Short-period multilayer X-ray mirrors†

S. S. Andreev,[a] M. S. Bibishkin,[a] N. I. Chkhalo,[a] E. B. Kluenkov,[a] K. A. Prokhorov,[a]* N. N. Salashchenko,[a] M. V. Zorina,[a] F. Schafers[b] and L. A. Shmaenok[c]

[a]Institute for Physics of Microstructures RAS, 603600, GSP 105, Nizhny Novgorod, Russia, [b]BESSY II, Albert Einstein Strasse 15, D-12489 Berlin, Germany, and [c]A. F. Ioffe Physico-Technical Institute RAS, Politechnicheskaya 26, 194021, St Petersburg, Russia. E-mail: kp@ipm.sci-nnov.ru

Multilayer structures with short periods have been systematically investigated using a tunable soft X-ray synchrotron, BESSY II, and X-ray tube radiation. Multilayer X-ray mirrors of W/B_4C, W/Sc, Mo/B_4C, Mo/C, La/B_4C, Cr/C and Cr/Sc, with periods from 0.8 nm to 3.5 nm and number of periods up to 300–400, were constructed and investigated. The high reflectivity and spectral resolution of the mirrors allow them to be used to create multimirror systems for X-ray diagnostics of high-temperature plasma, for X-ray astronomy and microscopy.

Keywords: multilayer mirrors; reflections; spectral resolutions; soft X-ray radiation; water windows; reflectometers.

1. Introduction

Multilayer X-ray mirrors with small (1–3 nm) periods are currently attracting considerable attention owing to the fact that they can be used for the development of optics in many important applications. These mirrors are used as radiation-stable dispersion elements and polarizers for synchrotron radiation, as dispersion elements for high-temperature plasma diagnostics, for the creation of normal-incidence optical systems for projection microscopy in the 'water window' spectral range and X-ray astronomy, and for the formation of X-ray radiation beams. As a rule, from 200 to 400 periods participate in effective reflection in such mirrors; therefore the fabrication process of mirrors should provide stability of the thickness of periods on the depth of a structure, $\delta d \simeq d/N$ (where d is the value of the period and N is the number of periods); usually $\delta d \simeq 5 \times 10^{-3}$ nm. Details of the fabrication process of such mirrors, and results of research on their properties are considered here.

In the given work, short-period multilayer X-ray mirrors were systematically investigated using a broad class of materials and with the help of tunable synchrotron radiation from BESSY II and radiation from a special dismountable X-ray tube. Multilayer X-ray mirrors, of W/B_4C, Mo/B_4C, Mo/C, La/B_4C, Cr/C and Cr/Sc with periods from 0.8 nm to 3.5 nm and number of periods up to 300–400, were constructed and investigated.

The multilayer mirrors were fabricated using a standard magnetron sputtering set-up comprising two 150 mm-diameter magnetrons and power and controlling supply. The targets were sputtered in Ar at a pressure of 8×10^{-2} to 9×10^{-2} Pa. The initial pressure of the residual gas at pumping was $\sim 10^{-4}$ Pa. Targets were sputtered by DC and RF current sources. In the sputter process the stability of the parameters of the magnetron discharge is the determining factor. A stability of better than 1% is achieved by deep modernization of the magnetron sputter sources and power control. The controlling system automatically provides a change in the gas pressure if some instability in the discharge occurs. Small-angle X-ray diffraction measurements were taken (in order to determine the parameters of the period of the mirrors) and the relationships between the layer thicknesses of different materials in a period and evaluations of the height of the interplanar interface roughness were investigated using a standard X-ray diffractometer.

2. Experimental results

The most detailed research was carried out using mirrors based on Cr/Sc and was first shown by Schafers *et al.* (1998, 2001). The Cr/Sc multilayer samples with periods in the 1.5–3 nm range were made by magnetron sputtering technology and were investigated by using s-polarized synchrotron radiation in the 3.1–6.3 nm spectral range. In the entire spectral range, normal-incidence mirrors have a reflectivity of 11–8% (Fig. 1). The multilayer mirrors were deposited on silicon substrates and on Si_3N_4 membranes with a thickness of 100 nm at the same time. The Cr/Sc mirrors are of special interest for applications in the area of the L absorption edges of Cr ($\lambda_L \simeq 2.16$ nm) and Sc ($\lambda_L \simeq 3.09$ nm). The Cr/Sc mirrors operating in reflection and in transmission were used as a basis for developing soft X-ray polarimeters at BESSY II (Schafers *et al.*, 1999).

A detailed research of the reflectivity of the mirrors in the 3.1–3.4 nm spectral region for angle and spectral dependences was carried out (Fig. 2). As was expected, outside the area of Sc anomalous dispersion the reflection of the Cr/Sc multilayer mirror is 1.5 times lower at a practically constant spectral resolution (about 250). However, at the same time, Cr/Sc is the optimal pair of materials for fabrication of dispersing and focusing elements for the practically interesting line of a hydrogen-like ion of carbon, $\lambda_{C\,VI}$ = 3.373 nm (Birch *et al.*, 2003; Eriksson *et al.*, 2002).

The mirrors were investigated in the 3–3.5 keV spectral range for application of Cr/Sc multilayer structures for spectrally selective soft X-ray tomography of argon ions in tokamak plasma (Shmaenok *et al.*, 2001). A multilayer mirror with period 1.57 nm and number of periods N = 250 also has a selectivity of about 250 at a reflectivity of about 6–8% (Fig. 3).

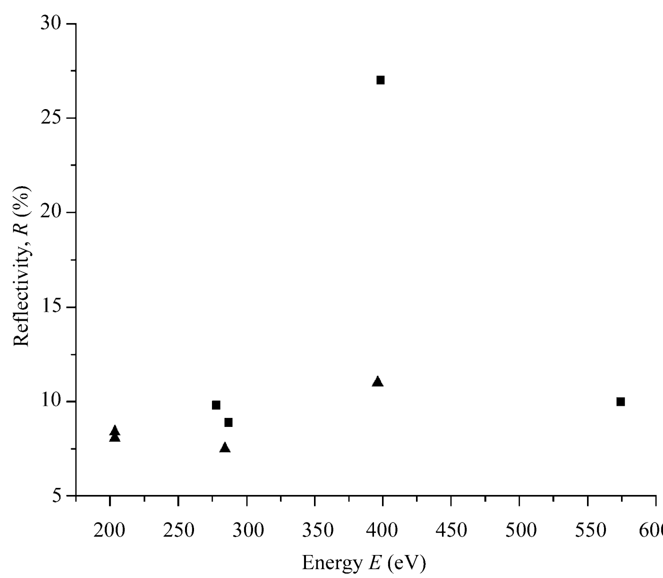

Figure 1
Short-period multilayer X-ray mirrors based on Cr/Sc. Triangles: measurements for $\theta = 85°$. Squares: measurements for $\theta = 45°$, where θ is the angle from the surface of the substrate and R is the reflectivity for s-polarization.

† Presented at the 'XIV Russian Synchrotron Radiation Conference SR2002', held at Novosibirsk, Russia, on 15–19 July 2002.

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Table 1
Parameters of multilayer mirrors optimized for specific impurity lines of X-ray radiation.

MLS is the multilayer structure, θ is the angle from the surface of the substrate, R is the reflectivity, $S = E/\Delta E$ is the selectivity of the MLS, and d is the period.

Ion	λ (nm)	MLS	d (nm)	θ (°)	R (%)	S
Ar XVIII	0.373	Cr/Sc	2.0	5.4	30	150
Ar XVII	0.395	Cr/Sc	2.0	5.7	30	150
Ne X	1.213	W/Si	2.1	17	16	96
Ne IX	1.345	W/Si	2.1	19	13	90
O VIII	1.9	W/Si	2.1	27	6	80
O VII	2.16	Cr/Sc	2.1	31	7	150
Ar XIV	2.74	Fe/Ti	2.5	33.6	13	140
C VI	3.37	Cr/Sc	2.5	41	7	140
C V	4.03	Cr/Sc	3.1	40	9	125
B V	4.86	Fe/C	3.8	40	12	80
B IV	6.03	Cr/C	4.7	40	10	75

The multilayer mirrors were fabricated and investigated (Table 1) for experiments in X-ray tomography of selected spectral lines of ions impurities in tokamak TEXTOR (Shmaenok *et al.*, 2001). The main demand for multilayer X-ray instrumentation for wavelength- and time-resolved plasma emission tomography is for obtaining good reflection at high spectral resolution, making it possible to select a useful investigated impurity signal from the background wide-band radiation of high-temperature plasma.

In solving some practical and fundamental problems, such as estimating the content of boron in various materials by the method of X-ray fluorescence element analysis ($\lambda_{B\,K\alpha} = 6.76$ nm) (Michaelsen *et al.*, 2001) and in the development of equipment for research into the element/charge states and spatial distribution of impurity in high-temperature tokamak plasma in the wavelength range $\lambda \simeq 7$–8 nm (for example, $\lambda_{\rm Be\,IV} = 7.59$ nm), one has to make use of X-ray dispersion elements optimized for this spectral region and the problems in question. Research into a La/B_4C mirror with period 3.5 nm was carried out on the BESSY II synchrotron. A reflectivity of 36.5% with selectivity $S = 140$ for s-polarized X-rays was obtained at a wavelength of 6.73 nm. On decreasing the wavelength in the area of the B anomalous dispersion ($\lambda = 6.66$ nm), the reflectivity increased by up to 42%, which once again confirms the practice of material choice for work behind the absorption edge of the light material included in the structure.

With the purpose of creating multilayer optics for collimation and focusing of hard X-ray radiation ($\lambda \simeq 0.02$–0.2 nm), multilayer mirrors W/B_4C and W/Sc with extremely short periods of 0.8–1.5 nm and numbers of period up to 400 were fabricated. Multilayer mirrors based on such pairs with short periods are of a special interest, because there is no increase of roughness when the period is reduced, as is shown experimentally (Andreev *et al.*, 1991; Christensen *et al.*, 1991). The cross sections of multilayer samples were investigated by electron microscopy. The characteristics of the mirrors were measured on the K-line radiation of molybdenum, copper and magnesium for non-polarized radiation (Fig. 4).

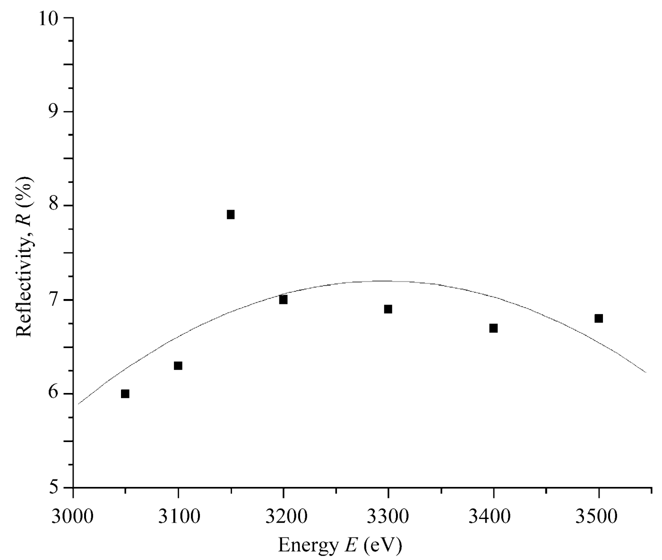

Figure 3
Reflectivity of Cr/Sc multilayer at $d = 1.57$ nm and $N = 250$ in the spectral area 3–3.5 keV (s-polarization).

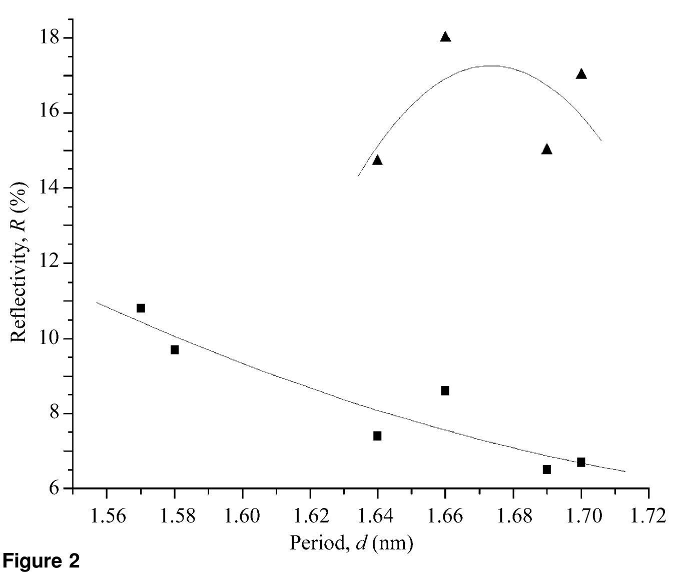

Figure 2
Reflectivity of Cr/Sc mirrors with periods of 1.57–1.70 nm in the spectral region 3.1–3.4 nm for spectral and angle measurements (s-polarization). Triangles: angle measurements at $\lambda = 3.14$ nm. Squares: spectral characteristics for $\theta = 85.6°$.

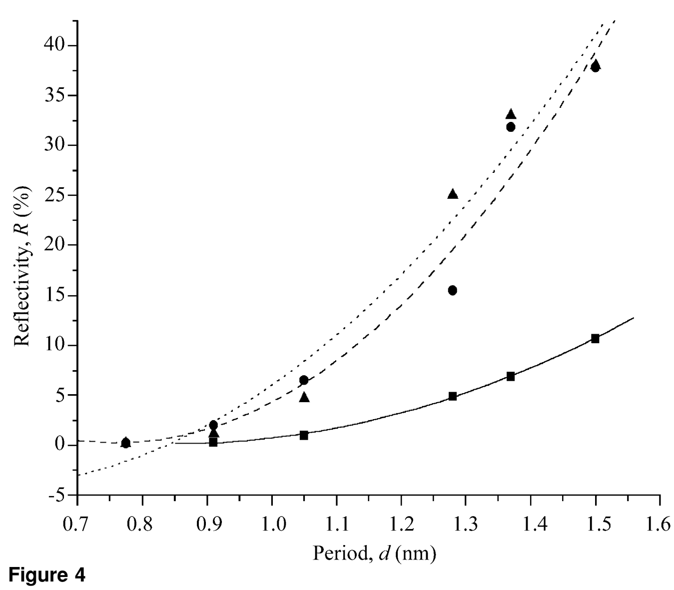

Figure 4
Reflectivity of short-period multilayer mirrors with periods of 0.775–1.5 nm based on W/B_4C (X-ray tube). Triangles: measurements at Mo $K\alpha$ (dotted line). Squares: measurements at Mg $K\alpha$ (solid line). Circles: measurements at Cu $K\alpha$ (dashed line).

Table 2
Multilayer mirrors based on pairs W/B_4C, Mo/C, Mo/B_4C and Cr/C.

MLS d (nm); N	λ (nm)	θ (°)	$\Delta\theta$	$S = \lambda/\Delta\lambda$	R (%)
W/B_4C 1.28; 280	0.989	23	0.11	220	4.9
W/B_4C 1.35; 200	0.989	21.4	0.1	225	6.9
W/B_4C 1.55; 250	0.989	18.94	0.11	180	9.1
Mo/C 1.45; 270	0.989	20.18	0.08	263	4.35
Mo/C 1.75; 200	0.989	16.66	0.09	191	12
Mo/C 2.09; 170	0.989	13.86	0.11	128	19.8
Mo/B_4C 1.32; 250	0.989	22.22	0.1	234	3.45
Mo/B_4C 1.59; 200	0.989	18.29	0.1	189	8.4
Mo/B_4C	0.989	15.68	0.12	133	12.5
1.86; 160	0.834	13.04	0.11	120	11.7
	0.712	11.04	0.1	111	9.9
Mo/B_4C	0.989	16.86	0.12	144	9.3
1.72; 170	0.834	14.01	0.12	119	7.7
	0.712	12.17	0.13	95	7.4
Mo/B_4C	0.989	24.24	0.1	257	2.42
1.22; 300	0.834	19.76	0.09	228	1.95
	0.712	16.96	0.08	218	1.2
Cr/C 2.19; 200	0.989	13.29	0.1	135	13.2

Multilayer mirrors of W/B_4C, Mo/C, Mo/B_4C and Cr/C with periods from 1.2 nm to 2.2 nm were constructed and investigated for the spectral regions of the ‘water window’ and hard X-ray radiation. The comparative characteristics of such structures measured in the wavelength range 0.7–1 nm using an X-ray spectrometer–monochromator are given in Table 2. The high selectivity of fabricated multilayer structures with good reflectivity show the opportunity of application of these multilayer mirrors as dispersing elements for X-ray spectroscopy.

3. Summary

We have presented a systematic investigation of magnetron-deposited short-period multilayer structures of different pairs of materials carried out on the synchrotron BESSY II and on a dismountable X-ray tube in the soft X-ray range. The multilayer X-ray mirrors based on W/B_4C, W/Sc, Mo/B_4C, Mo/C, La/B_4C, Cr/C and Cr/Sc with periods from 0.8 nm to 3.5 nm showed a high reflectivity and spectral resolution, which allows them to be used for various X-ray applications. Normal-incidence Cr/Sc mirrors have a reflectivity of 11–8% in the entire spectral range (3.1–6.3 nm). In the area of Sc anomalous dispersion the reflection of Cr/Sc multilayer mirrors was found to be 1.5 times higher than that outside the range. A wide range of X-ray multilayer mirrors consisting of different pairs of materials were prepared and measured for experiments on X-ray tomography of ion impurities in tokamak TEXTOR. A special investigation of Cr/Sc multilayer mirrors was made for spectrally selective soft X-ray tomography of argon ions in tokamak plasma in the spectral range 3–3.5 keV. Multilayer mirrors of W/B_4C and W/Sc with extremely short periods of 0.8–1.5 nm and number of period up to 400 have been fabricated and investigated in order to analyse the possibility of using them for collimation and focusing hard X-ray radiation. Multilayer mirrors of W/B_4C, Mo/C, Mo/B_4C and Cr/C were prepared and measured in the wavelength range 0.7–1 nm.

References

Andreev, S. S., Muller, M., Platonov, Yu. Ya., Polushkin, N. I., Salashchenko, N. N., Schafers, F., Shinkarev, S. I., Simanovsky, D. M. & Zuev, S. Yu. (1991). *Proc. SPIE*, **1800**, 195–208.

Birch, J., Eriksson, F., Johansson, G. A. & Hertz, H. M. (2003). *Vacuum*, **68**, 275–282.

Christensen, F. E., Zhu S.-H., Hornstrup, A., Schnopper, H. W., Plag, P. & Wood, J. (1991). *J. X-ray Sci. Technol.* **3**, 1–13.

Eriksson, F., Johansson, G. A., Hertz, H. M. & Birch, J. (2002). *Opt. Eng.* **41**, 2903–2907.

Michaelsen, C., Wiesmann, J., Bormann, R., Nowak, C., Dieker, C., Hollensteiner, S. & Jager, W. (2001). *Opt. Lett.* **26**, 792–794.

Schafers, F., Mertin, M., Abramsohn, D., Gaupp, A., Mertins, H.-Ch. & Salashchenko, N. N. (2001). *Nucl. Instrum. Methods Phys. Res. A*, **467**/**468**, 349–353.

Schafers, F., Mertins, H.-Ch., Gaupp, A., Gudat, W., Mertin, M., Packe, I., Schmolla, F., Di Fonzo, S., Soullie, G., Jark, W., Walker, R., Le Cann, X., Nyholm, R. & Eriksson, M. (1999). *Appl. Opt.* **38**, 4074–4079.

Schafers, F., Mertins, H.-Ch., Packe, I., Schmolla, I., Salashchenko, N. N. & Shamov, E. A. (1998). *Appl. Opt.* **37**, 719–722.

Shmaenok, L. A., Golovkin, S. V., Govorun, V. N., Ekimov, A. V., Salashchenko, N. N., Pikalov, V. V., Belik, V. P., Schuller, F. C., Donne, A. J. H., Oomens, A. A. M., Prokhorov, K. A., Andreev, S. S., Sorokin, A. A., Podlaskin, B. G. & Khasanov, L. V. (2001). *Rev. Sci. Instrum.* **72**, 1411–1415.

 Received 25 January 2003 • Accepted 7 July 2003