09

# Study of structural and reflective characteristics of multilayer X-ray mirrors based on a pair of Ru/B materials

© R.A. Shaposhnikov, V.N. Polkovnikov, N.I. Chkhalo, S.A. Garakhin

Institute of Physics of Microstructures, Russian Academy of Sciences, Nizhny Novgorod, Russia
E-mail: shaposhnikov-roma@mail.ru

Received July 30, 2024
Revised August 28, 2024
Accepted September 4, 2024

The paper presents the results of a study of the reflective characteristics at wavelengths of 0.154, 0.989 and 1.76 nm and the structural characteristics reconstructed from these data of multilayer Ru/B X-ray mirrors without interlayers and with carbon interlayers. The deposition of C interlayers on the B-on-Ru boundary resulted in a decrease of the transition region length from 0.69 to 0.37 nm. The calculated reflectivity of the Ru/C/B structure at a wavelength of 6.65 nm, performed using the obtained structural parameters of the mirrors, amounted to a record 68.9 %.

**Keywords:** Multilayer x-ray mirrors, synchrotron applications, X-ray monochromators, X-ray lithography.

DOI: 10.61011/TPL.2025.01.60138.20075

The 6.65−9 nm spectral range is one of the wavelength intervals of the greatest current interest. This interest stems from the fact that the indicated range is relevant to a number of important applications, such as next-generation projection lithography [1], X-ray fluorescence analysis [2], design of monochromators for synchrotron radiation stations [3], and X-ray spectroscopy [4]. The general rule for selection of materials for a multilayer X-ray mirror (MXM) is to choose a material with the lowest absorption in the operating wavelength range first. This material is then coupled with the one that provides the strongest contrast in permittivity, while also having as low absorption as possible. Boron or boron-containing materials (boron absorption $K$-edge is $\lambda_K = 6.63$ nm) are the best spacers within the $\lambda > 6.65$ nm wavelength range. Lanthanum and ruthenium appear to be the most efficient absorbers. Figure 1 presents the results of theoretical calculation of peak values of reflectance for La/B, $La/B_4C$, Ru/B, and $Ru/B_4C$ structures within the 6−9 nm spectral range under normal incidence. The case of ideal structures with zero roughness, tabular material densities, and zero period drift or fluctuations is considered here. It follows from these dependences that boron used as a spacer provides higher reflectance values than boron carbide. It should also be noted that the best reflectance values near $\lambda_K$ are obtained in La/B structures, while Ru/B structures provide the best results in the long-wavelength region of the examined spectral range. La-based structures were examined in [5–8]. Owing to mixing of materials at the interfaces between layers, the reflectance values obtained experimentally in these studies differed significantly from the maximum theoretically possible ones. One efficient method for enhancing the interface between materials is structure synthesis in a mixture of argon and nitrogen gases. In this case, nitrogen is introduced into the working volume only during the synthesis of lanthanum layers, which leads to the formation of a LaN/B structure. Reflectance $R = 64.1$ % at an incidence angle of 1.5° from the normal and a wavelength of 6.65 nm was obtained in [8] for this structure. This is the current reflectance record at the specified wavelength. The optical properties of $Ru/B_4C$ MXMs in the hard X-ray region were characterized in [9–12]. Ru/B MXMs have not been studied earlier. These are the MXMs examined in the present study.

The setup discussed in [13] was used for synthesis of multilayer structures. Ruthenium was deposited by magnetron sputtering (discharge parameters: $I = 300$ mA and $U = 290$ V), while ion-beam deposition was used for boron (beam current $I = 60$ mA and beam energy

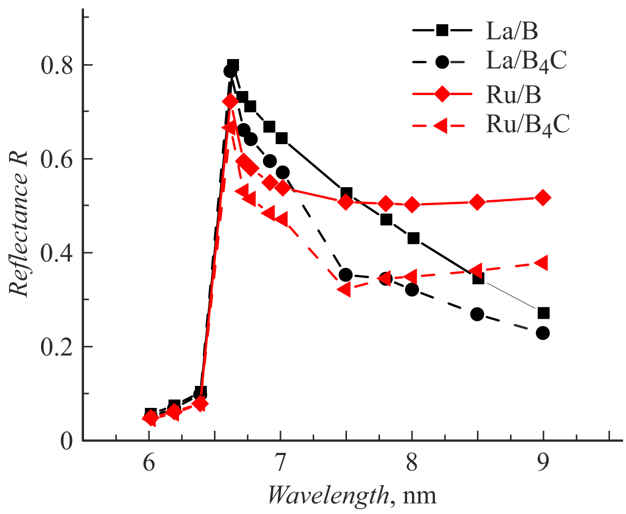


**Figure 1.** Reflectance dependences for ideal La/B, $La/B_4C$, Ru/B, and $Ru/B_4C$ structures within the range of 6−9 nm at a grazing angle of incidence of 90°. Optical constants from the CXRO database (https://henke.lbl.gov/optical_constants/) were used in calculations.

Structural parameters of the studied samples (number of periods for all structures $N = 50$)

| Compound of the structure | Period, nm | $\beta^*$ | Transition layer width, nm | | $R^{**}$, % | | $R^{***}$, % |
|---|---|---|---|---|---|---|---|
| | | | at the B-on-Ru boundary | at the Ru-on-B boundary | $\lambda = 0.989$ nm | $\lambda = 1.759$ nm | $\lambda = 6.65$ nm |
| Ru/B | 3.435 | 0.48 | 0.57 | 0.22 | 20 | 7.3 | 25.7 |
| Ru/B | 3.425 | 0.39 | 0.69 | 0.21 | 19.4 | 7.2 | 21.1 |
| Ru/B | 3.530 | 0.34 | 0.62 | 0.18 | 25.3 | 10.1 | 22.3 |
| Ru/B | 3.635 | 0.3 | 0.69 | 0.24 | 22.9 | 10 | 19.2 |
| Ru/B/C | 3.425 | 0.43 | 0.7 | 0.21 | 21.8 | – | 27.6 |
| Ru/C/B | 3.509 | 0.38 | 0.37 | 0.21 | 28.7 | 12.3 | 36.45 |

* Ruthenium fraction in a period.
** Measured reflectance values at wavelengths of 0.989 and 1.759 nm.
*** Theoretical value of the MXM reflectance at a wavelength of 6.65 nm calculated with account for the reconstructed structural parameters.

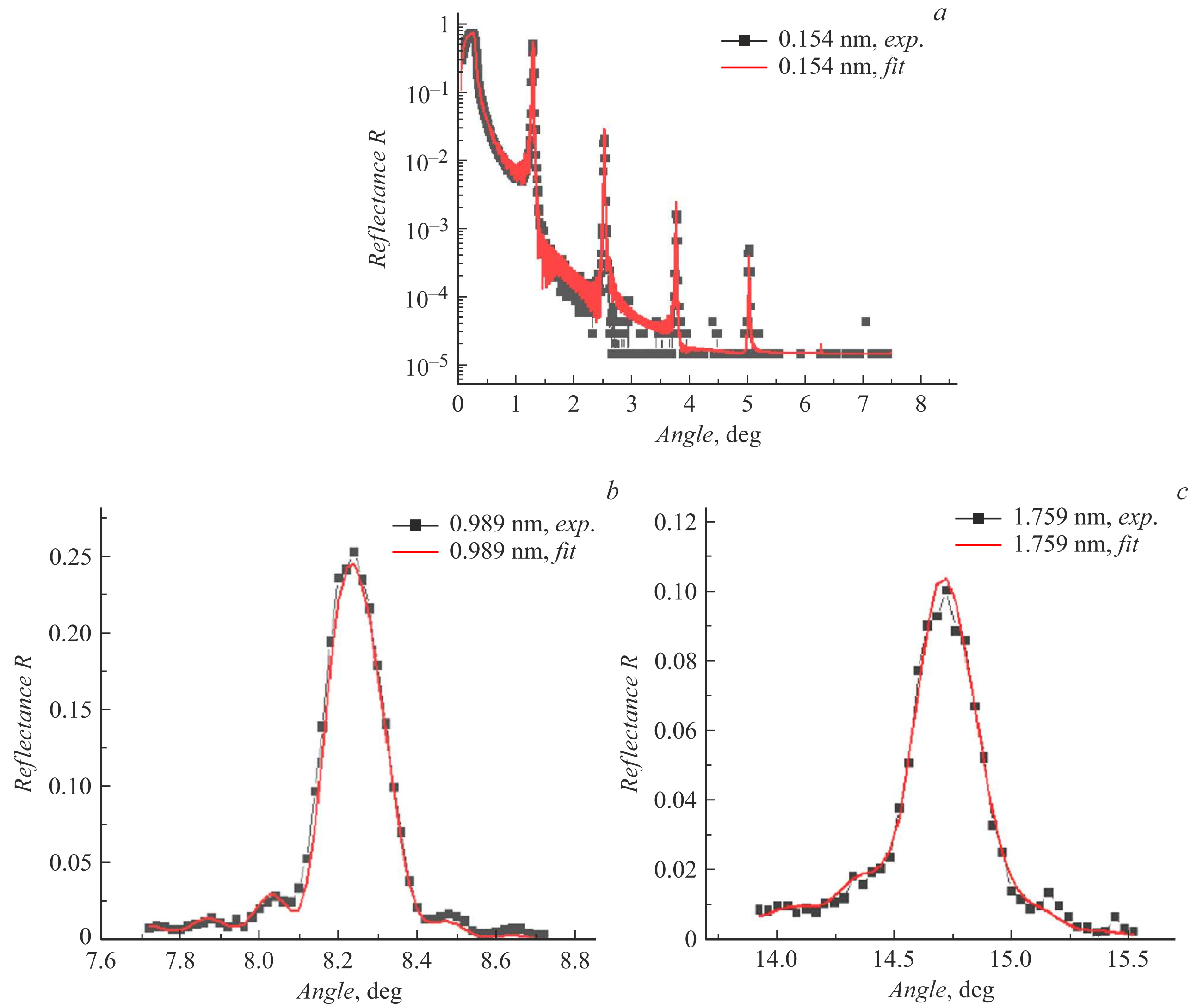


**Figure 2.** Dependences of the experimentally measured reflectance on the grazing angle of incidence and the results of fitting for wavelengths $\lambda = 0.154$ (*a*), 0.989 (*b*), and 1.759 nm (*c*).

$E = 800$ eV). Argon was the working gas; working pressure $p = 1.3 \cdot 10^{-3}$ Torr. Silicon wafers with roughness at the level of 0.2 nm served as substrates. The synthesized structures were examined both in the hard X-ray region at wavelength $\lambda = 0.154$ nm with a four-crystal PANalytical X'Pert PRO diffractometer and in the soft X-ray region at wavelengths $\lambda = 0.989$ and 1.759 nm with a laboratory reflectometer fitted with an RSM-500 [14] grating spectrometer/monochromator. The structural parameters of samples (structure period, layer thickness, material density, width of transition regions) were determined using the Multifitting software developed at the Institute of Physics of Microstructures of the Russian Academy of Sciences [15]. A series of samples with different values of parameter $\beta = d_{\mathrm{Ru}}/d$, where $d_{\mathrm{Ru}}$ is the thickness of ruthenium in a single structure period and $d$ is the multilayer mirror period, were synthesized for the study of structural parameters of multilayer Ru/B mirrors. The number of periods in all structures was $N = 50$. Figure 2 shows an example fit of experimental curves in Multifitting, which served as the basis for determination of the structural parameters. The composition of MXM samples and their main structural parameters are presented in the table. These data reveal an asymmetry in transition regions at different boundaries: the transition region at the B-on-Ru boundary is more extensive than at the Ru-on-B boundary. To reduce the extent of transition regions at the boundary, carbon barrier layers were added to the structure. The addition of carbon onto boron layers (Ru/B/C structure) did not produce a positive effect, while the deposition of carbon layers onto ruthenium (Ru/C/B structure) allowed us to reduce significantly the width of the transition region from 0.69 nm to 0.37 nm. The thickness of carbon layers was 0.33 nm. Figure 3 shows the dependence of reflectance on the grazing angle of incidence at wavelength $\lambda = 6.65$ nm for a multilayer Ru/B mirror with carbon barrier layers calculated numerically with account for the obtained values of the transition region width. The number of periods in this calculation was $N = 250$. It is evident that ruthenium/boron structures with barrier layers provide reflectance $R = 68.9\,\%$ at wavelength $\lambda = 6.65$ nm, which exceeds reflectance $R = 64.1\,\%$ obtained earlier in [8] for LaN/B structures. Thus, although the optical characteristics of ideal Ru/B structures are such that their theoretical reflectance in the vicinity of 6.7 nm is lower than the one of La/B structures, the experimentally attainable reflectance values for ruthenium-based structures are higher (owing to the small width of transition regions). However, despite the validity of the calculation model, this claim of multilayer Ru/B mirrors being advantageous over La/B ones requires experimental verification. Therefore, Ru/C/B MXMs potentially hold promise for use as reflective elements in optical circuits within the 6.65–9 nm spectral range.

The following important results were obtained. It was demonstrated that Ru/B structures have asymmetric thicknesses of transition layers: the B-on-Ru boundary is more extensive with a width of 0.57–0.70 nm, which makes it significantly more difficult to achieve high reflectance values. The use of barrier layers (carbon interlayers with a thickness of 0.3–0.4 nm applied to the most extensive boundary) allows one to reduce the width of the transition region by a factor of almost 2 (to 0.3–0.4 nm), which, in turn, leads to an increase in reflectance. Theoretical calculations revealed that multilayer Ru/C/B mirrors with the obtained structural parameters may provide a reflectance up to 68.9 % at a wavelength of 6.65 nm (a record-high value, if confirmed by measurements). Thus, the proposed structures have wide opportunities for application as reflective elements within the 6.65–9 nm wavelength range.

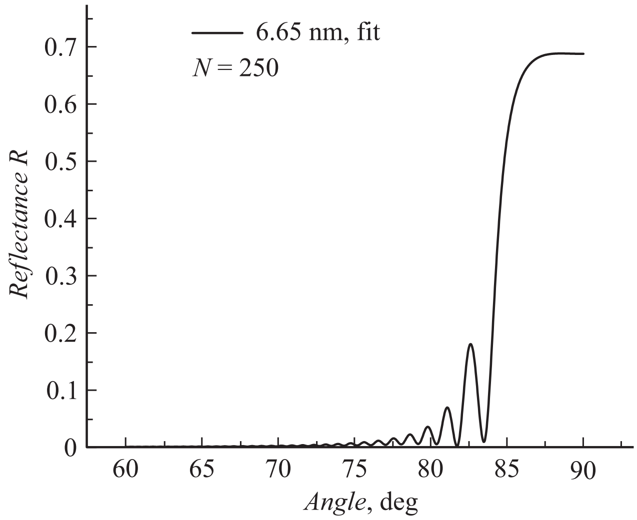


**Figure 3.** Theoretical dependence of reflectance on the grazing angle of incidence at wavelength $\lambda = 6.65$ nm for a multilayer Ru/C/B mirror calculated with account for the obtained structural parameters.

### Funding

The synthesis of samples and the measurement of angular dependences of reflectance in the X-ray range were supported by grant No. 21-72-30029 from the Russian Science Foundation. The work on reconstruction of structural MXM parameters based on X-ray reflection data was supported by grant No. 21-72-20108 from the Russian Science Foundation.

### Conflict of interest

The authors declare that they have no conflict of interest.

## References

[1] N.I. Chkhalo, N.N. Salashchenko, AIP Adv., **3** (8), 082130 (2013). DOI: 10.1063/1.4820354

[2] S.A. Garakhin, V.N. Polkovnikov, N.I. Chkhalo, J. Surf. Investig., **13** (2), 173 (2019). DOI: 10.1134/S1027451019020071.

[3] A.A. Akhsakhalyan, Yu.A. Vainer, S.A. Garakhin, K.A. Elina, P.S. Zavertkin, S.Yu. Zuev, D.V. Ivlyushkin, A.N. Nechay, A.D. Nikolenko, D.E. Pariev, R.S. Pleshkov, V.N. Polkovnikov, N.N. Salashchenko, M.V. Svechnikov, N.I. Chkhalo, J. Surf. Investig., **13** (1), 1 (2019). DOI: 10.1134/S1027451019010026.
[4] J.K. Lepson, P. Beiersdorfer, J. Clementson, M.F. Gu, M. Bitter, L. Roquemore, R. Kaita, P.G. Cox, A.S. Safronova, J. Phys. B, **43** (14), 144018 (2010). DOI: 10.1088/0953-4075/43/14/144018
[5] S.S. Andreev, M.M. Barysheva, N.I. Chkhalo, S.A. Gusev, A.E. Pestov, V.N. Polkovnikov, D.N. Rogachev, N.N. Salashchenko, Yu.A. Vainer, S.Yu. Zuev, Tech. Phys., **55** (8), 168 (2010). DOI: 10.1134/S1063784210080153.
[6] N.I. Chkhalo, S. Künstner, V.N. Polkovnikov, N.N. Salashchenko, F. Schäfers, S.D. Starikov, Appl. Phys. Lett., **102** (1), 011602 (2013). DOI: 10.1063/1.4774298
[7] P. Naujok, S. Yulin, N. Kaiser, A. Tünnermann, Proc. SPIE, **9422**, 94221K (2015). DOI: 10.1117/12.2085764
[8] D.S. Kuznetsov, A.E. Yakshin, J.M. Sturm, R.W.E. van de Kruijs, E. Louis, F. Bijkerk, Opt. Lett., **40** (16), 3778 (2015). DOI: 10.1364/OL.40.003778
[9] T.D. Nguyen, R. Gronsky, J.B. Kortricht, Mater. Res. Soc. Symp. Proc., **280**, 161 (1993). DOI: 10.1557/PROC-280-161
[10] C. Borel, C. Morawe, A. Rommeveaux, C. Huguenot, J.-C. Peffen, Proc. SPIE, **6317**, 63170I (2006). DOI: 10.1117/12.678472
[11] C. Borel, C. Morawe, E. Ziegler, T. Bigault, J.-Y. Massonnat, J.-C. Peffen, E. Debourg, Proc. SPIE, **5918**, 591801 (2005). DOI: 10.1117/12.613873
[12] Q. Huang, Y. Liu, Y. Yang, R. Qi, Y. Feng, I.V. Kozhevnikov, W. Li, Z. Zhang, H. Jiang, L. Zhang, A. Li, J. Wang, Z. Wang, Opt. Express, **26** (17), 21803 (2018). DOI: 10.1364/OE.26.021803
[13] I.G. Zabrodin, B.A. Zakalov, I.A. Kas'kov, E.B. Klyuenkov, V.N. Polkovnikov, N.N. Salashchenko, S.D. Starikov, L.A. Suslov, J. Surf. Investig., **7** (4), 637 (2013). DOI: 10.1134/S1027451013040204.
[14] M.S. Bibishkin, N.I. Chkhalo, A.A. Fraerman, A.E. Pestov, K.A. Prokhorov, N.N. Salashchenko, Yu.A. Vainer, Nucl. Instrum. Meth. Phys. Res. A, **543** (1), 333 (2005). DOI: 10.1016/j.nima.2005.01.251
[15] M.J. Svechnikov, Appl. Cryst., **53** (1), 244 (2020). DOI: 10.1107/S160057671901584X

*Translated by D.Safin*