$Mo_{0.5}Si_{0.5}$/Si multilayer soft x-ray mirrors, high thermal stability, and normal incidence reflectivity

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Multilayer soft x-ray mirrors with an absorber consisting of the mixture $Mo_{0.5}Si_{0.5}$ have been fabricated by electron-beam evaporation in UHV. This has been done to get soft x-ray normal incidence mirrors for 80–100 eV photon energy with enhanced thermal stability and still high reflectivity. The thermal stability is studied by baking them at temperatures between 600 and 950 °C. The results were compared with multilayers of pure Mo and Si, which were also fabricated by electron-beam evaporation. After each baking step the x-ray mirrors are characterized by small angle $Cu_{K\alpha}$ x-ray diffraction. The reflectivity of the first-order Bragg peak is nearly constant up to 20 min baking at 900 °C. Further we present the normal incidence soft x-ray reflectivity for wavelengths between 12 and 18 nm of a $Mo_{0.5}Si_{0.5}$/Si mirror with 12 double layers ($N=12$) and of a $Mo_{0.5}Si_{0.5}$/Si mirror as deposited with 33 double layers ($N=33$). With the latter a reflectivity of 46% is achieved.

For many applications the thermal stability of a multilayer stack is an important property. Optics at synchrotron radiation beamlines might, for example, be heated to a few hundred °C.[1] In the wavelength range between 13 and 30 nm the material combination of Mo and Si is most widely used for normal incidence multilayer mirrors. Reflectivities around 60% have been achieved,[2–8] but mirrors of this material combination are destroyed after baking at temperatures ~500 °C.[9–15] The studies in Refs. 11 and 12 gave hints that the reason for the breakdown of the reflectivity is the consumption of the silicon spacer due to interdiffusion of the Si and Mo layers and formation of Mo silicides. To slow down or stop this process Si is mixed into the absorber layers. This intermixing process results in an increase of the Si concentration in the absorber, decreasing the Mo concentration, and thus degrading the reflectivity. For this reason the absorber mixture $Mo_{0.5}Si_{0.5}$ was selected as a compromise between high thermal stability and reflectivity.

The multilayers are fabricated in UHV with two e^--beam evaporation sources, one for Mo and one for Si. The distance between the substrates and the evaporation sources is 65 cm. The base pressure of the deposition chamber is $\sim 1\times10^{-8}$ Pa and typical pressures during deposition are about 1×10^{-6} Pa. The deposition system possesses two quartz oscillators for measurements of deposition rate and layer thickness.

The absorber layers are prepared by simultaneous evaporation of both materials. The mixture is adjusted by rate measurements with the quartz oscillators. The layer thickness is controlled by an *in situ* reflectivity measurement[16,17] with C_K radiation ($\lambda=4.47$ nm) at an angle of $\alpha=70°$, with respect to the surface normal of the substrate. The double layer thickness (d spacing) is determined by the wavelength and angle of incidence of the used radiation. In order to smooth the interfaces the mirrors are thermally treated during deposition[2] at temperatures of about 175 °C.

The same mirror is baked to temperatures between 250 and 950 °C in steps of 50 °C. The baking is done under vacuum (5×10^{-3} Pa). The mirror is baked for 20 min at each temperature. The time for warming up to the nominal temperature is close to 10 min. The mirror needs around 1 h to cool down to room temperature. These times are in addition to the 20 min baking time at the nominal temperatures. The baking temperature is measured at the substrate holder with a NiCr/CuNi thermocouple. After each baking step the samples are characterized by means of small angle x-ray diffraction with a $Cu_{K\alpha}$ line source ($\lambda=0.154$ nm). Further details about the fabrication and characterization procedures are given in Ref. 18. The normal incidence reflectivity for soft x-rays is determined in the spectral region of 12–18 nm using a reflectometer at the electron storage ring BESSY.[19]

The multilayers used in this study had a d spacing of ~7 nm. They had nominal thicknesses of about 2.5 nm for the absorber layers and 4.5 nm for the spacer layers. The reflectivity of the first-order Bragg peak measured with the $Cu_{K\alpha}$ line source versus the baking temperature for a $Mo_{0.5}Si_{0.5}$/Si and a Mo/Si sample with 12 double layers is given in Fig. 1(a). The reflectivity for the $Mo_{0.5}Si_{0.5}$/Si mirror decreases from 64% for the sample as deposited to 56% for the same sample after a baking at 600 °C. In the baking temperature range between 600 and 850 °C the reflectivity remains almost constant. After baking at 900 °C the reflectivity decreases to 41%. Baking at 950 °C for 20 min destroys the multilayer completely: A first-order Bragg peaks does not occur. For the Mo/Si multilayer the reflectivity is nearly constant to about 70% up to a baking temperature of 550 °C. After baking at 600 °C the reflectivity decreases to 5%. At a baking temperature of 800 °C

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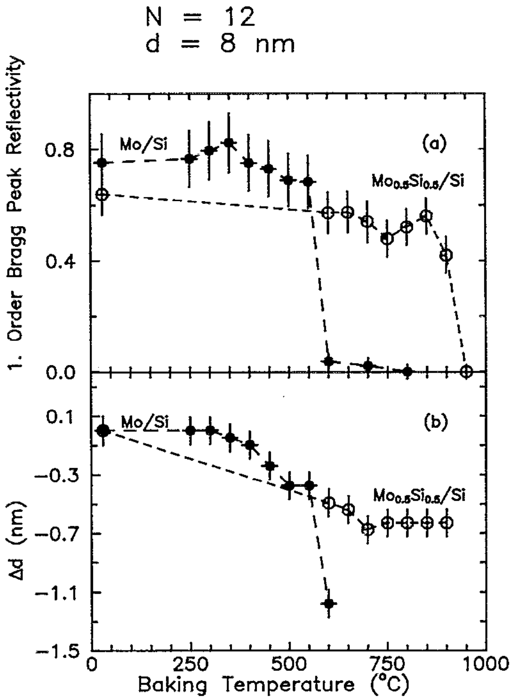

FIG. 1. Reflectivity of the first-order Bragg peak vs baking temperature for grazing incidence with a $Cu_{K\alpha}$ line (a) and the changing of the period thickness Δd vs baking temperature (b).

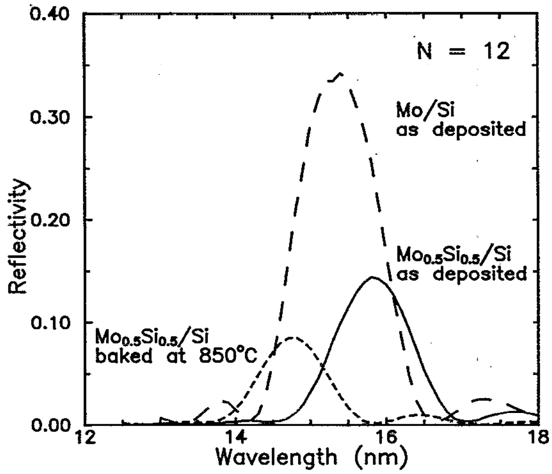

FIG. 2. Normal incidence reflectivity curves for the $Mo_{0.5}Si_{0.5}/Si$ mirror as deposited with 12 double layers (solid) and after baking at 850 °C (dashed) and for a Mo/Si mirror as deposited with 12 double layers. After baking at 850 °C no reflectivity is detectable.

the multilayer no longer shows a layer structure. Figure 1(b) displays the change of the period thicknesses Δd versus the baking temperature for both samples. They are deduced from the angular positions of the third-order Bragg maxima using a program that corrects for the refraction index of the multilayers. The period thickness of the Mo/Si mirror decreases above a baking temperature of 300 °C. At a temperature of 600 °C the change is 1.2 nm and at higher temperatures a third Bragg order peak is no longer detectable. The thickness of the $Mo_{0.5}Si_{0.5}/Si$ mirror decreases about 0.7 nm up to a baking temperature of 700 °C. At higher temperatures the period thicknesses keeps constant up to 900 °C.

The multilayers are designed as normal incidence mirrors for the wavelength range between 13 and 16 nm. Therefore we have also studied the reflectivity behavior of a $Mo_{0.5}Si_{0.5}/Si$ and a Mo/Si mirror with 12 double layers for the mirror as deposited and for the mirror which is baked at 850 °C for 20 min. The result is shown in Fig. 2. The $Mo_{0.5}Si_{0.5}/Si$ mirror as deposited has a reflectivity of 15% at a wavelength of 15.8 nm. After a 20 min baking at 850 °C, the reflectivity of the mirror decreases to 9%. The maximum of the reflectivity has shifted about 1 nm to 14.8 nm. The Mo/Si mirror as deposited has a higher reflectivity of about 33%, but after a 20 min baking at 850 °C the mirror has no detectable reflectivity.

The results of the measurements show that mixing of Si into the absorber layer enhances the thermal stability of Mo/Si multilayers: First, one obtains reflectivities around 10% for the $Mo_{0.5}Si_{0.5}/Si$ multilayers at baking temperatures up to 850 °C, while Mo/Si multilayers are already completely destroyed at 600 °C. Second, we observe no change in the *d* spacing for baking temperatures between 700 and 850 °C which is also very important for applications. These results are comparable with the work of Kondratenko *et al.*[9] who studied the thermal stability of $MoSi_2/Si$ multilayers, which were fabricated by sputtering, but did not report absolute reflectivity values.

In order to get higher reflectivity for this system we have raised the number of double layers. A reflectivity of 46% at a wavelength of 14.2 nm is achieved for a $Mo_{0.5}Si_{0.5}/Si$ mirror as deposited with 33 double layers (Fig. 3). With a Mo/Si mirror with 30 double layers, we achieve a reflectivity of 58% (Fig. 3).

Furthermore, we have measured the normal incidence reflectivity vs baking temperature of this mirror [Fig. 4(a)] and the wavelength of the peak maxima vs baking temperature [Fig. 4(b)]. The reflectivity decreases from 46% at 14.25 nm for the sample as deposited to 38% at 14.1 nm

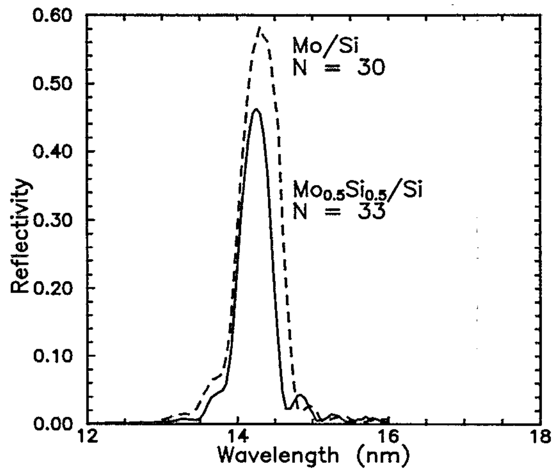

FIG. 3. Normal incidence reflectivity curve for a $Mo_{0.5}Si_{0.5}/Si$ mirror as deposited with 33 double layers and Mo/Si mirror with 30 double layers.

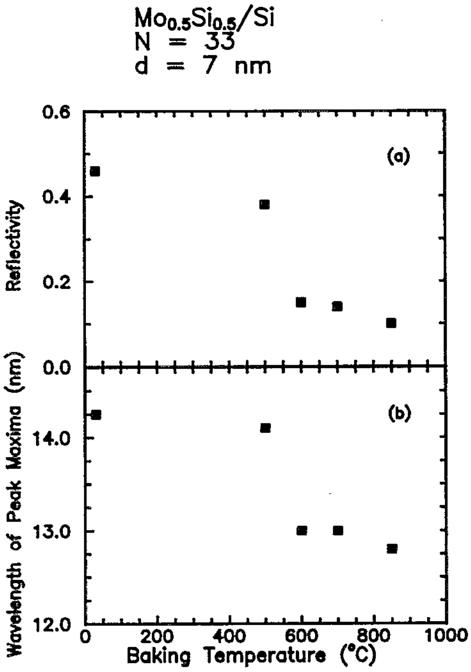

FIG. 4. Normal incidence reflectivity (a) and wavelength of peak maxima (b) vs baking temperature for the $Mo_{0.5}Si_{0.5}/Si$ mirror with 33 double layers.

after 20 min baking at 500 °C. After baking at 600 °C the reflectivity is decreased to 15% at 13 nm and remains almost constant up to 850 °C. Large angle x-ray scattering studies of the samples reveal the development of $MoSi_2$ crystallites at temperatures of 600 °C and higher. This is certainly at least partly responsible for the abrupt changes in reflectivity and *d* spacing between 500 and 600 °C. The fact that these changes are larger than for the sample with $N=12$ (Figs. 1 and 2) is probably due to slightly larger Mo content in the $N=33$ sample.

In summary, we have fabricated $Mo_{0.5}Si_{0.5}/Si$ multilayer soft x-ray mirrors by electron beam evaporation in UHV. The thermal stability of these mirrors was studied. After a 20 min baking at 600 °C the reflectivity of the mirrors decreases to about 15%. Mo/Si multilayers are completely destroyed at 600 °C. Further baking at temperatures between 600 and 850 °C hardly alters the reflectivity and *d* spacing of these x-ray mirrors, which makes them attractive for applications in high temperature surroundings.

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