Fabrication and characterization of ultra-high resolution multilayer-coated blazed gratings

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ABSTRACT

Multilayer coated blazed gratings with high groove density are the most promising candidate for ultra-high resolution soft x-ray spectroscopy. They combine the ability of blazed gratings to concentrate almost all diffraction energy in a desired high diffraction order with high reflectance soft x-ray multilayers. However in order to realize this potential, the grating fabrication process should provide a near perfect groove profile with an extremely smooth surface of the blazed facets. Here we report on successful fabrication and testing of ultra-dense saw-tooth substrates with 5,000 and 10,000 lines/mm.

Key words: diffraction grating, diffraction efficiency, blazed grating, multilayer coating, e-beam lithography, wet anisotropic etch, EUV, soft x-rays, atomic force microscopy.

1. Introduction

Soft x-ray spectroscopy techniques like Resonance Inelastic X-ray Scattering require a very high spectral resolution of 10^4-10^6 [1]. This imposes very high requirements for the diffraction gratings which are a key component of x-ray spectrometers. Traditionally used grazing incidence gratings operating in a 1st diffraction order have some limitation in terms of spectral resolution [2] due to slope error, size of the spectrograph and throughput, all of which are eliminated by high order high density gratings. The development of these ideas closely follows those adopted for high resolution spectroscopy in the optical regime.

These obstacles can be overcome with multilayer-coated blazed gratings (MLG), which according to simulations can deliver very high efficiency in a high diffraction order [3]. The high dispersion mitigates problems due to slope error, reduces geometric aberrations to the larger scattering angle, increases throughput and results in a compact design [4]. Recently we reported the successful fabrication of the 5,000 lines/mm MLG for EUV [5], which is a prototype of high resolution soft x-ray gratings. The saw-tooth substrate with the period of 200 nm was fabricated with a micro-fabrication process based on wet anisotropic etch of silicon single crystals, and then coated with a Mo/Si multilayer. In this work we scale the process towards a shorter period of 100 nm to obtain an MLG with a groove density of 10,000 lines/mm. We characterize the gratings and compare their quality to reveal the impact of the period scaling on performance. We also investigate the impact of different multilayers on grating parameters.

2. Experiment

Two saw-tooth substrates with the blaze angle of 6 degrees were fabricated form identical silicon wafers with a micro-fabrication process based on wet anisotropic etch of silicon [6]. The surface of the wafers had a 6 degree inclination from (111) planes of the silicon crystal lattice. High resolution lithography techniques were used to create periodical pattern on the surface of the wafers. Fabrication of the grating #1 with the period of 200 nm used scanning beam interference lithography

[7], and the 100 nm period pattern for the grating #2 was produced with e-beam lithography. The micro-fabrication process described earlier [8] was applied to the wafers to form a saw-tooth profile of the substrates.

The saw-tooth substrates were coated with multilayers using dc-magnetron sputtering. In order to provide the blazed conditions for a particular diffraction order, the d-spacing of the multilayers was chosen according to the ratio $d\sin\phi/\Delta=m$, where d is a grating period, ϕ is a blaze angle, Δ is a d-spacing of the multilayer, and m is a number of a blazed diffraction order of the grating. The grating #1 was covered by a Mo/Si ML with Δ=7 nm which is about 3 times smaller than the grating groove depth. In this way the grating #1 is optimized for the 3rd diffraction order under the blazed conditions. The grating #2 was deposited with a Al/Zr ML with Δ=10.25 nm, which equals to the grating groove depth, to provide blazing for the 1st diffraction order. The Mo/Si and Al/Zr multilayers consisted of 30 and 20 bi-layers respectively, so the total thickness of the MLs was approximately 200 nm. The witness Mo/Si and Al/Zr multilayers were deposited on flat silicon substrates simultaneously with the corresponding gratings.

3. Results and discussion

The micro-fabrication process results in triangular groove profiles for both the gratings. Three-dimensional AFM images of the saw-tooth substrates #1 and #2 are shown in Fig. 1a and 1b respectively. Both gratings have similar groove profiles shaped with 6-degree tilted blaze facet and short highly tilted anti-blazed facet. AFM measurements confirmed that surface roughness measured over a 1 μm^2 area does not exceed 0.3 nm rms for both the gratings. These data show that the micro-fabrication process was successfully scaled down to 100 nm period.

After the deposition of the Mo/Si multilayer the groove profile of the grating #1 underwent substantial changes (Fig. 2a). The grating grooves became smoothed and rounded especially in the vicinity of anti-blazed facets. Nevertheless the surface of the blazed facets remains stable and relatively flat and keeps the 6-degree slope.

Much more dramatic changes of the groove profile are observed for the grating #2 after the Al/Zr ML deposition (Fig. 2b). The grating surface became tremendously smoothed, and initially triangular grooves turned to sine-like ones, and the groove depth reduced significantly. Initially high asymmetry in slope and dimensions the blazed and anti-blazed facets is wiped out, making them almost indistinguishable.

The profile change is a result of a complicated interplay of smoothing and shadowing processes taking place during the ML deposition. Evolution of the Mo/Si stack structure during the ML growth onto the 200 nm saw-tooth substrate were discussed earlier [9].

The AFM data on groove profile (Fig. 1-2) can be used for characterization of smoothing properties of the multilayers. Although the gratings #1 and #2 have different periods, the smoothing ability of the Mo/Si and Al/Zr multilayers can be compared in a Power Spectral Density (PSD) domain where the difference in grating periods is not relevant. The PSD spectra for the grating #1 and #2 before and after ML deposition are shown in Fig. 3 and Fig. 4 respectively. The spectra have sharp peaks corresponding to Fourier harmonics of the groove profiles; the frequency of the peaks is multiple to the groove density of the gratings. Deposition of the multilayers results in suppression of high-frequency harmonics, which is much more pronounced for Al/Zr multilayer. Intensity of the

0.01 nm^{-1} harmonic corresponding to 100 nm spatial wavelength reduces by a factor of 2 for Mo/Si and by a factor of 5 for Al/Zr multilayers. High frequency harmonics still survive after the Mo/Si deposition, while Al/Zr multilayer suppress completely all spatial frequencies higher than 0.02 nm^{-1}. This difference indicates the much higher mobility of surface atoms during Al/Zr deposition compared to Mo/Si multilayer.

The smoothing ability is a well known property of the multilayers deposited on flat substrates [10, 11]. It is caused by surface diffusion which is a dominant relaxation process for the case of magnetron sputtering. The smoothing provides suppression of the surface roughness of flat substrates for high spatial frequencies, $f > 0.02$ nm^{-1} [11]. However, impact of the smoothing extends to much lower frequencies of 0.01 and 0.005 nm^{-1} for the saw-tooth substrates (Fig. 3 and Fig. 4). This can be explained by impact of substrate relief on surface atom mobility.

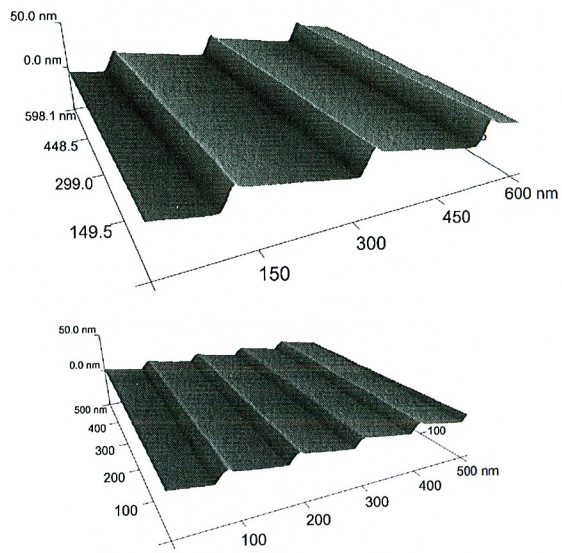

Fig. 1. AFM images of the saw-tooth silicon substrates #1 (a) and #2 (b) with groove density of 5,000 and 10,000 lines/mm respectively

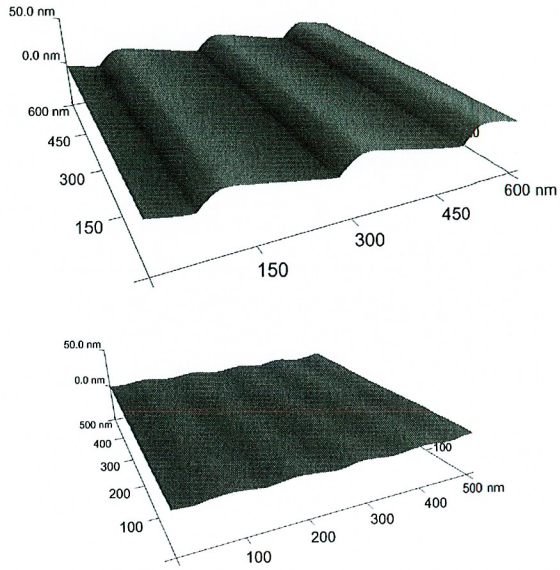

Fig. 2. AFM images of the blazed gratings #1 (a) and #2 (b) after deposition of Mo/Si and Al/Zr multilayers respectively

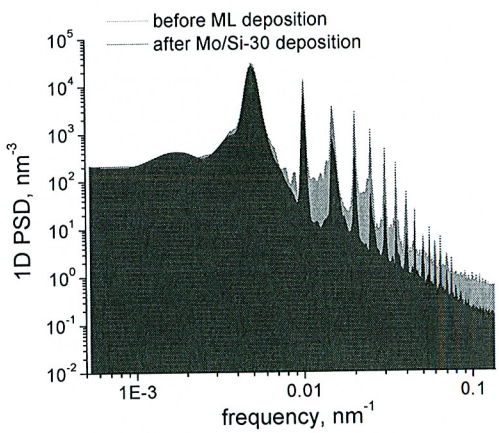

Fig. 3. One dimensional PSD spectra of the gratings #1 before and after deposition of the Mo/Si multilayer.

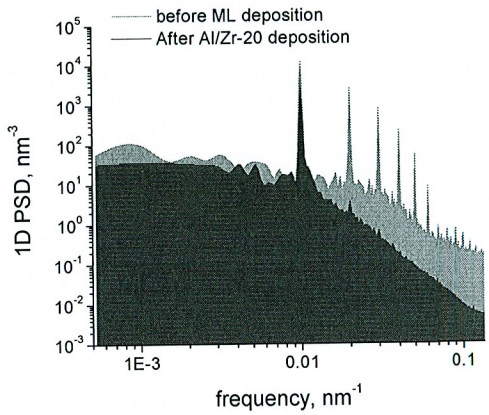

Fig. 4. One dimensional PSD spectra of the gratings #2 before and after deposition of the Al/Zr multilayer.

The smoothing of the groove profile results in deterioration of the blazing performance and a reduction of diffraction efficiency. A number of diffraction orders, which should be suppressed for an ideal blazed grating, appeared on the diffraction pattern of the grating #1 (Fig. 5). At the same time the efficiency of the 3rd blazed order reduces compared to the witness Mo/Si reflectance of 64% by a factor of 0.6, but it is still rather high because a large portion of the blazed facets survived after deposition.

The grating #2 that suffered much more from the smoothing demonstrates efficiency of the 1st blaze order of 13%, which is smaller than witness Al/Zr reflectance of 48% by a factor of 4. This is due to the large variation of the blaze angle across the grating groove so that the diffracted energy is directed to the zero and negative 1st orders, which have efficiency comparable to the one for the blazed 1st order.

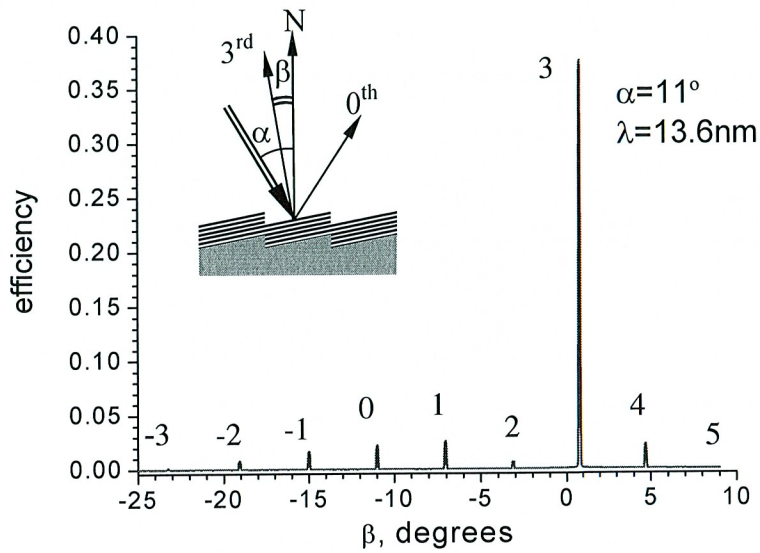

Fig. 5. Diffraction from grating #1 coated with a Mo/Si multilayer at the incident angle of 11°, and the wavelength of 13.6 nm.

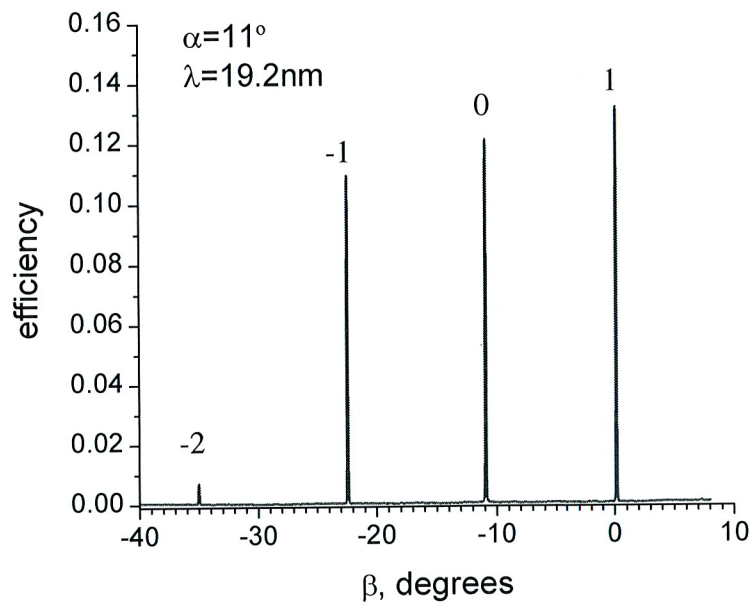

Fig. 6. Diffraction from grating #2 coated with an Al/Zr multilayer at the incident angle of 11°, and the wavelength of 19.2 nm.

4. Summary

The fabrication process for high quality saw-tooth substrates was successfully scaled towards ultra-short grating period of 100 nm. The gratings have near-perfect triangular groove profile and very smooth surface of blaze facets.

However the deposition of a multilayer on the saw-tooth substrates results in significant smoothing of the groove profile, which results in reduction of the MLG performance. It was found that Al/Zr multilayers have much more pronounced smoothing ability compared to Mo/Si ML. The process of the deposition of multilayers on the highly corrugated surfaces of the saw-tooth substrates imposes new conditions on deposition and elemental selection. Clearly some form of collimated deposition will reduce the transverse energy of atoms to a level much lower than in magnetron sputtering. However some degree of transverse energy is required for smoothing on the plane facet surfaces, so reduction of this energy to zero, if possible, would result in unacceptably rough surfaces. In addition, there is a finite limit to the transverse energy, given by the interaction of the depositing atom and the atomic structure of the surface. Achieving a balance between local roughness and local shape error will require a more detailed understanding of surface mobility on systems of relevance to multilayer reflectors.

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