Fabrication and characterization of Si-based soft x-ray mirrors

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ABSTRACT

Silicon-metal multilayers, which are designed as soft x-ray mirrors, have been fabricated with e^--beam evaporation in Bielefeld. Quartz oscillators and in situ soft x-ray reflection with C-k radiation were applied to control the fabrication process. The Mo/Si and Ta/Si layer systems were fabricated for wavelengths between 12 and 30 nm and normal radiation incidence. They were studied with synchrotron radiation soft x-ray reflection in the PTB laboratory at BESSY in Berlin and with Cu-K_α reflection as well as the surface analytical methods Rutherford-Backscattering (RBS) and Sputtering in combination with Auger electron spectroscopy (Sputter/AES) in Bielefeld. The optical data, C-k in situ reflectivity, Cu-k_α and synchrotron radiation reflection, are compared with calculations on the basis of the Fresnel equations . Tests were made with RBS and Sputter/AES to prove their suitability for a study of the multilayer microstructures and their changes after thermal treatments.

1. INTRODUCTION

It has been demonstrated already in [1,2] that multilayers consisting of alternate layers of a low and a high atomic number material can exhibit a high reflectivity for soft x-rays. They are thus a suitable basis for several soft x-ray optical applications, for example in the areas of x-ray microscopy[3-5], x-ray lithography[6-8], x-ray astronomy and synchrotron radiation optics.

A number of groups has started producing multilayers[9-11] with different goals. Some of them deal mainly with applications but from a number of works[12-18] and further references in[11] it is also clear that work must be done to improve the multilayer systems themselves, on the one hand in order to reach the theoretical limits for the reflectivity on the other hand to improve their long term and temperature stability.

This work describes first results of a project, in which multilayers are produced by e^--beam evaporation. The layer systems are then analyzed by surface analytical methods, Cu-k_α reflection and soft-x-ray optical methods in order to find relations between the microstructural and soft x-ray optical properties. Calculations on the basis of the Fresnel equations are used for the multilayer design, the support of the fabrication process, and for the x-ray optical analysis of the fabricated multilayer samples.

2. FABRICATION AND IN SITU C-k REFLECTION

The deposition is done in an ultra high vacuum vessel with 2kW e^--beam evaporation sources (Fig.1). They are complemented by quartz oscillators and a quadrupole mass spectrometer for rate and layer thickness monitoring. The pressure during deposition ranges down to about 5 times 10^{-10} mbar. To reduce the heat load on the system during evaporation the vessel walls are completely water cooled. The substrates, at present ten with a size of 14 x 25 mm², are mounted in a holder 65 cm above the evaporation sources; this holder can be rotated about a horizontal axis and leaves the center position in the vessel free for a reference substrate which is mounted in a separate adjustable holder at the same height. The reference substrate is part of an x-ray monitor system, which includes also an x-ray source and a proportional counter and is used for thickness control as in[1,10]. Both, the multilayer substrates and the evaporation materials can be exchanged by wobble sticks, i.e. without a venting of the system.

The soft x-ray reflection coefficient of the reference substrate is measured during deposition. The x-ray source and the proportional counter can be used at angles α of 70, 45 and 30 degrees with respect to the reference substrate surface normal (The exact value of α can deviate from these values by up to 2 degrees due to different adjustments of source, substrate and proportional counter.)

A result obtained with Carbon-k radiation and α = 68 degrees is shown in Fig. 2. The reflectivity vs thickness (as determined with the quartz oscillators) during the evaporation of a single thick molybdenum film on a silicon substrate exhibits strong oscillations occuring periodically with depth. They are due to interference between the reflected amplitude from the surface and bottom of the top Mo-film[1,10] as will also be illustrated by a calculation in the next section .They are damped with film thickness due to absorption in the film. Multilayer fabrication with in situ carbon-k reflection is done by evaporating molybdenum and silicon alternately changing from molybdenum to silicon evaporation when a maximum in reflectivity is reached and from silicon to molybdenum evaporation when the reflectivity exhibits a minimum. This procedure yields the C-k reflection curve shown in Fig. 3. Pronounced maxima and minima of the reflectivity are found which after a few layers occur periodically with depth within the accuracy of the quartz oscillator thickness measurement of a few percent. The detailed shape of this curve will be discussed together with a corresponding calculation in the next section. The calculation yields also equal double layer thickness within 2 percent accuracy after a few layers. In general we start therefore with the deposition of a few layers on the reference substrate before the samples in the sample holder are put into the vapour beam.

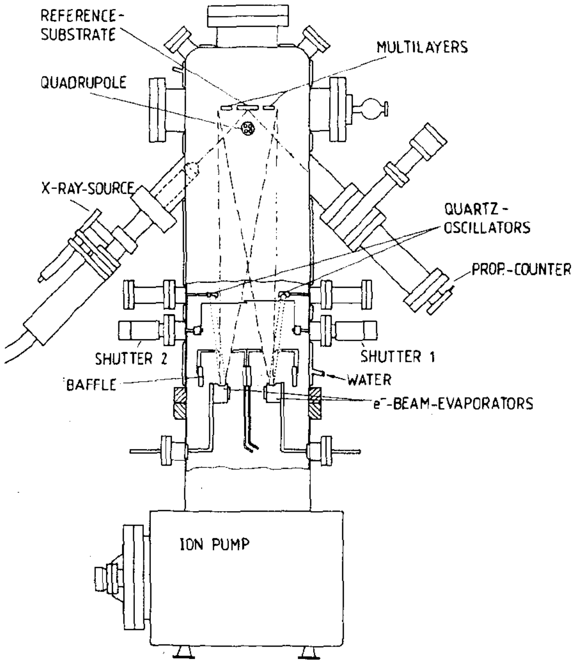

Fig. 1: Deposition system for the multilayer fabrication.

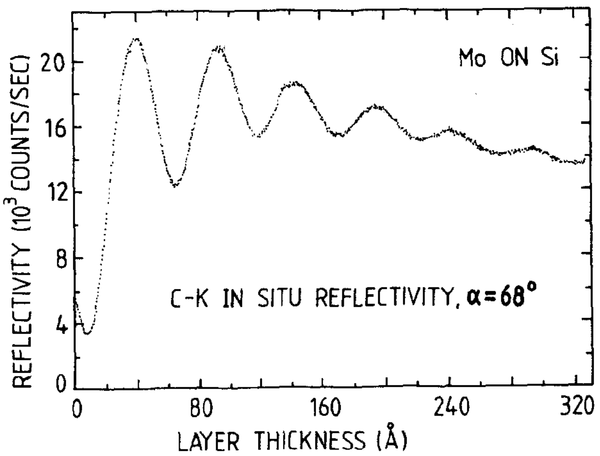

Fig. 2: Carbon-k in situ reflection curve collected during deposition of a single thick Mo-layer on a silicon substrate.

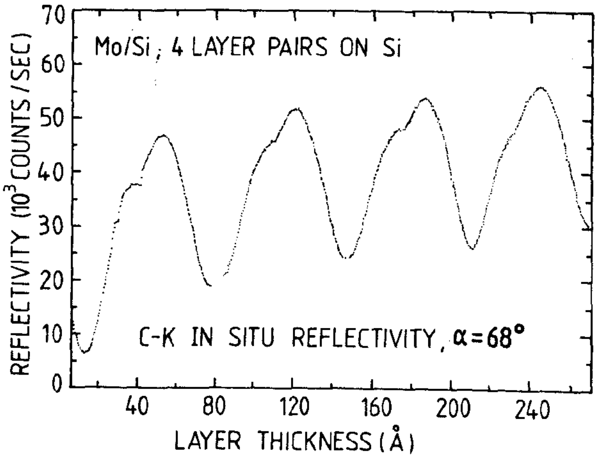

Fig. 3: Carbon-k in situ reflection curve collected during deposition of 4 layer pairs Mo/Si on a silicon substrate.

3. CALCULATIONS

Calculations of the reflectivity of the multilayers have been performed for different purposes. They allow the design of multilayers and can be used for an analysis and understanding of the experimental data obtained from in situ C-k reflection as well as the Cu-k_α and synchrotron radiation reflection curves. We perform the calculations on the basis of the Fresnel equations, a procedure which is in principle treated in textbooks on optical thin films[19] and has earlier been applied to multilayer soft x-ray reflectors[20]. Briefly[21], we start with the Fresnel coefficient r_{12} for the reflected amplitude from a single boundary between two materials of complex refractive indices n_1 and n_2 and propagation angles α_1, α_2 with respect to the interface normal. The reflected amplitude of a single film of thickness d with two boundaries (top and bottom) with reflected amplitude r_t, r_b and transmitted amplitude of the top boundary t_t^+, t_t^- as well as propagation angle α in the film is calculated from

$$r_f = r_t + \frac{r_b t_t^+ t_t^- \exp(2i\delta)}{1+r_t r_b \exp(2i\delta)} \quad \text{with } \delta = \frac{2\pi}{\lambda} nd \cos\alpha \qquad (1)$$

Recurrent application of this equation yields the reflected amplitude for a multilayer. Imperfect boundaries can be incorporated into the calculation by describing the reduction of the reflected amplitude at a boundary by a Debye-Waller factor[20]:

$$DW = \exp\{-2(\frac{2\pi\sigma\cos\alpha}{\lambda})^2\}. \qquad (2)$$

σ is the width of a smooth transition layer without scattering losses.

The procedure is illustrated by a calculation of the reflected amplitude corresponding to the in situ reflectivity curves in Figs. 2 and 3. For a single Mo film on silicon the calculation yields the spiral line for r in Fig. 4 converging to the reflected amplitude value of a single Mo-vacuum interface. It is obvious from this figure that due to absorption the oscillations of the reflectivity with depth are strongly damped in agreement with the finding in Fig. 2.

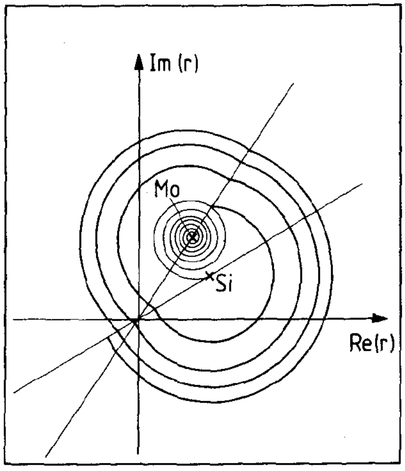

Fig. 4: Calculated reflected amplitude corresponding to the experimental in situ control curves of Figs. 2 and 3. The course of the thin line describes the reflectivity behaviour during evaporation of a thick Mo-layer. The heavy spiral line is obtained when the multilayer deposition process of Fig. 3 is simulated.

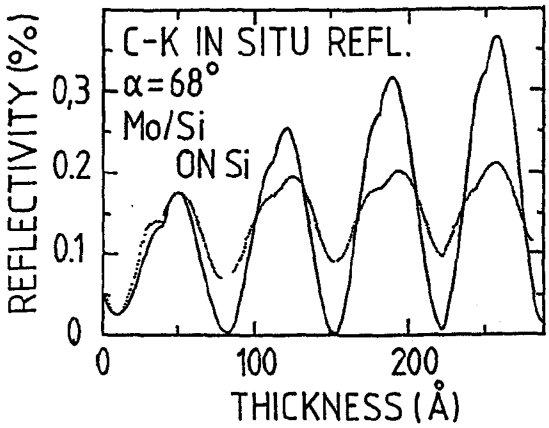

Fig. 5: Comparison of the experimental deposition curve of Fig. 3 (dots) with a corresponding calculated Carbon-k in situ reflection curve (solid line).

The calculated course of r for the multilayer deposition corresponding to Fig. 3 is given by the heavy line in Fig. 4. Change of the evaporation material induces a change of the center of the spiral and in case of silicon evaporation on molybdenum an increase in reflectivity before the strong reflectivity decrease to the minimum is found. This is exactly the shape which was found in the experiment in Fig. 3. Fig. 5 shows the corresponding calculated reflectivity curve compared quantitatively with the curve from Fig. 3. We find that the oscillations in the experimental curve are damped with thickness compared to the calculated curve. The damping regards on the one hand the average value of the reflectivity with thickness, an effect which can be due to roughness of the type described by the DW-factor in equation(2). We find, however, mainly a damping of the maximum to minimum ratio, which cannot be described by roughnesses of the Debye-Waller type. These may be due to thickness errors on a laterally microscopic scale, an effect which cannot be compensated by in situ soft x-ray reflectivity which minimizes the average thickness error of the whole stack. Minor parts of the damping of the curve are also due to the experimental resolution, namely the line width of the C-k line and the angular resolution of the in situ C-k setup of ±0.5 deg.

4. TESTS WITH RBS AND SPUTTER/AES

Rutherford Backscattering (RBS) and sputtering in combination with Auger electron spectroscopy have been applied to deduce microstructural information about the layer systems which were fabricated as described above[22]. The experimental setup for the RBS system is given in[23]. The Sputter/Auger system is part of the apparatus given in[24]. Fig. 6 shows results obtained with RBS for a 5 layer Ta/Si-system. For 350 keV He^+ ion incidence on the layer system we measure the energy distribution of the particles backscattered at three different angles a, b and c as indicated in the inset of Fig. 6 and obtain the three different backscattering spectra. All the spectra show 3 clearly resolved peaks at the high energy edge, two weak peaks at 200 keV and a structureless contribution at lower energies which increases continuously with decreasing energy. A more detailed data analysis shows that the latter contribution is due to ions backscattered from the silicon substrate while the two weak oscillations stem from Si and the three strong peaks from Ta in the layer stack, respectively. We get thus directly an impression regarding the regularity of the layers in the stack and also - within the experimental uncertainty - an information about the smoothness of the interfaces including that with the silicon substrate. The data analysis is more complicated in cases, where a larger number of layers is involved as for the data given in Fig. 7. For this 18 layer Mo/Si stack the oscillations at the high energy edge of the spectra which are due to backscattering of He-ions from Mo are only resolved for the first four layers. The contributions of the deeper layers are intermixed with the contributions from the Si layers. One gets thus from these spectra mainly a rough impression concerning the regularity of the multilayer stack as a whole while details about the individual layers cannot easily be deduced from these spectra. The data show, however, that it should be possible to study model systems as for example ultrathin metal films separated by about 100 Å silicon spacers. In order to improve the resolution of the system further, an electrostatic analyzer is built up within this project which should improve the energy and thus the depth resolution by a factor of 5.

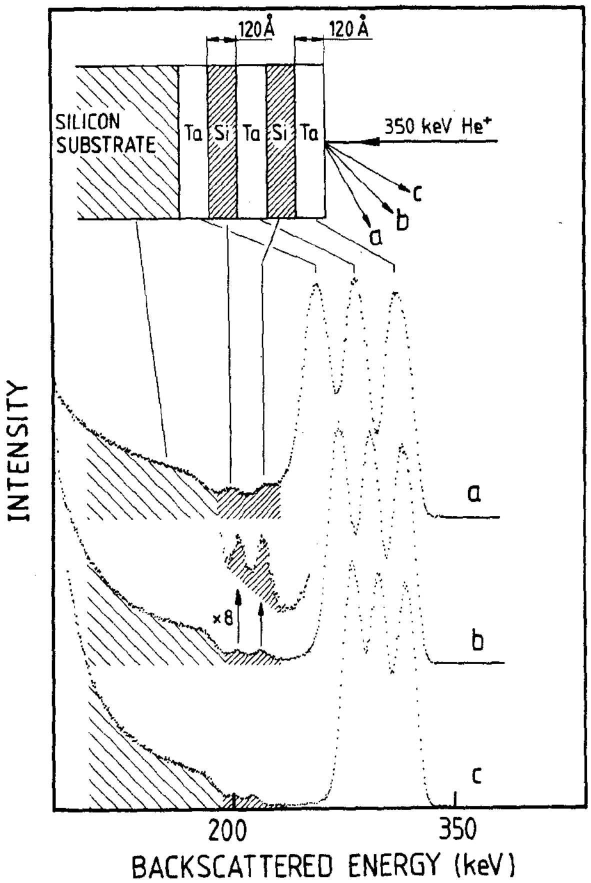

Fig. 6: RBS analysis of a Ta/Si stack with 5 layers.

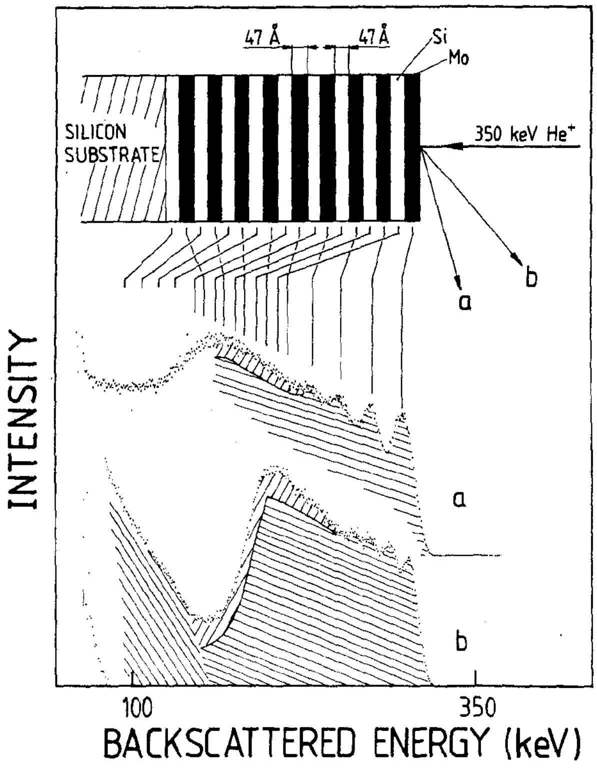

Fig. 7: RBS analysis of a Mo/Si multilayer with 9 layer pairs.

A better separation of the individual layers in the multilayer stack is already obtained with Sputter/AES, the second microstructural characterization technique which we apply to determine the element composition vs. depth in the stack. A typical result is given in Fig. 8, while the inset illustrates the technique: The layers are successively removed from the stack with 600 eV Ar^+ sputtering. The elemental composition is determined by Auger electron spectroscopy with a primary energy of 3 keV. Fig. 8 displays the Auger amplitudes vs. sputtering time for a Mo/Si system with a layer thickness of 30 Å each corresponding to a 100 eV soft x-ray mirror. The open circles denote the Si contribution, filled squares that of Mo. Both the Si and Mo Auger amplitudes vary periodically with sputter time as is expected for an ideal multilayer stack. The variation of the Mo signal is weaker. This result is similar to an earlier observation in ref. 25, where a weaker variation of the W signal in studies of W/Si multilayers was found.

The Sputter/Auger technique has also been applied to study thermally treated samples. Such studies are of interest for different reasons. Firstly, samples, which are stable at high temperature are in general also stable for a long time. Secondly multilayer optics is intended to be used also for applications with high radiation fluxes and thus high thermal loads as for example in synchrotron radiation applications and thirdly we want to find out if controlled diffusion processes of silicon into metals can create multilayers with higher thermal stability. Changes in Mo/Si and Ta/Si multilayers upon heating treatments between 200°C and 400°C have been studied with synchrotron radiation reflection and will be described in section 6. According to those studies the multilayers were stable up to 200°C; above this temperature diffusion processes occured. The microstructure of Mo/Si, W/Si and Ta/Si multilayers before and after heating to 500°C has been studied with Sputter/AES. Fig. 9 shows a typical result for a Mo/Si multilayer. First some layers of the as prepared multilayer were removed by sputtering to analyze their microstructure. After a 500°C heating the Sputter/AES analysis was continued. The data indicate that a large amount of silicon diffuses to the surface and that the silicon and metal layers intermix. A periodicity in the depth distribution of the elements seems to remain, however, even after this prolonged heating procedure (45 min). Similar results were obtained for the W/Si and Ta/Si multilayers. W/Si systems show even after heating to 750°C periodicity in the stack. At either temperature the surface diffusion of silicon is much weaker for the system Ta/Si than it is for Mo/Si and W/Si.

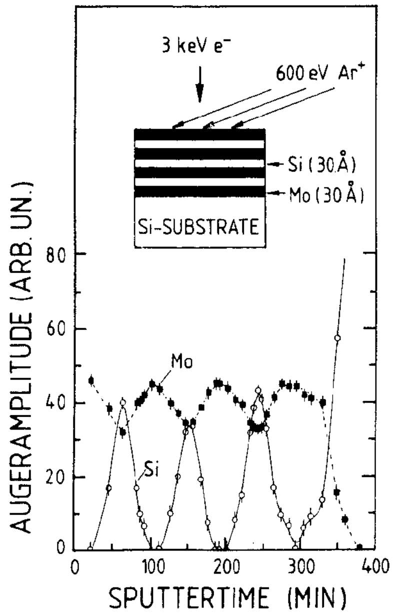

Fig. 8: Sputter/AES analysis of a 7 layer Mo/Si stack.
■: Mo-Augeramplitude
O: Si-Augeramplitude

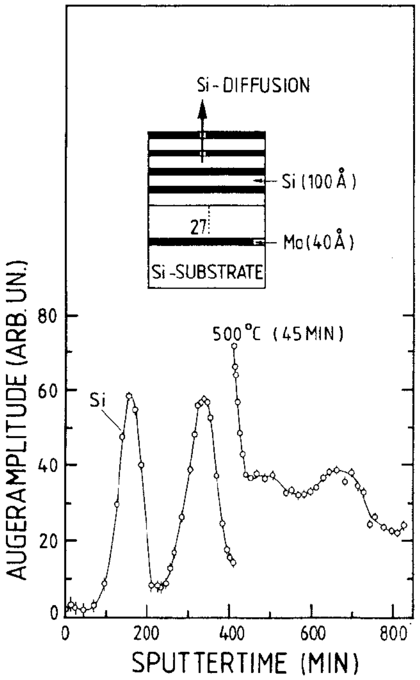

Fig. 9: Sputter/AES analysis of a Mo/Si multilayer before and after heating to 500°C.

5. CHARACTERIZATION WITH Cu-k_α REFLECTION

The characterization of multilayers with Cu-k_α radiation is done with an experimental setup which is similar to that described in[26]. It consists of a Cu-k_α x-ray source, a germanium monochromator crystal, a θ-2θ table, a proportional counter and collimating systems which reduce the angular widths of the incoming and outgoing beams. One determines the reflectivity of the multilayers for the 1.5407 Å radiation vs. angle between 0 degrees (direct beam) and the first and higher order Bragg reflections, which for soft x-ray reflectors are typically separated in the angular scan by grazing angles θ between 0.5 and 1.5 degrees. A detailed description of the method which is based on calculations with the Fresnel equations like those in section 3 is given in[27].

Mo/Si multilayers with different numbers of layer pairs and produced with in situ soft x-ray reflection have been analyzed with Cu-K_α reflection. Results are given by the solid lines in Fig. 10. In the left part data are given for N=6 layer pairs and in the right part for 12 layer pairs. In addition to the first and second order Bragg maxima these curves reveal a number of Fresnel maxima between the Bragg peaks as one would expect for a perfect multilayer.

The curves are also compared with calculations (dashed lines in Fig. 10). The calculations were performed for ideal multilayers with abrupt interfaces and the same layer thicknesses as produced in the experiment, i.e. for the stacks obtained by simulation of growth with in situ C-k control. Comparing the measured with the calculated curves in Fig. 10 we find very good agreement for grazing angles θ smaller than the first Bragg angles at about 0.7 deg, while considerably smaller experimental values are found at larger angles.This may be due to the interfacial imperfections, which were also found in the in situ C_k-control curves during deposition and which have been described above. The reflectivity values in the second Bragg maximum are by far more sensitive to interfacial roughness and thickness errors than those in the first Bragg maximum[2,27].

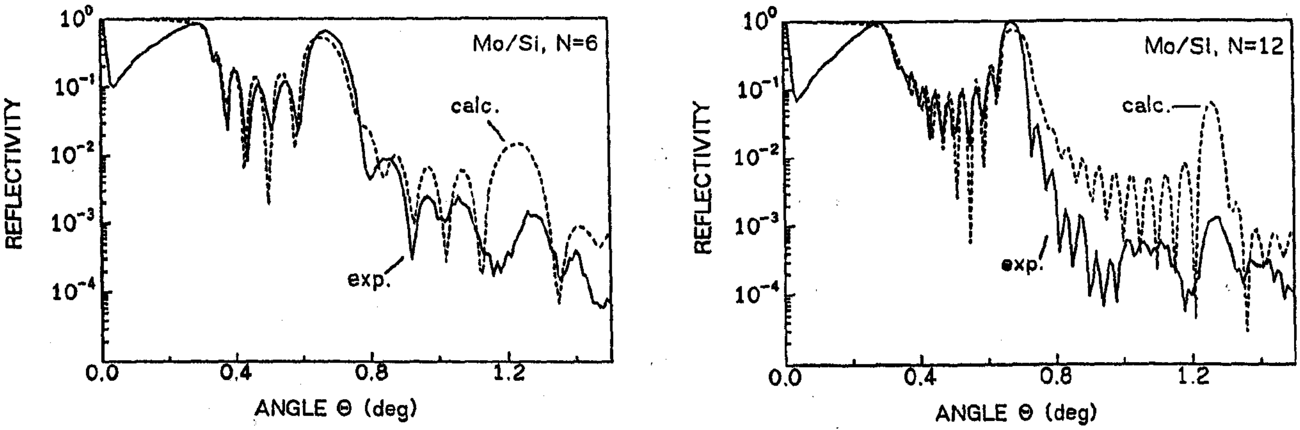

Fig.10: Cu-k_α reflection curves obtained for Mo/Si multilayers:
left: 12 layers, right: 24 layers. The experimental curves (—)
are compared with calculations (- -).

6. CHARACTERIZATION WITH SYNCHROTRON RADIATION

Synchrotron radiation soft x-ray reflection curves vs. wavelength were determined for a couple of multilayers at an experimental setup in the PTB laboratory at BESSY. This apparatus is described in[28]. Most of the data were obtained for normal incidence of the radiation. Typical results for 3 different layer systems with different layer pair thicknesses are given in Fig. 11. In the upper figure, which was obtained for a multilayer with a rather large layer pair thickness, we find the reflectivity maximum at the largest wavelength, while maxima for the multilayers with smaller periods are obtained at shorter wavelenghts. The width of the reflectivity peaks decreases also with decreasing wavelength, as the number of layers contributing to the peak reflectivity increases. The sharp peak in the upper curve is a second order reflection of the multilayer. In all three cases we obtain peak reflectivities of about 10 %. These multilayers were produced without in situ C-k control.

A result for a multilayer produced with C-k control is given in Fig.12. The maximum for this 12 layer pair stack occurs at 141 Å wavelength with a value of about 15%. The dashed line represents a calculation for a corresponding multilayer with smooth interfaces which was performed as described in section 3. Experiment and calculation show the same dependence on wavelength of the radiation with the first Bragg maximum at 141 Å and some oscillations at larger wavelengths. The absolute values differ by a factor of 1.8. Assuming an interfacial roughness σ of 9.5 Å we get the dash dotted curve in Fig 12, which agrees roughly with the experimental result.

In Fig. 13 the angular dependence is shown for the multilayer which corresponds to the reflectivity curve in the upper part of Fig. 11. The largest value for the reflectivity is found for normal radiation incidence, while the reflectivity is considerably lower for incidence angles around 40 deg. This is due to the fact that the measurements are performed with linearly polarized radiation with the E-vector parallel to the scattering plane. As the Brewster angle in the studied photon energy range is about 40 deg the reflectivity is that low around 40 deg. This shows, vice versa, that the multilayer is an efficient polarizer.

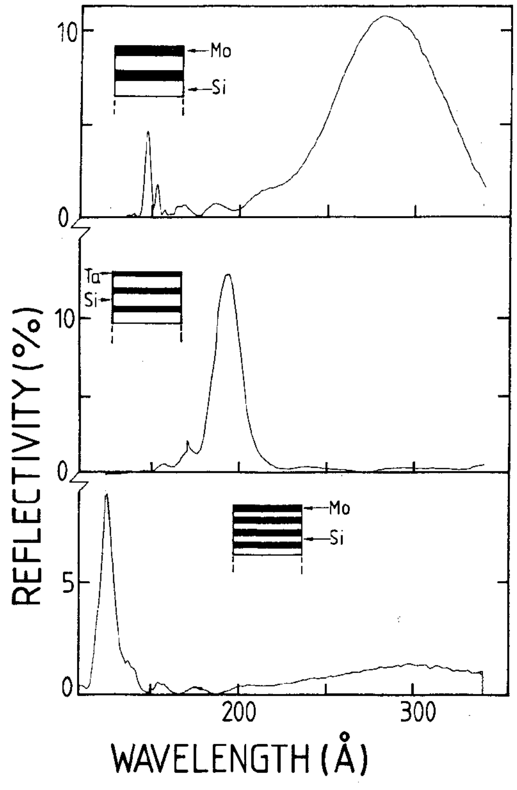

Fig.11: Three typical reflectivity curves for metal silicon multilayers obtained with soft x-ray synchrotron radiation for normal incidence. The multilayers were produced without in situ C-k control.

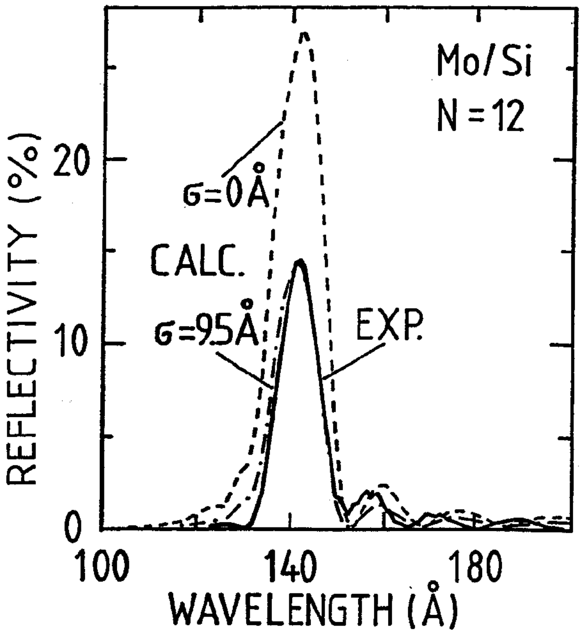

Fig.12: Reflectivity curve for a multilayer produced with in situ C-k control. The experimental curve is compared with calculations for smooth (dashed line) and rough (σ = 9.5 A) interfaces (dash-dotted line)

Synchrotron radiation soft x-ray reflection was also used to study the changes in reflectivity after heat treatments of the multilayers. Ta/Si multilayers with 7 layers were heated to 200°C, 300°C and 400°C for 20 minutes in vacuum. The corresponding reflectivity curves are displayed in Fig. 14. Heating to 200°C does hardly influence the reflectivity behaviour. Heating to 300°C and 400°C induces a substantial decrease in double layer spacing, which is most probably due to silicon diffusion into the Ta layers[29]. A substantial amount of material is thus transported across the interfaces. The reflectivity decreases also. We note, however, that even after 400°C heating and a double layer thickness change of more than ten percent, the reflected amplitude is not lower than two thirds of its original value.

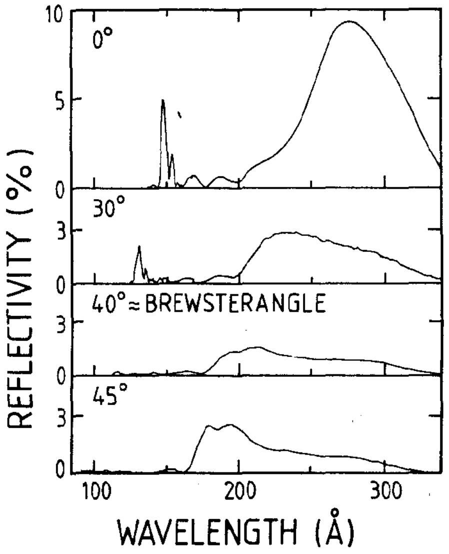

Fig.13: Angular dependence of a Mo/Si multilayer reflectivity for p-polarized synchrotron radiation.

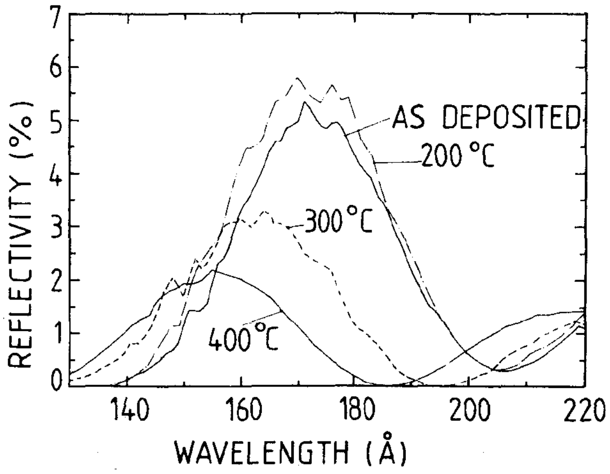

Fig.14: Reflectivity vs. wavelength curves for four identically deposited Ta/Si stacks with 7 layers after heating to 3 different temperatures.

7. CONCLUSIONS

Metal silicon multilayers have been fabricated with e⁻ beam evaporation and in situ C-k control. They have been studied with synchrotron-radiation soft x-ray and Cu-k_α reflection as well as the surface analytical methods RBS and Sputter/AES. Synchrotron radiation reflection yields typical reflectivity values between 10 and 15 % for wavelength between 125 and 300 Å. For one multilayer we studied the polarizing properties. Comparison of in situ C-k reflectivity curves with calculations shows that roughnesses at the interfaces exist which cannot completely be described by multiplying the reflected amplitude at each interface by a Debye Waller factor. We studied also changes in multilayer stacks which are induced by heating in vacuum. Heating of Ta/Si samples can induce a considerable change ($\geq$ 10 %) in d-spacing of the multilayers while the reflected amplitude is only reduced to two thirds of its original value.

8. ACKNOWLEDGEMENTS

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