Ion beam sputtering of x-ray multilayer mirrors

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ABSTRACT

Ion beam sputtering has been applied for polishing, figuring and multilayer coating on silicon and quartz glass substrates for the fabrication of x-ray mirrors. For high-performance x-ray optics extremely low microroughnesses of the substrates have to be achieved. Particularly for low d-spacing multilayers (d = 1...2 nm) even small improvements of the surface quality result in significant performance gains of the mirrors. By ion beam polishing silicon substrate surfaces could be smoothed from 0.18 nm rms to 0.11 nm rms (AFM scan length = 5 µm). Furthermore figuring of spherical substrates into elliptical or parabolic surface contours has been developed and applied. Spherical quartz glass substrates with initial rms roughnesses of 0.73 nm and 0.52 nm show reduced roughnesses after figuring and multilayer coating of 0.26 nm and 0.10 nm using AFM scan lengths of 20 µm and 5 µm, respectively. The testing of the ion beam figured mirrors for the application as parallel beam and focussing optics shows very promising results: The comparison of collimating mirrors, produced either by ion beam figuring or bending, shows very similar x-ray intensities. However, the ion beam figured mirrors open the perspective for further reduced figure errors, improved long-term stability and 2-dimensional focusing.

Keywords: X-ray optics, multilayer mirrors, ion beam polishing, figuring and sputtering, surface roughness, smoothing

1. INTRODUCTION

For many years nanometer multilayers have been used as mirrors and monochromators in the x-ray and EUV spectral range. Pioneering work in this field has started already in the 1970s [1-3]. X-ray mirrors consist of flat or curved substrates and reflection coatings deposited on top of the substrate surfaces. The reflection coatings can be single layers of high-Z materials (e. g. Au, Pt, Rh) or multilayers with single layer thicknesses in the nanometer range. Routinely used multilayers are for instance combinations of one of the absorber materials Cr, Ni, Mo, Ru, W with one of the spacer materials B_4C, C, Si. The shape of the substrate determines how the x-ray beams will be deflected, e. g. elliptical curvatures will result in focusing, parabolic curved mirrors collimate a divergent beam arising from a point source. The reflection coating defines the spectral answer of the mirror. The material of a single layer or the design of the multilayer determines which intensity is reflected for given values of the x-ray wavelength λ and the grazing angle Θ.

Typical x-ray mirror substrates have aspherical surface contours that are made by figuring bulk silicon or quartz glass substrates or by bending flat silicon wafers. Already in 1965, first attempts to figure optical substrates by energetic ion beams were reported [4]. In the meantime, ion beam figuring became an established technology for optics fabrication. However, it is still a challenge to obtain precisely figured aspherical surfaces with extremely low microroughnesses in the order of 0.1 nm rms as they are needed for x-ray mirrors. Therefore the elastic bending of silicon wafers has been developed as a second technology for one-dimensional manipulations of x-rays [5-7]. The advantages of the bending technology are the possibility to use highly polished and inexpensive silicon wafers and the high flexibility. Depending on the concrete application wafers can be bent in virtually any geometry needed for x-ray mirrors. However, bending is only possible in one direction. Therefore with a single mirror only one-dimensional focusing or collimating mirrors can be fabricated. Hence, for two-dimensional beam shaping optics two mirrors have to be combined. Furthermore the bending results in unavoidable figure errors because of the fact that in practice it is extremely difficult to get exactly the same mirror contour in the middle of a silicon stripe and in the near-edge regions.

Besides figuring of aspherical surfaces polishing is still a very important issue. Standard technologies like grinding and lapping result in surfaces with microroughnesses between 0.2 and 0.5 nm rms. However, particularly for low d-spacing multilayers with periods < 2 nm any further small improvements will result in significant gains of the optical performance. Polishing of surfaces by ion bombardment is well-known since more than 30 years [8]. Later it has been shown

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that also with low-energy ions (kinetic energies < 2 keV) surface topographies can be manipulated and roughness can be reduced [9].

The final step of x-ray mirror fabrication is the deposition of multilayer coatings on the mirror surface. In recent years huge progress has been made in the field of multilayer research and development. Many material combinations and deposition technologies have been pushed to the status that they can routinely be used in an industrial production process. Most of the groups dealing with nanometer multilayer coatings apply magnetron sputter deposition (MSD) as a very robust and productive coating method for nanometer multilayers [11-16]. However, besides MSD further technologies like ion beam sputter deposition (IBSD) [17-19], electron beam evaporation [20-21] and pulsed laser deposition [22] are successfully used for the fabrication of multilayer mirrors. Each method has its specific advantages. In many cases the concrete material combination triggers the choice of the appropriate deposition technology in order to obtain the optimal performance of the mirrors.

This paper presents latest research and development results obtained with our dual ion beam machine. Starting with commercial substrates, ion beam polishing and figuring has been applied in order to fabricate aspherical substrates with extremely smooth surfaces. The surface roughness before and after the process steps polishing and figuring has been compared using atomic force microscopy. Furthermore, multilayer depositions have been carried out using standard processes optimized for coatings on superpolished silicon wafers. The reflection spectra obtained on silicon wafers and on ion beam figured mirrors have been compared. Finally, different types of multilayer mirrors (1- and 2-dimensional beam shaping) have been tested.

2. METHODOLOGY

2.1 Polishing, figuring and multilayer deposition

Ion beam polishing, figuring and the multilayer deposition have been performed in a commercial dual ion beam tool. As main parts, the machine contains two linear ECR ion sources with grid lengths of 400 mm. The ion sources can be operated at energies between 50 and 2000 eV. Substrates with dimensions of up to 500 mm x 200 mm are arranged face-down and can be linearly moved across differently shaped slits. Further details of the ion beam machine can be found elsewhere [18].

For direct smoothing of substrate surfaces only the secondary ion source has been activated. In this case typical ion energies of 100-800 eV are used. The mean incidence angles of the ions bombarding the surface are 0-20 degree. Typical smoothing durations are between 10 and 30 minutes. During the smoothing process the substrate rotates around its symmetry axis or moves linearly across the fixed slits in front of it.

Ion beam figuring of surfaces is done in a similar way as ion beam polishing. The main difference is the energy of the ions. For figuring kinetic energies between 1200 and 1800 eV are applied. The main reason is the improved sputter yield at higher energies. Typical removal rates are 50-100 nm/min for quartz glass. With the known spatial profile of the ion beam the velocity profile of the substrate movement across fixed slits can be calculated in order to obtain a desired substrate figure. In a different working mode the substrates move with constant velocity and specific shapers are placed in front of the substrates in order to get the appropriate substrate figure (fig. 1).

Multilayer deposition has been carried out using the primary ion source which delivers ions that are 1-dimensionally focused onto the target surfaces. For target sputtering ion energies between 600 and 1500 eV are applied. The precise energy depends on the film properties needed. Using higher energies smoother and denser films can be obtained. However, higher energies also result in larger intermixing at the multilayer interfaces. Therefore there is always a certain trade-off between smooth single layers and steep concentration gradients at the interfaces. Additionally, the secondary ion source can be used for assisting the growth of the films on the substrate surface during multilayer deposition. For this purpose the ion energies of the secondary source are on the lower limit at around 50-200 eV.

The different operation modes described so far are schematically shown in fig. 2. It should be mentioned that the processes can easily be scaled in the direction perpendicular to the linear substrate movement since linear ion sources are used. Currently the machine allows the treatment of maximum substrate areas of 500 x 200 mm^2.

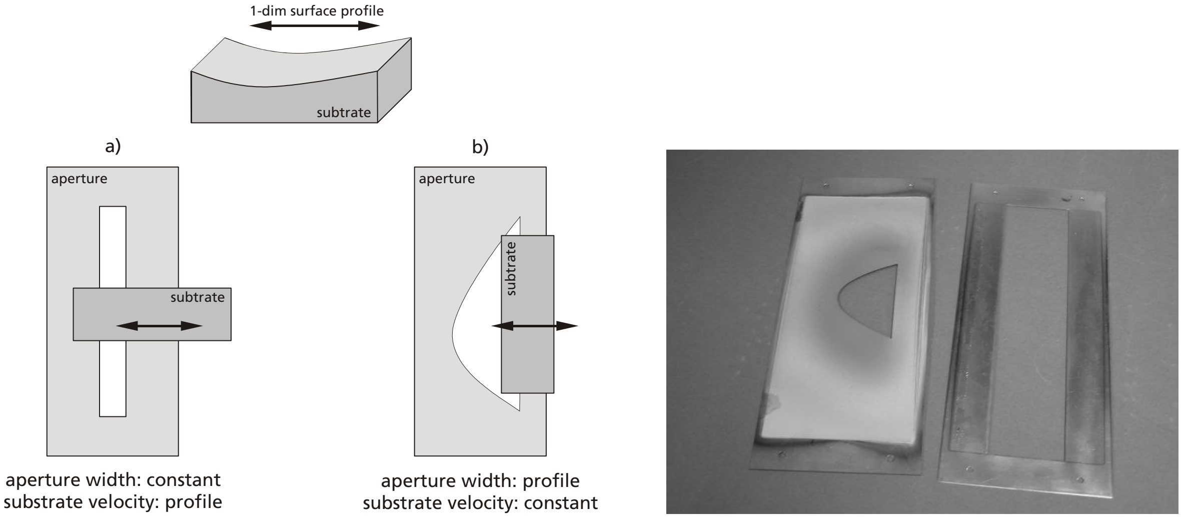

Fig. 1. Different operation modes of the 1-dim ion figuring process: a) movement of the substrate parallel to the profile direction with a well-defined velocity profile above a fixed aperture with constant width or b) movement of the substrate perpendicular to the profile direction with constant velocity above an aperture with a certain width profile. The right photograph shows examples of used apertures.

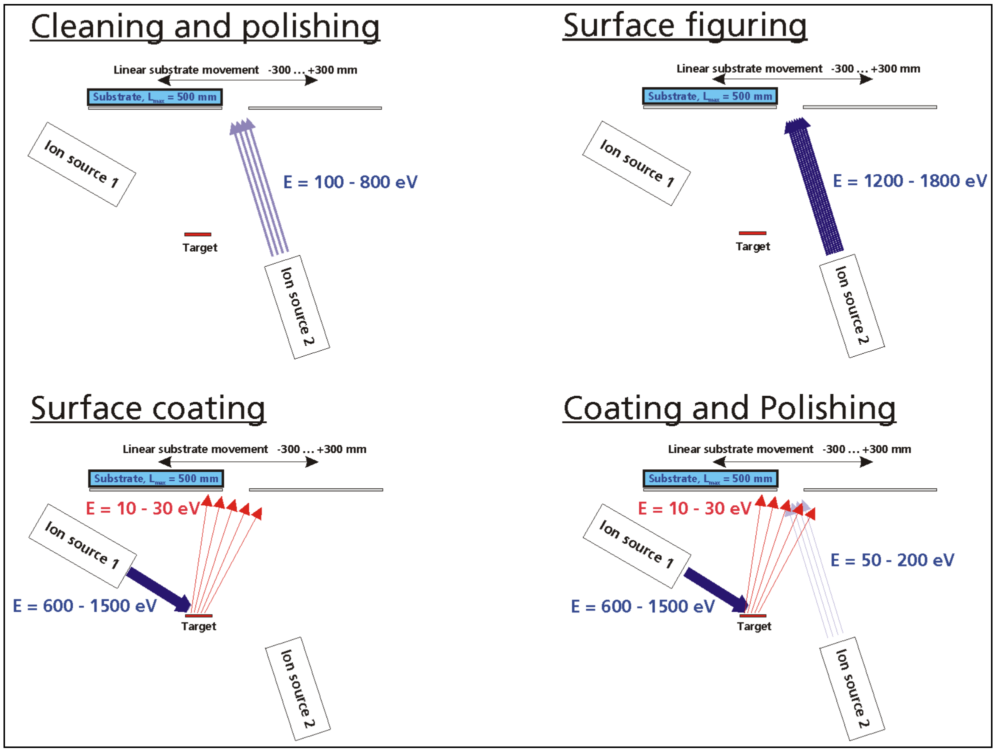

Fig. 2. Working modes of the dual ion beam machine. Cleaning and polishing of substrate surfaces is performed using ion energies in the range of 100 – 800 eV. For substrate figuring typically energies between 1200 – 1800 eV are used. For the multilayer coating the primary ion source is operated at ion energies in the range of 600 – 1500 eV.

For the experiments described in this work several different substrate geometries have been used. For 1-dimensional beam shaping mirrors spherical or cylindrical substrates with curvature radii in the order of 10 m have been processed (fig. 3a). In the case that they have to be combined for 2-dimensional beam shaping (Montel geometry) a special 45 degree cut has been applied (fig. 3b). For 2-dimensional beam shaping mirrors cylindrical substrates with curvature radii in the order of 4 mm have been used (fig. 3c). In this case the ion beam figuring has to be started from a flat (cylindrical) geometry (perpendicular to the curvature). Hence the material removal in the center of the mirror has to be in the order of ~100 µm (see also fig. 7).

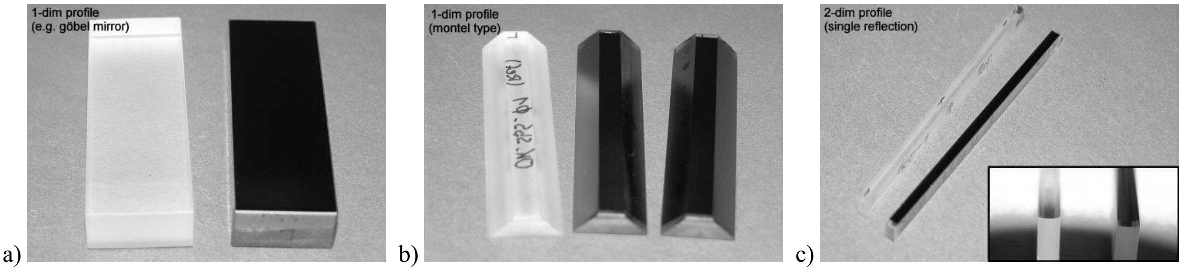

Fig. 3. Various ion beam treated x-ray optics

a) “standard” göbel mirror, 1-dimensionally focusing or collimating. Left hand side: original substrate, Right hand side: example for a figured and multilayer coated mirror.

b) mirrors for “side-by-side” arrangements (Montel geometry), 1-dimensionally focusing or collimating. Left hand side: uncoated substrate

c) 2-dimensionally focusing optics (single reflection), the original substrates (left hand side) have cylindrical shapes with curvature radii of 4 mm in the short direction. The inset shows the front sides in higher magnification.

2.2 Characterization of figure, roughness and reflectivity of x-ray mirrors

Figure characterization of the mirrors has been performed by laser triangulation using a cyberscan vantage unit, model DRS-500. This tool collects a series of z-heights with sub-micron resolution in order to produce a 2-dimensional surface profile. The spot size of the laser beam is in the range of 16-23 µm, the minimum step size in x- and y-direction is 1 µm and the z-resolution is 0.125 µm. The maximum size of the measurement area is 305 mm x 305 mm.

The surface topography has been characterized by atomic force microscopy (AFM) operated in tapping mode using a Dimension 3100 with a Nanoscope V controller (Veeco Instruments) installed in an air environment. The AFM measurements have been performed with different scan length between 1 µm x 1 µm and 20 µm x 20 µm having 256 x 256 or 512 x 512 pixels. After the measurements we typically applied the flatten algorithm delivered by the Veeco software in the following manner: 0th order for 1 µm scan lengths, 1st order for 5 µm and 2nd order for 20 µm scan lengths. From the height profiles, the rms surface roughness σ_r and the 2-dimensional power spectral densities PSD have been calculated using standard formulas (see e. g. [23]).

The reflectivity of the multilayer mirrors has been measured by x-ray reflectometry (XRR). A parallel beam of Cu- or Mo-Kα radiation hits the sample surface under grazing incidence and is reflected into the detector. The intensity of the reflected beam as a function of the incidence angle Θ delivers information about the mirror reflectivity, the multilayer period and the interface quality including roughness. The measurements have been performed using two machines, a Siemens D5005 and a Bruker AXS D8, each having different angular resolutions δ of approximately 0.006 and 0.01 deg. The finite angular resolutions of the reflectometers result in a convolution of the inherent multilayer reflectivity spectrum with a specific tool function. Particularly for high resolution multilayers having small periods d_p and high period numbers N, increased values of δ result in broader and less intensive reflection peaks compared to measurements at synchrotron sources with $\delta \sim 0$. However, by measuring the same multilayer at both machines and applying model calculations, an accurate extrapolation of the intrinsic multilayer performance (reflectivity, bandwidth) for $\delta \sim 0$ becomes possible [18].

3. RESULTS

3.1 Ion polishing of silicon

The main challenge dealing with nanometer multilayer coatings is the optimization of the interfaces. They have to be smooth and chemically abrupt at the same time. Both effects – roughness and diffuseness – have a negative impact on the mirror performance. Both contributions together are often denoted as interface width $\sigma_{interface}$ with the following relation to interface roughness σ_r and diffuseness σ_d: $\sigma^2_{interface} = \sigma^2_r + \sigma^2_d$. In specular reflection scans it can not be distinguished between roughness and diffuseness, both have to be minimized in order to obtain the optimal performance. In fig. 4 calculations are shown how the interface width affects the multilayer performance. The calculations were carried out for the reflection of Cu-Kα radiation (wavelength $\lambda = 0.154$ nm) at W/B$_4$C multilayers with d-spacings of d = 1.5 nm and 500 periods. The comparison of the maximum reflectances shows a significant decrease with increasing interface widths: -30 % for $\sigma_{interface} = 0.3$ nm compared to perfect multilayer structures. The intensity loss is even stronger if the values of the integrated intensities are compared: -63 % for $\sigma_{interface} = 0.3$ nm.

The diffuseness at the interfaces of nanometer multilayers is mainly chemically driven. Additional contributions can arise from particle implantation during thin film growth. In any case the diffuseness can only be altered by the coating process or by the design of the multilayer. In difference to diffuseness the interface roughness is strongly influenced by the initial substrate roughness. Typically, during the coating process lower roughness frequencies are replicated whereas higher spatial frequencies can be smoothed out. Hence, the deposition process is partially able to reduce roughness but only to a certain degree. Therefore the substrates have to be as smooth as possible, especially for low d-spacing multilayers. Provided that the diffuseness is fixed at $\sigma_d = 0.2$ nm, a decrease of the roughness from $\sigma_r = 0.2$ nm to 0.1 nm results in a reduction of $\sigma_{interface}$ from 0.283 nm to 0.224 nm.

The smoothing experiments described in this paper are performed at an ion energy of 600 eV with a polishing time of 25 minutes. A silicon 100 crystal were used as substrate. The initial roughness of 0.18 nm rms, measured by AFM with a scan length of 5 µm, could be reduced to 0.11 nm rms (fig. 5). The analysis of the 2-dimensional power spectrum PSD shows that particularly roughness with spatial frequencies 1 – 10 μm^{-1} can effectively be smoothed by ion beam polishing (fig. 6).

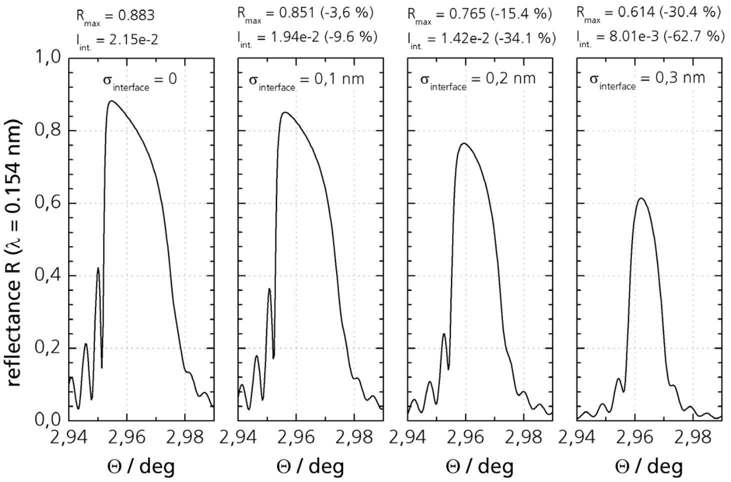

Fig. 4. Calculations of the reflectance spectra of W/B$_4$C multilayers with d-spacings of 1.5 nm for different values of the interface widths $\sigma_{interface}$. Particularly the integrated intensity $I_{int.}$ decreases dramatically for higher values of $\sigma_{interface}$.

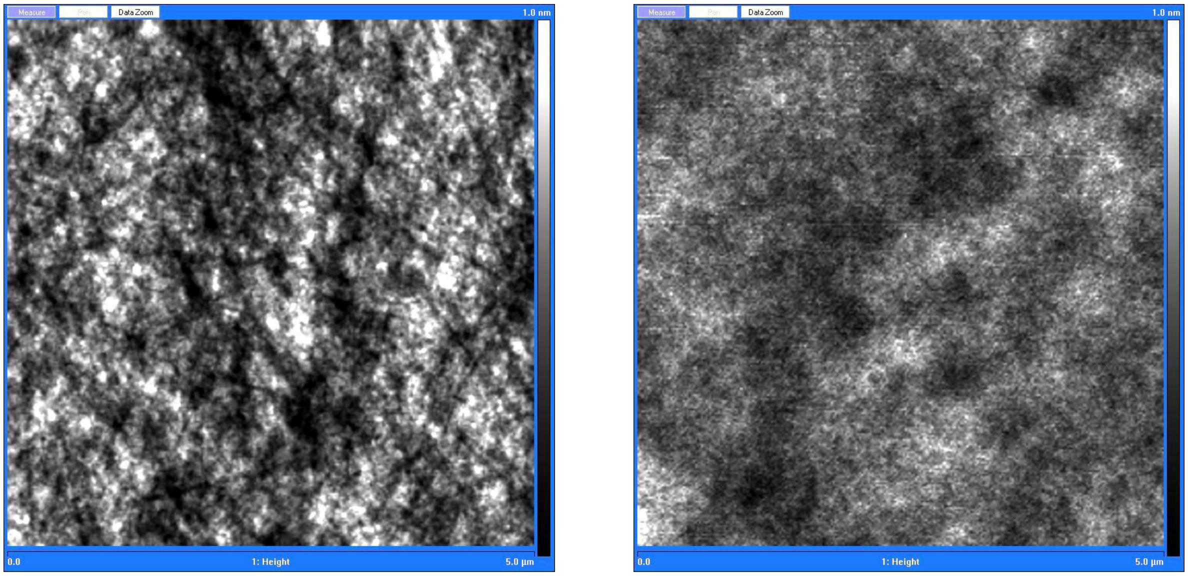

Fig. 5. AFM pictures of silicon surfaces before (left hand side) and after (right hand side) ion beam polishing. The microroughness could be reduced from 0.18 nm rms to 0.11 nm rms (AFM scan length = 5 µm).

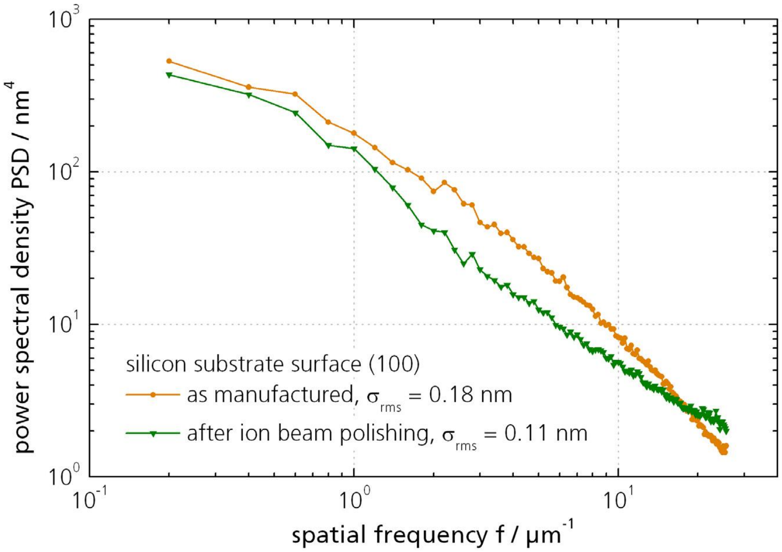

Fig. 6. Power spectral density of the surfaces shown in fig. 5. Even for rather smooth silicon surfaces with rms roughness values < 0.2 nm, spatial frequencies in the range of 1 – 10 µm^{-1} can effectively be smoothed by ion beam polishing.

3.2 Ion figuring of quartz glass

Beside the well known bending technology of flat and already coated silicon wafers figuring of compact substrates prior the deposition of the optical (multi)layer is an often used alternative technique. As described above the application of ion beams for this process, at least for the final finish of the optical surface, is essential to achieve the required surface roughness in the order of 0.1 nm rms. Additionally ion beam treatment of substrates is characterized by an excellent performance in terms of accuracy, reproducibility and flexibility. Hence prefiguring and smoothing of initial flat or even spherical curved surfaces in one technological process would be a huge advantage of the ion beam technique.

For typical 1-dimensional collimating or focusing x-ray optical mirrors for laboratory applications the figure of the surface is characterized by an aspherical shape with a depression in the order of 50-100 µm from the flat contour (fig. 7 left). The typical length of such a mirror is in the order of 50-100 mm. In fig. 7 (left) spherical approximations with radii of about 10 m and 7.2 m are given. Spherical quartz glass substrates with these shapes are commercially available with sufficient surface quality and for moderate financial costs. In these cases the amount of material to be removed during figuring is in the order 5 to some 10 µm.

In our figuring experiments we used both movement approaches described in fig. 1a)+b), the velocity profile of the substrate with a constant slit width (10-50 mm) and the constant substrate velocity above a profile mask. With both techniques we achieved similar results, slight differences can be obtained concerning accuracy (figure error) vs. process time. For the figuring we used source parameters of 1000 eV / 340 mA resulting in an ion beam density on the substrate surface of about 0.4 mA/cm² and a removal rate of about 1 nm/s.

Fig. 7 (right) shows the measured surface profiles of a spherical substrate with a radius of curvature of about R = 10 m and the final result of the figured elliptical shaped mirror. In this case the figure error was in the range of 0.5 µm with the highest deviations near the mirror edges. From the measured height data we calculated the slope error to $\Delta\Theta < 0.01°$ (0.2 mrad). This error corresponds to the typical angular width of the Bragg reflections of the multilayer to be coated onto the figured substrate.

Fig. 8 shows AFM pictures of the initial substrate (left) and the mirror surface after figuring and multilayer deposition (middle + right). Scan lengths were 20x20 µm². We obtained a strong smoothing effect of the surface during the ion beam treatment. The microroughness could be decreased from 0.74 nm rms to 0.26 nm rms. In the higher spatial frequencies (1x1 µm² AFM, not shown here) from 0.52 nm rms to 0.10 nm rms. Fig. 9 shows the 2-dim power spectral densities of the AFM images of both scan lengths. In the spectral range of 0.3 to 20 μm^{-1} a significant reduction of the roughness can be achieved.

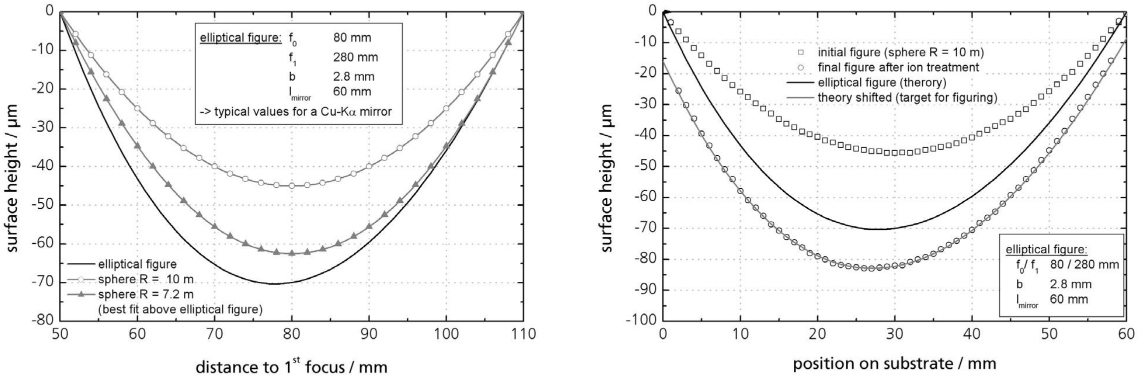

Fig. 7. Theoretical and experimental surface profiles for an elliptical mirror with focal distances of f_0/f_1 = 80/280 mm and a short axis value b of 2.8 mm. These values are typical for Cu-Kα focusing optics.
Left picture shows the calculated surface profile for a 60 mm mirror and some spherical approximations. If the initial substrate shape is flat one have to remove about 70 µm at the deepest point of the mirror. Using spherical (preprocessed) substrates this value can be reduced down to 10 µm.
The right picture shows experimental results obtained by laser triangulation. Initial substrate was a spherical quartz glass with R = 10 m. Finally we removed about 35 µm material in the middle of the mirror.

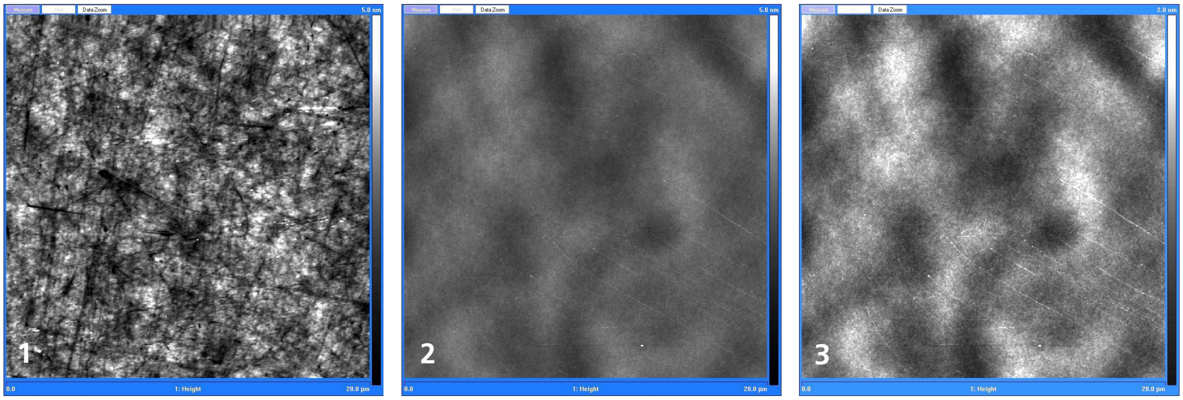

Fig. 8. AFM pictures with scan lengths of 20 µm x 20 µm of quartz glass surfaces before (picture 1) and after (pictures 2 and 3) ion beam figuring and multilayer coating. Pictures 2 and 3 show the same AFM scan just with different z scalings. The material removal by ion beam etching results in significant decreases of the microroughness: for scan length of 20 µm x 20 µm from 0.74 nm rms to 0.26 nm rms and for scan length of 1 µm x 1 µm from 0.52 nm rms to 0.10 nm rms.

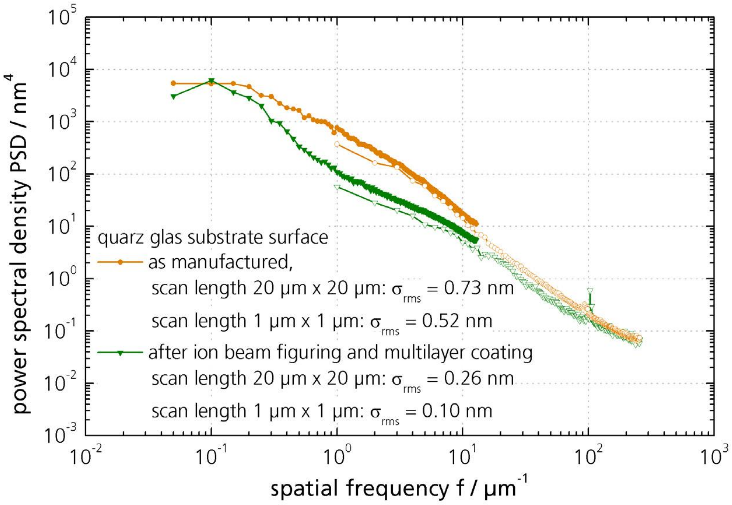

Fig. 9. Power spectral density of the surfaces shown in fig. 8. Applying figuring processes with higher ion energies and etch durations in the order of ~10 h, spatial frequencies in the range of 0.3 – 20 µm^{-1} can significantly be reduced.

3.3 Multilayer deposition

Final step of the fabrication of x-ray optical mirrors is the deposition of the reflective multilayer coating. In our case the same ion beam machine can be used for both, the ion beam pre-treatment of the substrates and the ion beam deposition itself. Advantages are the possibility to produce tailored mirror figures with appropriate multilayer coatings on top and secondly to avoid contamination of the substrate between figuring, smoothing and deposition.

A prominent material system for Cu-Kα optics is Ni/C because of the high reflectance and rather low divergence of the reflected beam and the possibility to suppress Cu-Kβ radiation of the laboratory source. Best reflectances of > 90 % at d-spacings around 4 nm can be achieved by ion beam deposition on superpolished silicon substrates. In this case the initial substrate roughness is in the range of 0.1 nm rms or better.

Fig. 10 shows a comparison of the XRR reflection patterns of the same Ni/C multilayer on a flat silicon substrate and a quartz glass substrate figured and coated with the dual ion beam machine. Intensity and shape of the Bragg reflections are very similar, also the decay of the maximum values for the higher Bragg orders. The optical performance of the multilayer on the figured quartz glass coating should therefore be comparable to that of the multilayer on superpolished silicon wafers. Differences can be seen in between some Bragg reflections and at higher angles in the background noise. These effects result from different interlayer on the substrate surface and the more intense backscattering of the quartz glass substrate.

For shaped collimating or focusing mirrors the reflective multilayers have to be deposited with certain thickness profiles along the substrate length to fulfill the Bragg condition for constructive interference. The deposition of these lateral gradients were done similar to the figuring by using of certain slits and velocity profiles of the substrate above the slits. For details of graded multilayer deposition with the dual ion beam machine see ref. [18].

In fig. 11 the resulting Bragg angles of a graded Ni/C multilayer at different mirror positions are shown. The corresponding period thicknesses were calculated and are displayed on the right hand axis. The thickness gradient follows an elliptical behavior, even if the resulting elliptical parameters of the best fit (dashed line) differ somewhat from the intended ones (solid line). Although this deposition has not the optimum gradient for the prefigured elliptical mirror (see fig. 7) the deviations of the Bragg peaks are less then 0.01° from the ideal case. Because of the width of the Bragg peaks of around $\Delta\Theta = 0.03°$ (FWHM) this deposition should be of sufficient quality with some limitations in the optical performance (integral reflectance, divergence, spot size). This (first) experimental result demonstrates furthermore the need of precise and reproducible deposition techniques in case of prefigured mirrors.

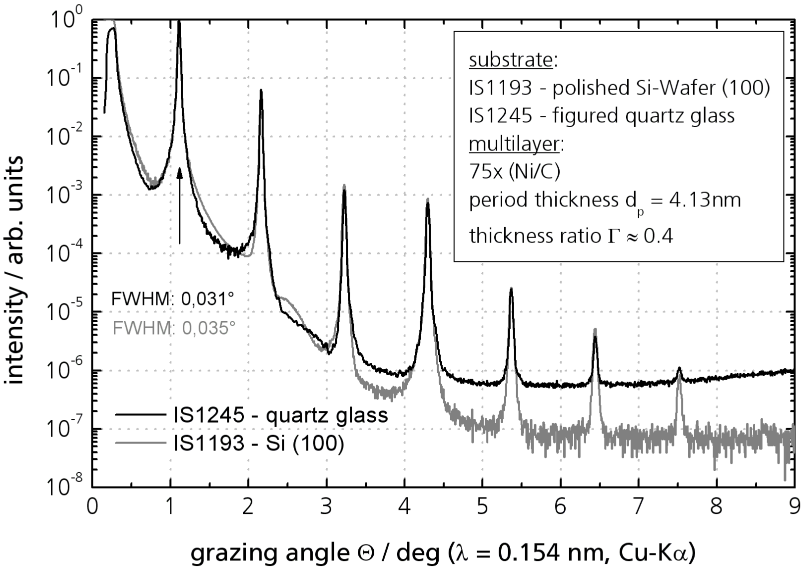

Fig. 10. Comparison of the reflection patterns of two identical multilayers on a figured quartz glass and on a flat 100-silicon wafer.

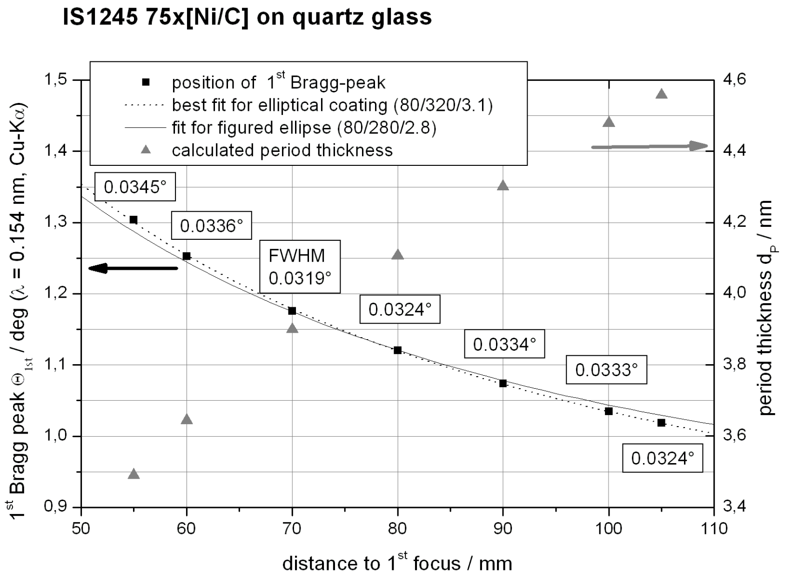

Fig. 11. Measured thickness profile of a Ni/C-multilayer on an elliptical prefigured quartz glass. The positions of the first order Bragg reflections fit quite well to the theoretical line for an elliptical gradient. The width of the reflections are noted for each point and are in the range of 0.033° (theta). This value is typical for this kind of multilayer and defines the accuracy needed during pre-figuring of the substrate and the deposition of the multilayer.

3.4 X-ray optics testing

For the performance test of our multilayer mirrors the Siemens D5005 and the Bruker AXS D8 have been used. For the testing of the one-dimensional mirrors (Göbel mirrors) the D5005 has been operated with the line focus of a size of approximately 0.04 mm x 10 mm. The experiments with the two-dimensional beam shaping mirrors (Montel geometry and single-reflection mirror) the D8 has been used with the point focus of a size of approximately 0.4 mm x 1 mm.

Preliminary results of one-dimensional parallel beam mirrors are shown in tab. 1. It is shown that the reflected intensity of the ion beam figured mirrors is somewhat higher in comparison to that one obtained by a standard Göbel mirror (bended silicon type). Using an alternative material system for the reflective multilayer with broader Bragg reflections (e.g. W/Si) can result in even higher reflectance (and higher beam divergence).

Measurements of the divergence of the reflected beam as well as the quantitative characterization of the intensity and the spot size of 1-dimensional and 2-dimensional mirrors are underway and should be publishes in the near future.

Tab. 1. Comparison of beam intensities obtained with different parallel beam optics. The results show that ion beam figured parallel beam optics deliver comparable x-ray intensities like the reference mirrors produces by bending.

Mirror type	*Multilayer*	*Slit configuration*	*Intensity / arb. units*
Silicon bended, reference	Ni/B_4C	1 mm / XRM + absorber	186
		1 mm / XRM / 1 mm + absorber	165
Quartz glass, ion beam figured	Ni/B_4C	1 mm / XRM + absorber	205 (+10 %)
		1 mm / XRM / 1 mm + absorber	185 (+12 %)
Quartz glass, ion beam figured	W/Si	1 mm / XRM + absorber	230
		1 mm / XRM / 1 mm + absorber	207

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