates that the stresses are of a tensile nature. For comparison, the table shows data from the paper [15], which studied the Mo/Be multilayer mirrors with a greater period $d = 5.8$ nm and greater total thickness of 403 nm. The internal stress in this mirror turned out to be comparable to the stresses in the studied mirrors.

Sample No. 8 was selected (Table. 2) with a period of $d = 2.25$ nm to study the thermal stability at temperatures of 100, 200 and 300°C. Annealing at each temperature took

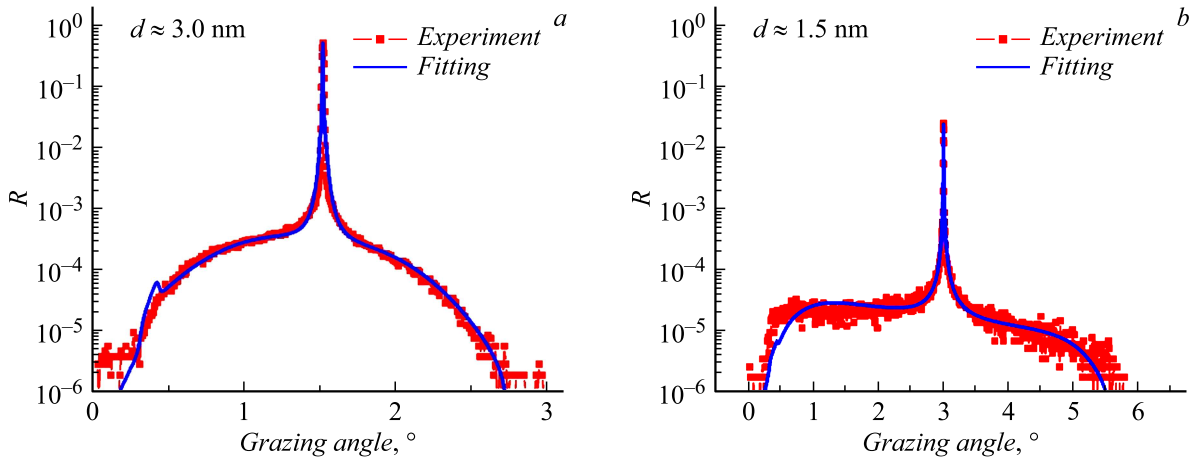

Figure 9. Experimental and fitting swing curves of multilayer mirrors with periods of $d = 2.97$ (a) and 1.48 nm (b). The mirror position for each sample corresponds to the first Bragg peak. Experimentally measured curves are shown by lines with dots, results of fitting are shown by solid lines.

Table 4. The period and X-ray optical characteristics of the studied multilayer mirrors before and after annealing at 300°C for 1 h. The measurements were performed at the wavelength of $\lambda = 0.154$ nm

Before annealing					After annealing			
Sample	d, nm	R_{1br}, %	Θ_{1br}	$\Delta\theta_{br}/\mathrm{tg}(\theta_{br})$, %	d, nm	R_{1br}, %	Θ_{1br}	$\Delta\theta_{br}/\mathrm{tg}(\theta_{br})$, %
4	3.65	70.8	1.25	2.4	3.64	55.4	1.25	2.2
6	2.97	59.0	1.51	1.7	2.95	43.6	1.52	1.6
7	2.50	41.9	1.79	1.3	2.49	23.8	1.80	1.2
8	2.25	35.0	1.98	1.1	2.24	15.8	2.00	1.0
9	1.99	19.2	2.24	1.0	1.98	8.3	2.26	1.1
10	1.69	10.3	2.63	0.7	1.69	2.6	2.64	1.2
12	1.48	2.6	3.00	0.7	1.48	0.7	3.01	1.2

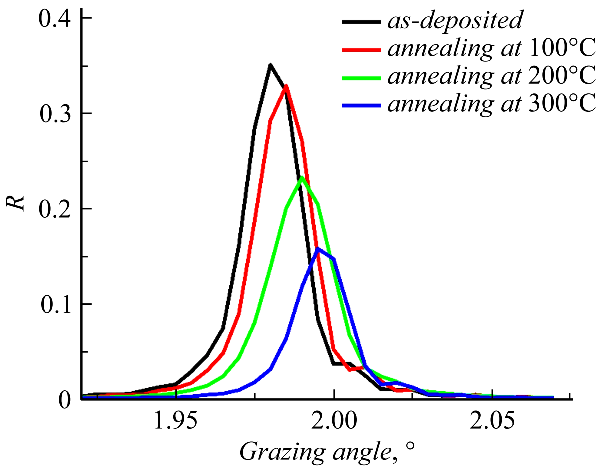

Figure 10. The first Bragg peaks of the measured angular dependences of the reflection coefficient before and after annealing. The measurements were performed at the wavelength of $\lambda = 0.154$ nm.

place for 1 h, the complete time dependence of the sample temperature on the time during annealing is shown in Fig. 3. The angular dependences of the reflection coefficient at a wavelength of 0.154 nm were measured before and after annealing. Figure 10 shows the first Bragg peaks of the angular dependence of the reflection coefficient before and after annealing.

The height and position of the Bragg peak change during annealing as can be seen from Fig. 10: the reflection coefficient and the multilayer mirror period decrease, respectively. Also, annealing was performed for all samples from Table 2 at a temperature of 300°C for 1 h. Reflection curves were also measured for all samples before and after annealing, after which the mirror parameters were restored using „Multifitting“̇The period and X-ray optical characteristics of the studied structures before and after annealing are given in Table 4. For clarity, the dependences of the reflection coefficient and spectral selectivity on the period are shown in Fig. 11.

The reflection coefficient decreases even faster after annealing at 300°C with a reduction of the period (Fig. 11, *a*). Spectral selectivity becomes slightly better after annealing, but not over the entire studied range of periods as can be seen in Fig. 11, *b*: its sharp degradation is observed starting from the period of $d = 1.99$ nm.

Conclusion

We studied the internal structure and thermal stability of short-period Mo/Be multilayer mirrors with periods from 3.65 to 1.29 nm. A total of 17 samples were used in the study. 9 samples were used in the study with the reduction of the multilayer mirror period. Such a relatively long series was chosen in order to see the intrastructural trends associated with an extreme reduction of the thickness of the main layers. Structures with ultra-short periods can be very useful for the use of focusing and collimating X-ray optical elements, but they should not result in the contamination of the working beam with scattered radiation.

We can draw the following conclusions as a result. The width of the transition region in Mo/Be mirrors strongly depends on the thickness of the period in the range of thicknesses of periods 3.65–1.29 nm and its sharp increase by 2 Åis observed starting from the period value of 1.64 nm. Once again, we note that this result is an observable trend and is significantly based on the observation of a large (nine) number of samples with a gradually changing period. The selection of roughness makes it possible to estimate the interpenetration of the materials of the layers and the resulting drop of optical contrast being the most significant at a period thickness of < 2 nm.

The determination of numerical values of stresses and the extent of the transition layer in the Mo/Be multilayer mirrors was an important result of the study from the point of view of X-ray optical applications. Certain widths of the

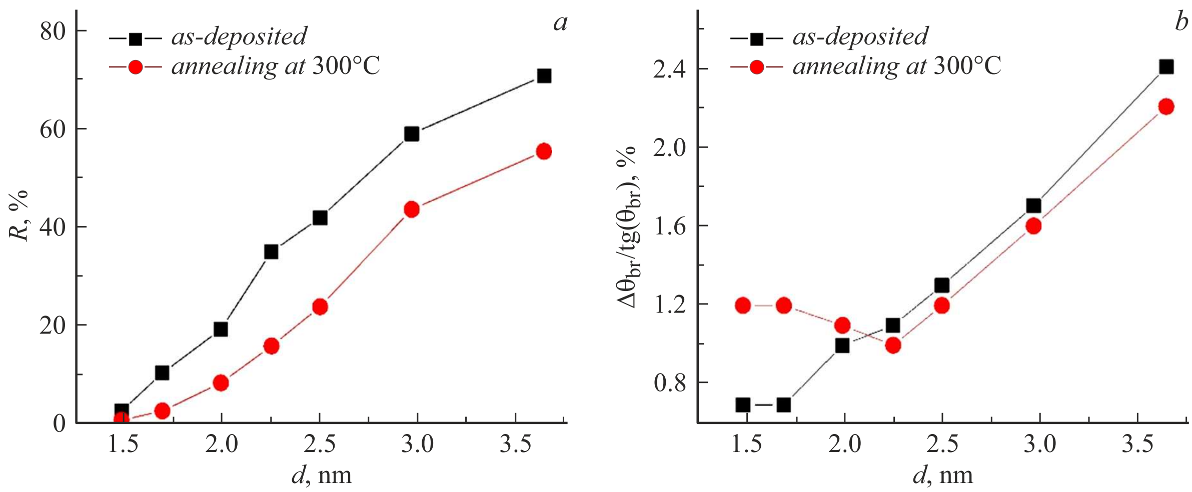

Figure 11. Experimental dependences of the reflection coefficient (a) and spectral selectivity (b) on the multilayer mirror period before and after annealing at 300°C for 1 h. The measurements were performed at the wavelength of $\lambda = 0.154$ nm.

transition layers make it possible to predict the X-ray optical characteristics of the multilayer mirror in a wide range of periods and wavelengths. The stress values obtained are at the level of $0.1-0.45$ GPa. However, the Mo/Be multilayer mirrors structure begins to degrade during annealing, at least starting at a temperature of 100°C, which results in a degradation of the X-ray optical characteristics of the mirror. The decrease of the reflection coefficient with a further increase of the annealing temperature, up to 300° C, is critical.

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Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] P.C. Pradhan, A. Majhi, M. Nayak. J. Appl. Phys., **123**, 095302 (2018). DOI: 10.1063/1.5018266

[2] M.S. Bibishkin, N.I. Chkhalo, A.A. Fraerman, A.E. Pestov, K.A. Prokhorov, N.N. Salashchenko, Y.A. Vainer. Nucl. Instrum. Methods Phys. Res. A, **543**, 333 (2005). DOI: 10.1016/j.nima.2005.01.251

[3] Yu.A. Vainer, A.E. Pestov, K.A. Prokhorov, N.N. Salashchenko, A.A. Fraerman, V.V. Chernov, N.I. Chkhalo. J. Exp. Theor. Phys., **103** (3), 346 (2006). DOI: 10.1134/S1063776106090020

[4] S.V. Rashchenko, M.A. Skamarokha, G.N. Baranov, Y.V. Zubavichus, I.V. Rakshun. AIP Conf. Proc., **2299**, 060001 (2020). DOI: 10.1063/5.0030346

[5] S. Roling, B. Siemer, F. Wahlert, M. Wöstmann, H. Zacharias, L. Samoylova, H. Sinn, S. Braun, P. Gawlitza, E. Schneidmiller, M. Yurkov, F. Siewert, E. Ziegler, O. Chubar. Proc. FEL-2013, WEPSO56, 627 (2013).

[6] R.A. Shaposhnikov, S.A. Garakhin, K.V. Durov, V.N. Polkovnikov, N.I. Chkhalo. Tech. Phys., **68** (7), 866 (2023). DOI: 10.61011/TP.2023.07.56629.77-23

[7] R. Shaposhnikov, V. Polkovnikov, S. Garakhin, Y. Vainer, N. Chkhalo, R. Smertin, K. Durov, E. Glushkov, S. Yakunin, M. Borisov. J. Synchrotron Radiat., **31** (2), 286 (2024). DOI: 10.1107/S1600577524000419

[8] M.V. Svechnikov, N.I. Chkhalo, S.A. Gusev, A.N. Nechay, D.E. Pariev, A.E. Pestov, V.N. Polkovnikov, D.A. Tatarskiy, N.N. Salashchenko, F. Schäfers, M.G. Sertsu, A. Sokolov, Y.A. Vainer, M.V. Zorina. Opt. Express, **26** (26), 33718 (2018). DOI: 10.1364/OE.26.033718

[9] R.M. Smertin, S.A. Garakhin, S.Yu. Zuev, A.N. Nechai, V.N. Polkovnikov, N.N. Salashchenko, M.V. Svechnikov, M.G. Sertsu, A. Sokolov, N.I. Chkhalo, F. Schäfers, P.A. Yunin. Tech. Phys., **64** (11), 1692 (2019). DOI: 10.1134/S1063784219110252

[10] M. Svechnikov. J. Appl. Crystallogr., **53** (1), 244 (2020). DOI: 10.1107/S160057671901584X

[11] M.S. Bibishkin, D.P. Chekhonadskih, N.I. Chkhalo, E.B. Kluyenkov, A.E. Pestov, N.N. Salashchenko, L.A. Shmaenok, I.G. Zabrodin, S.Y. Zuev. Proc. SPIE, **5401**, 8 (2004). DOI: 10.1117/12.556949

[12] R.M. Smertin, N.I. Chkhalo, V.N. Polkovnikov, N.N. Salashchenko, R.A. Shaposhnikov, S.Y. Zuev. Thin Solid Films, **782**, 140044 (2023). DOI: 10.1016/j.tsf.2023.140044

[13] G.G. Stoney. Proc. R. Soc. Lond. A., **82**, 172 (1909). DOI: 10.1098/rspa.1909.0021

[14] R. Pleshkov, N. Chkhalo, V. Polkovnikov, M. Svechnikov, M. Zorina. J. Appl. Crystallogr., **54** (6), 1747 (2021). DOI: 10.1107/S160057672101027X

[15] P.B. Mirkarimi. Opt. Eng., **38** (7), 1246 (1999). DOI: 10.1117/1.602170

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