X-ray Schwarzschild objective for the carbon window ($\lambda \sim 4.5$ nm)

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We deal with the recent progress in the fabrication of the graded Co/C multilayer mirrors to be used in a 21× Schwarzschild objective (SO) operating at the wavelengths near 4.5 nm ("carbon window" region). The peak reflectivity of flat Co/C mirrors was measured to be 14.8% (wavelength of 4.48 nm, incidence angle of 5°). The reflectivity curves of the spherical mirrors achieved 3%–6%, with the spectral matching accuracy being $\Delta d \sim 0.008$ nm ($\Delta d/d \sim 0.3\%$). As a result the SO demonstrates a full working aperture ($N_A \sim 0.2$) operation with the total throughput of 0.25%. © 2009 Optical Society of America

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Soft x-ray microscopy in the spectral "carbon window" region ($4.5 < \lambda < 5$ nm) proves to be a noncomplicated and fruitful technique for studying thick ($\sim 5-100\ \mu$m) carbon-containing objects with soft x rays. It benefits by a relatively low absorbing dose in transparent proteins, lipids, and so on; differential contrast of different carbon compounds near the K edge; histological paraffin-based fixing of samples without an expensive cryogenic technique; and less severe vacuum requirements [1,2].

In contrast with the typical working wavelengths of "water window" microscopy $\lambda = 2.3-2.4$ nm, the carbon window wavelengths are fully accessible by reflective multilayer-based optics that operates at near-normal incidence angles: the reflectivity of the multilayer mirrors measured at the carbon K_α line ($\lambda = 4.47$ nm) is about 10% for Fe/C and Co/C [3], 8% for V/C, and 12.2% for Cr/C [4] mirrors. However, there proved to be too low a throughput of normal incidence multimirror optical systems (see, for example, [5]) for a practical application in soft x-ray microscopy.

In the present work the x-ray multilayer mirrors were based on Co/C reflective coating, which possesses a relatively high integral reflectivity. The multilayers were fabricated using Ar^+ dc-magnetron sputtering with a base vacuum of 10^{-4} Pa and a working pressure of 0.2 Pa [2]. The sputtering setup has been modified to enable high rate depositing of ferromagnetic materials for the production of smooth and uniform cobalt layers with thicknesses down to 1 nm. The deposition rate of the carbon target was kept close to the minimum value to hinder cobalt carbide formation.

Flat Co/C multilayer mirrors were fabricated on standard silicon wafers with the surface roughness of ~ 0.15 nm rms. Properties of the deposited multilayer structures were studied by using a number of different experimental techniques: cross-sectional transmission electron microscopy (TEM) imaging, atomic force microscope (AFM) scanning, and small-angle x-ray diffraction (SAXD) ($\Theta-2\Theta$) measurements at the wavelengths of Cu K_α ($\lambda = 0.154$ nm) and Ni K_α ($\lambda = 0.166$ nm) lines. The mirror's reflectivity curves versus the wavelength in the working spectral region of the carbon window were measured using synchrotron radiation facilities.

Short-period Co/C multilayer structures were found to turn into cobalt carbide/carbon composition owing to the active interlayer interaction [6] and the formation of metastable carbon-enriched cobalt carbide $\mathrm{Co_2C}$ with the density of about 6.2 g/cm^3 [7]. From the viewpoint of its optical performance, the multilayer structure $\mathrm{Co_2C/C}$ has a lower integral and peak reflectivity with the resonance wavelength shifted toward longer wavelengths. Note that similar carbide forming interlayer interaction is known to occur in many short-period multilayer metal/carbon compositions, e.g., in Cr/C structures [8].

The mentioned cobalt–carbon interaction should be taken into account in the design and fabrication of the reflective Co/C coatings: to obtain the period of $d = 2.25-2.3$ nm and the thickness ratio of $\Gamma = 0.5$, the deposited layer thicknesses were chosen to be 0.7 and 1.8 nm for cobalt and carbon layers, respectively. A control set of five flat Co/C mirrors (we call the structures as Co/C but not $\mathrm{Co_2C/C}$ for the sake of simple notation) were fabricated with the number of periods $N = 80-200$ (see Table 1). After coating deposition, sample 4 went under annealing at the temperature of 240°C for 1 h in a special vacuum chamber. Calibrated annealing of Co/C mirrors was used for precise changing of the multilayer's period up to 0.05 nm.

Table 1 and Fig. 1 present the results of soft x-ray reflectivity measurements of the Co/C mirrors carried out at the near-normal incidence angle (5°) using the BESSY synchrotron beamline [3]. One can see that the achieved reflectivity values are from $R = 8.1\%$ ($N = 80$) to $R = 14.3\%$ ($N = 200$), with the correspondent reflectivity bandwidths being $\Delta\lambda = 0.021-0.05$ nm. As the result of the annealing, the

Table 1. Parameters of the Co/C Multilayer Mirrors[a]

Sample of Co/C Multilayer	1	2	3	4	4 (ann.)
Period (nm)	2.322	2.275	2.28	2.245	2.29
Number of periods N	80	100	150	200	200
Normal incidence reflectivity R (%)	8.1	9.7	13.0	14.3	14.8
Wavelength λ_{peak} (nm)	4.606	4.522	4.535	4.466	4.555
Top layer roughness (nm)	0.142	0.135	0.137	0.154	0.146
Interface roughness (nm)	0.327	0.330	0.323	0.340	0.315

[a] Sample 4 was annealed at the temperature of 250°C; the correspondent column is marked as "4 (ann.)." The rms roughness of the top (carbon) layer was measured using an AFM scan in the field 0.25 μm × 1 μm [7].

multilayer period d was found to grow by 0.045 nm with the raise of the reflectivity up to $R = 14.8\%$.

The developed deposition technique has enabled the fabrication of high-quality Co/C multilayer coatings with low period error and supersmooth interfaces (interface roughness was found to be 0.3–0.35 nm rms on the flat samples and it did not grow with a large period number). High normal incidence reflectivity of the developed Co/C coatings has enabled starting a series of imaging experiments in the spectral region of the carbon window [7]. The next step is to improve the spatial resolution with the help of multimirror low aberration x-ray optics, such as a Schwarzschild objective (SO).

SO consists of spherical convex and concave mirrors. Owing to its simplicity and good optical performance, this two-mirror system is rather popular in soft x-ray/extreme UV imaging. In this work the basic geometric parameters of the mirrors for the SO were chosen to be the same as those the authors have used before [9]: large (concave) mirror has the diameter of $d_1 = 54$ mm and a curvature radius of $r_1 = 100$ mm; small (convex) mirror has the diameter of $d_2 = 10.5$ mm and a curvature radius of $r_2 = 35$ mm. To get a full aperture working objective, the reflectivity bands of the mirrors are to be locally coincident with the accuracy of $\Delta\lambda/\lambda < 0.5\%$ over all the working range of incidence angles, 2°–9°. This condition compels one to use an r-graded multilayer coating with the variable period deposited in agreement with both the primary reflected wavelength and the incidence angle.

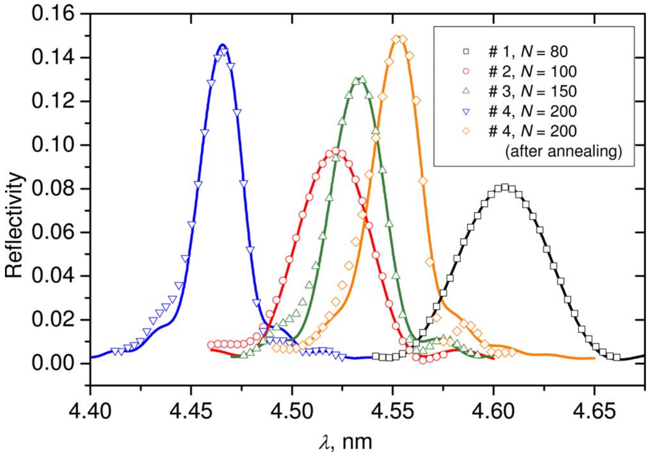

Fig. 1. (Color online) Measured (symbols) and calculated (curves) reflectivity of Co/C multilayer mirrors versus wavelength: incidence angle is 5°. Note that $\lambda/\Delta\lambda$ strongly corresponds to N for all measured samples. See also Table 1.

To produce the graded multilayer mirrors for SO operating in the carbon window, 100 periods ($N = 100$) of Co/C coating were deposited onto supersmooth spherical substrates (roughness of 0.23–0.25 nm rms), following the improved technology that has been used for the fabrication of flat Co/C mirrors. No special masks were implemented for the deposition of the small (convex) mirror, and the axial symmetry of the multilayer structure was achieved by rotation of the substrate. SAXD measurements at the wavelength of $\lambda = 0.15405$ nm (Θ–2Θ scans) were applied to determine the local parameters of the deposited Co/C coating on the surface of the convex mirror. Additionally, the multilayer parameters were controlled by a set of small silicon wafer plates (dimensions of 3 mm × 3 mm) fixed for twin deposition on the spherical surface, resembling the small mirror figure.

The information about the convex mirror coating was used for the design and fabrication of the conjugated reflective structure on the surface of the large (concave) mirror of the SO. The mask-assisted deposition and the variable rate movement of the concave substrate have proven to provide the needed gradient period distribution over the multilayer mirror (for more details, see [2]). The SAXD measurements of coated curved thin mica stripes and small wafer plates were also used to control the local period distribution on the concave mirror. For additional adjustment of the mirror periods, the deposited structure might be treated by medium temperature annealing under conditions of low vacuum ($P = 10^{-3}$ Pa) and low gradient heating [8].

The soft x-ray reflectivity measurements of the graded multilayer mirrors in the working spectral region ($\lambda \sim 4.5$ nm) were performed on the advanced light source (ALS) beamline 6.3.2 [10]: the wavelength-coordinate scanning mode was used with the incidence angle of 5°, a wavelength step of 0.02 nm, and a beam spot size of 0.3 mm × 0.05 mm.

Local multilayer's period (resonance wavelength) was derived by fitting of the measured reflectivity spectra $R(\lambda)$ (Fig. 2). The fitting procedure also revealed the interface roughness/diffusion layer value: the Co/C multilayer interfaces on curved surfaces were found to be more rough ($\sigma \sim 0.43-0.46$ nm rms) than in the case of the flat mirrors, resulting in lower values of the peak reflectivity, $R = 4.5\%-6\%$ (Fig. 3).

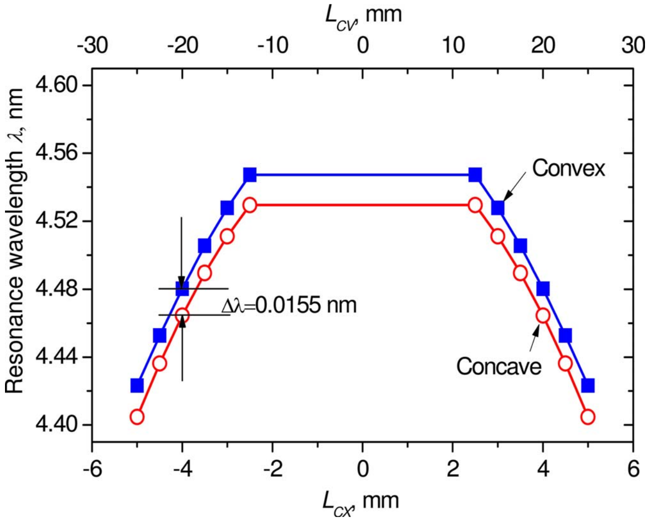

Fig. 2. (Color online) Resonance wavelengths (of maximum reflectivity) measured locally across the diameters of concave (empty circles, L_{CV}) and convex (filled squares, L_{CX}) mirrors to be used in the soft x-ray SO. The X axes are scaled to take into account ray tracing relation of the mirrors in the SO.

Figure 2 shows that the spectral matching of the reflectivity bandwidths of SO mirrors has been achieved with the accuracy (offset) of $\Delta\lambda = 0.0155$ nm that corresponds to the period matching accuracy of $\Delta d = 0.008$ nm ($\Delta d/d \sim 0.3\%$). The similarity of the gradient functions looks to be even better ($\Delta d/d \sim 0.1\%$), which enables additional posttuning of the mirror bandwidths with the help of the mentioned annealing treatment. Comparison of data obtained on the ALS beamline with the correspondent SAXD measurements demonstrates that laboratory hard x-ray characterization of the multilayers can be carried out with the accuracy of 0.004 nm and $\pm 5 \times 10^{-4}$ nm/mm, for the local period and gradient values correspondingly.

The normal incidence multilayer optics based on the structure Co/C provides the peak reflectivity as high as $R \sim 15\%$ for the flat and $R \sim 6\%$ for the figured substrates in the spectral region of the carbon window. Developed techniques of precisely graded short-period multilayer deposition and control enabled producing the variable period multilayer spherical mirrors with the accuracy needed for the construction of a full aperture two-mirror objective at the working wavelength of $\lambda \sim 4.5$ nm. The reflectivity matching of the fabricated mirrors limits the system throughput by 0.25%, but this value can be easily increased up to 1% using the currently available technology by deposition of larger number of periods (up to $N = 200$).

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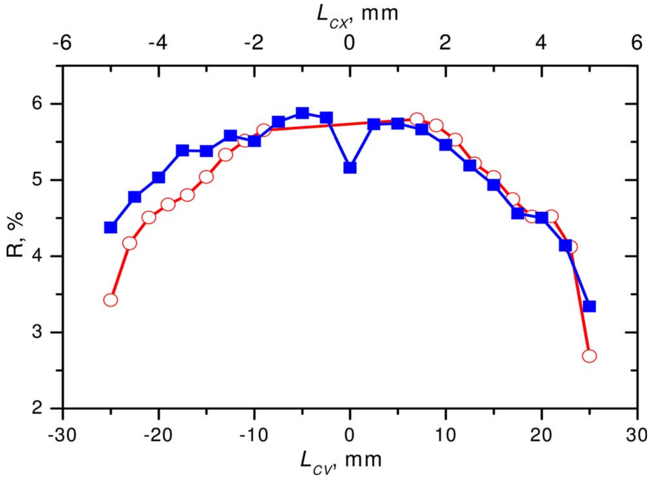

Fig. 3. (Color online) Peak reflectivity measured locally across the diameters of concave (empty circles, L_{CV}) and convex (filled squares, L_{CX}) mirrors. Pronounced drop in relative reflectance at the center of the convex mirror is believed to result from increased surface roughness of the substrate used.

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