Structure, thermal stability and reflectivity of Sc/Si and Sc/W/Si/W multilayer X-ray mirrors.

A.V. Vinogradov[a], Yu.P. Pershin[b], E.N. Zubarev[b],
D.L. Voronov[b], A.V. Pen'kov[b], V.V. Kondratenko[b],
Yu.A. Uspenskii[a], I.A. Artioukov[a], and J.F. Seely[c]

[a] Lebedev Physical Institute, Leninski prosp. 53, Moscow 119991, Russia
[b]Kharkov State Polytechnic University, Frunze str., Kharkiv 310002, Ukraine
[c] Naval Research Laboratory, Washingthon DC 20375, USA

ABSTRACT

Processes going on at elevated temperatures between Sc and Si layers in Sc/Si coatings are studied by X-ray scattering and cross-sectional transmission electron microscopy. It is shown that the W layers of 0.5-0.8 nm placed at Sc-Si interfaces form effective barriers preventing the penetration of Si into Sc. The effects of Si-Sc diffusion and W-barriers on the reflectivity of coatings are calculated in good agreement with experimental results. Presented measurements show that the Sc/W/Si/W multilayers with the period of 20.5 nm fabricated by dc-magnetron sputtering possess thermal stability up to 250 C and the normal incidence reflectivity of 24% at wavelengths about 40 nm.

Keywords: EUV optics, multilayer, thermal stability

1. INTRODUCTION

The high reflectivity of Sc/Si multilayers in an EUV interval λ= 35-50 nm[1,2] made them very promising for many applications. These multilayers have been successfully employed in normal incidence optics for the table-top capillary discharge soft X-ray laser and in several important experiments.[3–5] In addition to high reflectivity, some applications, for example space EUV studies of the Sun and stellar objects, require thermal stability of coatings at elevated temperatures T$\geq$100 C. The last requirement is difficult for Sc/Si mirrors, since the interaction of Sc and Si components increases with temperature and causes the degradation of multilayer parameters.[6]

To improve the thermal stability of multilayer mirrors, two strategies are used. The first, which is applicable to short-period structures, places special emphasis on selection of components being in phase equilibrium one with another.[7] For this aim pure components are frequently replaced by alloys or compounds. The second utilizes anti-diffusion barriers between layers to prevent the dissolving and chemical reactions of components. This strategy, which looks preferable for long-period multilayers, is used in the present work to stop the penetration of Si into Sc layers. Fabricated Sc/W/Si/W multilayers are tested at elevated temperatures and their thermal stability is compared with that of Sc/Si multilayers. Obtained results show processes going on between structure components, their dependence on layer thicknesses and temperature. The material science analysis is added by the calculations and experimental studies of reflectivity in the Sc-based multilayers.

2. FABRICATION AND TESTING OF MULTILAYERS

A number of Sc/Si and Sc/W/Si/W multilayers with a period H=20-35 nm was prepared by dc-magnetron sputtering at $3 \cdot 10^{-3}$ Torr of Ar. All multilayers were designed as having 20 periods, the equal thicknesses of Sc and Si and capped by the Si layer of 5 nm. The thickness of W barriers was varied from 0 nm to 0.8 nm.

The period and structure of fabricated multilayers were controlled by CuK_α (λ=0.154 nm) X-ray scattering at small and large angles, as well as by cross-sectional transmission electron microscopy (TEM). To test the thermal stability, the annealing of varied duration at 210 C and 420 C in vacuum of 10^{-5} Torr was used. The reflectivity of coatings was measured at the incidence angle 5^o in the interval 25-50 nm at Brookhaven synchrotron.

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3. PROCESSES IN SC/SI MULTILAYERS AT ELEVATED TEMPERATURES

Earlier studies[1,2,6] have shown that the mixed amorphous regions of 3 nm are formed at interfaces between polycrystalline Sc and amorphous Si in the process of Sc/Si multilayer deposition (Fig. 1ab). The composition of these regions is close to ScSi.[8] In contrast to Mo/Si multilayers,[9] no significant asymmetry between Sc/Si and Si/Sc interfaces was observed. The TEM studies of Sc/Si coatings showed that the interaction between Sc and Si layers increases with temperature. This increasing manifests itself as the growth of mixed amorphous regions at the cost of the thinning of pure Sc and Si layers, while the composition of the regions holds close to ScSi. After the 41-hour annealing at 210 C, all scandium in the H=34 nm multilayer was consumed for the Sc-Si amorphous region formation (Fig. 1ab). At this point the process of mixing slows down, but does not stop. To approach to the Sc_3Si_5 composition, the mixed region takes more and more Si. The annealing at 420 C forms the Sc_3Si_5 compound and is accompanied by a significant decreasing of the period (Fig. 2).

4. STRUCTURE AND THERMAL RESISTANCE OF SC/W/SI/W MULTILAYERS

High melting temperature makes W capable to form ultrathin continuous layers. Tungsten does not practically interact with Sc. W/Si multilayers fabricated by dc-magnetron sputtering have the dense symmerical regions of the WSi_2 composition with the thickness of 0.9-1.4 nm.[10] These facts defined our choise of W as antidiffusion barrier between Sc and Si. The TEM images of Sc/W/Si/W multilayers with H(W)=0.8 nm showed no interaction between Sc and Si layers as is seen in Fig. 1cd. The observed width of W-containing layers is 1.5-1.8 nm, that is twice of the W thickness. The barriers contains about 0.4 nm of pure tungsten and $\sim$1.1 nm of WSi_2. This follows from the total thickness of the barrier and the specific volume of WSi_2, which is 2.6 times larger (per one W atom) than of pure tungsten. The direct identification of W and WSi_2 layers in TEM images is difficult, because of high electron density of both materials.[10]

Our experiments showed that the neighbouring layers of Sc and Si are not practically mixed when H(W)$\geq$0.5-0.6 nm, that is, the barrier is of 1.2-1.3 nm or larger. A smaller thickness of W results in the arising of mixed regions between Sc and W, which have the width of 1.2-1.4 nm for H(W)=0.24 nm and of 2.0-2.6 nm for H(W)=0.14 nm. The mixed regions, which are clearly seen in Fig. 1cd, are wider at the Si/W/Sc barrier (Si over W and Sc) than at the Sc/W/Si. Different efficiency of these barriers is a result of dissimilar growth of W on Sc and Si: the lack of interaction between W and Sc leads to the island-type growth of W on Sc, while thin films of W on Si are more continuous. The asymmetry in the region width is 12% for H(W)=0.24 nm and achieves 20% for H(W)=0.14 nm.

The deposition of W barriers with H(W)$\geq$0.5-0.8 nm significantly improves the thermal resistance of Sc/Si coatings. Small angle X-ray diffraction evidenced that the 700-hours annealing at 150 C does not change the period and structure of the Sc/W/Si/W multilayer with H=23 nm (Fig. 3). At the same time, the period of the Sc/Si multilayer essentially decreases even under shorter annealing at this temperature. The one-hour annealing at varied temperature showed that the Sc/W/Si/W coatings with H(W)=0.8 nm remain thermal resistant up to 250 C (Fig. 2). At higher temperatures the W barrier of 0.8 nm occurs no longer effective to prevent the interaction of Sc and Si.

5. REFLECTIVITY OF SC/SI AND SC/W/SI/W MIRRORS

Our earlier studies[1,2] have shown that the reflectivity of Sc/Si coatings fastly decreases with the growth of interaction between Sc and Si layers. To better understand this effect, we calculated the normal incidence reflectivity (R) of the Sc/Si multilayer mirror with H= 20.5 nm as a function of the mixed region width (l_{mix}). In this calculation, the spatial distribution of Sc and Si atoms in the mixed region was modelled with the diffusion equation, the dielectric function of Si was taken from the handbook[11] and that of Sc from our recent measurement.[12] Results presented in Fig. 4 show that even the reflectivity of as-deposited Sc/Si mirrors (l_{mix}=3 nm) is significantly lowered by the Sc-Si mixing in comparison with the case of ideally sharp interfaces. The growth of l_{mix} to 5 nm, which approximately corresponds to the 3-hour annealing at 210 C, reduces R from 24% to 13%. Similar calculation with l_{mix}=7 nm evidences that the 8-hour annealing at 210 C completely destroys the mirror ($R \approx$5%).

The deposition of W barriers affects on the reflectivity of Sc/Si multilayers by two ways: removing the Sc-Si mixed regions and providing more EUV absorption, as W has high absorption at the wavelengths of interest. These factors significantly balance each other. For example, the calculated reflectivity of the Sc/W/Si/W multilayer mirror with H=20.5 nm and H(W)=0.55 nm is approximately equal to that of the as-deposited Sc/Si mirror with the same period.

Our measurements of R excellently agree with the calculations (Fig. 5) and evidence that the Sc/Si multilayers with W barriers allow to obtain both the good thermal stability and rather high performance of reflectors.

6. SUMMARY

Our studies of Sc/Si multilayers revealed processes going on between Sc and Si components at elevated temperatures. Tungsten placed at Sc-Si interfaces forms effective barriers, which stop the penetration of Si into Sc. By this way it is possible to obtain the good thermal stability of layers and receive multilayer mirrors with rather high reflectivity.

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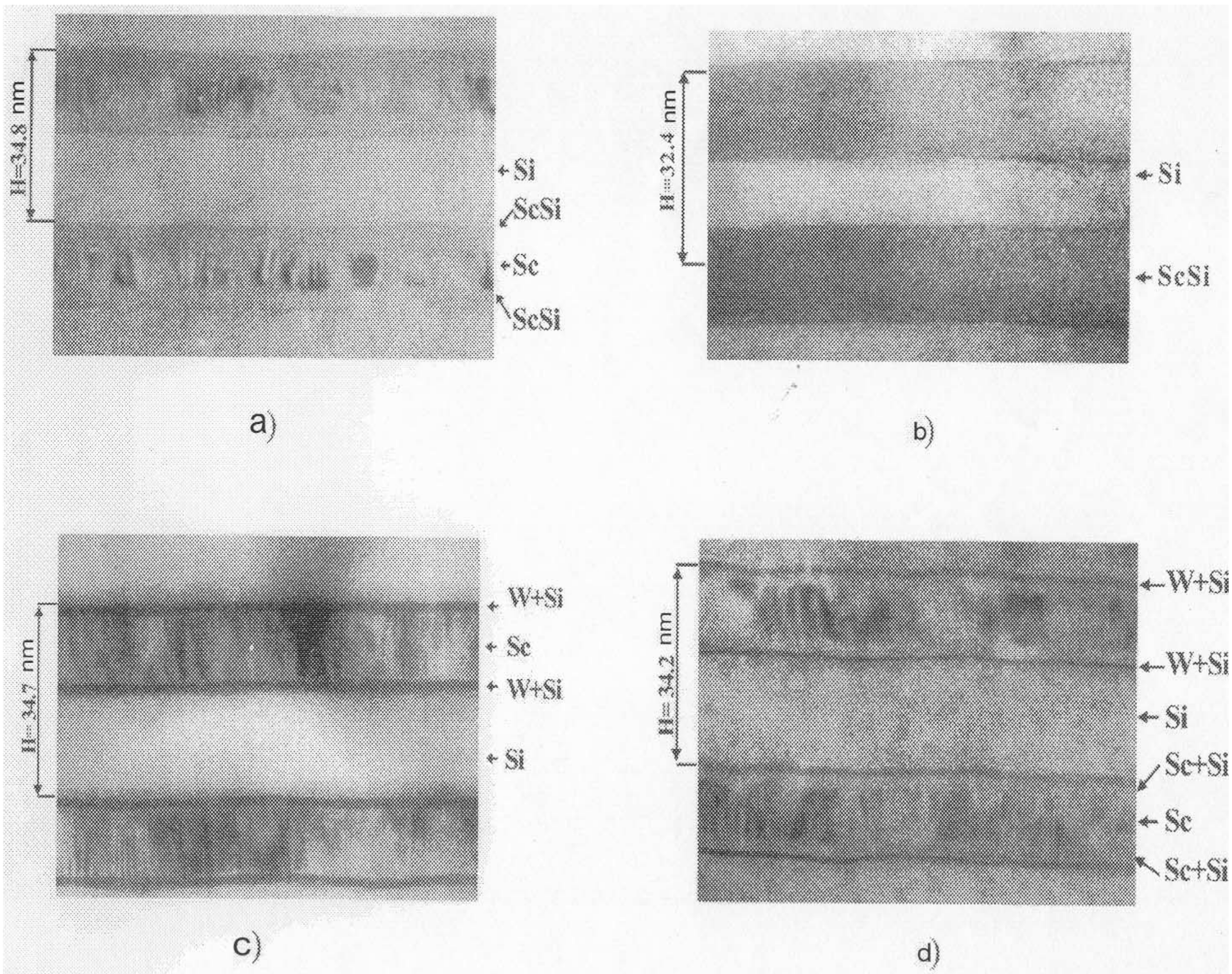

Figure 1. TEM images of Sc-based multilayers: (a) as-deposited Sc/Si, (b) Sc/Si after 41-hour annealing at 210 ^{o}C, (c) Sc/W/Si/W with H(W)=0.8 nm, (d) Sc/W/Si/W with H(W)=0.14 nm.

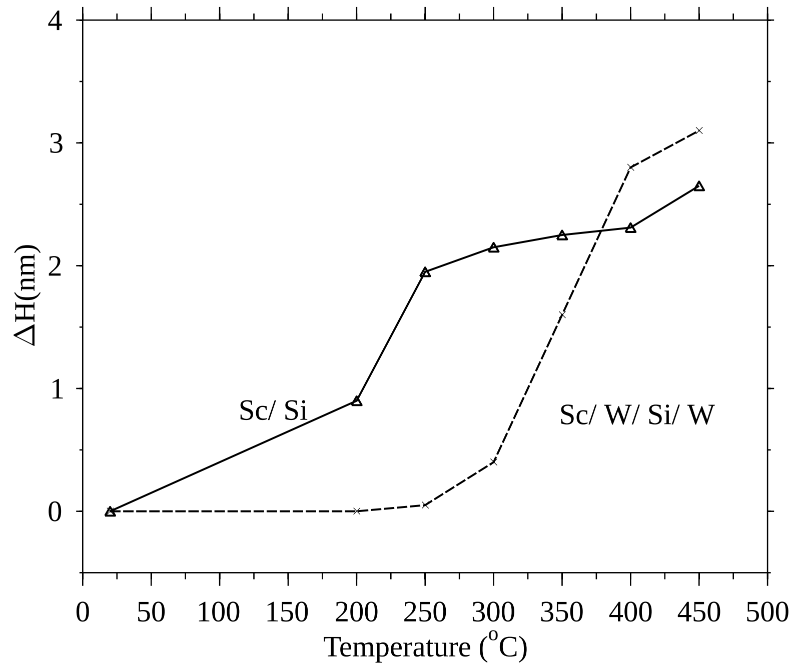

Figure 2. Changing of the period of Sc/Si and Sc/W/Si/W multilayers (H(W)=0.8 nm) with the temperature of one-hour annealing.

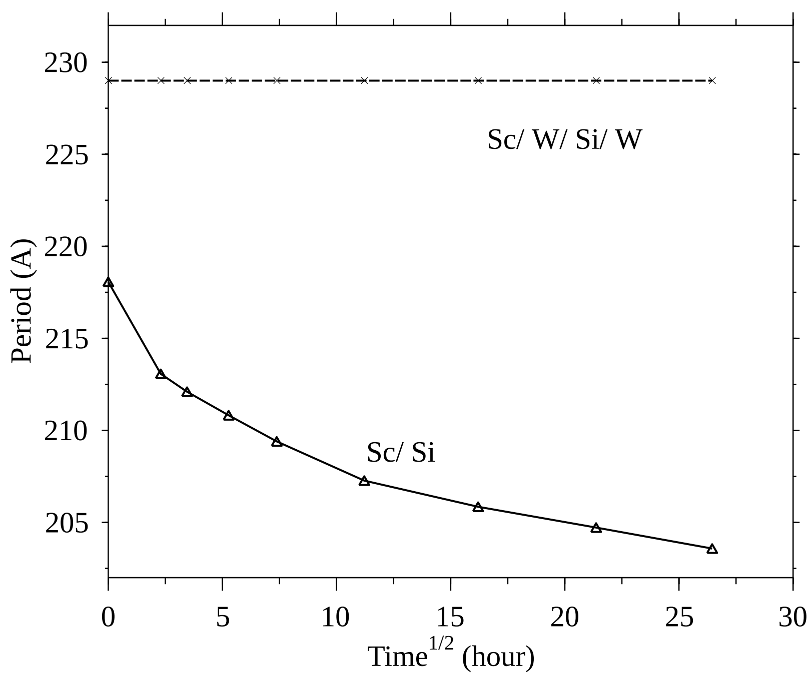

Figure 3. Changing of the period of Sc/Si and Sc/W/Si/W multilayers (H(W)=0.8 nm) with time at the temperature 150 C.

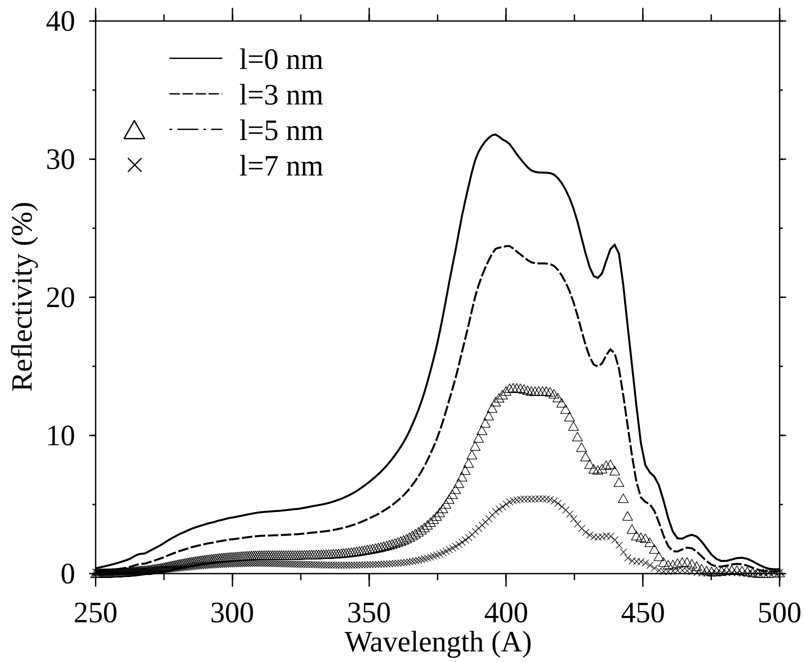

Figure 4. Calculated normal incidence reflectivity of Sc/Si coatings for a varied width of the Sc-Si mixed region (l_{mix}).

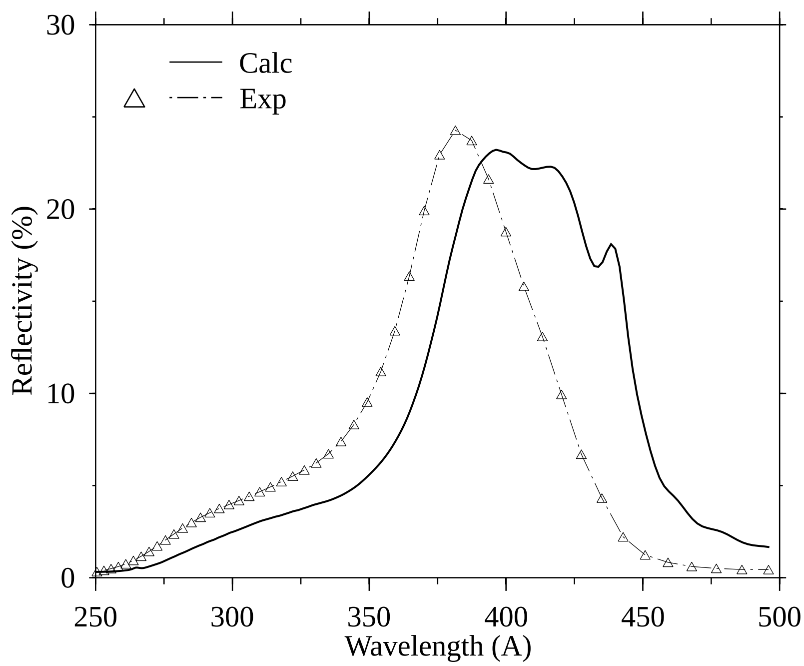

Figure 5. Calculated (line) and measured (symbols) reflectivity of the Sc/W/Si/W coating with H=20.5 nm and H(W)=0.55 nm.