

Highly reflective Ru/Y multilayer mirrors for the spectral range of 9-11 nm

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Abstract: The results of the investigation of the reflective characteristics of multilayer mirrors based on Ru/Y are presented. Reflection coefficients at the level of 38.5% at an operating wavelength of 9.4 nm. It is shown that the deposition of B₄C barrier layers onto Y layers makes it possible to significantly increase the reflection coefficient compared to structures without barrier layers. A reflectance of 54% was obtained for mirrors optimized for 11.4 nm, which is close to the theoretical limit for these materials.

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1. Introduction

The spectral region 9-11 nm is of interest in relation to a number of scientific and technological applications. In particular, it is important for solar astronomy due to the presence of a strong emission line of the FeXVIII ion in the corona spectrum at $\lambda = 9.34$ nm [1]. This range is also interesting for the next generation EUV lithography with a wavelength shorter than 13.5 nm [2], as this range contains the maxima of krypton and xenon plasma radiation [3,4]. The efficiency of these applications largely depends on the reflectivity of the multilayer mirrors (MLMs) used in the construction of X-ray optical schemes. Theoretical calculations demonstrate that strontium (up to 70%) and yttrium (Fig. 1) are the best materials for weakly absorbing layers in this wavelength range, whereas Pd, Ag, Ru, Mo are the most suitable for scattering layers. The results of calculations for Pd/Y, Ru/Y, Mo/Y, Ag/Y and Ru/Sr multilayer mirrors are presented in Data File 11, Data File 12, Data File 13, Data File 14 and Data File 15 correspondingly.

The Mo/Sr MLMs were experimentally investigated in the paper [5]. Mirrors with a reflectivity of 40.8% were obtained at theoretically achievable values of more than 70% (wavelength 9.4 nm). However, the thin-film structures based on strontium were rapidly oxidized due to its high chemical activity. The reflectivity coefficient values dropped from the original ones 40.8% to below 1% after 24 hours from the moment of synthesis, which makes them unsuitable for practical applications. Attempts of the multilayer structure passivating and the producing of a protective carbon layer were unsuccessful. The synthesis technology used for these structures was not developed further.

A much larger number of works, including those currently being conducted, are devoted to yttrium-containing MLMs. The Mo/Y MLMs were investigated in [6–13]. The maximum reflection of about 30% was experimentally obtained for a theoretical value of 42% (at $\lambda = 9.34$ nm). The asymmetry of the interfaces is shown in [8] (the Y-on-Mo border has a length of about 0.33 nm, and the Mo-on-Y border is 0.7 nm). Use of the barrier deposition method allowed to reduce the length of the Mo-to-Y transition and to increase the reflection coefficient to 32% (for measurements at a wavelength of 9.34 nm). At the moment, the highest reflection value for Mo/Y mirrors optimized for a wavelength of 9.34 nm is 34% [9]. In [11] for Mo/Y, a



Fig. 1. Theoretical values of the peak reflection coefficient for ideal multilayer structures of Pd/Y [Data File 14], Ru/Y [Data File 11], Mo/Y [Data File 13], Ag/Y [Data File 15] and Ru/Sr [Data File 12] MMs for the spectral range of 8–12 nm. Here and below, the optical constants of the database https://henke.lbl.gov/optical_constants/ were used.

reflectance of 34.7% at a wavelength of 9.7 nm was obtained, and in [12] 38.4% at 9.48 nm. In the long-wavelength part of the range, 46% were obtained at 11.4 nm [10] and 47% at 11.8 nm [13].

The MLMs based on Pd/Y, Ag/Y and Ru/Y have the highest theoretically achievable reflection coefficients in the vicinity of a wavelength of 9.34 nm. However, values of the reflection coefficient close to the maximum have not been obtained experimentally. Thus, the best experimental result was obtained for the MLM based on Pd/Y with B4C barrier layers and reached 43% at the wavelength of 9.4 nm [14]. The degradation of the reflective characteristics of Pd/Y MLM was also noted in the paper. Periodic Pd/B4C/Y multilayers have been shown to have reasonably good temporal stability, with a drop in peak reflectance from 38% to 34% over a period of 16 months.

The Pd/Y structures synthesized in a mixture of argon and nitrogen were studied in [15]. It was shown that the reflection coefficient of the Pd(N)/Y(N) mirrors was 30% with a nitrogen fraction in the mixture of 6%. The article notes that although the use of nitrogen, on the one hand, reduces the mixing of the layers, but, at the same time, increases the absorption of the YN layers in comparison with the Y layers produced in pure argon. Similar experiments with a mixture containing nitrogen were performed in the article [16]. The results obtained in this work were similar to [15]. The Pd/B4C structures were synthesized with a reflection coefficient of 32% for a proportion of nitrogen in the mixture with argon of 15% [17]. Thus, we can conclude that the use of reactive sputtering did not allow to approach significantly the theoretical maximum of the reflection coefficient, and the best way to eliminate mixing of layers in such a structure is the use of B4C barrier interlayers.

We can also note the papers devoted to the study of Ag/Y MLM [18,19]. The sensitivity of the reflective characteristics of Ag/Y MLM to the material of the upper layer was established and the widths of the transition boundaries were determined in these works. The best reflectance at the wavelength of 9.34 nm was obtained for the Ag/Si/Y MLM (where Si is a thin barrier layer) and

amounted to 18%. Nevertheless, it can be seen that MLM based on Ag/Y is noticeably inferior to Mo/Y and Pd/Y ones in terms of reflection.

The Ru/Y mirrors were studied in [14,20,21]. However, the best experimentally obtained reflectance at a wavelength of 9.34 nm was only 34%. The paper [14] shows a positive effect from the use of B4C barrier layers (thickness 0.6 nm). The measurements were made in the range of 8.5-9 nm. It is shown that the roughness decreased from 0.835 nm to 0.75 nm. The measured reflection coefficient increased from 21.5% to 25%. There are no data in the literature on the reflective characteristics of Ru/Y MLMs in the vicinity of $\lambda = 11-12$ nm. There is an interest in the reflective characteristics of yttrium containing mirrors in this range due to the possibility of replacing beryllium-containing multilayer mirrors, because of the toxicity of beryllium. Theoretical calculations show that in this range Ru/Y MLMs are extremely promising with a reflection coefficient of more than 60%, which surpasses even Pd/Y mirrors.

The systematic study of the Ru/Y MLMs is presented in this paper. The reflective characteristics and the internal structure of Ru/Y MLSs, as well as the effect of B4C interlayers on them, were studied. The record values of the reflection coefficients of the Ru/Y structures compared with previously studied ones were obtained.

2. Experiment

During the experiments Ru/Y MLMs were synthesized by magnetron sputtering using a facility equipped with four planar magnetrons. This number of sources allows to work with both two-component structures and barrier layers deposited between the layers of base materials. The pressure of residual gases in the chamber before the start of the structures synthesis was at the level of $5 \cdot 10^{-5}$ Pa. MLMs were deposited on silicon substrates (with rms roughness 0.1–0.2 nm). The targets sputtering of was carried out at direct current in an environment of high-purity (99.998%) argon. The working gas pressure in the technological process is about 0.1 Pa. The characteristic values of the power supplied to the magnetrons were 108 W for Y and 78 W for Ru.

The main parameters of MLMs, such as the period, the thicknesses of the individual layers, and the length of the interlayer interfaces, were determined from the results of small-angle X-ray diffraction and reflectometry in the vicinity of wavelengths of 9.34 and 11.4 nm. A description of the studied samples is given in Table 1.

Measurements of X-ray radiation at a wavelength of $\lambda = 0.154$ nm were carried out using a high-resolution four-crystal diffractometer PANalitycal X'Pert Pro. Measurements in the vicinity of wavelengths 9.34 and 11.4 nm were carried out on a laboratory reflectometer with an RSM-500 spectrometer-monochromator. More details of the deposition technique and measuring equipment are given in [22]. In order to calibrate the laboratory reflectometer, sample #10 was examined using both a laboratory reflectometer and the reflectometer installed on the Optics Beamline at the BESSY-2 synchrotron facility (Berliner Elektronenspeicherring-Gesellschaft fur Synchrotronstrahlung, Berlin) [23,24].

In the Fig. 2 the solid lines show the results of measurements on the BESSY-II (presented in Data File 18), while the lines with symbols show the results from a laboratory reflectometer (presented in Data File 16). It can clearly be seen from the data that the results of measurements obtained with the different reflectometers are in good agreement. As it was expected the reflectance values obtained using the laboratory reflectometer are approximately 4% lower, and the angular half-widths are 9% wider compared to the curves measured at the synchrotron setup, due to the larger spectral width and angular divergence of the probe beam of the laboratory reflectometer.

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Mirror number	Composition (order of materials from substrate to surface)	d, nm	$\gamma = d_{Ru}/d$	Thickness B ₄ C, nm	λ, nm	$\Theta_{\rm inc}$, °	R, abs. un.
#1	Y/Ru	5.12	0.35	-	9.34	22	0.18
#2	Y/Ru	5.05	0.39	-	9.34	19.5	0.19
#3	Y/Ru	5.06	0.43	-	9.34	20	0.23
#4	Y/Ru	4.85	0.45	-	9.34	7	0.27
#5	Y/Ru	4,88	0.5	-	9.34	10	0.285
#6	Y/Ru	4.84	0.54	-	9.34	6	0.29
#7	Y/Ru	4.9	0.57	-	9.34	12.5	0.28
#8	Y/Ru	4.84	0.6	-	9.34	6	0.24
#9	Y/B ₄ C/Ru	4.86	0.54	0.1	9.34	8	0.345
#10	Y/B ₄ C/Ru	4.9	0.54	0.2	9.34	12.5	0.36
#11	Y/B ₄ C/Ru	4.84	0.54	0.3	9.34	6	0.375
#12	Y/B ₄ C/Ru	4.85	0.54	0.4	9.34	7	0.385
#13	Y/B ₄ C/Ru	4.84	0.54	0.5	9.34	6	0.34
#14	B ₄ C/Y/Ru	4.84	0.54	0.4	9.34	6	0.33
#15	B ₄ C/Y/B ₄ C/Ru	4.84	0.5	0.4 + 0.4	9.34	6	0.38
#16	Y/B ₄ C/Ru	5.95	0.54	0.4	11.4	6	0.54

Table	1.	Main experimental	parameters	and character	istics of the	studied samples
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Fig. 2. Comparison of measurements of the reflection coefficient angular dependencies for an MLM, performed using a synchrotron (solid line) [Data File 18] and a laboratory reflectometer (curve with symbols) [Data File 16].

3. Results and discussion

The key parameters of the synthesized structures were determined by solving the inverse problem of X-ray optics using open-source software called Multifitting [25], which was written in the C ++ programming language and developed at the IPM RAS. This code is based on the so-called extended model for the reconstruction of periodic multilayers from extreme ultraviolet and X-ray reflectivity data taking into account the contributions of various physicochemical processes occurring at the boundaries during the growth of the MLM [26]. The experimentally measured angular dependences of the reflection coefficient for each structure at hard X-ray ($\lambda = 0.154$ nm) and at the working wavelength ($\lambda = 9.34$ nm) were fitted with the calculated dependences at the corresponding wavelengths within the framework of this method. The influence of the interfaces was taken into account by dividing the transition region into thin homogeneous layers. The profile of the transition region was taken as the sum of the error function and a linear profile with weights, which made it possible to describe the entire reflection curve (formula (1) [26]):

$$f(z,\sigma) = \frac{\alpha_1 f_1(z,\sigma) + \alpha_2 f_2(z,\sigma)}{\alpha_1 + \alpha_2}, \quad \alpha_j \ge 0, \quad \sum_j \alpha_j > 0 \tag{1}$$

$$f_1(z,\sigma) = \frac{1}{2} \left(1 + erf(\frac{z}{\sqrt{2}\sigma}) \right)$$

$$f_2(z,\sigma) = \begin{cases} 0, \quad z \le -\sqrt{3} \ \sigma \\ \frac{1}{2} + \frac{z}{2\sqrt{3}\sigma}, \quad -\sqrt{3} \ \sigma < z < \sqrt{3} \ \sigma \\ 1, \qquad z \ge \sqrt{3} \ \sigma \end{cases}$$

, where α_1 and α_2 are weight coefficients, f1 is error function and f2 is linear function.

An example of a fitting for the Ru/Y structure #6 is shown in Fig. 3(a) and (b). The corresponding profile of the dielectric constant ε for 3 periods of the structure is shown in Fig. 3(c)). The profile sampling corresponds to that used in the simulation. The meander represents the case of an ideal structure. The data in Fig. 3 is presented in Data File 3, Data File 4, Data File 5 and Data File 6.

From the given profile it is possible to determine the values of the interfaces as $\sigma \approx 0.2$ nm for Y-on-Ru and $\sigma \approx 1.3$ nm for Ru-on-Y. Weight coefficients α_1 and α_2 are chosen equal to each other for the best fitting. The relatively high value of σ for Ru-on-Y explains the difference between the experimentally obtained reflection and the theoretical limit. The best fit between the total external reflection region and the first Bragg peak is achieved by assuming complete oxidation of the films of the first MLM period. From the presented profile, the fraction of ruthenium in the period (the parameter $\gamma = d_{Ru}/d$) as the full width at half maximum (FWHM) of the profile ϵ (meander width) was determined.

In the first set of experiments, the dependence of the peak reflection coefficient R on γ for the Ru/Y MLM series (samples #1–8) was studied. The total number of periods in the studied structures was 90. The measurements were conducted using a laboratory reflectometer. The results are shown in Fig. 4 (the data presented in Data File 20).

As can be seen from Fig. 4, the reflection coefficient of such structures increases with growth of the parameter γ and reaches a maximum at $\gamma = 0.54$, after which a decrease is observed. The maximum reflection coefficient of Ru/Y MLM was 29%. Further studies of the effect of B₄C interlayers on the ε profile were conducted for samples with a value of the parameter γ close to optimal.

The B_4C barrier layer technique was applied in order to reduce the width of the interfaces. Boron carbide was introduced into the structure by a corresponding reduction in the yttrium



Fig. 3. Experimental data for the reflection coefficient and fitted reflection curves for sample #6 [Data File 3, Data File 4, Data File 5 and Data File 6]: a) angular dependence at $\lambda = 0.154$ nm; b) angular dependence at $\lambda = 9.34$ nm; c) reconstructed profile of the dielectric constant ε at $\lambda = 0.154$ nm.



Fig. 4. The dependence of the reflection coefficient on the thickness of the ruthenium layer in the period. The total number of periods is 90. Measurements were taken using a laboratory reflectometer at the wavelength of 9.34 nm [Data File 20].

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layer. First, the barrier was applied to the most problematic boundary, that is, boron carbide was deposited on the yttrium surface. A series of experiments in which the thickness of the boron carbide layer varied from 0.1 to 0.5 nm were conducted in order to determine the optimal thickness of the B₄C layers (samples #9–13). The ruthenium thickness d_{Ru} and the total period of the structure d remained almost unchanged, thus, the parameter γ remained fixed. Boron carbide was introduced into the structure by a corresponding reduction in the yttrium layer. The resulting dependence is shown in Fig. 5 (the data presented in Data File 17). The measurements were carried out on a laboratory reflectometer.



Fig. 5. Dependence of the reflection coefficient for a $Y/B_4C/Ru$ MLM on the thickness of the boron carbide layer. The measurements were performed on a laboratory reflectometer at a wavelength of 9.34 nm [Data File 17].

The presented dependence demonstrates that the reflection coefficient of the structure reaches a maximum value of 38.5% at a boron carbide layer thickness of 0.4 nm. A further increase in the thickness of boron carbide leads to a decrease in the reflection coefficient. It should be noted that the obtained reflection coefficient of 38.5% significantly exceeds the previously published corresponding value for the Mo/Y and the Ru/Y mirrors and approaches the value for Pd/Y with B₄C barrier layers (43%).

Figure 6 shows the fitting results and the reconstructed profile of the dielectric permittivity of the Y/B4C/Ru sample (sample #10) (the data on Fig. 6 is presented in Data File 7, Data File 8, Data File 9 and Data File 10), which are similar to those shown in Fig. 3.

As can be seen from the figure, the introduction of the B₄C barrier layer led to a noticeable reduction in the transition region from Y to Ru, which had a positive effect on the measured reflection coefficient. The extent of the Ru-on-Y interface decreased to 0.6 nm with 0.2 nm of the B4C barrier layer. In this case, the weight coefficient α_2 is an order of magnitude less than the coefficient α_1 . For definiteness, 0.1 and 1, respectively.

The Fig. 7 demonstrates the comparison of the spectral dependencies of reflection coefficient of the samples #6 (Ru/Y) (the data presented in Data File 2) and #10 (Ru/Y/B4C) (the data presented in Data File 1), measured at the incidence angle 2° at the synchrotron BESSY II. The



Fig. 6. Experimental data on reflection and fitted reflection curves for sample #10 [Data File 7, Data File 8, Data File 9 and Data File 10]: a) angular dependence at $\lambda = 0.154$ nm; b) angular dependence at $\lambda = 9.34$ nm; c) reconstructed profile of the dielectric constant ε at $\lambda = 0.154$ nm.



Fig. 7. Spectral dependencies of the reflectance coefficients for the samples #6 (Ru/Y) and #10 (Ru/Y/B4C), measured at the incidence angle 2° at the synchrotron BESSY II [Data File 1, Data File 2].



Fig. 8. Angular dependence of the reflection coefficient for sample #16 at a wavelength of $\lambda = 11.4$ nm (measurements taken on a laboratory reflectometer) [Data File 19].

maximum reflection coefficient value is 32% for the sample #6 and 39% for the sample #10. The measured value of FWHM is 0.16 nm.

The deposition of B_4C on the surface of Ru layers only slightly affects the reflection coefficient (sample #14). An MLM composed of $B_4C/Y/Ru$ (representing the order of the materials from substrate to surface) has a reflectivity of 33% at a wavelength of 9.34 nm.

The MLS (sample #16) with the previously obtained optimal values of the B₄C thickness and γ for $\lambda = 11.4$ nm was synthesized and its reflection coefficients were studied. The investigated MLS with a period of d = 5.95 nm (d_{Ru} = 3.2 nm, d_{B4C} = 0.4 nm, d_Y = 2.35 nm) demonstrated the reflection coefficient R = 54% at a wavelength of 11.4 nm (the position of the Bragg peak 6 ° from the normal), as shown in Fig. 8 (the data presented in Data File 19).

4. Conclusion

In the framework of the presented paper the multilayer Ru/Y and Ru/Y mirrors with B4C barrier layers optimized for working wavelengths of 9.4 nm and 11.4 nm were studied. The interfaces widths were defined. It was shown that at the Y-on-Ru boundary, $\sigma \approx 0.2$ nm, while at the Ru-on-Y boundary, $\sigma \approx 1.3$ nm. It was found that the use of boron carbide barrier layers and the selection of their optimal thickness in the structure makes it possible to reduce the length of the interlayer regions to the 0.6 nm and to achieve a reflection coefficient of 38.5% at a wavelength of 9.34 nm. The spectral half-width of the reflection peak measured at the synchrotron was $\Delta\lambda = 0.16$ nm. There were also investigated the structures optimized for the working wavelength of 11.2 nm, for which the measured reflectance was 54%.

The practical significance of the obtained results lies in the fact that the investigated yttriumcontaining multilayer mirrors can be successfully used as the basis of optical schemes for operation in the range 9–12 nm. Despite the fact that the reflection coefficient for Ru/Y MLMs in the vicinity of 9.34 nm was slightly lower than for Pd/Y mirrors (\approx 3%), Ru/Y MLMs have better spectral selectivity. The FWHM measured in this work for Y/B₄C/Ru was 0.16 nm, while for Pd/B₄C/Y, the FWHM was 0.2 nm [14], which may be important for spectroscopic research.

Although there are no available experimental data for Pd/Y MLMs in the vicinity of 11 nm, it could be argued that Ru/Y MLMs are preferable for this working wavelength because the experimentally obtained data presented here reached the theoretical limit for Pd/Y.

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Data availability. Data underlying the results presented in this paper are available in Data File 1, Data File 2, Data File 3, Data File 4, Data File 5, Data File 6, Data File 7, Data File 8, Data File 9, Data File 10, Data File 11, Data File 12, Data File 13, Data File 14, Data File 15, Data File 16, Data File 17, Data File 18, Data File 19, and Data File 20.

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