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Control of surface mobility for conformal deposition of Mo-Si multilayers on saw-tooth substrates

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Abstract

Multilayer-coated Blazed Gratings (MBG) with high groove density are the most promising solution for ultra-high resolution soft x-ray spectroscopy. Unlike traditional grazing incidence x-ray gratings, MBGs can provide a much higher spectral resolution while maintaining good efficiency, due to the three dimensional nature of the diffraction process that allows use of a high groove density and high spectral order. The performance of MBGs however depends on the conformal deposition of the multilayer (ML) stack on a saw-tooth substrate, which results in special requirements for the deposition process. The ML needs to be grown conformally to preserve the saw-tooth shape of the grating grooves through typically hundreds of interfaces, while maintaining near atomic-scale interfacial roughness. Such growth on flat substrates has been largely perfected, but here, the deposition process is complicated by the presence of the periodic variation of the surface height of the grating substrate. Coatings deposited by conventional processes tend to smooth out the grooves, which results in degradation of the blazing ability of a MBG and consequent loss of diffraction efficiency. The deposition process has to be reconsidered and deposition methods need to be optimized to provide perfect replication of the grooves by the ML interfaces, at the same time avoiding significant roughening of these interfaces. In this work we present an analysis of the roughening and smoothing processes during growth of Mo/Si multilayers deposited over a range of pressures of Ar sputtering gas. The MBG coated under the optimal deposition conditions demonstrated high diffraction efficiency in the EUV and soft x-ray wavelength ranges. A Linear Continuum Model (LCM) of film growth allows us to understand the interplay between smoothing and roughening of interfaces on a grating surface and may be used to predict the optimum conditions for deposition.

Keywords: EUV, soft x-rays, diffraction grating, multilayer, film growth, continuum growth model, surface diffusion

1. Introduction

Fabrication of multilayer mirrors for EUV, soft and hard x-rays is currently a well established process. Key applications of multilayer mirrors such as EUV lithography, x-ray astronomy and spectroscopy, and synchrotron beamline optics require flat or slightly curved surfaces with a very low figure error (< 1 μrads), and with high reflectivity, and therefore demands interfacial roughness less than typically 0.3 nm. A number of deposition methods such as dc-magnetron sputtering, e-beam evaporation, ion-beam sputtering, combined with sophisticated technological adaptations such as ion-assisted deposition, deposition followed by a partial sputtering of layers, and application of diffusion barriers are aimed at providing high quality interfaces of the multilayer stack, a pre-requisite for high reflectivity. Whatever deposition technique is used, it follows a common strategy. A deposition process should provide an effective relaxation of high frequency interface roughness generated during the deposition due to stochastic time-space variations of the deposition flux, polycrystalline structure of a film, shadowing effects etc., and at the same time prevent or reduce intermixing of the materials at the interfaces. Bulk diffusion across the interfaces should be suppressed while surface diffusion along the interfaces should be promoted as much as possible. This approach was successfully implemented by a number of groups for flat or slightly curved substrates. As a result of these efforts, high quality multilayers have been developed with performance approaching theoretical limits [1].

However, the approach does not always work well for substrates which have a complicated surface relief. Deposition of the MLs on saw-tooth substrates which are of great importance for dense Multilayer-coated Blazed Grating (MBG) [2,3] applications is extremely challenging. A Fourier spectrum of a saw-tooth surface contains high order harmonics which are of the same frequency as the interfacial roughness. Since traditional deposition techniques aim to suppress high frequency imperfections of a surface, the high Fourier harmonics of the saw-tooth profile are affected by the smoothing as well. As a result, the highly corrugated surface of a saw-tooth substrate undergoes smoothing by multilayers deposited on it [4-6]. This dramatically affects the blazing performance of a MBG resulting in a significant reduction of diffraction efficiency.

To avoid groove profile degradation, a new deposition methodology needs to be developed specifically for MBG applications. We reported recently [7] on improvement of the diffraction efficiency of a MBG by deposition of an optimized Mo/Si multilayer on a saw-tooth substrate with a groove density of 5250 lines/mm. In this paper, we present our ML deposition optimization procedure. A detailed study of surface morphology and internal structure of the MLs has been performed and optimal deposition conditions have been found. As a result of this optimization, a record diffraction efficiency in the EUV and soft x-ray spectral ranges was achieved.

2. Experiment

2.1 ML deposition and characterization

Mo/Si multilayers composed of 30-40 pairs of Mo and Si layers and target d-spacing of 6.63 nm were deposited by dc-magnetron sputtering in an Ar gas pressure varied within a range of 1-5 mTorr in the multilayer coating facility at the Center for X-ray Optics at LBNL. The deposition of the layers was performed by continuous motion of the substrates over the magnetron sources at a source/substrate distance of 75 mm. Specially shaped apertures placed in between the sources and the substrates provided high uniformity of the layer thickness over the substrate area. The velocity of the motion and the power of the sources were adjusted to obtain the desired thickness of the layers.

The MLs were deposited on prime quality Si wafers which served as flat reference substrates and saw-tooth gratings were fabricated with an anisotropic etching technique as described in Si [8-10]. The triangular groove gratings had a period of 190 nm with working facets tilted at an angle of 2º with respect to the grating plane. The height of 6.63 nm of the saw-tooth grooves corresponded to the d-spacing of the multilayers providing a blazed condition for the 1st diffraction order of the MBG.

The surface of the substrates was extensively characterized before and after deposition with a Veeco Dimension 3100 atomic force microscope (AFM). Based on the AFM data, an analysis of the growth kinetics was performed using a Linear Continuum Model (see the section 2.2) in order to find an optimal growth regime for saw-tooth substrates.

Measurements of the reflectivity of the multilayers and diffraction efficiency of the MBGs were performed at beamline 6.3.2 of the Advanced Light Source [11]. The internal structure of the multilayer stacks was investigated with cross-sectional TEM.

2.2 Growth analysis with the Linear Continuum Model

To investigate processes of roughening and smoothing of the ML interfaces during the growth, a surface kinetics analysis has been performed. Since the multilayers considered in this paper had a very low surface roughness, the Linear Continuum Model (LCM) [12-14] can be used for the analysis. According to this model, the two-dimensional isotropic Power Spectral Density (PSD) function of the top surface of the coating (PSD_{top}) can be expressed as:

$$PSD_{top} = PSD_{film} + a\ PSD_{sub} \tag{1}$$

$$PSD_{film} = \Omega \frac{1-a}{\sum 2\nu_n f^n}, \tag{2}$$

$$a = \exp(2d\sum_n \nu_n f^n), \tag{3}$$

where PSD_{sub} is the power spectral density of the substrate. The replication factor, a, characterizes smoothing of a substrate relief by the film, and PSD_{film} characterizes the intrinsic roughening/smoothing dynamics of the film surface during the growth process. The replication factor is a spatial frequency (f) dependent function, and a characteristic of the dependence is defined by a relaxation exponent, n, which can range from 1 to 4 for different relaxation mechanisms [12,15,16]. The contribution of a particular relaxation mechanism is characterized by strength parameter, ν_n, and depends on the film thickness, d.

Due to the random nature of a deposition process, stochastic roughening occurs during the growth. This depends on the thickness of a coating and the volume of deposited particles, Ω, which in the simplest case of an amorphous film growth corresponds to the atomic volume of the deposited material [13,14]. Non-stochastic roughening caused by 3D island growth of the film might also occur. This roughening is also frequency dependent and can be taken into account by adding respective terms to the sum in the formula (2) but with an opposite sign as compared to the smoothing parameters [12]. For our simulations we assumed that surface diffusion and desorption, re-sputtering, or ballistic mechanisms characterized with the exponents n=4 and n=2, and negative strength parameters ν_4 and ν_2 respectively are relevant for the room temperature growth conditions while contributions of viscous flow and bulk diffusion can be neglected ($\nu_1=\nu_3=0$). Also, following earlier work [12] we took into account a non-stochastic roughening caused by island growth at the early stages of deposition of the layers. Such a process is characterized with an exponent n=1 and a strength parameter, ν_1, having a positive sign.

3. Results and discussion

3.1. Impact of the sputtering gas pressure on Mo/Si structure and growth dynamics

Dependence of the surface roughness of the Mo/Si multilayers on the sputtering gas pressure was investigated by AFM. AFM images of the top surface of the multilayers deposited at Ar pressure of 1-5 mTorr (Fig. 1) show that the pressure dramatically affects the surface roughness which progressively increases with the pressure. Surface morphology remains structureless in the range of 1-4 mTorr, but at a pressure of around 5 mTorr, a transition to well-shaped grain morphology occurs which is accompanied by a sharp rise in the surface roughness (Fig. 1f).

Cross-sectional TEM images of the ML deposited at 1, 3.5, and 5 mTorr reveals an internal structure of the multilayer stacks and shows evolution of the interface roughness in the course of the deposition (Fig. 2). No roughness buildup is observed for the deposition at a pressure of 1 mTorr. Non-correlated roughness of the interfaces of the multilayer is fairly low and stays at the same level through the entire thickness of the ML stack. A moderate progressive increase of interface roughness from the substrate towards the top surface of the ML stack is observed for the 3.5 mTorr multilayer, and some roughness correlation for adjacent interfaces is also clearly visible. A strong interface roughness correlation is observed for the 5 mTorr deposition resulting in the formation of well-known column-like structure of the stack [17-19] and a dramatic roughness increase.

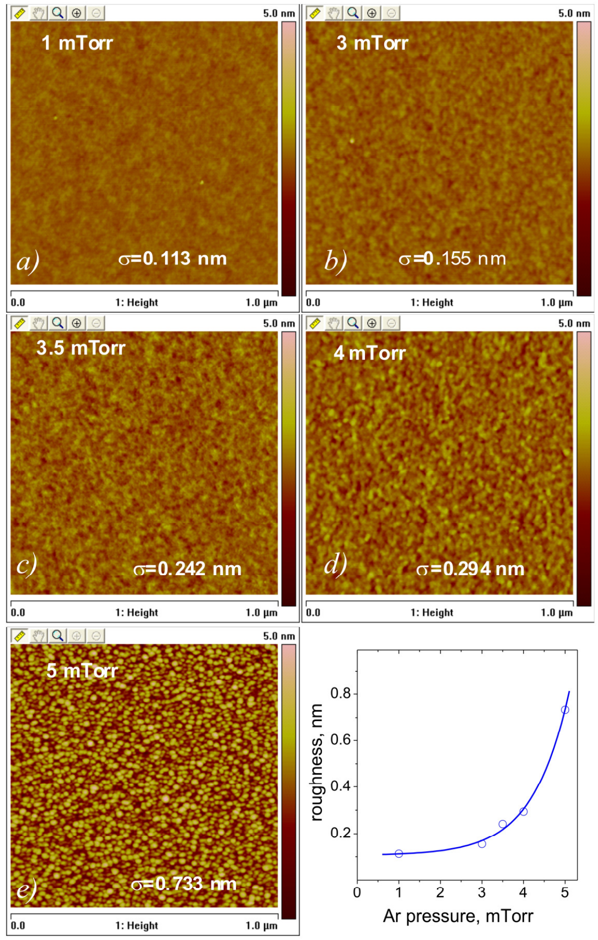

Fig. 1. AFM images of the top surface of Mo/Si multilayers deposited at different pressures of the Ar sputtering gas (a-e), and the dependence of roughness of the top surface on Ar pressure (f).

Such evolution of structure is the result of the dependence of the dynamics of a growing surface on deposition conditions. Analysis of the PSD spectra of the top surface of the multilayers gives us an insight into the interplay of roughening and relaxation processes taking place during ML growth. The PSD spectra of the top surface of the Mo/Si MLs deposited at Ar pressures between 1 and 5 mTorr are shown in Fig. 3 (symbols). The best fits obtained with the LCM (formulas (1-3)) are shown with solid lines. Values of the fitting parameters for the ML PSDs are listed in Table 1. To take into account the contribution of substrate roughness to the PSDs, one of the silicon substrates was inspected with AFM before the deposition and an empirical PSD_{sub} function was generated (not shown in Fig. 3) and used in the simulation.

A few observations regarding changes of the PSDs with the pressure can be made. The parameter Ω which characterizes the strength of a stochastic roughening process remains the same for the multilayers deposited at 1-4 mTorr. The value of the Ω parameter is within an order of magnitude of the average volume of Mo and Si atoms and consistent with values obtained earlier by other authors for Mo/Si multilayers [13,14]. An abrupt increase of Ω at a pressure of 5 mTorr is a result of a transition from a linear growth regime to a non-local columnar growth driven by shadowing (Fig. 2c). Since non-local growth cannot easily be described with a linear model, the physical meaning of the parameter Ω as an atomic volume cannot be readily applied.

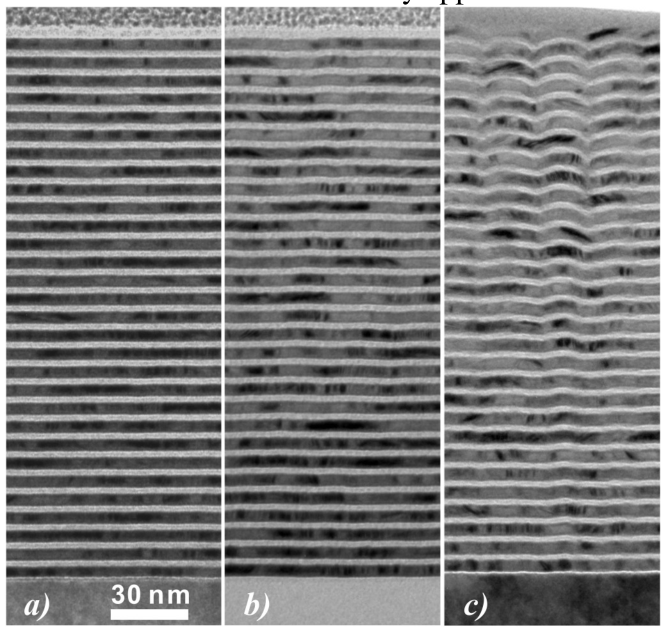

Fig. 2. Cross-sectional TEM images of Mo/Si multilayers (a,b,c) deposited at an Ar gas pressure of 1, 3.5, and 5 mTorr, respectively.

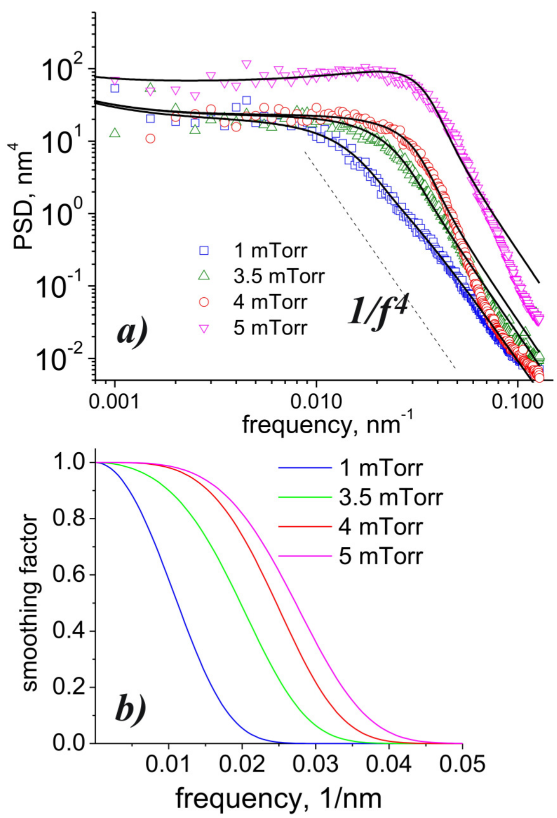

Fig. 3. PSD functions calculated for the top surface of Mo/Si multilayers deposited at an Ar pressure of 1, 3.5, 4, and 5 mTorr are shown with square, triangular, circle, and inverted triangular symbols, respectively (a). The solid lines show the best LCM fits: the fit parameters are listed in Table 1. Smoothing factors (b) were calculated for the multilayers using parameters ν_2 and ν_4 from the Table 1.

Table 1. Parameters of simulated PSD spectra shown in Fig. 3

Ar pressure, mTorr	Ω, nm^3	ν_1, nm	ν_2, nm^2	ν_4, nm^4
1	0.03	0.02	-0.3	-10
3.5	0.03	0.02	-0.06	-4.5
4	0.03	0.02	0	-3
5	0.18	0.02	0	-2

The high frequency slope of the PSD of the 1 mTorr multilayer case is close to a f^{-4} dependence. This is a signature of surface diffusion as the dominant relaxation mechanism. A slight deviation of the slope from an ideal f^{-4} dependence (shown in Fig. 3a with a dashed line) indicates some contribution from a f^{-2} relaxation process. Originally such a process was identified as desorption [15], but more recently a ballistic mass transfer mechanism (downhill currents mechanism) induced by bombardment of a growing surface by energetic particles has been suggested [16]. Molecular dynamics simulations showed that such a kind of surface relaxation leads to an f^{-2} decay of the PSD at high frequencies.

The PSD spectra undergo significant evolution with Ar pressure. The correlated part (the high-frequency slope) of the PSD curves shifts towards higher frequencies indicating attenuation of relaxation processes. The simulations reveal reduction of the strength of the relaxation parameters ν_2 and ν_4 with pressure (see Table 1). At the same time the high frequency slope increases up to $\sim f^{-4}$ for 3.5 mTorr and even more for 4 mTorr deposition. This is a signature of a non-stochastic roughening process caused by three-dimensional island growth at an early stage of the deposition of each individual layer [12,20]. According to [12] such a roughening is frequency dependent and results in some increase in the PSD spectrum at high frequencies (PSD $\sim f$). A contribution of the non-stochastic roughening process is unambiguously identified for the 4 mTorr multilayer, but it is masked for the lower pressure depositions due to the strong $\sim f^{-2}$ and $\sim f^{-4}$ relaxation processes. However, since no evident difference in microstructure of the multilayers is observed in the TEM images (Fig. 2) we have assumed an island growth roughening process with the same strength parameter, ν_1, for all the multilayers (see Table 1).

3.2. Estimation of the smoothing ability of MLs via PSD analysis.

The smoothing ability of the Mo/Si multilayers shown in Fig. 1 can be extracted from their PSD spectra (Fig. 3a). While the PSD spectra contain information on both roughening and smoothing processes, the later can be separated by excluding roughening terms and keeping only relaxation components in the replication factor:

$$a_{smooth.} = \exp 2d(\nu_2 f^2 + \nu_4 f^4)$$

Such a replication factor characterizes solely the smoothing ability of a film. The smoothing factors of the Mo/Si MLs show that the smoothing ability of the multilayers progressively reduces with Ar pressure (Fig. 3b). For example, a sinusoidal surface with a spatial period of 50 nm (f=0.02 nm^{-1}) will be 90% smoothed out by a ML deposited at the pressure of 1 mTorr, while only 25% reduction of amplitude of the same sinusoidal surface would be observed after deposition of the multilayer at a pressure of 4 mTorr.

Multilayers with reduced smoothing power are vital for MBG applications. A ML with reduced smoothing ability is expected to provide much better replication of the saw-tooth profile by ML interfaces resulting in a gain of grating efficiency. However, AFM and TEM data show that the roughness of the MLs interfaces increases with the sputtering gas pressure. This will have a negative impact on ML reflectance. Direct measurements of the MLs reflectance have been performed to find the point at which ML reflectance rolls off as a function of pressure. Fig. 4a shows the reflectance of the multilayers as a function of wavelength, and Fig. 4b shows the dependence of the peak ML reflectance on the Ar pressure. The highest reflectance of 67.2% was measured for the 1 mTorr M case, but then as the pressure increases, the reflectance reduces slightly, down to 63.5% measured for the 4 mTorr sample. A sharp drop of the reflectance at 5 mTorr makes this regime unacceptable for MBG application. The 4 mTorr ML has the lowest smoothing power as compared to 1 and 3.5 mTorr (Fig. 3b). However, using this regime is too risky for a saw-tooth substrate as it can cause shadowing and trigger a transition to column-like growth. Based on these considerations a pressure of 3.5 mTorr was chosen for the deposition of a Mo/Si multilayer on a saw-tooth substrate (see the section 3.3).

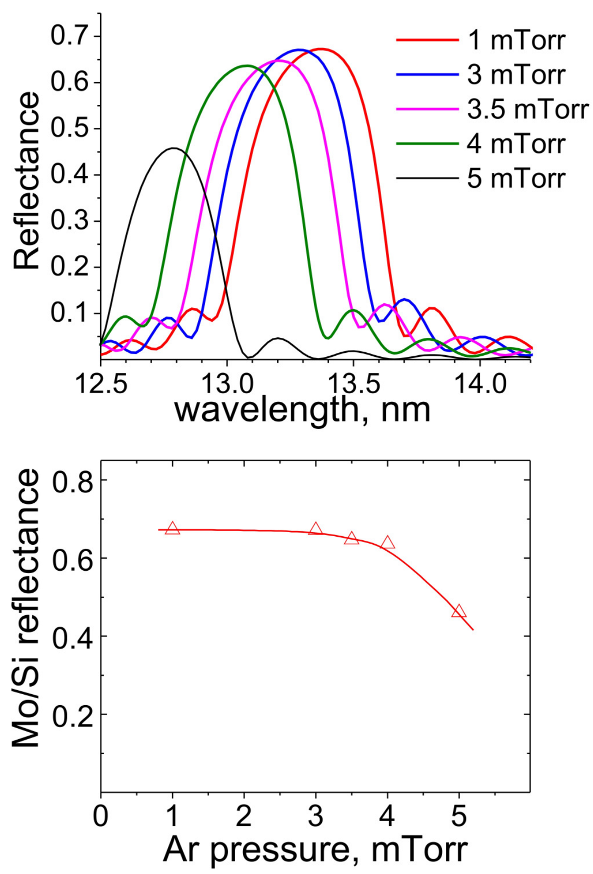

Fig. 4. Reflectance of Mo/Si multilayers versus wavelength (a), and dependence of peak reflectance of the multilayer on the Ar pressure (b).

3.3. Growth of Mo/Si MLs on saw-tooth substrates

AFM images of a saw-tooth substrate before and after Mo/Si deposition at Ar pressures of 1 and 3.5 mTorr are shown in Fig. 5. Averaged groove profiles of the substrate and the MBGs are shown in Fig. 5c. Some roughening of the surface of the blazed facets of the gratings occurs at higher Ar pressure in accord with the tendency observed for the multilayers deposited on flat substrates under the same conditions (see Fig. 1a and 1c). At the same time the saw-tooth grooves of the substrate undergo a substantial smoothing by the multilayer deposited at 1 mTorr while the high pressure deposition provides much better replication of the saw-tooth profile by the multilayer as expected. This confirms that the smoothing of saw-tooth substrates can be qualitatively predicted from the analysis made above (section 3.1) of the growth of the multilayers on flat substrates.

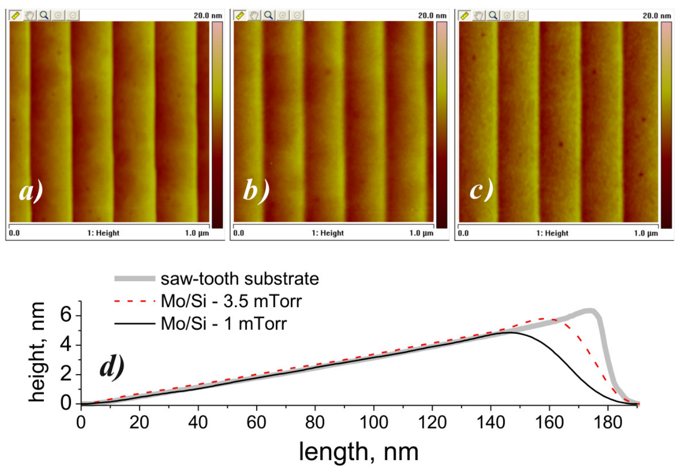

Fig. 5. AFM images of a saw-tooth substrate (a) and Multilayer blazed gratings coated with Mo/Si multilayers deposited at the Ar pressure of 1 and 3.5 mTorr ((b) and (c) respectively). (d) Average groove profiles of the gratings are shown with light grey, red dashed, and black curves respectively.

The AFM data of the MBG surface shown in Fig. 5 allows quantitative characterization of the smoothing of a saw-tooth substrate by a multilayer deposition. The replication factor as a measure of smoothing power can be found from changes of the PSD function of a saw-tooth surface caused by the deposition. Since the roughness of the blazed facets is much smaller than the surface variations of the saw-tooth gratings, the term related to PSD_{film} in formula (1) can be neglected, and the smoothing factor can be found as a ratio of the PSDs: $a = PSD_{top} / PSD_{sub}$.

We found, however, that experimental PSD curves calculated directly from the images (Fig. 5a-c) are affected by noise generated by grating and image imperfections such as surface roughness, errors in groove periodicity, nonlinearity and drift of the piezo scanner etc. For smoothing analysis we used synthetic “ideal” gratings with the average groove profiles shown in Fig. 5d. PSD spectra of the gratings consist of a number of peaks which correspond to harmonics of the Fourier transform of the saw-tooth profile

(Fig. 6). The smoothing of the grooves results in decay of the high-order harmonics. Replication factors found from the ratio of the amplitudes of the harmonics before and after deposition are shown in Fig. 6b for 1 and 3.5 mTorr depositions, shown with star and circle symbols respectively.

It is interesting to compare the saw-tooth replication factors with those obtained above for flat multilayers for the same deposition conditions. The latter are shown in Fig. 6b with solid curves. The comparison shows good agreement for low spatial frequencies (f<0.01 nm^{-1}) but there is a small discrepancy for higher frequencies. High frequency harmonics of the PSD spectra decay more slowly in the case of saw-tooth substrates than the LCM predicts. A similar trend was reported by Roder et al. for successive smoothing of rippled surfaces in the course of pulsed laser deposition of ZrO_2 coatings [21]. We also observed similar behavior for Al/Zr multilayer growth on a saw-tooth substrate [22]. The discrepancy is caused by limitations of the LCM which is based on the small surface slope approximation and is not applicable for the steep gradients of a saw-tooth surface. For a highly corrugated surface, non-linear effects should be taken into account for a more precise quantitative prediction of the smoothing processes as was discussed earlier [22,23].

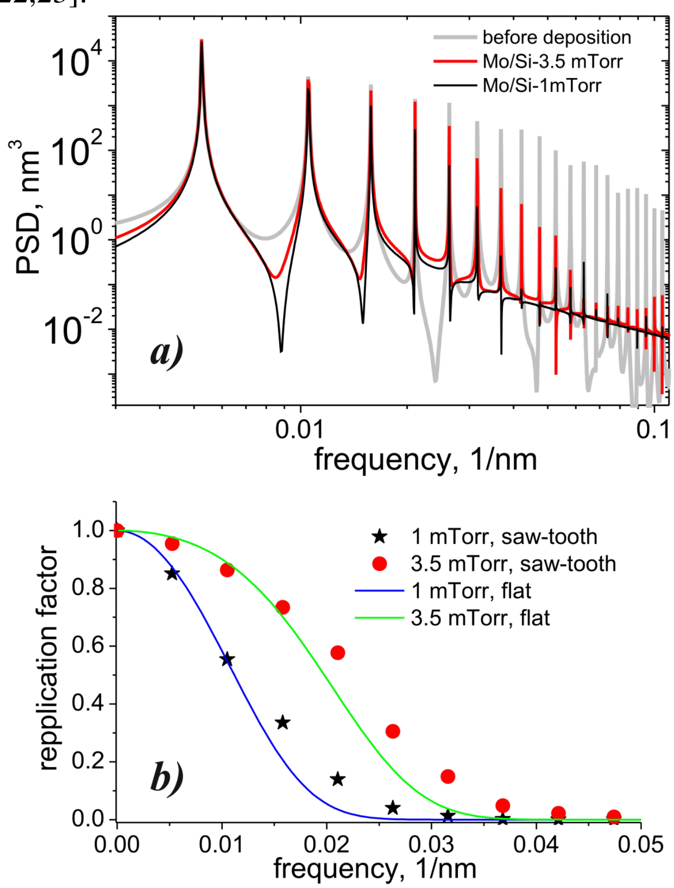

Fig. 6. (a) Synthetic PSD functions for the saw-tooth substrate before (light grey curve) and after deposition of the Mo/Si ML at Ar pressures of 1 and 3.5 mTorr (black and red curves respectively); (b) replication factors calculated for smoothing of the saw-tooth substrates with ML deposition (star and circle symbols respectively). The replication factors calculated with the LCM for the multilayers deposited on flat substrates under the same conditions are shown by solid lines.

3.4. Diffraction efficiency of a soft x-ray MBG with optimized Mo/Si multilayer

A cross-sectional TEM image of a MBG with the Mo/Si coating deposited at 3.5 mTorr is shown in Fig. 7a. The MBG with the optimized Mo/Si multilayer provides near perfect conformal replication of the saw-tooth grooves of the substrate. Due to the near perfect structure of the ML stack, the grating demonstrated a record absolute diffraction efficiency of 44% at near normal incidence at a wavelength around 13.5 nm as recently reported [7]. This wavelength range is relevant for EUV spectroscopy, lithography and astrophysics.

The same grating can be used with soft x-rays at oblique illumination. Diffraction from the grating at a wavelength of 1.36 nm and an incidence angle of 85.4º from the grating normal is shown in Fig. 7b. An absolute efficiency of 13.1% was measured for the 1st diffraction order under the blazed condition. The diffraction efficiency is limited substantially by the performance of the multilayer which reflects only about 20% at the wavelength considered. As the ML reflectance increases in the energy range of 850-1200 eV the diffraction efficiency increases to 17.6% (Fig. 8c). Compared to Lamellar Multilayer Coated Gratings [24-26] the MBG demonstrates a much higher efficiency due to the blaze effect and at the same time has much higher dispersion and better spectral resolution due to the much higher groove density. Superior performance of the MBG confirms the high degree of perfection achieved for the ML stack deposited under the optimal conditions.

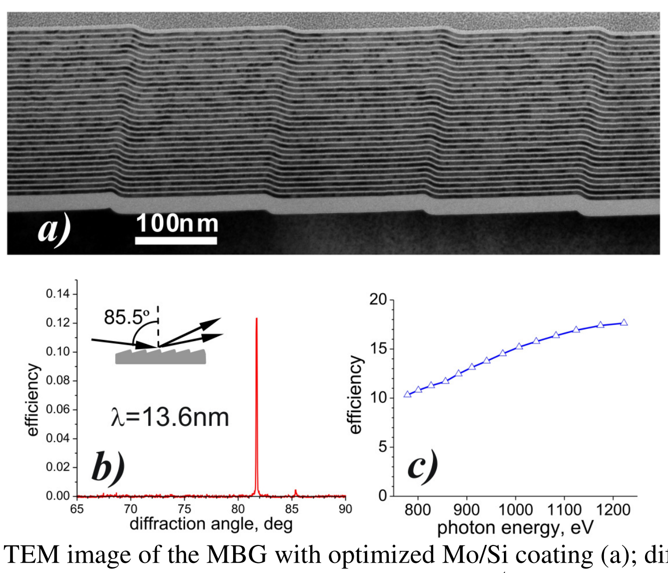

Fig. 7. Cross-sectional TEM image of the MBG with optimized Mo/Si coating (a); diffraction from the grating at a wavelength of 1.36 nm (b); diffraction efficiency of the 1st positive order of the MBG in the soft x-ray wavelength range of 900-1200 eV (c).

Summary

The smoothing ability of Mo/Si multilayers deposited under different conditions has been investigated. Analysis of roughening/smoothing dynamics of the growing surface shows that the mechanisms of surface relaxation decay gradually with the pressure, which results in reduction of the smoothing power of the MLs and roughening of the interfaces. We found that a pressure of 3.5 mTorr of Ar gas provides an optimal balance between roughening and smoothing processes, for ML deposition on saw-tooth substrates. The optimal deposition conditions provide almost perfect replication of the saw-tooth grooves and keep the roughness of the ML interfaces at an acceptable level. As a result of the optimization, a very high quality MBG could be fabricated. The grating demonstrated record diffraction efficiency in the EUV and soft x-ray wavelength ranges. High diffraction efficiency combined with a high groove density and the unique capability of blazed multilayer gratings to operate in a high diffraction order opens great prospects for ultra-high resolution soft x-ray spectroscopy.

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