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Structural and reflective characteristics of Cr/C multilayer mirrors synthesized by reactive sputtering method

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The paper presents the results of a study of the reflective characteristics and structural parameters of multilayer X-ray mirrors based on a pair of Cr/C materials optimized for the spectral range of wavelengths of 44–66 Å. The article also presents the results of a study of Cr/C structures synthesized in an argon + nitrogen gas mixture.

Keywords: Multilayer X-ray mirrors, magnetron sputtering, X-ray microscopy, carbon transparency window, biological research.

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Introduction

Multilayer X-ray mirrors comprise layers of different materials applied alternately to a substrate. At the same time, the period of the structure can vary very widely: from several angstroms [1] to hundreds of angstroms [2]. This fact distinguishes mirrors from crystals. One of the main laws describing multilayer X-ray mirrors is the Bragg-Wulff condition:

$$2 \cdot d \cdot \sin(\theta) = m \cdot \lambda, \quad (1)$$

where d is the period of the structure, θ is the angle of radiation slip, λ is the wavelength of radiation, m is the order of the Bragg peak. This equation establishes the relationship between the operating wavelength and the angular position of the reflection maximum of the mth order. Since the period is fixed for crystals, the optical circuit can only operate at certain angles for a given operating wavelength. At the same time, the use of multilayer X-ray mirrors makes it possible to design an optical circuit with a high degree of freedom for the operating wavelength, since the mirror period can be selected in such a way as to provide the operating angle most suitable for a specific task. Thus, in the case of imaging optics, such as X-ray telescopes [3], microscopes [4], lithographs [5], normal incidence mirrors are used to ensure the best resolution of the studied objects. In other applications, such as synchrotron radiation monochromatization systems, where it is required to ensure the narrowest possible width of the Bragg peak, sliding incidence mirrors are used [6]. Thus, the choice of multilayer mirrors instead of crystals makes it possible to significantly simplify the solution of various applied tasks.

One of the important practical applications of multilayer X-ray mirrors is X-ray microscopy, aimed at studying biological samples. At the same time, various spectral ranges are promising for this task. Thus, one of the wavelength ranges of interest for X-ray microscopy is the „water window" [7]. This is the wavelength range from 2.3 to 4.4 nm, in which oxygen, which is the absorbing component of water, has a relatively low absorption of X-ray radiation. When examining biological objects in this wavelength range, a high contrast is achieved between carbon-containing compounds, such as proteins, and water, which makes it possible to obtain images with a contrast approaching that achieved using fluorescent dyes.

Another important spectral region for microscopy of biological objects is the „carbon transparency window", with wavelengths of 4.4–6.6 nm. The advantages of this range for histological studies are discussed in Ref. [8]. The boundaries of the range are defined as follows. Fig. 1 shows the dependences of the real (δ) and imaginary (γ) parts of the refractive index n carbon and boron. The relationship of the refractive index with the parameters δ and γ is expressed by the following formula:

$$n = 1 - \delta + i \cdot \gamma. \quad (2)$$

Based on the presented dependencies, it can be seen that the carbon absorption coefficient drops sharply at wavelengths greater than 4.4 nm, which is due to the presence of the K-edge of carbon absorption. At the same time, boron has even lower absorption at wavelengths greater than 6.6 nm, which is due to the presence of the K-edge of boron absorption in this region. Thus, the wavelength range limited by the carbon and boron absorption edges is called the carbon transparency window, in which this material has one of the lowest absorption values. This paper is devoted to the study of the multilayer Cr/C structure, which is promising for this wavelength range.

The choice of materials for a multilayer mirror is extremely important. The general rule for choosing materials

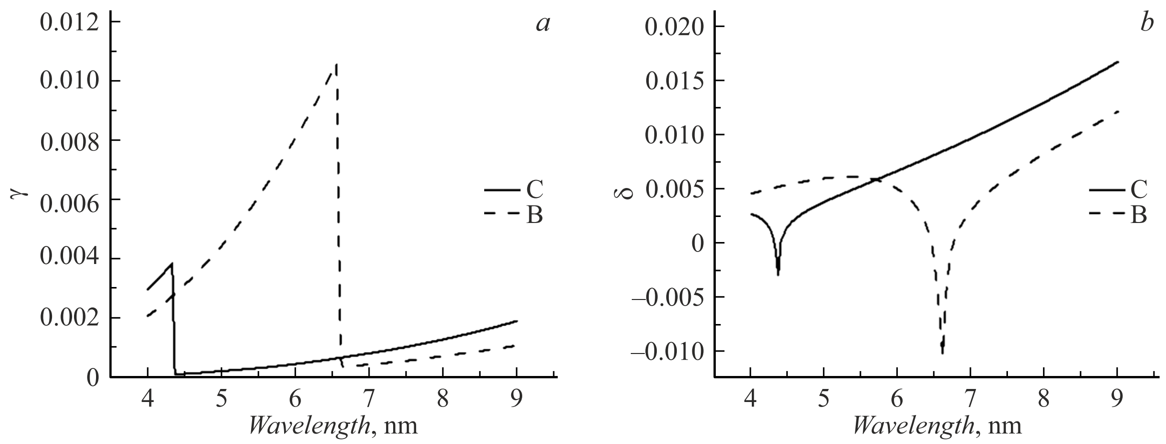

Figure 1. Dependence of the imaginary γ (a) and the real δ (b) parts of the refractive index of carbon (solid curve) and boron (dashed curve).

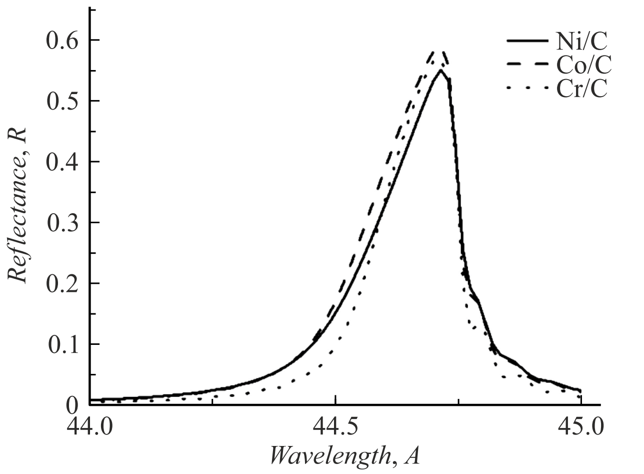

Figure 2. Dependences of the calculated reflection coefficient for ideal structures with grazing angles of 90° based on materials Ni/C (solid curve), Co/C (dashed curve) and Cr/C (dotted curve).

is described below [9]. First, a material is selected that has minimal absorption in the spectral region for which the mirror should be optimized. This material is called light, as well as spacer or weakly absorbent. It is paired with a material that, in the working wavelength range, would provide, on the one hand, a good optical contrast, i.e., a large jump in dielectric permittivity compared to the first material, and, on the other, would have low absorption. Since the frequency of X-ray radiation is significantly higher than the natural frequencies of most electrons in atoms, X-ray radiation actually interacts with a set of free electrons.

Carbon should be chosen as a weakly absorbing material for multilayer X-ray mirrors optimized for the wavelength range corresponding to the carbon transparency window, since it has the lowest absorption in this spectral region (Fig. 1). Calculations show that such materials as nickel, chromium, and cobalt can be selected as a highly absorbent material. Fig. 2 shows a graph of the wavelength dependence of the reflection coefficient of multilayer X-ray mirrors based on pairs of Ni/C, Cr/C, and Co/C materials. The calculation was carried out for mirrors with working grazing angles of 90°. Also, as part of the calculation, it was assumed that the structures had zero interlayer roughness and tabular values of material densities, i.e., the calculation was carried out for ideal structures.

The presented graphs show that theoretically the structures based on Cr/C and Co/C have the best reflection: $R = 56.9\,\%$ and $58.9\,\%$ respectively. Thus, it is the above pairs of materials that should be considered promising for creating multilayer X-ray mirrors based on them, optimized for operation in the spectral range of 4.4–6.6 nm. The advantage of Cr/C mirrors is the absence of Cr magnetic properties, which greatly simplifies their production.

It is worth noting that multilayer X-ray mirrors based on Cr/C pairs of materials have already been studied by various research groups. For instance, structures with a period of $d = 11.64$ nm were synthesized in Ref. [10]. The number of periods was $N = 20$. The studies presented in this paper found that the roughness increases from 0.32 to 0.49 nm with an increase in the parameter β, which is defined as the ratio of the thickness of the highly absorbent material in the period to the value of the period (in this case, $\beta = d_{\mathrm{Cr}}/d$, where d_{Cr} is the thickness of the chromium layers, d is the period of the multilayer structure). The samples were measured in the field of hard X-ray radiation with an energy of 8.04 keV. It is also shown in this paper that the synthesized structures have zero internal stresses at a value of $\beta = 0.45$. Also, the reflection coefficient was measured for a structure with $\beta = 0.37$, which was equal to $R = 26.6\,\%$ at a radiation energy of 1.04 keV. The magnitude of the internal stresses of such a structure was 261.72 MPa.

The presence of internal stresses in the structures leads to a change in their surface shape, which, in turn, leads to a deviation of the beam path from the calculated one.

Specifically, a flat quartz substrate with a diameter of 200 mm and a thickness of 20 mm will have a deflection arrow of about 20 nm after sputtering of the mirror for a value of 261 MPa. This value is quite large for diffraction-quality optics, in which standard deviations of the surface shape are considered acceptable no more than $\lambda/14$, where λ is the operating wavelength. Thus, when designing an optical circuit, it is necessary to take into account the presence of internal stresses in multilayer structures. Also, if the internal value exceeds a critical value, the reflective coating may peel off from the substrate.

1. Multilayer mirrors based on Cr/C

Reflection coefficient values were obtained in Ref. [11] for multilayer X-ray mirrors based on Cr/C pairs of materials $R = 7.5\,\%$ at a grazing angle of 85° and $R = 13.61\,\%$ for s-polarized radiation at a grazing angle of 44°.

Cr/C multilayer X-ray mirrors with a period of $d = 3.86$ nm, $\beta = 0.6$, $N = 100$ were synthesized in Ref. [12]. The reflection coefficient was $R = 21.8\,\%$ for s-polarized radiation with an energy of 250 eV (wavelength is 4.96 nm) at a grazing angle of 40.7°. The roughness of the chromium and carbon layers had the following values: 0.4 and 0.32 nm, respectively.

Cr/C multilayer structures with the period of $d = 9$ nm, $\beta = 0.33$ and the number of periods of $N = 10$ were synthesized [13]. A reflection coefficient of $R = 23\,\%$ was obtained at a wavelength of 4.48 nm at a grazing angle of 14°. The roughness values for the chromium and carbon layers were 0.42 and 0.26 nm, respectively.

Cr/C mirrors with periods of $d = 3.25$ nm, $\beta = 0.4$ and the number of periods of $N = 150$ were synthesized in Ref. [14]. The reflection coefficient of $R = 18.9\,\%$ was obtained in this study at a wavelength of 6.42 nm and a grazing angle of 88°. The values of interlayer roughness were 0.35 nm.

The value of the reflection coefficient of $R = 7\,\%$ was obtained in Ref. [15] for Cr/C multilayer X-ray mirrors with a period value of $d = 2.5$ nm and the number of periods of $N = 110$ at the wavelength 5 nm. The radiation grazing angle was 78°. The recovered roughness values were 0.35 nm.

Multilayer X-ray mirrors were synthesized in Ref. [16] based on a pair of Cr/C materials with a period value of $d = 11.51$ nm, $\beta = 0.37$ and corresponding layer thicknesses of $d_{\mathrm{Cr}} = 3.25$ nm and $d_{\mathrm{C}} = 6.16$ nm. The values of transition regions (interlayer interfaces) were 1.3 nm for the Cr-to-C boundary and 0.8 nm for the C-to-Cr boundary. The synthesized structures were irradiated with synchrotron radiation and heated to fixed temperatures. Irradiation with synchrotron radiation with a power density of 0.1 W/mm^2 and an energy of 1183.6 eV for 18 h led to contamination of the surface: a spot appeared in the place where the beam fell. At the same time, measurements of the reflective characteristics in the hard X-ray wavelength range (radiation energy $E = 8.04$ keV) showed no difference for the area where the beam fell and outside it. At the same time, the measurement of the reflection coefficient for radiation with an energy of 1183.6 eV showed that in the irradiated area the reflection coefficient was about 20 %, and outside the spot about 26 %. The reflective characteristics of mirrors were also measured at incident radiation energies from 1120 to 1400 eV at a grazing angle of 87° and at energies from 900 to 1250 eV at an angle of 86.4°. The reflection coefficient during annealing increased from $R = 27.9\,\%$ in the first case and from $R = 21.6\,\%$ in the second case by several percent. After annealing at 200 °C the values of the reflection coefficient were equal to $R = 31.7\,\%$ and 25.6 %, respectively. A decrease in the reflection coefficient was observed with further heating. The reflection coefficient was equal to $R = 29.2\,\%$ and 22.4 % in case of annealing at a temperature of 700 °C, however, this is even greater than the original value. This effect is explained by an increase in chromium density and a decrease in carbon density. Thus, this paper demonstrates that Cr/C mirrors have excellent temperature stability and can be used in synchrotron radiation monochromators.

At the moment, the highest experimentally obtained reflection coefficient for Cr/C multilayer X-ray mirrors of normal incidence in the vicinity of the carbon absorption edge was published in Ref. [17] and was equal to $R = 15.4\,\%$ at a radiation grazing angle of 81.62°. The measurements were carried out at a wavelength of 4.47 nm.

The reflection coefficient values obtained experimentally for real structures are significantly lower than the theoretical values for ideal structures. There are several factors that negatively affect the reflective characteristics. Firstly, such factors include the formation of transition regions at the boundaries between layers of different materials, which are obtained due to mixing, chemical interaction, and mutual diffusion of atoms of different layers. This effect leads to the fact that the value of the dielectric constant jump at the boundaries of the multilayer structure decreases, which, in turn, leads to a deterioration in reflection from each boundary, and, consequently, the structure as a whole has a lower reflection coefficient compared to the ideal one with absolutely sharp boundaries. Secondly, in addition to blurring the boundaries, there is also the effect of their distortion as a whole, i.e. the formation of so-called interlayer roughness. It leads to the fact that part of the radiation is scattered into angles other than the mirror angle, which, in turn, leads to a decrease in the reflection coefficient in the direction of the mirror angle. This effect is also called diffuse scattering. Thirdly, the densities of materials in thin films, as well as in multilayer structures, may differ significantly from the tabular values. This fact can have both a positive effect on the reflective characteristics and lead to an increase in the reflection coefficient, and a negative effect and lead to a decrease in the reflection coefficient. The reflection from each boundary is determined by a jump in the dielectric constant, which for the X-ray wavelength range means a jump in the electron density. Thus, in the case when the density of the light material is less than tabular, the optical contrast increases, and, consequently, the reflection coefficient increases. If the density of heavy material decreases in the multilayer structure, then the optical contrast, on the contrary, deteriorates, and the reflection coefficient decreases.

Interface engineering techniques are used to reduce the impact of these effects in real conditions. Among these techniques, there are 3 main ones that are most often used to improve the reflective characteristics of multilayer X-ray mirrors. Firstly, passivation of layers with nitrogen is a common method of interface engineering. This method is as follows. The layers in the structure are synthesized in pure argon. In this case, nitrogen is injected into the working vacuum volume after application of the material layer. Nitrogen injection stops before the next layer of material is applied. This method has been successfully applied in a number of studies [18,19]. In particular, passivation of chromium layers allowed obtaining record values of the reflection coefficient $R = 23.8\,\%$ at a wavelength of 3.14 nm in Ref. [20] for multilayer structures based on a pair of Cr/Sc materials.

Another well-developed method of interface engineering related to the use of nitrogen is the so-called reactive sputtering, which consists in the synthesis of a multilayer structure in a mixture of gases „argon + nitrogen“. The use of this method for LaN/B structures optimized for operation in the vicinity of a wavelength of 6.7 nm allowed us to obtain a record reflection coefficient for this spectral region $R = 64.1\%$ at an angle of incidence of 1.5° from the normal at a wavelength of 6.65 nm [21].

The use of barrier layers consisting in applying thin layers of a third material into a two-component structure is another well-developed method of interface engineering. Carbon [22] and boron carbide [23–25] are most often used as materials for barrier layers.

Multilayer X-ray mirrors based on a pair of chromium and carbon materials are studied in this paper, and the effect of reactive sputtering of materials in a mixture of gases „argon,+,nitrogen“ on the reflective characteristics and structural parameters of Cr/C mirrors is studied.

2. Experimental methodology

Multilayer X-ray mirrors were synthesized by magnetron sputtering in a facility equipped with four planar magnetrons [26]. The scheme of this setup is shown in Fig. 3.

The magnetron discharge parameters had the following values $I_{\mathrm{Cr}} = 360$ mA, $U_{\mathrm{Cr}} = 303$ V, $I_{\mathrm{C}} = 800$ mA, $U_{\mathrm{C}} = 302$ V. Prior to the start of synthesis, residual gases were pumped out of the vacuum volume to a pressure value at the level of $5 \cdot 10^{-7}$ Torr. High-purity (99.998 %) argon was used as the working gas. The pressure of the working gas during the synthesis was at the level of $1.3 \cdot 10^{-3}$ Torr. The inlet of the working gas into the vacuum volume was carried out through a special gas flow

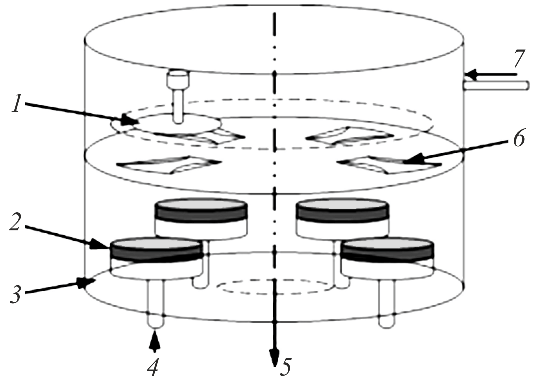

Figure 3. Schematic representation of a magnetron sputtering facility for the synthesis of multilayer X-ray mirrors: *1* — substrate with motor, *2* — target, *3* — magnetron, *4* — cooling, *5* — pumping system, *6* — shaped orifices, *7* — gas supply.

regulator, which allowed for a time-stable gas flow from the cylinder into the vacuum volume. The reflective coating was applied to silicon substrates with a RMS roughness value of 2 A. During the synthesis of the structure, the substrate alternately passed over magnetrons equipped with chromium and carbon targets. The uniform distribution of the substance flow along the lateral coordinate was ensured by shaped diaphragms located on special casings between the target and the substrate. The uniformity of the distribution over the period along the lateral coordinate is at the level of 0.5

Nitrogen was injected into the vacuum volume in the same way as argon was injected through the gas flow regulator (GFR). Variation of the degree of opening of the GFR flap allows changing the partial pressure of nitrogen in a mixture of gases „argon + nitrogen“. Also, software specially developed at IPM RAS allows for both constant nitrogen injection and addition to a vacuum volume at specified times, as a result of which both materials and individual layers can be sputtered into a mixture of gases.

Samples are loaded and unloaded from the working volume using a special vacuum lock, which is cut off from the working volume by a gate. The use of a vacuum lock is very important for several reasons. Firstly, it allows maintaining a high vacuum in the main volume, which is necessary to obtain highly reflective structures. Secondly, when atmospheric air is injected into a vacuum volume, it takes about a day to reach the pressure of residual gases at the level of 10^{-7} Torr. At the same time, the air is pumped out from the airlock chamber to a high vacuum within about 30–40 min. Thus, in the case of a reloading through the main vacuum volume, productivity would be limited to one sample per day. The use of a vacuum lock makes it possible to increase the productivity of the installation up to 2–3 samples per day. Thirdly, the targets of chemically active materials such as lanthanum, strontium, yttrium, and boron begin to degrade dramatically upon contact with the atmosphere. The use of an airlock for sample reloading

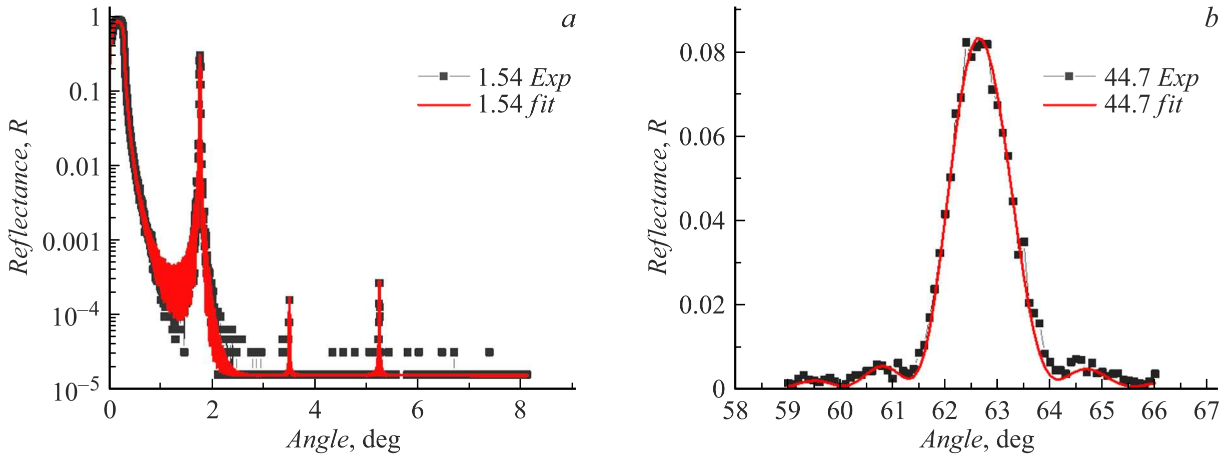

Figure 4. Dependence of the reflection coefficients of the multilayer Cr/C structure on the radiation grazing angle at wavelengths of 1.54 (*a*) and 44.7 (*b*). Curves with dots correspond to experimental measurements, solid curves correspond to fitting.

makes it possible to avoid prolonged contact of targets with the atmosphere, which makes experiments with chemically active materials possible in principle.

Magnetrons are powered by stabilized current sources developed at IPM RAS. The installation management system, software, and user graphical interface were developed at IPM RAS. The stepper motor is controlled by applying sync pulses to the stepper motor driver. The frequency of the synchro pulses determines the speed of passage of the substrate over the target of the sprayed substance. The gas flow rate and the turbomolecular pump are controlled by sending commands via the RS-485 interface in accordance with the data exchange protocol established by the equipment manufacturer.

The dependences of the reflection coefficient on the angle of radiation slip were measured both in the field of hard X-ray radiation with a wavelength of 1.54 Å on a four-crystal diffractometer Panalitical X'Pert PRO and at a working wavelength of 44.7 Å on a laboratory reflectometer equipped with a monochromator RSM-500 [27].

The structural parameters of the synthesized samples were reconstructed using the software Multifitting [28], developed in IPM RAS. This software allows simultaneous adjustment of several experimentally measured curves of the dependence of the reflection coefficient on the angle of radiation slip and on the wavelength. Based on this adjustment, structural parameters such as the period of the multilayer structure, the individual thicknesses of the layers of different materials in the period, the density of the materials, as well as the magnitude of the interlayer interfaces are restored. Simultaneous adjustment of the experimental curves measured in both the hard and soft X-ray wavelength ranges makes it possible to reconstruct the structural parameters with high accuracy. The method of recurrence relations is used to calculate the reflection curves. In this case, the transition region at the boundaries of the multilayer structure is defined by a linear combination of functions with different weight coefficients, which improves the quality of the fit. An example of such a fit is shown in Fig. 4. The Multifitting program also provides the ability to analyze diffuse scattering curves, which makes it possible to determine the amount of geometric roughness that leads to scattering of incident radiation into angles other than specular.

3. Results and their discussion

At the first stage of the experiments, the synthesis of multilayer normal-incidence X-ray mirrors based on a pair of Cr/C materials in pure argon with a different proportion of chromium thickness per period was conducted. The period value for all samples was $d = 25$ Å. The number of periods for each structure was $N = 80$. Fig. 5 shows the dependence

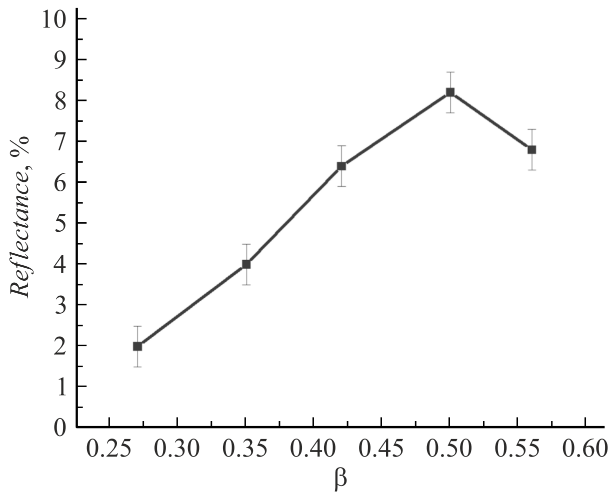

Figure 5. Measured dependence of the reflection coefficient of multilayer X-ray mirrors Cr/C on the parameter β.

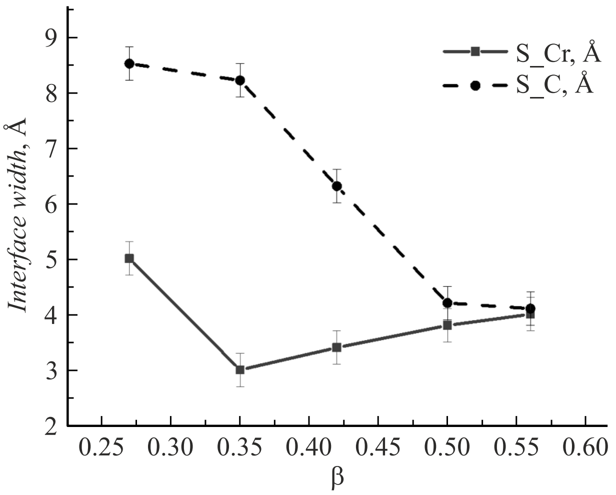

Figure 6. Dependence of the magnitude of the transition regions on the parameter β for the boundaries of chromium (solid curve) and carbon (dashed curve).

of the measured reflection coefficient at a wavelength of 44.7 A on the parameter β.

Despite the fact that theoretical calculations show that the optimal value of β in terms of obtaining the maximum reflection coefficient is about 0.4, the experimental optimum is around 0.5. This result is explained by the fact that a sharp increase in the transition region at the Cr-to-C boundary is observed with a decrease in the chromium fraction in the period starting from the values of $\beta = 0.5$, which leads to a decrease in the reflection coefficient of the mirror. At the other boundary, C-to-Cr, there is a slight decrease in the transition region with a decrease in the chromium fraction with a sharp increase, starting from the values of $\beta = 0.35$. The graph of the dependence of the magnitude of the transition regions on the parameter β is shown in Fig. 6. Next, a structure was synthesized based on a pair of Cr/C materials, with the number of periods of $N = 200$, the period value of $d = 22.78$ A and the value $\beta = 0.5$. The reflection coefficient of such a structure was $R = 15\,\%$ at a wavelength of 44.7 Å.

At the second stage of the experiments, the reflective characteristics and structural parameters of multilayer Cr/C X-ray mirrors synthesized in a mixture of gases „argon,+,nitrogen“ were studied. The period of the structures was $d = 25$ Å, the parameter value was $\beta = 0.5$, the number of periods was $N = 80$. Fig. 7 shows the dependence of chromium density on the partial pressure of nitrogen in a mixture of gases. Fig. 8 shows the dependence of the size of the transition regions at the boundaries of the structure on the proportion of nitrogen in the gas mixture.

It can be seen from the presented dependencies that the transition region at the C-to-Cr boundary begins to decrease with an increase in the proportion of nitrogen in the mixture. The size of the transition region at the Cr-to-C boundary does not change. However, an increase in the partial pressure of nitrogen also leads to a decrease in the density of chromium layers in the multilayer structure, as a result of which the optical contrast at the boundaries deteriorates. Thus, despite the fact that the boundaries become sharper with an increase in the proportion of nitrogen, the overall reflection coefficient of the structure begins to fall. Therefore, chromium must be sputtered in pure argon.

Based on the above, Cr/CN structures were synthesized. During the synthesis of these structures, argon was continuously injected into the working volume. In this case, nitrogen was added to the chamber only during the passage of the substrate over the carbon target. Thus, the chromium layers were sputtered in pure argon, and the carbon layers

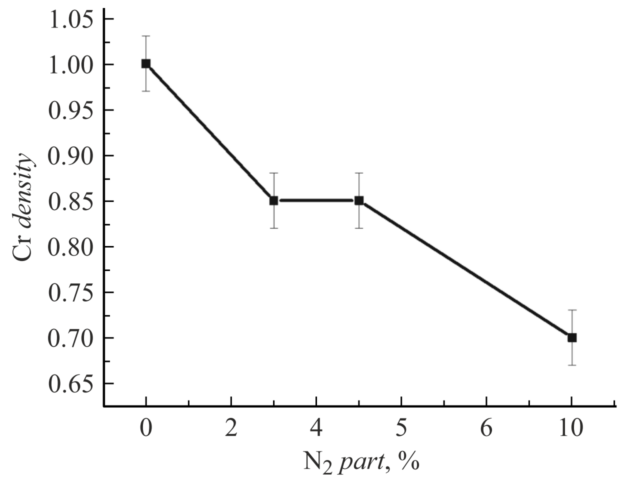

Figure 7. Dependence of chromium density (values are given in fractions relative to the tabular value of metallic chromium) on the partial pressure of nitrogen in a mixture of gases „argon + nitrogen“.

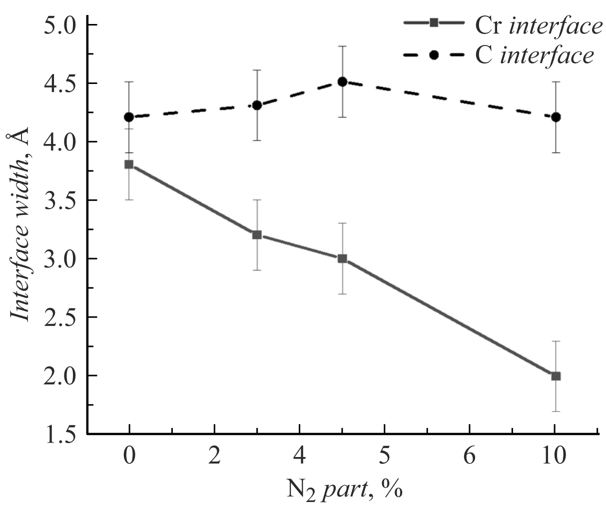

Figure 8. Dependence of the magnitude of the transition regions of chromium and carbon on the partial pressure of nitrogen in a mixture of gases „argon + nitrogen“. The solid curve corresponds to chromium, the dashed line corresponds to carbon.

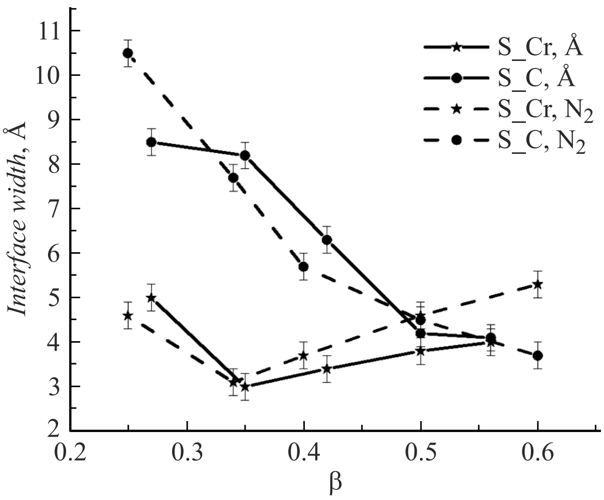

Figure 9. Dependences of the magnitude of the transition regions of chromium (curves with stars) and carbon (red with circles) for the case when structures were synthesized in pure argon (solid curves), as well as for the case when carbon layers were synthesized in a mixture of gases „argon + nitrogen“ (dashed curves).

in a mixture of gases. Fig. 9 shows the dependences of the magnitude of the transition regions on the parameter β for the case when both materials were sprayed in pure argon and for Cr/CN structures. From the presented dependencies, it can be concluded that spraying in a mixture of gases does not lead to an improvement in the transition regions.

Thus, it can be concluded that reactive carbon spraying did not allow to increase the reflection coefficient relative to the case when nitrogen was not added to the system.

Conclusion

As part of this work, the reflective characteristics and structural parameters of multilayer X-ray mirrors based on a pair of Cr/C materials were studied. It was found that the size of the transition region at the Cr-to-C boundary increases with a decrease in the chromium fraction in the period at values $\beta < 0.5$. Thus, $\beta = 0.5$ is optimal for obtaining the maximum reflection coefficient. The reflection coefficient of the complete structure with the number of layers $d = 200$ and $\beta = 0.5$ was $R = 15\,\%$. Synthesis of both layers of the structure in a mixture of gases „argon + nitrogen“ leads to a decrease in the transition region at the C-to-Cr boundary, as well as to a decrease in chromium density. The negative effect of reducing the optical contrast is more significant, as a result of which the reflection coefficient decreases with increasing partial pressure of nitrogen in the gas mixture. During the synthesis of chromium layers in pure argon, and carbon layers in a mixture of gases „argon + nitrogen“ the values of the transition regions did not decrease compared to the case when both materials were sprayed in pure argon. Based on the above, it can be concluded that interface engineering, which consists in reactive spraying of materials, does not allow increasing the reflection coefficient of multilayer X-ray mirrors based on a pair of Cr/C materials.

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Conflict of interest

The authors declare that they have no conflict of interest.

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