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ABSTRACT
The effect of ion polishing in sputter deposited W/Si multilayer mirrors with a d-spacing of 2.5 nm was studied. 0.1 to 0.5 nm of Si were etched with 100 eV Ar+ ions. This process resulted in a pronounced reduction in diffused scattering, measured at wavelengths about 0.1 nm. However, CuKa X-ray specular reflectivity and AFM showed only a marginal reduction of the roughness amplitude in the systems. Furthermore, the soft X-ray reflectivity at 0.84 and 2.4 nm did not show any changes after the ion polishing as compared to the nonpolished structures. Grazing incidence X-ray reflectivity (GIXR) analysis revealed that there was no pure W present in the deposited multilayers, with WSi2 being formed instead. As a result, it was concluded that the initial roughness in W/Si multilayers grown by magnetron sputtering is not the major factor in the reflectivity deviation from the calculated value for an ideal system. Nevertheless, the grazing incidence small-angle X-ray scattering (GISAXS) analysis revealed that ion polishing reduces the vertical propagation of roughness from layer to layer by a factor of two, as well as favorably affecting the lateral correlation length and Hurst parameter. These improvements explain the reduction of diffused X-ray scattering at 0.1 nm by more than an order of magnitude, which is relevant for applications like high resolution XRD analysis.

I. INTRODUCTION
Multilayers with nanoscale thick layers are used in a large variety of applications, for the reason that they can be tuned to a specific wavelength and working angle required for a particular task. One of the applications is X-ray fluorescence spectroscopy, where multilayers serve as analyzing crystals to resolve fluorescence emission lines from the investigated material. In this work, such analyzers are considered to resolve spectral lines of O Ka to Al Ka (525 eV–1486 eV) in order to produce a qualitative analysis of materials in this range. This corresponds to the range of wavelengths 0.84–2.4 nm. The focus in this work is on W/Si multilayers prepared by magnetron sputtering, which have a high reflectivity at these wavelengths. Multilayers are designed following Bragg’s law in order to reflect the incident radiation by constructive interference. Therefore, to reflect at 0.84 nm and 2.4 nm wavelengths at specific angles of relevance for the application (9.7° and 29.5° grazing incidence, correspondingly), a period of the multilayer of 2.5 nm is required. Apart from the optical indices of materials used, the reflectivity is determined by the quality of the interfaces between the layers. The shorter the period, the more sensitive the reflectivity is to imperfections at the interfaces. Interdiffusion and interfacial roughness are two limiting factors to achieve the maximum reflectivity. For instance, Liu et al. studied interface formations in W/Si and WSi2/Si multilayers where they showed that interdiffusion between W and Si in W/Si multilayers leads to a formation of less sharper interfaces than in WSi2/Si multilayers.1,2 One of the advantages of magnetron sputtering is that it can inherently produce low interfacial roughness. However, even a relatively low roughness might affect reflectivity of short period multilayers. Moreover, intermixing of the elements can contribute to an additional interfacial roughness formation, which will further reduce reflectivity. Therefore, even the multilayers produced by magnetron sputtering might need an additional roughness control. Ion beam polishing...
was reported to be an effective tool to reduce interfacial roughness in multilayers and single surfaces.\(^3\)–\(^4\) Soyama et al.\(^5\) reported about a strong reduction of the interfacial roughness from 0.7 nm to 0.35 nm in Ni/Ti multilayers deposited by ion beam sputtering with a period \(d = 12 \text{ nm}\) after applying an ion beam polishing of 100 eV after deposition on either a Ni or Ti layer. Another successful experience of ion polishing was demonstrated by Puik et al.\(^6\) for W/C and Ni/C short period \(d = 7.4 \text{ nm}\) multilayers. Metal layers of W and Ni were deposited by e-beam evaporation and etched afterwards using Ar\(^+\) ions at 200–300 eV energies. Etching a thickness of 2.8 nm of metal layers resulted in a significant gain in reflectivity detected by an \textit{in situ} soft X-ray monitoring system. Spiller\(^7\) obtained an increase in the reflectivity from 8\% to 16\% at \(\lambda = 4.8 \text{ nm}\) by using ion beam polishing in Rh-C, and around the same gain from 9\% to 20\% at \(\lambda = 4.78 \text{ nm}\) in ReWCo-C multilayer mirrors deposited by electron beam evaporation for normal incidence X-ray telescopes. Ar\(^+\) ions with energies of 300 eV and 500 eV were used to polish Rh-C with \(d = 6.17 \text{ nm}\) and ReWCo-C with \(d = 3.64 \text{ nm}\) multilayers, respectively.

Ion beam polishing was reported to be an effective tool to reduce the interfacial roughness in Mo/Si multilayers with a period \(d = 7 \text{ nm}\).\(^1\)–\(^6\) In these works, about 4 nm thick Si was exposed to Kr\(^+\) ions with energies in the range of 100–2000 eV to achieve smoothening of interfaces. The etched thicknesses of Si were in the range from 0.12 to 0.75 nm. It was found that low energies in the range of 100–150 eV are more favorable for reducing interfacial roughness. W/Si multilayers with a period of 4.1 nm were studied by Kessels et al.\(^8\) who applied Kr\(^+\) ions on W and WRe layers with a thickness of 1.7 nm. The energy of 100 eV was found to be optimal to reduce interfacial roughness and increase the reflectivity of carbon-K radiation from 43\% to 65\% for 10-period stacks. Applying higher energies led to intermixing of layers and an increase of the interfacial roughness, hence a reduced reflectivity for all multilayers.

Thus, ion polishing was shown to be a useful tool to increase the reflectivity by reducing the roughness at the interfaces for multilayers having a relatively long period. The main goal of this work was to investigate the possibility of increasing the reflectivity of multilayers having a much shorter periodicity. We investigated the effect of ion polishing of Si in W/Si multilayer mirrors with a period of 2.5 nm, deposited by magnetron sputtering.

II. EXPERIMENT

A. Multilayer deposition

All multilayers were deposited on super-polished silicon wafers 25× 25 mm\(^2\) (RMS roughness ~ 0.14 nm) using a DC magnetron sputtering system with a base pressure of 1 × 10\(^{-9}\) mbar. Ar\(^+\) gas was used as a working gas at a pressure of about 1 × 10\(^{-4}\) mbar. The multilayers consisted of 200 periods with the period thickness \(d = 2.5 \text{ nm}\) and gamma values varying from 0.06 to 0.24. Every Si layer was polished with an ion source after its deposition, with the etched thickness being 0.1–0.5 nm. The ion source was mounted such that the ions arrive at the normal incidence with respect to the substrate surface. The energy of ions was chosen to be 100 eV in order to keep the penetration of ions into the layer as low as possible to avoid intermixing with the underlying layer, but at the same time practical to avoid an extended polishing process. The etching rate was calibrated by depositing stacks by varying the amount of etched Si. These stacks were measured with CuKa (\(\lambda = 0.154 \text{ nm}\)) reflectometry to determine the resulting periods and subsequently determine the etching rate. To avoid oxidation of the surface after exposure to the air, every multilayer was finished with a Si layer with the same thickness as in the rest of the structure. Depositions were performed at a power of 30 W for W and 260 W for Si. Such a low power for W was chosen to minimize the deposition rate of W in order to have a better thickness control over thin W layers. The uniformity was achieved by spinning the holder with substrates at 90 rpm during the deposition process (see Fig. 1 for the configuration of the deposition setup).

B. Multilayer characterization

The multilayers were characterized by \(\theta \sim 20^\circ\) grazing incidence reflectivity measurement at CuKa wavelength (\(\lambda = 0.154 \text{ nm}\)) using a Malvern Panalytical Empyrean diffractometer to determine the period. A hybrid monochromator consisting of a combination of an X-ray mirror and a 2-crystal Ge(220) 2-bounce monochromator was used.

For a fast qualitative assessment of multilayers, X-ray diffused scattering rocking curves were measured with a conventional X-ray tube at the wavelength of 0.154 nm, with the procedure of the measurement being described elsewhere.\(^1\) The rocking curves were taken at the second Bragg peak.
For quantitative analysis of X-ray diffused scattering, grazing incidence small-angle X-ray scattering (GISAXS) measurements were implemented. These measurements were carried out at the bending magnet beamline Langmuir of the synchrotron radiation source Siberia-2 at the Kurchatov Institute. Monochromatization at the beamline was arranged by a thermally stabilized two bounce Si monochromator with the (111) reflection. Higher harmonics of the monochromatized beam were suppressed with quartz and tungsten X-ray mirrors. The synchrotron beam was collimated with three sets of slits. The resulting beam size was 50 × 200 μm², with the corresponding average direct beam intensity being approximately 2 × 10⁷ cps. The vertical beam divergence was 4 arc sec and the horizontal divergence was 20 arc sec. The diffuse scattering intensity was measured with a Pilatus100 k 2D detector. Measurement settings for both samples (sample with and without ion treatment) were identical. The measurements were taken at the wavelength \( \lambda = 0.1 \) nm in three exposures of 15 min each, in order to avoid detector saturation.

The angle of incidence was set to \( \theta_0 = 0.3° \), in between the total external reflection and the first Bragg peak. The incident synchrotron beam intensity was monitored with an ionization chamber FMB Oxford IC plus 50.

To measure the top surface roughness of the samples, a Bruker Dimension Edge™ Atomic Force Microscope (AFM) was used. A high resolution tip HiRes-C14/Cr-Au by MikroMasch was used (radius of the tip was \( \sim 1 \) nm).

Soft X-ray reflectivity at specific wavelengths and grazing angles of incidence required for the application (0.84 nm, \( \theta \approx 9.7° \) and 2.4 nm, \( \theta \approx 28.7° \)) were measured at the storage ring BESSY II at Physikalisch-Technische Bundesanstalt (PTB) in Berlin-Adlershof, Germany. Since the reflectivity was measured at the grazing incidence angle, a 0.5 mm exit slit on the monochromator was used in order to reduce the size of the beam footprint on a sample.

### III. RESULTS

#### A. Diffuse scattering

To determine the impact of ion polishing on the roughness for the deposited structures, x-ray diffuse scattering at CuKa wavelength (\( \lambda = 0.154 \) nm) was measured by means of the rocking curve scan. This measurement is done by fixing the detector at the center of the Bragg reflection and rocking the sample in a wide angular range. A perfect structure will produce a very sharp peak in the specular direction only, while a structure with diffused scattering will show wide wings around it. The rocking curve measurements from multilayer structures are described in more detail elsewhere. It is common to measure diffuse scattering at higher orders to observe a wider range of angles; however, due to the low intensity of the high-order Bragg peaks, all scans in this work were performed at the second Bragg peak of 3.2° (2\( \theta = 6.4° \)). Figure 2 shows the rocking curve scans for the multilayers deposited with and without ion polishing. The peaks at 3.2° correspond to the specular reflectivity at the second Bragg order, with the diffused scattering being on the left and right sides of the specular peak.

As seen, a substantial decrease in diffuse scattering is observed as the amount of etched Si during the ion-polishing process increases. This implies that the interfacial roughness was affected significantly by the polishing process.

The integral diffuse scattering does not differ significantly between the samples with 0.3 nm and 0.5 nm of Si etched that indicates that the effect of ion polishing reaches saturation at 0.3 nm. For this reason, we proceeded with a 0.3 nm ion polishing for further analysis.

#### B. Grazing incidence X-ray reflectivity

In Fig. 3, grazing incidence X-ray reflectivity (GIXR) at CuKa wavelength \( \lambda = 0.154 \) nm of the initial (unpolished) and 0.3 nm polished W/Si multilayers deposited with and without ion polishing is shown. The enhanced reflectance at high orders indicates an improvement in the structures with ion polishing.
polished multilayers are shown. The main difference between the two multilayers is that the high-order peaks are more pronounced in the multilayers after polishing, which usually indicates a reduced roughness, which is in line with the diffused scattering measurement shown in Sec. III A that suggests reduced roughness.

C. Soft X-ray reflectivity

Simulations in IMD\textsuperscript{17} show that the optimum gamma value \( \Gamma = \frac{d_W}{d_W + d_{Si}} \) for the maximum reflectivity of W/Si multilayers at 0.84 nm wavelength and the angle of incidence \( \theta \approx 9.7^\circ \) is \( \Gamma = 0.23 \). For an ideal multilayer (no interfacial roughness), the maximum reflectivity is 60%. It was reported that W and Si tend to form a silicide \( W_xSi_x \) in W/Si multilayer structures, which means that certain changes in the period and thus in gamma values occur during deposition, depending on the amount of deposited materials.\textsuperscript{7,13} Therefore, in order to find the optimum gamma value resulting in the maximum reflectivity in our experiments, a series of multilayers in the range of gamma from 0.06 to 0.24 was deposited for every set of multilayers with polished and unpolished Si layers. Figure 4 shows the reflectivity for these structures at 0.84 nm and at the angle of incidence \( \theta \approx 9.7^\circ \). As seen in Fig. 4(a) the optimum gamma value obtained was \( \Gamma = 0.12 \), which is about a factor of two smaller than in our IMD calculations. Furthermore, the obtained maximum reflectivity did not differ for both types of structures.

The multilayers were also measured at 2.4 nm and showed the same dependency of reflectivity on gamma value. The same maximum reflectivity of 9.5% was obtained at 2.4 nm for the unpolished and polished structures at a gamma value of 0.12. This way both soft x-ray measurements showed that the applied ion polishing did not result in any improvements in reflectivity. Spiller et al.\textsuperscript{18} reported that implantation of Ar\textsuperscript{+} species in Si layers could reduce the reflectivity in their work. Similarly, the presence of Ar\textsuperscript{+} could counterbalance the possible reflectivity gain in our polished multilayers. However, XPS analysis did not reveal the presence of Ar\textsuperscript{+} in our samples.

Further analysis was needed to understand the differences in the results obtained at 0.154 nm, 0.84 nm, and at 2.4 nm. For that, the studied structures were measured with AFM and GISAXS, which allowed us to extract parameters of roughness quantitatively.

D. Atomic force microscopy

In Fig. 5, a comparison of AFM measurements is shown for both the unpolished and polished structures with 0.3 nm etched Si. Both surfaces were measured several times at different spots for better statistics. The rms roughness value for the unpolished sample was \( r_q = 0.134 \) nm with a standard deviation of 0.01 nm. The rms roughness value for the polished sample was \( r_q = 0.116 \) nm with a standard deviation of 0.008 nm. This way the AFM measurements confirmed some reduction in the roughness of the polished
structures, although the difference in the surface roughness was found to be only about 0.02 nm.

E. Grazing incidence small-angle X-ray scattering

AFM allows for direct measurements of the roughness of the outermost surface of the structure. However, the surface is oxidized after exposure to the atmosphere; consequently, the morphology of its roughness is different from that of the inner interfaces. Therefore, imperfections of the multilayer cannot be characterized in full by the sole use of AFM. For the characterization of the interface roughness, grazing incidence small-angle X-ray scattering (GISAXS) has been employed.

The GISAXS method is based on the measurement of the diffuse scattering of the incident radiation. The GISAXS measurements for not polished (a) and polished (b) W/Si multilayers are shown in Fig. 6 at a logarithmic scale. In Fig. 6, one can note distinct features: sets of maxima which are commonly referred to as Bragg sheets in the literature. The nature of the Bragg sheets is attributed to the scattering on the correlated roughness. This scattering produces coherent superposition of the scattered radiation which subsequently diffracts on the multilayer structure, resulting in the Bragg sheets. In the opposite case of the not correlated roughness, radiation scatters with a random phase. No diffraction from the multilayer structure appears in the scattered radiation in that case. The shape of the Bragg sheets is dependent on the statistical parameters of the interface roughness, as opposed to AFM which allows for direct roughness measurements but only for the outermost surface of the structure.

The interface roughness in multilayers can be represented as a self-affine surface. Within that approach, the interface roughness morphology is defined with the following parameters: $\xi_v$ is the vertical correlation length, $\xi_l$ is the lateral correlation length, and Hurst parameter $H$. The lateral correlation length is the averaged characteristic size of the interface roughness in the lateral direction. The vertical correlation is the effective depth within which the shape of roughness is maintained from an interface to an interface. The Hurst parameter defines the jaggedness of the interface. Limiting cases: $H \to 1$ corresponds to a smooth roughness profile, while $H \to 0$ corresponds to a jagged roughness profile. Concise explanation of the physical meaning of these parameters is given in Ref. 23. Qualitatively, a decrease of lateral correlation leads to a broadening of the Bragg sheet in the $q_x$ direction. Similarly, the increase of the vertical correlation leads to the broadening in the $q_x$ direction. It is apparent in Fig. 6 that the polished sample has a bigger lateral correlation length than the not polished one because of its narrower Bragg sheets. We estimated statistical parameters of the interface roughness using an approach, similar to what was used in Ref. 19. The vertical correlation length was estimated using the equation $\xi_v = 2\pi \sigma_w$, where $\sigma_w$ is a full width half maximum (FWHM) parameter. The FWHMs were analyzed by fitting line extractions of the second Bragg peak along the $q_x$ axis taken in $q_x = 0$, see Figs. 7(a) and 7(b). The second Bragg peak was chosen due to the low penetration depth of X-rays at the first Bragg peak. These line extractions were fitted using the pseudo-Voigt profile, which is conventionally used in spectroscopy and diffraction data analysis. Thus, the FWHMs of the second Bragg peak were estimated.

The parameters of Hurst and lateral correlation length were estimated by fitting line extractions of the second Bragg sheet taken along the $q_x$ axis [see Figs. 7(c) and 7(d)]. These line extractions were fitted with the use of a K-correlation model. There, the correlation function is given by

$$C(r) = \frac{\sigma^H}{2^{H-1} \Gamma(H+1)} \gamma^H K_H(\gamma),$$

where $\gamma = r \sqrt{2H/\xi_v}$, $\Gamma$ is the gamma function, $K_H$ is the modified Bessel function of $H$-th order of the second kind, and $\sigma$ is the roughness rms amplitude. We approximated a corresponding power spectral density function as

$$P(q_x) \propto \int_0^\infty r^H J_0(q_x r) \exp(C(r) - 1) dr,$$

where $J_0$ is the 0-th order Bessel function of the first kind. Finally, we assumed that the scattering cross section in the region of line extraction along the $q_x$ axis in the second Bragg peak is proportional to the PSD function $\frac{dr}{d\Omega} = \Re P_0(q_x)$. This assumption imposes a restriction on the estimation of the absolute value of the roughness amplitude $\sigma$. Indeed, for the sufficiently small argument of exponent in Eq. (2), the diffuse scattering intensity is linearly scaled with the value of $\sigma^2$ (due to the Taylor expansion of exponent). Thereby, in this model, the intensity of the incident beam $I_0$ is strongly correlated with $\sigma$. However, due to the normalization on the incident beam intensity, we can cross-compare roughness amplitudes of the measured samples. We estimate $\sigma_1 = 0.28$ nm (without ion polishing) and $\sigma_2 = 0.21$ nm (with ion polishing). These are effective parameters of roughness amplitude, nevertheless, we can conclude that the parameter of roughness changed insignificantly due to the polishing procedure. The results of the fit are presented in Table 1.
Intensities of the diffuse scattering on the sample prepared with ion assistance were lower; therefore, uncertainties of the parameters for this sample are higher.

Finally, it should be noticed that in Fig. 6(b) an enhancement of the scattering outside the Bragg sheets can be seen. This effect is more apparent in Fig. 8.

In Fig. 8, line extractions along $q_x$ directions averaged in between Bragg sheets in the range between $q_z = 3.4 \text{ nm}^{-1}$ and $q_z = 3.7 \text{ nm}^{-1}$ for both the polished and not polished samples are shown. Line extraction for polished samples have maxima at $q_x \approx \pm 0.5 \text{ nm}$. The peaks at $q_x \approx \pm 0.5 \text{ nm}$ correspond to the distribution of density fluctuations with the mean neighbors distance of about 10 nm. In our recent work, we showed that this scattering is associated with slight density fluctuations of Si in W/Si multilayers where the Si layers were polished with Ar$^+$ ions. Line extraction for the not polished sample in Fig. 8 has no visible peaks outside the center $q_x = 0$, which indicates the absence of the density fluctuations in this sample. Note that similar density fluctuations were also observed inside the Ge layer in Fe/Ge multilayer structures and were attributed to the trapping of Ar$^+$ inside the Ge layer. Both observations indicate that Ar$^+$ ion treatment may stimulate the formation of density fluctuations inside the Si layers that can be studied further in more details.

### F. Free-form analysis of GIXR

To obtain additional information about the multilayer structure from GIXR curves presented in Fig. 3, we performed a model-independent (free-form) reconstruction of the structural profile, using the approach described in Ref. 28. Compared to

![Fig. 7. Line extractions calculated from Fig. 6: (a) and (b) are the line extractions for the second Bragg peak along the $q_z$ axis taken at $q_x = 0 \text{ nm}^{-1}$ for the unpolished and polished multilayer, respectively (red curve is a measurement, blue curve is a fitting including error bars); (c) and (d) are the line extractions for the second Bragg peak along the $q_x$ axis for the unpolished (taken at $q_z = 4.9 \text{ nm}^{-1}$) and polished multilayer (taken at $q_z = 4.7 \text{ nm}^{-1}$), respectively (red curve is a measurement, blue curve is a fitting including error bars).](image)

![Fig. 8. Line extractions in between Bragg sheets along the $q_x$ direction averaged in between Bragg sheets in the range between $q_z = 3.4 \text{ nm}^{-1}$ and $q_z = 3.7 \text{ nm}^{-1}$.](image)

| TABLE I. Results of the line extraction fit. |
|---------------------------------------------|
| Not polished | Polished |
| $\xi_v$ (nm) | $92.9 \pm 0.9$ | $36.5 \pm 2$ |
| $\xi_l$ (nm) | $10 \pm 1$ | $25 \pm 2$ |
| $H$ | $0.54 \pm 0.01$ | $0.76 \pm 0.04$ |
| $\sigma$ (nm) | $0.28$ | $0.21$ |
conventional methods of fitting XRR data, it allows for more freedom in modeling an interface profile, which is crucial for very thin multilayer structures where an interface profile width is comparable to the period thickness.

For treatment of GIXR curves from Fig. 3, the periodic part of the multilayer was divided into 13 sublayers, with the top layer being treated separately. The reconstruction started from a steplike profile and finished in about 200 steps with the results shown in Fig. 9. As can be seen from Fig. 9(a), the fit quality is good, with all the Bragg peaks and the shape of the XRR curve in between being properly described. The results of reconstruction are shown as delta-profiles where \( \delta = 1 - \text{Real}(n) \), \( n \) is an optical constant. As seen, the shape of the interface profiles between W and Si layers is almost identical in both samples. However, the width of the interface profile for the polished sample is slightly smaller. This can be attributed to somewhat lower roughness in the polished sample. It is important to note that this reconstruction also allows us to conclude that Ar\(^+\) ion bombardment of thin Si layers at 100 eV energy did not result in the ion penetration through the Si layer. Clearly, no ballistic intermixing takes place with the underlying W layer as a result of the applied ion beam polishing.

An interesting observation can be made about the composition of the W layers. The delta value for pure W based on the tabulated values\(^{28}\) is \( 4.6 \times 10^{-5} \). However, the delta value of the highest density material in the stack was found to be about \( 2.4 \times 10^{-5} \), which corresponds to a W fraction of only 44%. It means that there is no pure W material within the multilayers. The obtained delta value is close to the value of WSi\(_2\), which is a consequence of the interaction between W and Si resulting in the formation of silicide as it was described by Refs. 7 and 13.

**G. IMD model simulation**

The model-independent approach resulted in a smooth delta profile that effectively describes the interface profile but does not allow to discriminate between the contribution of roughness and intermixing individually. Nevertheless, the carried out reconstruction could now be used as an input for some further estimations. Based on the reconstructed density profile, a two-layer model WSi\(_2\)/Si (\( d = 2.5 \) nm, \( N = 200 \), and \( \Gamma = 0.33 \)) was used to fit our reflectivity data at 0.154 and 0.84 nm with IMD to estimate the roughness values before and after applied polishing. A parameter of the Debye-Waller factor was used to take into account the roughness values at interfaces. The obtained results (Fig. 10) are in a good agreement with experimental data: fittings at 0.154 nm look relatively good and reflectivity at 0.84 nm match the experimental reflectivity of 40%. Changes of the interface widths from \( \sigma_{\text{WSi}_2/\text{Si}} = 0.23 \) nm and \( \sigma_{\text{Si}/\text{WSi}_2} = 0.2 \) nm to \( \sigma_{\text{WSi}_2/\text{Si}} = 0.22 \) nm and \( \sigma_{\text{Si}/\text{WSi}_2} = 0.18 \) nm explain the high-order changes at 0.154 nm without affecting reflectivity in the soft x-ray range.

**IV. DISCUSSION**

We observed a substantial reduction in diffused scatter in the 0.154 nm X-ray rocking curve measurements of the multilayers.
when polishing the Si layers. From that, it could be expected that the roughness of the multilayers was reduced essentially as a result of the polishing process. The specular reflectivity from these multilayers was qualitatively in agreement with the diffuse scatter measurement results. However, the reflectance of these multilayers at soft X-rays of 0.84 nm and 2.4 nm does not show any improvements. The highest reflectivity achieved was 40% and 9.5%, respectively, regardless of whether the multilayers received Si ion treatment or not, with the calculated maximum reflectivity for ideal structures to be 60% and 20.5%, correspondingly. Further AFM measurements of the multilayer surfaces suggested only a marginal smoothening effect for the polished structures of about 0.02 nm.

It is necessary to explain why so insignificant changes in roughness resulted in such a substantial reduction in 0.154 nm diffuse scattering. Subsequent 0.154 nm GISAXS measurements allowed us to estimate parameters of roughness quantitatively. The lateral correlation length increased from 10 nm to 25 nm, and the Hurst parameter increased from 0.54 to 0.76 as a result of Si layer polishing. The effective parameters for roughness amplitude were estimated to be: $\sigma_1 = 0.28 \text{ nm}$ (without ion polishing) and $\sigma_2 = 0.21 \text{ nm}$ (with ion polishing). This indicates some smoothening effect, although these changes are indeed insignificant. More importantly, it was found that polishing also resulted in a substantial reduction in the vertical correlation length from 92.9 nm to 36.5 nm. This reduction means that the constrictive interference of diffused scattering from interfaces was significantly reduced. According to our simulations in IMD with the data obtained from GISAXS, the changes in the roughness parameters could explain the strong reduction in the diffused scattering observed in the 0.154 nm X-ray rocking curve measurements. Consequently, we conclude that the amplitude of the initial roughness in W/Si multilayers grown by magnetron sputtering is not sufficiently high to be further reduced by the applied ion polishing. Furthermore, this roughness did not seem to be the major factor resulting in a reflectivity loss in the deposited multilayers. Trying to identify this factor, a model-independent X-ray analysis was applied to the measured GIXR curves from both the unpolished and polished multilayers. The reconstructed refractive index profiles suggested that the W layers in the deposited multilayers do not reach bulk density. In fact, effectively only the presence of 44% of bulk W density could explain the observed refractive index of the reflective layer. If we presume that the reduced density is due to a formation of a compound between W and Si, notably the formation of WSi2, this reduction in density could explain the observed refractive index of the reflective layer. As a result, we conclude that the initial roughness in W/Si multilayers grown by magnetron sputtering is not high enough to be further reduced by ion polishing.

FIG. 10. The measured and fitted reflectivity of unpolished (a) and polished (b) W/Si multilayers at 0.154 nm (left) and 0.84 nm (right). WSi2/Si model was used for the fit. The parameters were: $d = 2.5 \text{ nm}$, $N = 200$, and $\Gamma = 0.33$. The roughness values (Debye-Waller factor) $\sigma_{WSi2}/Si = 0.23 \text{ nm}$ and $\sigma_{WSi2}/Si = 0.2 \text{ nm}$ for the unpolished structures, and $\sigma_{WSi2}/Si = 0.22 \text{ nm}$ and $\sigma_{WSi2}/Si = 0.18 \text{ nm}$ for the polished structures. The dip in reflectivity below $\theta = 1^\circ$ is caused by oxidation of the surface. Surface oxidation only affects the reflectivity between the Bragg peaks and does not affect the reflectivity from the periodical part of the multilayer.
is an important mirror property, Si ion polishing can reduce the fraction of scattered radiation by more than an order of magnitude.

V. CONCLUSIONS

Previously, ion polishing was shown to be a useful tool to increase reflectivity by reducing roughness at the interfaces for multilayer soft X-ray reflectors with period thicknesses above 3.5 nm. The main goal of this work was to investigate a possibility to reduce roughness and thus increase the reflectivity of shorter, 2.5 nm period, W/Si multilayers. The multilayers were deposited by magnetron sputtering and were polished with 100 eV Ar+ ions for every Si layer directly after its deposition, with the amount of etched Si being varied from 0.1 to 0.5 nm.

The procedure resulted in a pronounced reduction in diffused scattering measured with rocking scans at 0.154 nm and with GISAXS at 0.1 nm. Analysis of GISAXS revealed a strong reduction of the vertical interfacial roughness correlation length from 91 nm to 36 nm. Additionally, the lateral correlation length and the Hurst parameter were improved. The lateral correlation length increased from 10 nm to 25 nm, and the Hurst parameter increased from 0.54 to 0.76. The effective parameters of the roughness amplitude reduced from 0.28 to 0.21 nm. AFM measurements showed only a marginal reduction in the surface roughness of about 0.02 nm for the polished multilayers.

Soft X-ray reflectivity at 0.84 and 2.4 nm does not show any changes after ion polishing. A maximum reflectivity of 40% at 0.84 nm (AOI = 9.7° grazing incidence) and 9.5% at 2.4 nm (AOI = 29.5° grazing incidence) was achieved.

Free-form analysis of 0.154 nm GIXR measurements revealed there is no pure W present in the deposited multilayers. The obtained delta value of the optical index of the highest density material in the stack corresponded to a WSi2 compound. Based on this, a two-layer model of WSi2/Si (d = 2.5 nm, N = 200, Γ = 0.33) was used in IMD to successfully fit both our reflectivity data at 0.154 and 0.84 nm. The small change in the roughness amplitude of 0.015 nm could explain high-order changes for the X-ray reflectivity at 0.154 nm, while not affecting the reflectivity at 0.84 and 2.4 nm.

The initial goal of this work was to increase the reflectivity of W/Si multilayers deposited by magnetron sputtering applying ion beam polishing. However, 100 eV Ar+ ion polishing of Si in W/Si multilayers did not result in a significant reduction of the interfacial roughness due to the already small roughness present in the initial (unpolished) structures. However, roughness characteristics like the vertical and lateral correlation lengths and the Hurst parameter were significantly improved, which resulted in a reduction of diffused scattering by more than an order of magnitude at 0.1–0.15 nm. Such a strong reduction of diffuse scattering is very beneficial, for example, for applications of a multilayer mirror as a collimator or focusing element, e.g., for high resolution X-ray diffraction analysis.

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