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Short-Period Multilayer X-ray Mirrors for “Water” and “Carbon Windows” Wavelengths

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This review paper summarizes and provides an overview of our recent studies related to two types of short-period multilayer X-ray mirrors, W/B₄C and Co/C. It deals with the experimental observation of the layer intermixing effects and how they affect the X-ray mirror's optical performance. The paper presents also some examples of using the fabricated X-ray mirrors in focusing and imaging experiments at the working wavelengths 2.48 nm and 4.47 nm.

Keywords: X-ray Multilayer Mirrors, X-ray Optics, Thin Films, Interlayer Interaction, Short-Period Multilayer Structures, Metal-Carbon Compositions.

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1. INTRODUCTION

Development of X-ray microscopy for studying biological objects in the spectral regions of “water” and “carbon windows” (the wavelengths $\lambda = 2.37$ to 4.4 nm and $\lambda = 4.4$ to 5.5 nm, correspondingly) depends on an availability of the effective optical systems. X-ray multilayer mirrors (XMMs) are known to be one of the most popular X-ray optical elements that have been used in a lot of imaging and focusing X-ray experiments.^{1,2} The obvious advantages of XMMs, such as large clear aperture, near-normal incidence operation mode, spectral selectivity and mechanical and thermal stability make them to be the best choice in many X-ray optical applications based on a compact X-ray source.

Evidently, a near-normal incidence XMM operating at the wavelengths 2 to 5 nm needs a very short-period multilayer reflective coating deposited with a nanometer-scale structure period 1.2 to 2.8 nm. The formation of these short-period structures meets a number of serious problems which do not reside in fabricating longer period structures. First of all, there is a problem of mixed zones which were formed on the layer interfaces during deposition^{3–5} with their dimensions being comparable to the main material layers. The formation of the mixed zones is known to strongly affect the layer thickness and chemical composition. Another problem is an influence of the interface roughness upon the reflectivity of short-period XMMs. In early stages of film formation the thin layers are observed in the form of almost isolated islands. Of course, such non-continuous layers give rise to a significant interlayer roughness and a distortion of the interference pattern in the multilayer structure.^{6,7}

In other words, the real properties and structure of a deposited short-period XMM can be quite different from not only ideal model but also the same larger period multilayer. Therefore, a successful practical realization of high reflection short-period XMMs requires careful investigation of film formation processes and interlayer structures to compose an optimal design of the mirror. It is important to note here that this investigation is usually specific for

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the given material-pair and multilayer structure and cannot be generalized to all types of short-period structures.

The paper presents an overview and synthesis of the results related to our studies of the structure, density and chemical composition of two selected short-period XMMs. The X-ray mirrors have been fabricated and used in a series of successful imaging and focusing experiments.

There is a well-known problem of reliable characterization of the interlayer interactions, especially the interface mixed zones formed during deposition. Particular challenging is an identification of the crystalline phases and chemical analysis inside thin individual layer due to its amorphous state and standard X-ray diffraction methods cannot be used. On the other hand, nanoscale thickness of the layers makes an application of other analytical techniques (ICP-AES, XPS, SIMS) almost impractical affected by intermixing effects of etching ion beam and limited escape depth of the electrons.^{8,9}

Nevertheless, the small-angle X-ray diffractometry can provide important information on a periodic structures consisting of nanometer-scale layers. X-ray diffraction pattern analysis is known to reveal the multilayer structure period and thickness ratio as well to estimate the interface roughness height.¹⁰ As we will show below, the study of a series of the multilayers with slightly modified parameters can empower this method and yield more reliable results.

This review covers our recent experimental studies of short-period Co/C and W/B₄C multilayers deposited by magnetron sputtering. These multilayer coatings were intended for use as XMMs in the spectral regions of the “carbon window” ($\lambda = 4.4$ to 5.5 nm) and the “water window” ($\lambda = 2.7$ to 3.1 nm), correspondingly. Our choice

of these material-pairs is motivated by their optical constants and ability to form ultra-thin continuous films of sub-nanometer thickness.^{11,12}

2. STRUCTURAL CHARACTERISTICS OF SHORT-PERIOD W/B₄C MIRRORS

To investigate the specific features of short-period W/B₄C multilayer structures we fabricated a series of the multilayers with the same thickness of B₄C layers (5.3 nm) but different thickness of W layers. All the structures consisted of from 17 to 20 periods (bi-layers). The nominal thickness of tungsten layers in every sample was calculated as the product of the tungsten deposition time and its deposition rate. Obviously, the nominal multilayer period could be found as a sum of the calculated thicknesses of W and B₄C layers (for more details see Kopylets et al.¹³). The dependences of both calculated (nominal) and measured values of the structure period upon the nominal tungsten layer thickness H_W are presented in Figure 1. One can see clearly that the measured dependence unlike the calculated one consists of two linear segments. The second segment $H_W = 0.9$ to 3 nm is almost parallel to the nominal curve. However, at small thicknesses $H_W < 0.9$ nm the experimental curve reclines by a different angle. To make the mentioned change more visible Figure 2 shows the difference between the measured and nominal values of the period. The different slope angle means decreased growth rate of the W layers in the early stages of their deposition despite of the constant deposition rate. Note that the breakpoint of the growth rate corresponds to the nominal tungsten thickness $H_W \approx 0.85$ nm.

Figure 1. The dependence of both measured and nominal (calculated) values of the period H upon a nominal thickness H_W of tungsten layers in the W/B_4C multilayer.¹³ Dashed lines depict two linear approximations used.

Other multilayer structures, e.g., W/Si , Co/C , Mo/Si are known to demonstrate a similar change of the layer growth rate for thin layers,^{5,14–16} that is usually related to various interlayer interaction processes during deposition, e.g., the formation of new chemical compounds and solid solutions accompanied by a volume change. In the particular case of W/B_4C samples the breakpoint at $H_W \approx 0.85$ nm can be explained as follows. Below this point whole tungsten layer turns into a B_4C -based mixed layer, while grown tungsten layer would contain both mixed and pure tungsten layers. In other words, an extended tungsten deposition leads to a growth of the pure tungsten layer.¹³

The measured period of W/B_4C multilayers is found to be always less than its nominal value predicted by the deposition rates, and the period diminution is up to 0.25 nm. We suggest that specific interaction of W and B_4C in the tungsten layers during deposition result in new chemical compounds and metastable solid solutions but not a simple mixture consisting of the particles of pure tungsten and boron carbide.

Our simulation and fitting of the corresponding small-angle X-ray diffraction (SAXD) curves prove that the very interlayer interaction during deposition makes the layer

Figure 2. The difference between measured and nominal values of the period ΔH (see Fig. 1) versus a nominal thickness H_W of the tungsten layer.¹³

thickness different from the predicted one and changes the layer density.

The difficulty of studying the structure of short-period multilayers stems also from too few peaks on the measured SAXD curves (see Fig. 3(a)). Usually an analysis of the peak amplitude damping enables one to calculate the thickness ratio of spacer/absorber layers in a periodic multilayer or the thickness ratio of absorber/period (β) (see, for example, Refs. [17, 18]). The absence of the third diffraction order peak observed in our case makes it impossible to determine the parameter β , while a diminution of the second peak may be the result of the roughness and not the thickness ratio $\beta = 0.5$. As was mentioned above, to determine the real parameters of W/B_4C multilayers we used a new approach. It is based on the analysis of X-ray diffraction patterns measured on a family of similar multilayer structures with the same periods but variable thickness ratio. Of course, this series would include a sample with the minimum second peak intensity due its absorber/period thickness ratio 0.5.

We deposited a series of W/B_4C structures with about the same period values of 1.70 to 1.73 nm (400 periods) but different portions of the tungsten layer, i.e., its

Figure 3. (a) Small-angle X-ray diffraction curve measured with $Cu-K\alpha$ radiation on W/B_4C structure with the period 1.7 nm; (b) relative intensity of the second diffraction peak R_2/R_1 as a function of the critical angle θ_c measured on the W/B_4C structures with about the same structure period ~ 1.7 nm but different thickness ratio.¹³

thickness to the period ratio.¹³ Then, for each sample the critical angle of total reflection (TR) and the intensities of two diffraction peaks of the SAXD curve were measured. The critical angle depends upon the average refraction index of the entire multilayer structure¹⁹ and, consequently, is proportional to the tungsten content.

Figure 3 shows the ratio of the intensities of two SAXD diffraction peaks as a function of the critical angle measured on the W/B₄C structures. Each point of the plot corresponds to one sample in the series. The ratio of intensities of two first diffraction peaks has been proven to weaken the impact of the measurement errors and low intensities of the multilayers with the thickness ratio that is far from optimal.

The lowest value of the second peak intensity was observed at a critical angle of $\theta_c = 0.372^\circ$ (see Fig. 3). Consequently, the W/B₄C multilayer with a thickness ratio of 0.5 and period 1.7 nm should have the critical angle 0.372° . Then, with using relation¹⁹ between the critical angle and averaged reflective index of the multilayer, one can estimate the density of the tungsten layer as $13.4 \pm 0.7 \text{ g/cm}^3$. The obtained value is much less than that of pure tungsten. The tungsten density is known to be 19.26 g/cm^3 for a bulk and 18.4 g/cm^3 for a thin film. The observed discrepancy indicates a presence of a significant amount of low density compounds in the tungsten layer.

In other words, a deposition of short-period W/B₄C multilayer with the period 2.5 nm or less, results in a complex structure consisting of the products of reactions between the tungsten and boron carbide but not pure tungsten and boron carbide layers. Below we will call new layers as “tungsten-containing” noting that there are no pure tungsten layers.

To study interlayer reactions related to a disappearance of pure tungsten layer we used methods of X-ray phase analysis. Figure 4 shows that there are two wide peaks observable in a large-angle X-ray diffraction curve measured on the samples with the structure periods 1.35 nm

and 2.5 nm. The peak at an angle of 69.2° corresponds to (004) reflection of silicon monocrystalline substrate used (see the diffraction curve of blank silicon substrate). Another peak at an angle of 37.2° corresponds to neither pure tungsten nor boron carbide. Observation of strong lines related to tungsten carbides and borides in the angle range of 25 to 50° allows suggesting a presence of the corresponding products of interlayer reactions. Earlier we have reported the first signs of the tungsten carbide lines appear just after start of the W/B₄C structure annealing²⁰ (Fig. 5). However, the carbon atoms are known to be unable to replace the boron in transition metal compounds²¹ and such an appearance of the carbide lines under heating means that sufficient part of tungsten atoms was initially either unbounded or bounded in amorphous boron carbides. In other words, this experiment indicates that the main part of tungsten atoms did not bind with boron and the peak at 37.2° can be explained as a superposition of multiple peaks formed by various tungsten carbides. It is also an evidence of the absence of a pure tungsten layer in short-period W/B₄C multilayers deposited with the period 2.5 nm or less, where the tungsten occurs only in the form of carbon-containing compounds.

The Auger analysis of a W/B₄C structure containing slightly thicker tungsten layers is known to recover a presence of chemical bonds W–C.⁹ So, with no pure tungsten detected in the layers, we can suggest that thin tungsten-containing layers in short-period W/B₄C multilayers cannot be described as a mechanical mixture of pure tungsten islands and boron carbides filling the space between them.

To understand the mixed layer parameters and its behavior under elevated temperature we have given serious consideration to structural and phase transformations of the W/B₄C multilayer structures under heating (annealing). Siffalovic et al.²² investigated the phase transformations in W/B₄C structures with the period 1.37 nm at high temperature. The X-ray diffraction analysis showed a series of new peaks appearing under heating to 900°C against a halo of initial amorphous materials. These peaks indicate a production of tungsten-boron compounds with the high content of boron as a final result of the W/B₄C interlayer interaction. However, in that work the corresponding intermediate phase transformations at lower temperature were not identified.

The works^{20,23} dealing with the W/B₄C structures with the periods 1.35 nm and 2.5 nm are more informative on this subject. In this case the 2.5-nm period structure, which consisted of thicker layers, demonstrated much wider regions of the coherent X-ray scattering. The distinct peaks of the X-ray diffraction enabled a relatively easy identification of the crystal phases even at low temperature. The X-ray analysis has proved a formation of tungsten carbides, first of all, W₃C, under moderate heating at temperature 100 to 600°C (see Fig. 5).²⁰ However, at higher temperature a formation of various tungsten borides took

Figure 4. Small-angle X-ray diffraction curves of two W/B₄C structures with the periods 1.35 nm and 2.5 nm measured with Cu-K α radiation ($\lambda = 0.154 \text{ nm}$).^{20, 23} X-ray diffraction curve of a blank silicon wafer is plotted as well.

Figure 5. Small-angle X-ray diffraction curves of W/B_4C structures with the periods 2.5 nm (a) and 1.35 nm (b). The curves were measured with $Cu-K\alpha$ radiation before and after annealing at the following temperatures: 620 °C, 800 °C and 950 °C (a), 500 °C and 1000 °C (b).^{20,23} In order to suppress relatively intense substrate peaks, the measurements were carried out with a ω -offset of 3°. The substrate is (001) monocrystalline Si wafer.

place including high-tungsten borides W_2B/WB and high-boron borides W_2B_5/WB_4 . Note that similar processes are reported to happen in $Mo/(B + C)$ multilayer structures under annealing.²⁴

The X-ray diffraction curves of W/B_4C structures with a period of 1.35 nm (see Fig. 5) follow the curves of 2.5-nm structures but in this case their phase peaks are wider and less pronounced. Like some longer-period structures these multilayers show the peaks of high-boron tungsten borides that were suggested to be a final product of the chemical reactions under annealing at temperature 1000 °C. Despite of the analysis of the X-ray diffraction curves on 1.35-nm period structures is not so evident, we can assert that these samples pass through the same sequence of the chemical processes with carbide and boride formations as 2.5-nm period structure does but here these processes start at lower temperature.

This assumption is confirmed also by overlapping of the period-versus-temperature curves obtained for both W/B_4C structures (Fig. 6). The dependence of 2.5-nm period structure has a maximum at a temperature of 600 °C. The observable volumetric change indicates phase transformations causing formation of new compounds and another density. Thus, the structure period rises by 1.7% under moderate heating to 120 to 600 °C. Our volumetric analysis of all possible chemical reactions in $W-B-C$ compositions (with using the density values for bulk materials) shows that a formation of tungsten carbide CW_3 is the

only possible process that is accompanied by a volume increase.²⁰ Other chemical reactions were found to lead to decreasing volume. It is a CW_3 carbide formation that explains the diffraction peaks appearance after annealing at temperature 400 to 620 °C. Under further heating to higher temperatures the structure begins to compress back to its initial volume, and the X-ray diffraction peaks related to the tungsten carbide disappear totally (Fig. 5). The boron atoms displace the carbon in tungsten-compounds and turn them into the borides. Finally, the structure returns to its

Figure 6. The dependence of relative change of the period H/H_0 versus an annealing temperature. The curves are plotted for two different W/B_4C structures with the periods $H = 2.5$ nm and $H = 1.35$ nm.²³

initial period and unextended volume because of this formation of tungsten borides.¹⁵ Note that unlike the structures with the period 2.5 nm, 1.35-nm multilayers show a relatively weak rise in the period value (only 1.3%) but the volume compression starts earlier at a temperature which is about 100 °C lower.

As a result, a W/B₄C-based XMM with 1.35-nm period would be operational and preserve its periodical structure at relatively high temperature (up to 600 °C). However, its reflectivity would degrade dramatically at a temperature of 750 °C, which is 150 °C lower compared to a 2.5-nm period structure.

TEM images (Fig. 7) shows that the mentioned degradation of the X-ray reflectivity and structure of the W/B₄C multilayers with the periods 1.35 nm and 2.5 nm is a result of agglomeration of the tungsten-containing layers and formation of the globules.²³ However, in the case of 1.35-nm-period structures the globules begin to appear at temperatures as low as 800 °C (Fig. 7). In other words, the destruction of this structure is observed at temperature 200 °C lower compared to that of 2.5-nm period structure due to thinner layers of B₄C. One possible explanation is that the atoms of tungsten-containing layers need to pass a shorter way to form the globule and, consequently, the destructive process of globule formation can be initiated by less mobile atoms at lower temperatures. Besides that, small thickness of B₄C layers can also facilitate a critical breach in the film continuity leading to a coagulation of neighboring tungsten-containing layers.

Figure 7. TEM images of W/B₄C multilayer structures with the period of 1.35 nm before (a) and after (b) annealing at a temperature of 800 °C.²³

To confirm the influence of the thickness of B₄C layers on the critical temperature we deposited and annealed one more sample of the W/B₄C multilayer with an increased thickness of B₄C layer (3.5 nm) and the same thickness of the W layer as compared to the previous 2.5-nm period structure.²⁰ Figure 8 shows the X-ray diffraction curve measured on this sample before and after the annealing at a temperature of 1000 °C. One can see the high temperature does not affect the structure regularity. The first order diffraction peak holds its position and height, and the number of the peaks remains.

The X-ray phase analysis shows no tungsten boride related peaks on the curve and no large globules exceeding the period length at temperatures below or equal 1000 °C. Nevertheless, as temperature rises to 1150 °C the structure regularity gets destroyed as one can see from the reflectivity fall (Fig. 8). So, the deposition of thickened B₄C layers improves the thermal stability of W/B₄C multilayers by approximately 100 °C.

Thus, our experimental studies have demonstrated that an annealing of W/B₄C multilayers would results in notable interlayer mixing processes that change the thickness, density and phase composition of the tungsten-containing layers.

Obviously, a measurement of the X-ray reflection coefficient can be used as a criterion of the X-ray optical performance of multilayer structures with taking into account the structure quality and interface roughness. Figure 9 plots the measured X-ray reflectivity of a series of W/B₄C XMMs as a function of their structure period.²³ The number of periods of the samples was varied from 90 to 1100 to meet a change of the effective number of the reflecting layers due to different period values. The tungsten-containing layer shared 0.35 to 0.45 of the structure period.

The X-ray reflectivity was measured to drop significantly for the period lengths less than 2.5 nm (see Fig. 9). It should be mentioned that this behavior of the reflectivity is common for all types of the XMMs^{12,25} due to the same kind of influence of structure imperfections, such as interface roughness, heterogeneous layer morphology, etc. One can see from Figure 9 that the reflectivity curve calculated with assumption of the interface roughness 0.285 nm (rms) and the density of tungsten-containing layer 13.4 g/cm³ coincides well with the experimental data obtained for the XMMs with the periods less than 2.5 nm.

A decline of the reflectivity observable for the structures with the period 1.2 to 1.3 nm does not mark a destruction of their periodic structure (Fig. 7), while the reflectivity is still around 40% (Fig. 9). We assume this reduction of the X-ray reflectivity is due to effects of both changing density and chemical composition of the absorber layer and related degradation of the optical properties of the materials in the reflective coating. For example, a decrease of the density of tungsten-containing layers in 2.5-nm period W/B₄C structure down to 13.4 g/cm³ would result in ~20% drop

Figure 8. Small-angle X-ray diffraction curves of two W/B₄C multilayers before and after annealing at 1000 °C: (a) The structure has the period 4.3 nm and increased thickness (3.5 nm) of B₄C layers; (b) the structure has the period 2.5 nm with ordinary B₄C layers. The data were measured at Cu-K α line.²³

of the X-ray reflectivity in both hard X-ray (Cu-K α line) and “water window” spectral regions.

One more problem in design and fabrication of a normal-incidence XMM operating in the spectral region of “water” or “carbon windows” is known to be a very narrow bandwidth of the reflectivity peaks. Therefore, an accurate design of the mirrors requires reliable information about the chemical composition and density of the material-pairs in the deposited reflective coating to determine its real X-ray optical properties including correct position of reflectivity peak.

The work²⁶ presents an example of successful solution of this problem in production of W/B₄C mirrors to be used in the experiments on focusing of soft X-ray wavelength $\lambda = 2.48$ nm in “water window” spectral region. The multilayer mirrors with the period 1.25 nm were deposited by magnetron sputtering with taking into account of all

the mentioned information on the density, composition and interface roughness of tungsten-containing layers. Using the corrected period and thickness ratio of the deposited multilayer enabled us to meet the required mirror’s optical specification including its spectral and angular properties. These short-period X-ray mirrors were applied in experiments to produce a quasi-monochromatic X-ray beam from laser plasma X-ray source at the wavelength $\lambda = 2.48$ nm.²⁶ The results of the work²⁶ show that we have succeeded in a fabrication of the X-ray multilayer mirror with very narrow reflectivity peak fixed at the specified position with an accuracy of 0.5° close to the normal incidence. And this mirror has been used successfully for X-ray optical experiments in the water window spectral region.

3. STRUCTURAL CHARACTERISTICS OF SHORT-PERIOD Co/C MULTILAYER MIRRORS

As was mentioned above, the highest X-ray reflection coefficient of a short-period mirror can be achieved only with the help of the multilayer structures consisting of nanometer-scale continuous layers. Thus, a normal-incidence Co/C XMM operating at the wavelength $\lambda = 4.5$ nm should contain cobalt layers with the thickness about 0.5 nm. However, such ultra-thin cobalt layers deposited by magnetron sputtering are known to be non-continuous.

Figure 10 presents TEM images of the Co/C multilayer structure deposited with slightly thicker cobalt layers. One can see that there is a film coalescence in 0.8-nm thick cobalt layers, while the roughness heights of both C/Co

Figure 9. Peak reflectivity R of the W/B₄C multilayers measured in the first diffraction order with Cu-K α radiation as a function of the period H . Dashed curve presents the calculated reflectivity with the interface roughness 0.285 nm (rms).²³

Figure 10. TEM images of Co/C multilayer structures with the following thicknesses of cobalt layers (a) 0.8 nm, (b) 1.1 nm and (c) 1.4 nm. (d) Shows electron diffraction pattern obtained in the selected area of (c). The carbon layer thickness is always equal to 6 nm.

and Co/C interfaces rise up to the thickness of the cobalt layer (Fig. 10(a)). As the cobalt layer thickness increases up to ~ 1.1 nm, the coalescence looks to be over. Finally, it leads to a formation of the continuous cobalt layer (Fig. 10(b)). The correspondent SAXD curves show also a decrease of the interface roughness down to 0.45 nm (rms). Further thickening of the cobalt layer up to 1.4 nm reduces the interface roughness to 0.36 nm. Figure 11 shows TEM images of Co/C multilayers with the thicknesses of cobalt and carbon continuous layers 1.7 nm and 0.7 nm.²⁷

As was mentioned above, another common defect of thin layer structures is an interlayer mixing during deposition. According to the phase diagram, the cobalt and carbon can form only two metastable cobalt carbides Co_2C and Co_3C but not any stable compound.^{28,29}

Figure 11. TEM images of the Co/C multilayer structure deposited with a skipped cobalt layer. The structure period is about ~ 2.4 nm. The arrow indicates position of a missing cobalt layer.²⁷

Similar to short-period W/B₄C structures mentioned above, crystallography analysis (e.g., X-ray diffraction, electron diffraction, etc.) of a short-period Co/C interlayer mixing is rather difficult because of amorphous state of both layers.^{14,30–33} Neither TEM imaging is effective in reliable identification of mixed zones of the cobalt and carbon¹⁴ in contrast to, for example, mixed zones of Mo/Si and Sc/Si structures which usually form crystalline metal layers.^{34,35}

However, there are other experimental methods which can be used for investigation of interlayer interactions of short-period Co/C multilayer structures. Chernov et al.^{36,37} successfully used EXAFS spectroscopy to study thin cobalt layers with the thickness 1.2 nm. This method provided the number of the nearest cobalt atoms around the central one in the first coordination sphere. This number was determined to be much less than that measured for the thicker cobalt layers with the thickness 5 nm. Moreover, in thin layers some carbon atoms were found also among the neighbors.³⁷ It was suggested that the growth of cobalt layers starts with a formation of the mixed layer for the thicknesses less than ~ 0.6 nm.³⁶ Krishnan et al.³⁸ suggested also a presence of an amorphous 1-nm-thick carbide layer in all interfaces of Co/C structure, if thickness of the cobalt layers is less than 4 nm.

More detailed description and experimental studies of the interlayer interactions of Co and C layers deposited by magnetron sputtering has been published before.^{14,27} Figure 11 shows TEM images of the Co/C structure with variable thickness ratio of cobalt and carbon layers.²⁷ One can see the ratio is about 2.5:1, although but it should be only 1:1.7 according to the established deposition rates.

Moreover, when we intentionally skipped the cobalt layer in the multilayer deposition sequence (Fig. 11), we have got tripled but not doubled thickness of the carbon layer. This significant extension of two neighbouring carbon layers is related to a disappearance of both interfaces C–Co and Co–C that control the interlayer mixing.

Figure 12. The structure period H of Co/C multilayers as a function of the nominal thickness H_{Co} of the cobalt layer.¹⁴

Figure 13. Soft X-ray reflectivity calculated for different Co/C multilayer mirrors as a function of a wavelength in the spectral region of “carbon window.” The parameters of the mirrors are presented in Table I. The incident angle is 2.5° .

We found out that the mixing processes and related increase of the thickness ratio result in a shortening of the structure period. For example, there is a 5% difference between the calculated (nominal) and measured values of the structure period in a Co/C multilayer with the nominal period 2.2 nm and nominal thickness ratio 1:1.

Figure 12 shows two linear sections in the dependence of the period versus the nominal cobalt layer thickness.^{14,27} As well as in the case of the W/B₄C multilayers, to obtain this plot we produced a series of Co/C structures with equal thicknesses of the carbon layers (corresponding to the nominal layer thickness 6.2 nm) and different thicknesses of the cobalt layers.

As was mentioned above, this kind of two-linear segments dependence is a common feature of many multilayer periodic structures containing interlayer mixed zones.¹⁵ Slow growth of the structure period in the initial stage (when the nominal cobalt layer thickness is less than $H_{Co} \sim 1.6$ nm) results from an active mixing of cobalt and carbon layers¹⁴ and a contraction of the deposited multilayer volume. To note a complex composition of thin cobalt layers they will be called as “cobalt-containing” similar to tungsten layers of W/B₄C structures.

To determine a chemical composition of the cobalt-containing layer we fabricated also a set of samples coated by a single cobalt or carbon layer deposited under the

experimental conditions of the corresponding Co/C multilayer structures. The total thickness of a single-layer coating was equal to the product of the carbon (or cobalt) layer thickness and the number of periods. These thick single layer films consisting of one material provided us with the thickness and density of the layers formed without interlayer mixing. These data can be used for a comparison with the corresponding parameters measured on the Co/C multilayer structures.

We used the obtained values of the layer thickness and density to estimate a chemical composition of the cobalt-containing layer with the help of well-known chemical Eq. (1) and relation (2):³⁹

$$x\text{Co} + y\text{C} = \text{Co}_x\text{C}_y \quad (1)$$

$$x:y = \frac{m_1(\text{Co})}{M(\text{Co})} : \frac{m_1(\text{C}) - m_2(\text{C})}{M(\text{C})} \quad (2)$$

where $(x:y)$ is the ratio between the numbers of cobalt and carbon atoms in a cobalt-containing layer, $M(\text{Co})$ and $M(\text{C})$ are the molar mass of cobalt and carbon, respectively, $m_1(\text{Co})$ and $m_1(\text{C})$ are the masses of cobalt and carbon in a thick single layer film divided by the number of periods, $m_2(\text{C})$ is the mass of one carbon layer in the Co/C multilayer structure.

Note that the relation (2) contains only the mass $m_1(\text{Co})$ of a pure cobalt layer in a thick cobalt layer but not the mass $m_2(\text{Co})$ in the Co/C multilayer. We assume that a thin (thickness < than 1.8 nm) cobalt-containing layer in the Co/C multilayer would consume all the pure cobalt during deposition.¹⁴ The corresponding calculations showed that increasing of the cobalt-containing layer thickness from 1.2 nm to 1.8 nm would result in a change of the cobalt/carbon atoms ratio in this layer from 1:1 to 2:1.

The SAXD study of Co/C multilayer structures revealed the density of the cobalt-containing layer increases from 6 g/cm³ up to 7–7.5 g/cm³,¹⁴ i.e., to the density 7.66 g/cm³ of metastable cobalt carbide Co₂C.⁴⁰ By contrast, the density of the carbon in the single layer and Co/C multilayer structure was found to be the same, about 2 g/cm³. These experimental results are in a good agreement with Spiller et al.⁴¹ where the authors hypothesized a presence

Table I. Parameters of the Co/C multilayer structures used for calculation of the soft X-ray reflectivity curves in Figure 13 (“carbon window” mirrors).

Sample no.	Cobalt layer thickness, nm	Number of periods	Multilayer period, nm	Cobalt layer density, g/cm ³	Interface roughness (rms), nm	Element ratio in cobalt-containing layers
8	0.80	200	2.25	5.2	0.80	Co _{0.3} C
11	0.11	200	2.25	5.8	0.45	Co _{0.8} C
12	0.12	200	2.25	6.0	0.42	CoC
14	0.14	200	2.25	6.4	0.36	Co _{1.3} C
15	0.15	200	2.25	6.6	0.33	Co _{1.5} C

Figure 14. TEM images of Co/C multilayer structures with different thicknesses of cobalt layers: (a) 2 nm, (b) 3 nm and (c) 4 nm.⁴⁴

of an amorphous mixture of cobalt and carbon in the form of cobalt carbide Co_2C with the density 6.75 g/cm^3 in carbon-containing layers. In other words, a short-period cobalt/carbon multilayer structure is in fact $(\text{Co} + \text{C})/\text{C}$.

To illustrate the influence of the interface mixing processes on the soft X-ray optical performance of XMMs (see Fig. 13 and Table I) we calculated near-normal incidence X-ray reflectivity curves at the wavelength $\lambda = 4.47 \text{ nm}$ (“carbon window” spectral region) for a number of multilayer structures with the same period 2.25 nm but various structure parameters, such as the thickness ratio, density, chemical composition of the cobalt-containing layers and interface roughness (some parameters of the cobalt-containing layers were calculated with using linear interpolation and extrapolation).

One can see that a Co/C multilayer deposited with the close-to-optimum thickness ratio value would possess the lowest reflectivity 0.23% at the working wavelength. Evident explanation is the cobalt-containing layer is too thin. It is still in the early stages of growing with clear island morphology and high roughness comparable to the layer thickness (see sample 8 with the cobalt layer thickness 0.8 nm). For the significant reflectivity to be achieved, the cobalt-containing layers should be continuous. The better quality of the layer seems to prevail under its non-optimal thickness 1.2 nm to 1.5 nm (samples 12, 14 and 15). Figure 13 shows also a displacement of the reflectivity curves of Co/C XMMs with changing the cobalt-containing layers thickness.

It is interesting to note that interlayer mixing reactions lead to amorphous structure of the cobalt-containing layers.¹⁴ Pure cobalt layers are usually amorphous only at a condensation temperature below $33\text{--}38 \text{ K}$ but some amorphism-inducing additives, e.g., boron or carbon, can increase the temperature of amorphization significantly.^{42,43} A cobalt-containing layer remains in the amorphous state until the thickness achieves 2 nm (Fig. 14). So, the TEM images demonstrate high absorption contrast of the cobalt-containing and carbon layers that is typical for amorphous materials. One can see thicker cobalt-containing layers with the thickness

$\sim 4 \text{ nm}$ contain crystalline cobalt with evident crystal planes. More detailed description of composition and structural transformations of the cobalt layers can be found elsewhere.^{14, 27, 31, 44}

4. OPTICAL PERFORMANCE OF Co/C MIRRORS AT THE “CARBON WINDOW” WAVELENGTHS

We deposited a number of flat XMMs consisting of 200 Co/C periods by magnetron sputtering onto silicon wafers with the surface roughness about 0.15 nm (rms).⁴⁵ The multilayer period was chosen to be 2.3 nm with the thickness ratio about 1:1. The multilayer design was specified with taking into account the results of our previous investigations of the Co/C interlayer mixing effects concerning the thickness ratio, period and the layer density.

The measured reflection coefficient was $R = 14.3\%$ at the wavelength 4.5 nm at near-normal incidence. A follow-up annealing of the mirror at a temperature of 240°C improved the reflectivity up to 14.8% (Fig. 15). We observed also that annealing increased the structure period and, consequently, resulted in a shift of the reflectivity peak towards longer wavelengths. Simulation of the measured reflectivity curve indicated thickening of the carbon layers as the main contributor to the observable growth of the structure period (see Fig. 15). At the time of the measurements the reflectivity values $R = 14.3\%$ and $R = 14.8\%$ were the highest at this particular wavelength $\sim 4.5 \text{ nm}$ over the world. It should be mentioned here that Andreev et al.^{46,47} also studied the short-period metal/carbon multilayer structures at similar wavelengths and obtained relatively high values of the soft X-ray reflectivity.

We believe that an annealing treatment of such metal/carbon structures can be a convenient tool for a tuning of the structure period (and the reflectivity peak position) to a specified value of the wavelength and incidence angle. Of course, this technique needs to know a dependence of the reflectivity coefficient and the structure period as a function of annealing temperature. Figure 16 presents such

Figure 15. Measured (symbols) and calculated (dash lines) reflectivity curves of Co/C multilayer mirrors as a function of working wavelength: (1) Before and (2) after annealing at a temperature 240 °C. Near-normal incidence angle is about 5°. ⁴⁵

kind of dependence obtained for Co/C multilayer with the period 2.3 nm.

Temperature 270 to 280 °C is found to be maximum allowable for a short-period Co/C XMM. At higher temperatures the Co/C reflective coatings would be destroyed through a cobalt globule formation process. More details on this destruction process can be found in Refs. [31, 32].

For various X-ray optical applications in the “carbon window” spectral region we have deposited a number of multilayer mirrors onto spherical substrates with the curvature radii from 35 to 110 mm. The most challenging task was to deposit two Co/C spherical mirrors to be installed into layout of the Schwarzschild objective (SO). This sort of high spatial resolution optical systems based on normal-incidence XMMs is rather popular in various soft X-ray imaging and focusing experiments.^{1,2,48–52} This deposition work required special efforts to get the mirrors’ reflectivity bands overlapped (see below).

Figure 16. Change of the structure period $H-H_0$ (1) and hard X-ray reflectivity (2) as a function of annealing temperature. The X-ray reflectivity was measured in the first Bragg peak with using Cu- $K\alpha$ radiation. The nominal period is 2.3 nm. ²⁷

The deposited Co/C mirrors of SO consisted of only 100 periods to get the spectral reflectivity bandwidth increased. They were produced by magnetron sputtering with a shadow masking onto concave (curvature radius 100 mm, diameter about 54 mm) and convex (curvature radius 35 mm, diameter about 10 mm) spherical substrates. Due to variable incident angle on both mirrors it was critical to follow the calculated distribution of the structure period in all points of the mirror’s working area. Moreover, the conjugation of both mirrors in the SO ensemble required also a mutual tuning of their reflectivity curves over the all working aperture to achieve the spectral overlapping.^{49,50}

The “at-wavelength” measurements of these deposited Co/C mirrors showed the near-normal incidence reflectivity coefficient 4.5–6% for both concave and convex mirrors (Fig. 17).^{48,52} It is two times less than the reflection coefficient of the corresponding flat mirror.⁴⁵ The loss of the reflectivity can be explained by higher interface roughness in depositing curved multilayer mirrors: 0.48 nm against 0.36 nm (rms). This roughness growth appears to be a result of inclined magnetron sputtering. Due to magnetron is not a point source of the particles the complex geometry of the deposition needs more detailed consideration of particle kinetics and thin film formation. Torre et al.⁵³ demonstrated an increase of the roughness of films sputtered onto inclined substrates. In our case, the inclination of the magnetron plane to the substrate surface was 16° and 8° for concave and convex mirrors, correspondingly.

It should be mentioned that there is a reserve for a further enhancement of the reflectivity through an increasing the number of the periods up to 200. New methods of

Figure 17. The soft X-ray reflectivity curves of: (1) concave and (2) convex Co/C mirrors of the Schwarzschild objective measured in proper points of the ray trajectory.⁴⁸

accurate gradient deposition and control as well as post-deposition adjustment of the reflectivity curve allow one to use this way now.⁴⁸

5. CONCLUSION

A real structure of short-period W/B₄C and Co/C multilayers significantly differs from an ideal model mostly due to strong interlayer interactions. As a result, thin tungsten-containing layers of W/B₄C structures with a nominal thickness less than 0.85 nm acquire new reduced density ($13.4 \pm 0.7 \text{ g/cm}^3$) and turn into a mixture of the boron and tungsten carbides. Further growth of a thicker tungsten-containing layer will be supported by formation of the pure tungsten layer.

In the W/B₄C structures the layer intermixing at the deposition is found to result in the following: (a) the structure period contracts, a share of the tungsten-containing layer rises; (b) the reflection coefficient drops by 14–23% in the spectral regions of both “water window” and hard X-rays.

In Co/C XMMs the thickness of the mixed layer can reach 1.8 nm. An increase of the cobalt-containing layer thickness from 1.2 nm to 1.8 nm changes the cobalt/carbon atom ratio and the layer density from 6 g/cm^3 to 7.0 to 7.5 g/cm^3 . Similar to the W/B₄C structures the interlayer interaction results in a compression of the Co/C structure period. Thus, a 2.23-nm period Co/C structure shows about 5% contraction.

To achieve high soft X-ray reflectivity in the “carbon windows” spectral range the thickness of cobalt-containing layer should be 1.2 to 1.6 nm. It turns out to be 2.5–3 times thicker than the optimum design calculated on the basis of ideal model of the Co/C structure. Larger thickness of the cobalt-containing layer is needed to get appropriate layer continuity and interface roughness.

The real parameters of deposited short-period multilayer structures have enabled a more practical design which was used in manufacturing of Co/C XMMs for imaging and focusing experiments at the “carbon window” wavelengths. The reflectivity coefficient of the normal-incidence Co/C mirror was measured to be 14.3% at the wavelength $\lambda = 4.5 \text{ nm}$. An annealing of the Co/C mirror at a temperature of 240 °C can improve the reflectivity up to record value 14.8%.

The structural specifics of the multilayer coatings define their working temperature range too. Thus, short-period W/B₄C and Co/C structures would remain stable under heating up to 600 °C and 270 °C, respectively. The larger structure period, the higher temperatures of the multilayer degradation. For example, a W/B₄C structure with a period of 2.5 nm will be stable at temperatures below 750 °C.

We would have hoped that the information presented here on short-period multilayer structures has proved to be very useful and even critical for a practical design and fab-

rication of the soft X-ray mirrors operating in the spectral regions of “water window” and “carbon window.”

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