Scandium-silicon Multilayer X-ray Mirrors with CrB_2 Barrier Layers

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(Received 18 July 2018; revised manuscript received 24 October 2018; published online 29 October 2018)

Methods of X-ray reflectometry (λ = 0.154 nm), cross-sectional transmission electron microscopy and reflectometry in the EUV region (λ = 41-51 nm) were used to investigate the barrier properties of CrB_2 layers 0.3-1.3 nm thick in $Sc/CrB_2/Si$ multilayer X-ray mirrors (MXMs) deposited by DC magnetron sputtering. It is shown that barrier layers of ~ 0.3 nm separate Sc and Si layers completely and prevent interacting the Sc and Si layers. Thinner chromium diboride layers interact with the matrix layers forming interlayers containing mostly ScB_2 on the Si-on-Sc interfaces and $CrSi_2$ on the Sc-on-Si ones. Scandium-silicon MXMs with barrier layers on the both interfaces are shown to retain high reflectivity at the wavelength of λ ~ 47 nm.

Keywords: Multilayer X-ray mirror, Interlayer, Barrier layer, Reflectivity, Extreme ultraviolet.

DOI: 10.21272/jnep.10(5).05025

PACS numbers: 61.05.cm, 61.43.Dq, 68.65.Ac, 41.50. + h, 07.85.Fv

1. INTRODUCTION

Silicon based multilayer X-ray mirrors (MXMs) have the highest realized reflectivity in the EUV and soft X-ray regions with respect to their theoretical values: $R_E/R_{Th} > 0.8$ [1-4]. The main multilayer defect that hinders further increase in their efficiency is presence of silicide interlayers at the interfaces formed during the mirror fabrication [5-7]. These interlayers begin growing under external influence (for example, heat or irradiation) resulting in either moderate deterioration [8] or complete degradation [9, 10] of the MXM optical properties.

Thickness of the mixed interlayers in Sc/Si multilayer system reaches ~5 nm and further heating even up to ~ 370 K can result in their growth [11]. Thus retaining or even improving the optical properties is a topical problem for the current system.

One of the ways to stabilize the structure of the multilayer system and reduce the width of the interlayers is to use the barrier layers. In this connection, barrier layers of B_4C [8, 12-14], ScN [8], W [15, 16] were used for the ScSi MXMs. An introduction of barrier layers impedes mixing of matrix layers, but the efficiency of the Sc/Si optical pair decreases because generally the barrier layers have higher absorption in the region of extreme ultraviolet. Therefore, the selection of material for the barrier layers, taking into account the peculiarities of its deposition, as well as controlling the interaction with matrix layers, are important for increasing the efficiency of a multilayer mirror.

In this work we applied CrB_2 barrier layers to the Sc-Si interfaces in order to reduce the interaction of Sc and Si matrix layers and increase the efficiency of the ScSi MXMs in as-deposited state. This material was selected for the following reasons. Firstly, CrB_2 has a sufficiently high melting temperature (~2500 K), in relation to the above-mentioned materials of the barrier layers. That contributes to a decrease in the activity of diffusion processes at the interfaces. Secondly, on the one hand, Cr does not interact with scandium [17], unlike B_4C, and, on the other hand, boron can provide a chemical bond between diboride and scandium. On this basis, thin and continuous barrier layers can be grown at the Si-on-Sc interfaces, which is fundamental for this type of mirrors. We expect to achieve continuity for CrB_2 barrier at earlier stage in comparison to W which grows in island-type mode up to thickness of t_W ~ 0.6 nm [16]. In addition, CrB_2 should be less active in the interaction with silicon than chromium, since borides have already proven themselves as diffusion barriers in silicon technology [18-20]. Previously, we used this material to increase the thermal stability of the Sc/Si MXMs [21]. This work is focused on the study of the growth and the structure of the barrier layers. We will monitor how the thin layers of CrB_2 will interact with Sc and Si, i.e. how they will perform their barrier functions.

2. EXPERIMENTAL

Multilayer mirrors were prepared by direct-current magnetron sputtering. The targets were scandium, silicon and CrB_2 plates ~ 100 mm in diameter with purity of 99.3%, 99.99% and 99.7% respectively. The silicon target was a (111) Si single crystal wafer. During each experiment, the currents at all magnetrons and the argon pressure (2.4 mTorr) were kept constant to ensure a constant rate of deposition. The deposition rates for scandium, silicon and chromium diboride were ~ 0.26 nm/s, ~ 0.37 nm/s and ~ 0.131 nm/s, respectively. Mirrors were deposited onto silicon or super-smooth glass substrates with RMS surface roughness of 0.3-0.5 nm.

The samples were measured at X-ray diffractometer DRON-3M equipped with a primary crystal-monochromator (110) Si to select Cu$K_{\alpha 1}$ radiation (λ = 0.154 nm) only from the X-ray spectrum created by a copper tube. The use of such a scheme ensures the formation of an X-ray beam with a divergence of ~ 0.15° and the intensity of ~ 10^6 cps, which allows recording reflected radiation in a dynamic range of intensities covering 6 orders of magnitude. The phase composition of the samples was estimated at another diffractometer with a graphite analyzer that provided the emission of CuK_α radiation without separation of the K_α-doublet

($\lambda = 0.1542$ nm) with a divergence of ~ 0.5° and the intensity of ~ 10^7 cps.

Cross sections of multilayer samples for investigation in a transmission electron microscope (TEM) were prepared using mechanical and subsequent ion thinning. The investigations were carried out using the «PEM-U» microscope (Ukraine) with a resolution of ~ 0.2 nm.

Measurements in the EUV region were carried out in the 6.3.2 synchrotron ring ALS line of the Berkeley National Laboratory (USA) [22]. The line design provides a spectral width of the incident radiation to ensure $\lambda/\Delta\lambda < 7000$ and a high accuracy of the reflectivity measurements ($\Delta R/R$ ~ 0.2 %).

3. RESULTS AND DISCUSSION

Since chromium and boron can interact with silicon while boron can interact with scandium during the deposition process, we fabricated a series of Sc/CrB_2/Si multilayer samples in which the nominal scandium and silicon thicknesses were constant, and the thickness of the CrB_2 barrier layers was successively changed, and its influence on the MXM period was tracked step-by step. In order to minimize the effect of random deviations of Sc and Si layer thicknesses associated with the fluctuation of their deposition rates in different experiments, we deposited an additional multilayer stack without barriers to each substrate. Then the difference in the periods, Δd, between the multilayer stacks with barriers (d) and without barriers (d_0) will compensate the influence of random deviations of the matrix layers thicknesses in a particular experiment and will characterize only the effect of the barrier layers on the volume changes in the Sc/CrB_2/Si MXMs.

To reduce the number of samples, three different stacks were deposited at each substrate at once: one without barriers and two with barriers of different thicknesses. The thickness range of the applied barriers was 0.3-1.3 nm.

3.1 Investigations in the Hard X-ray Region ($\lambda = 0.154$ nm)

X-ray diffraction patterns were recorded for each sample. An example of an experimental diffractogram for a sample with three multilayer stacks, two of which have barrier layers of ~ 0.3 nm and ~ 0.8 nm, is shown in Fig. 1. As it can be seen from the figure, a rather large number of maxima ($n > 35$) is observed in the diffractogram. The first 3 maxima for all the stacks in the diffractogram are not distinguished. Peaks from different stacks are seen separated beginning from the 4th maximum. We sorted out the peaks into the stacks (indicated by numbers with respect to the stack) and used them to determine the periods of the deposited multilayer stacks.

The features of the interaction of the barrier layer with the Si and Sc layers should be revealed in the Δd dependence on the time (τ-CrB_2) of barrier layer deposition, $\Delta d = f(\tau\text{-}CrB_2)$, since the deposition time is also a measure of thickness. In Fig. 2 we plotted two such dependencies for different interfaces: Si-on-Sc (Fig. 2a) and Sc-on-Si (Fig. 2b). For each interface type two identical samples (A, B series and C, D ones) were prepared in each experiment to retrace a possible fluctuation in the results.

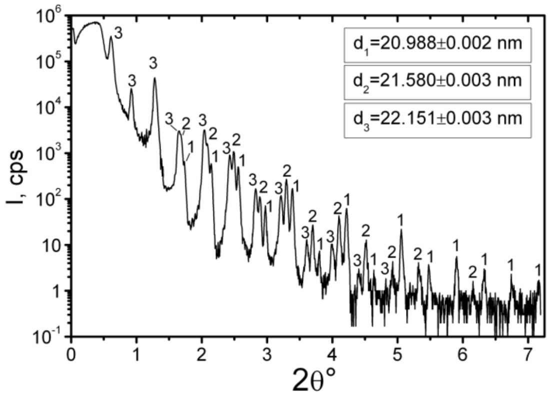

Fig. 1 – Experimental diffraction pattern (θ-2θ scan) of a sample with three Sc/Si multilayer stacks and CrB_2 barrier layers 0 nm, 0.3 nm and 0.8 nm thick

In Fig. 2 we can observe dependencies close to linear for both interfaces. Linearity can be caused by three reasons: 1) the absence of any interaction; 2) there is an interaction, but it is fixed for all barrier layers; 3) the interaction in the Sc/CrB_2/Si multilayer system is still ongoing. The barrier layer deposition rate can be estimated by the slope of these dependences. For the Si-on-Sc interfaces, it is $Pr_A = 0.128 \pm 0.002$ nm/s and $Pr_B = 0.127 \pm 0.003$ nm/s, and for the Sc-on-Si ones, it is $Pr_C = 0.134 \pm 0.003$ nm/s and $Pr_D = 0.133 \pm 0.002$ nm/s. These values are close to the nominal deposition rate of CrB_2 (0.131 nm/s), which may indicate an absences of interaction in the multilayer system or a fixed nature of the interaction.

It was previously found that Sc/Si MXMs have mixed interlayers at both interfaces with the composition of ScSi accompanied by a period shrinkage of 0.64 ± 0.08 nm [23]. The formation of one such interlayer with the same scale of mixing at a particular type of the interface should lead to a period shrinkage of Δd_{ScSi} ~ 0.32 nm. So the dependences of $\Delta d = f(\tau\text{-}CrB_2)$ for both interfaces in Fig. 2 should cross the ordinate axis (i.e. when τ-$CrB_2 = 0$) at the point of Δd ~ 0.32 nm, which indicates the separation of the matrix elements. However, they cross the Y axis at a point below 0.32 nm, which indicates that the barrier layers interact with the matrix materials. This interaction is accompanied by larger volume shrinkage at the Sc-on-Si interfaces (the intersection with the Y axis at ~ 0.14 nm) compared to the Si-on-Sc ones (the intersection with the Y axis at ~ 0.24 nm); shrinkage volume for them is ~ 0.18 and ~ 0.08 nm, respectively, i.e. the difference is ~ 0.1 nm.

We calculated the expected period (d_E) of the Sc/CrB_2/Si MXMs in the absence of any interaction in the multilayer system, as the sum of the nominal thicknesses of the barrier layer (t_{CrB2}), the experimental period value (d_{exp}) and the shrinkage value in the barrier-free sample (Δd_{ScSi}):

$$d_E = t_{CrB2} + d_{exp} + \Delta d_{ScSi}. \quad (1)$$

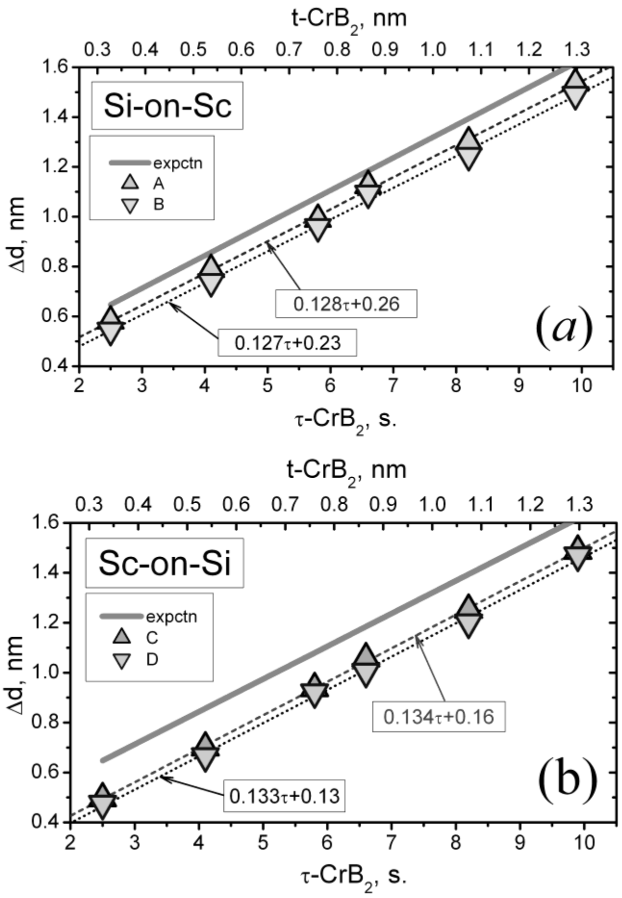

Fig. 2 – Experimental dependence of Δd (the difference of the Sc/Si MXM periods with and without barriers) on the CrB_2 deposition time for the Si-on-Sc (a) and Sc-on-Si (b) interfaces

The calculated value of d_E is plotted in Fig. 2 as wide straight lines. Both graphs show that d_E are slightly higher than the experimental values: by 0.097 ± 0.016 nm for the Si-on-Sc interfaces and by 0.158 ± 0.013 nm for the Sc-on-Si ones. Thus, the version with zero interaction in the mirrors is not viable. It remains only a version of interacting barrier layers with matrix materials, and the difference between d_E and d_{exp} is related precisely to this interaction.

The linearity for the experimental values of Δd in Fig. 2 indicates that barrier layers of $t_{CrB2} < 0.3$ nm interact with the matrix layers. In other words, in the $Sc/CrB_2/Si$ mirrors with barrier layers of $t_{CrB2} \sim 0.3$ nm, pure CrB_2 layers are absent, and the barrier layers are almost completely consist of reaction products.

3.2 Transmission Electron Microscopy

Sc/Si multilayer sample ($d \sim 27$ nm) on a Si-substrate with barrier layers at both interfaces was fabricated for TEM studies. At the top interlayers (Si-on-Sc), the nominal barrier thickness was ~ 0.76 nm, and at the bottom ones (Sc-on-Si) it was ~ 0.33 nm. The cross section of the sample is shown in Fig. 3. In this image, layers of crystalline scandium are visible in the form of wide dark bands and layers of amorphous silicon in the form of lighter broad bands. Sc-grains have a columnar structure and extend through the entire layer from the bottom till the top interfaces. Narrow dark bands are the denser barrier layers of CrB_2 that lie between the Sc and Si layers. They are also amorphous.

In the picture we measured thicknesses of all layers relying on the multilayer period. The period was calibrated according to a diffractogram recorded in X-rays. The ratio of the high-absorbing layers, including the CrB_2 layers, in the period is ~ 0.46, which is close to the optimal value for the effective performance of the MXM in the EUV region. The measured thickness of CrB2 for the bottom interfaces was 0.89 ± 0.08 nm and for the top ones was 1.29 ± 0.06 nm. These thicknesses are larger than the nominal values (~ 0.33 and ~ 0.76 nm respectively). Such discrepancies may be associated, on the one hand, with a slight slope of the sample in the column of the microscope, which should result in broadening of the thin barrier layers. Then the measured values define the maximal thickness of the formed barrier layers. On the other hand, this discrepancy may be due to the interaction of the barrier and the matrix layers. In this case, the measured value will characterize the thickness of the reaction products.

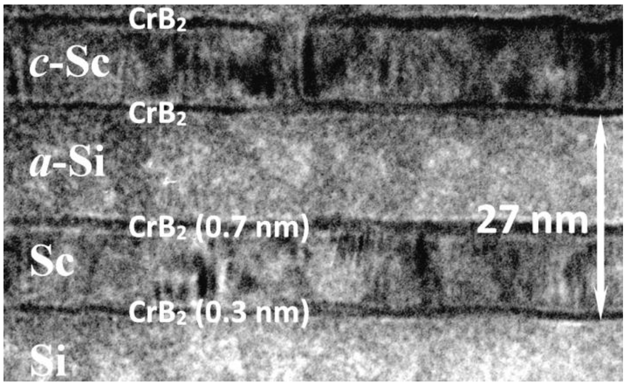

Fig. 3. – Transmission electron microscope image of a Sc/Si MXM with CrB_2 barrier layers of nominal thickness 0.3 nm and 0.7 nm (the substrate is at the bottom)

The linear dependence in $\Delta d(\tau\text{-}CrB_2)$ for the Sc-on-Si interfaces at thicknesses $t_{CrB2} > 0.33$ nm (Fig. 2 b) indicates that the top barriers ($t_{CrB2} \sim 0.76$ nm) react with the matrix layers incompletely. If we assume that a CrB_2 layer of ~ 0.3 nm thick only reacts at the top interfaces, then the top barriers should consist of two sublayers, with the reaction products accounting for $1.29 - (0.76\text{-}0.33) = 1.29 - 0.43 \approx 0.86 \pm 0.06$ nm.

3.3 Estimation of the Barrier Layer Composition in the Sc/Si MXMs

To estimate the composition of the barrier layers of the $Sc/CrB_2/Si$ MXMs due to the interaction of CrB_2 with the matrix layers, we calculated the volumes of the initial components and final products for various reactions that can occur with the participation of existing types of atoms, and also determined some useful ratios. A total of 24 reactions were considered. We assumed that at a minimum barrier thickness of ~ 0.33 nm, CrB_2 interacts with the MXM material completely. The results of the study in the hard X-ray region (subsection 3.1) showed that the deposition of the barrier layers is accompanied by period shrinkage, therefore, the reactions that occur with increased volume were omitted from the consideration. The results of calculations are presented in the table. We sorted the reactions by the interface type for the convenience of analysis: the first four reactions refer to the bottom interfaces (Sc-on-Si), when CrB_2 is deposited on Si; the 5-th reaction refers to the Si-on-Sc interfaces, when CrB_2 is deposited on Sc; and the remaining reactions refer to the case where the barrier layer reacts with both matrix elements. The molar volume of boride, V_B, is an analog to the barrier layer thickness, t_{CrB2}, and volumetric shrinkage, ΔV, in each reaction corresponds to shrinkage of the period, Δd.

We calculated the ratios of $\Delta V/V_B$ for each reaction, and they are presented in the fourth column of the table. As can be seen, these ratios are individual for each reaction. The experimental ratios $\Delta d/t_{CrB2}$, as follows from subsection 3.1, for the first experimental point are $0.085/0.33 \approx 0.26$ for the Si-on-Sc interfaces and $0.162/0.33 \approx 0.49$ for the Sc-on-Si ones. Comparing these data with the calculations in the table and taking into account the error in the determination we can distinguish the following reactions: No. 1-4, for which $\Delta V/V_B$ is close to 0.49; and reactions No. 5 and 10, for which $\Delta V/V_B$ is close to 0.26.

From the TEM data we obtained that the ratio of the apparent thickness of the barrier layer to the nominal at the top interfaces (Si-on-Sc) is $0.86/0.33 \sim (2.6 \pm 0.2)$. Similar calculated data are presented in the penultimate column of the table, as the ratio of the reaction products volume (V_F) to the volume of CrB_2. Here the reactions closest to the experimental values are No. 6-9. In all these reactions, chromium silicides and scandium borides (ScB_{12} or ScB_2) are present. The $\Delta d/t_{CrB2}$ ratios for the Sc-on-Si interfaces have close values (2.7 ± 0.2), and reactions No. 1-4 are also suitable here.

A small difference between the experimental and calculated values may be due to some difference between the real densities and tabular ones, incompleteness of reaction or non-stoichiometry of the compounds obtained. Thus, we can make a preliminary conclusion that the structure of the top barrier layers can include scandium borides and chromium silicides, and the bottom ones include $CrSi_2$ and silicon borides.

These data are supplemented by calculations of the thermal effects, which are given in the last column of the table. The enthalpies of formation in all reactions are reduced to one mole of CrB_2. It can be seen from these calculations that reaction No. 4 is unlikely, since it occurs with the heat absorption. Although a sufficient amount of energy is supplied when sputtered atoms arrives onto the substrate, yet other exothermic reactions have an advantage. Here, from the thermodynamic point of view, the most probable reactions are No. 2, 5, 7-9.

X-ray measurements show that for Sc-on-Si interfaces preferred reactions are No. 1-4, and for Si-on-Sc ones these are No. 5 and 10. TEM data confirms the results of X-ray studies for Sc-on-Si interfaces, but give an alternative manifested in reactions No. 6-9 for Si-on-Sc interfaces. Calculations of thermal effects with the exception of reaction No. 4 support both versions for Si-on-Sc interfaces. Since TEM studies, as indicated above, are more qualitative by nature, we tend to rely on the results of X-ray studies.

From the data obtained in this subsection, it can be concluded that at the Sc-on-Si interfaces the main reaction product is $CrSi_2$ and one of the silicon borides (B_4Si or B_6Si). It is primarily ScB_2 and Cr for Si-on-Sc interfaces; although ScB_{12} and CrSi may also appear here according to our estimates, but this is unlikely from the thermodynamic point of view.

Table 1 – The results of the calculation for chemical reactions expected at interfaces in the ScSi MXMs with CrB_2 barrier layers: ΔV are volume changes, V_B is the molar volume of CrB_2, V_F is the volume of final products, ΔH_{298} is the enthalpy of the chemical reaction per mole of CrB_2. (r) and (c) refer to the rhombic and cubic modification of B_6Si, respectively

№	Reaction	Molar volumes, cm^3	$\Delta V/V_B$	V_F/V_B	ΔH_{298} kJ/moll/K
1	$3CrB_2 + 7Si = 3CrSi_2 + B_6Si$(r)	42.3+84.4→65.2+39.1	– 0.53	2.47	– 12.6
2	$2CrB_2 + 5Si = 2CrSi_2 + B_4Si$	28.2+60.3→43.5+29.6	– 0.55	2.59	-17.3
3	$3CrB_2 + 7Si = 3CrSi_2 + B_6Si$(c)	42.3+84.4→65.2+43.2	– 0.43	2.56	– 12.6
4	$CrB_2 + 2Si = CrSi_2 + 2B$	14.1+24.1→21.7+9.2	– 0.52	2.19	+28.1
5	$CrB_2 + Sc = ScB_2 + Cr$	14.1+15→18.2+7.2	– 0.26	1.80	– 178
6	$6CrB_2 + 12Si + Sc = 6CrSi_2 + ScB_{12}$	84.6+144.6+15→109.8+61.6	– 0.86	2.03	– 52.0
7	$CrB_2 + 2Si + Sc = CrSi_2 + ScB_2$	14.1+24.1+15→21.7+18.2	– 0.94	2.83	– 279
8	$CrB_2 + Si + Sc = CrSi + ScB_2$	14.1+12.1+15→14.9+18.2	– 0.57	2.35	– 250
9	$5CrB_2 + 3Si + 5Sc = Cr_5Si_3 + 5ScB_2$	70.5+36.2+75.2→60.3+90.9	– 0.44	2.14	– 238
10	$6CrB_2 + 6Si + Sc = 6CrSi + ScB_{12}$	84.6+72.3+15→89.64+61.58	– 0.24	1.79	– 22.7

3.4 Measurements in the Soft X-ray Area

We measured the reflectivity of the $CrB_2/Sc/CrB_2/Si$ MXM with parameters close to the sample described in subsection 3.2, at near normal angle of incidence ($\theta = 85°$) in the wavelength range of 41-51 nm. We used measurements for Sc/Si mirrors with mixed zones of ~ 1.5 nm and ~ 5 nm thickness as a reference. The results are shown in Fig. 4. The peak reflection coefficient for MXM with barriers is 0.443, which is slightly inferior to the sample with interlayers of 1.5 nm (0.447) and one and a half times higher compared to a mirror having 5-nm interlayers (0.279). The main purpose of the barriers application is to increase the stability of the multilayer system, and as a rule, this is achieved due to deterioration of properties, i.e. at the expense of a decrease in reflectivity. We see that with the insertion of the CrB_2 barriers, the loss in the reflectivity is less than 1%, which is a very encouraging result.

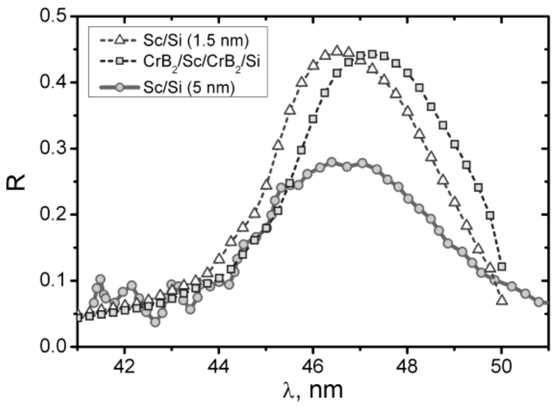

Fig. 4. – The reflectivity of the Sc/Si mirrors with the interlayers of t_{ScSi}~1.5 nm (triangles), $CrB_2/Sc/CrB_2/Si$ (squares) and the Sc/Si mirrors with the interlayers of t_{ScSi} ~ 5 nm (circles) in the 41-51 nm wavelengths range at near normal incidence ($\theta = 85°$)

It was earlier shown that the Sc/Si MXMs with barrier layers are more stable if compared to mirrors without barriers [21]. An increase in thermal stability of ~ 100° has been obtained on multilayer samples with the same barrier layers. Considering the fact that the optical characteristics of the MXM deteriorate insignificantly, it can be concluded that chromium diboride is promising material as the barrier layer.

4. CONCLUSIONS

X-ray reflectometry (λ = 0.154 nm) and cross-section transmission electron microscopy were used to study the effect of CrB_2 barrier layers 0.3-1.3 nm thick on the interface interaction in the Sc/Si multilayer X-ray mirrors. At thicknesses of t-CrB_2 < 0.3 nm there is no interaction between the matrix layers. Multilayer shrinkage of the period is observed due to the interaction of the barrier layers with the matrix material, which is ~0.08 nm for the Si-on-Sc interfaces and ~ 0.18 nm for the Sc-on-Si ones. The interaction is accompanied by the barrier layers broadening by ~ 0.6 nm in average. The reaction products at different interfaces were evaluated. For the Si-on-Sc interfaces the reaction products mainly consist of ScB_2 and Cr, and for Sc-on-Si interfaces they are $CrSi_2$ and silicon borides (B_4Si and/or B_6Si).

Measurements in the EUV (λ = 41-51 nm) showed that the reflectivity for the MXM with CrB_2-barriers is inferior to the Sc/Si MXM with 1.5 nm interlayers by only 1% and is ~ 0.443 at a wavelength of ~ 47 nm. This is a record result for a mirror with barrier layers on both interfaces at the given wavelength.

These studies have shown that chromium diboride is a promising material as a barrier for Sc/Si multilayer mirrors.

ACKNOWLEDGEMENT

YPP is acknowledged to ISKCON for the help in understanding the place and the significance of this study.

Багатошарові рентгенівські дзеркала Sc / Si з бар'єрними шарами CrB_2

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Методами рентгенівської дифракції (λ = 0,154 нм), просвічувачої електронної мікроскопії поперечних зрізів і рефлектометрії в області екстремального ультрафіолету (λ = 41-51 нм) досліджені бар'єрні властивості шарів CrB_2 товщиною 0.3-1.3 нм в багатошарових рентгенівських дзеркалах (БРД) $Sc/CrB_2/Si$, виготовлених методом прямоточного магнетронного розпилення. Показано, що бар'єрні шари товщиною ~ 0,3 нм повністю розділяють шари Sc і Si і перешкоджають утворенню перемішаних зон ScSi. Більш тонкі шари діборида хрому взаємодіють з матричними шарами, формуючи шари з переважним вмістом ScB_2 на кордонах Si-on-Sc і $CrSi_2$ на кордонах Sc-on-Si. Показано, що БРД Sc/Si з бар'єрами на обох кордонах зберігають високу відбивну здатність на довжині хвилі λ ~ 47 нм.

Ключові слова: Багатошарове рентгенівське дзеркало, Перемішані зони, Бар'єрні шари, Відбивна здатність, Екстремальний ультрафіолет.

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