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Highly efficient ultra-low blaze angle multilayer grating

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Abstract: We have developed an advanced process for blaze angle reduction of x-ray gratings for the soft, tender, and EUV spectral ranges. The process is based on planarization of an anisotropically etched Si blazed grating followed by a chemically selective plasma etch. This provides a way to adjust the blaze angle to any lower value with high accuracy. Here we demonstrate the reduction of the blaze angle to an extremely low value of 0.04°±0.004°. For a 100 lines/mm grating with a Mo/Si multilayer coating, the grating exhibits diffraction efficiency of 58% in the 1st diffraction order at a wavelength of 13.3 nm. This technique will be applicable to a wide range of uses of high efficiency gratings for synchrotron sources, as well as for Free Electron Lasers (FEL).

1. Introduction

Synchrotron x-ray facilities around the world are being upgraded at present by replacement of the magnet lattice of the accelerator with a multi-bend achromat system. This type of design gives a large reduction in the electron beam emittance, and consequently an increase of x-ray brightness by up to three orders of magnitude. In the case of the upgrade of the Advanced Light Source (ALS) in Berkeley, the reduction in electron beam emittance is sufficient to ensure that the source is close to transversely coherent throughput the soft x-ray energy range [1]. As well as replacement of the storage ring and addition of an accumulator ring, the ALS-U project includes 2 new and 2 upgraded undulator-based beamlines designed to exploit the new high-brightness source [2]. The beamlines are to be equipped with new advanced x-ray optics including x-ray diffraction gratings that maximize the beamline efficiency. Due to the small source size and the large source to grating distances, the plane grating monochromator design adopted for these beamlines requires that most of the gratings should have a low groove density down to 170 lines/mm and very low blaze angles down to 0.3°.

Another recent trend in the field of synchrotron x-ray optics is extension of the operational energy range of grating monochromators towards the tender x-ray regime. This can be achieved by use of multilayer blazed gratings [3] for monochromators operating in collimated light. However, for quasi coherent sources, the preferred solution is to use a monochromator design that is based on plane optics, due to the extreme accuracy with which the optics have to be fabricated. In this case the variable included angle, varied line space plane grating monochromator (VIA-VLS-PGM) is the preferred solution. Due to the non-co-planar geometry of the two plane surfaces, multilayer coatings cannot be used, and we must use single layer coatings. For the tender x-ray domain (defined as 1–5 keV), this results in a requirement for very small blaze angles. In addition, the advent of soft x-ray FEL sources has also resulted in an evolution of grating design. In this case, the extreme peak power of the FEL requires the use of extreme grazing incidence to avoid optical damage. As in the case of the tender x-ray regime case, this results in the need for gratings with very small blaze angles.

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Low blaze angle gratings can also be used as efficient spectral purity filters for EUV lithography to separate EUV radiation of wavelength of 13.5 nm from undesirable out-of-band radiation from the deep ultraviolet to the infrared resulting from the use of laser plasma x-ray sources. Currently the most practical solution is to use lamellar grooves made by diamond turning on top of the collector mirror to divert the undesirable out-of-band radiation outside the exit aperture by diffraction [4,5]. The efficiency of such an approach is limited since the lamellar grooves can provide suppression of the zero order diffraction for a certain wavelength only and some portion of the out-of-band radiation still can propagate through the exit aperture of the EUV source. An alternative approach based on multilayer blazed gratings, which provides 100% filtering was suggested by K. C. Jonson [6]. A Mo/Si multilayer blazed grating optimized for the wavelength of 13.5 nm can provide diffraction of the EUV radiation into the exit aperture while all of the out-of-band radiation will be angular separated outside the aperture. To avoid excessive dispersion of EUV radiation over the exit aperture the grating should have a period of about 10 µm and operate in a 1st diffraction order. Such a grating should have a groove depth of about 7 nm and an extremely low blaze angle of 0.04°.

Fabrication of such low aspect ratio structures composed of grooves as wide as 5-10 µm and with only a depth of 5-10 nm is very challenging. The small groove depth implies nanometer scale tolerances for the groove shape. Classical diamond ruling typically provides blaze angles of a few degrees. Although significant progress in making low blaze angle gratings was reported in recent years [7,8], it is still difficult to achieve a perfect saw-tooth shape of grating grooves. Typically, the apex of the ruled grooves is substantiality rounded, blazed facets are curved, and the anti-blaze facets are rather wide. These imperfections as well as surface roughness all affect diffraction efficiency of the grating and the production of scattered light.

A high quality groove shape can be achieved using a nanofabrication approach based on lithography and wet anisotropic etching of a single crystal Si surface in aqueous KOH solution [9,10]. The groove facets are defined by the (111) planes of the Si crystal lattice. These planes are tilted slightly with respect to the crystal surface by off-cutting of the surface with respect to the silicon lattice. This method works very well for blazed angles down to a few degrees but for very low blaze angle gratings the process is rather problematic since the anisotropy of the wet etching drops dramatically. Moreover, residual curvature of the blaze facets becomes comparable to the groove depth and affects diffraction efficiency [11].

To extend our fabrication method to very low blaze angles we have investigated a blaze angle reduction process based on planarization of a coarse Si grating by a polymer material followed by plasma etching of both polymer and silicon with a certain differential etch rate for the two

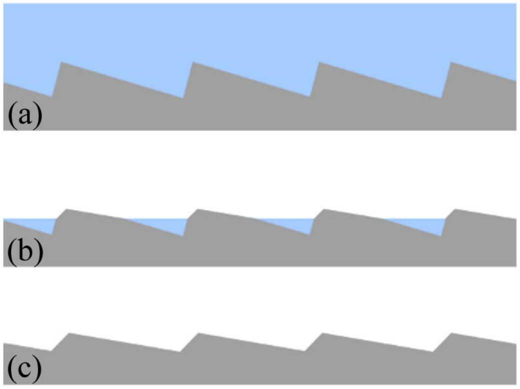

Fig. 1. Fabrication of low blazed gratings via planarization (a) and selective plasma etching (b, c).

materials (Fig. 1). A proof-of-principle experiment demonstrating reduction by a factor of 2 was reported earlier [12]. To achieve a low blaze angle the reduction process would have to be repeated many times which is not practical due to the risk of the generation of defects, which increases with the number of the cycles. In this paper we report on an advanced planarization/reduction process which provides the goal blaze angles of 0.4° for ALS-U gratings in a single step. We also demonstrate achievement of an extremely low blaze angle of 0.04° for a 100 lines/mm multilayer grating. The process provides the goal of a groove depth of 7 nm with sub-nanometer precision to perfectly match the d-spacing of the Mo/Si multilayer. We performed diffraction efficiency measurements of the multilayer grating in the EUV range to confirm the near perfect quality of the grating.

2. Coarse blazed grating fabrication and planarization

A 100 lines/mm blazed grating was fabricated by optical lithography and KOH anisotropic wet etching at a temperature of 40°C. We used 100 mm diameter (111) Si wafers with a miscut angle of 4°. The details of the fabrication process are described elsewhere [9,10]. AFM measurements showed a groove depth of 620 nm and an average blaze angle of 3.7° which is somewhat smaller than the miscut angle due to the slightly concave shape of the blazed facets (more details in Section 3). A photograph and an AFM image of the wafer-size blazed grating are shown in Fig. 2.

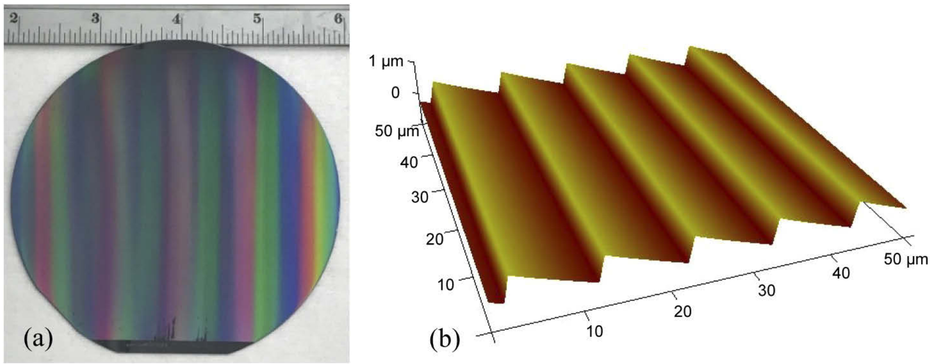

Fig. 2. A photograph of the anisotropically etched Si grating (a); AFM image of the coarse saw-tooth grating with groove depth of 620 nm and a blaze angle of 3.7° (b).

Planarization of the coarse grating can be performed by spin-coating with a polymer material such as resist, antireflective coating etc. It is very important to achieve planarization of the saw-tooth grating close to 100% to preserve the triangular groove shape during the following reduction process. Any residual non-planarity or waviness of the resist surface will be transferred to the facets of the Si grating during the plasma etch. The residual resist surface undulations should be much smaller than the final groove depth, otherwise the groove profile can be significantly compromised.

It is difficult to achieve 100% planarization by a single layer coating. We found earlier that centrifugal forces have a minor impact on the planarization, and residual non-planarity is mainly caused by shrinkage of the resist, caused by solvent evaporation [13]. According to the simplest planarization model, the surface of a liquid resist is perfectly plane owing to surface tension and the low viscosity of the resist. A substrate topography feature such as a depression of depth d_0 is perfectly planarized due to redistribution of the highly mobile material driven by capillary forces (Fig. 3). As the spin-coating proceeds the resist layer thins due to solvent evaporation while the viscosity of the resist increases, and at some thickness of h_0 the resist becomes immobile (Fig. 3(a)). Further evaporation of the solvent results in extra contraction of the material down to a final thickness of h_1 by a contraction factor $C = h_0/h_1$, (Fig. 3(b)). Since the resist layer is thicker over the substrate depression, the shrinkage results in residual non-planarity $d_1 = d_0 \times (C - 1)$. To achieve a high degree of planarization one needs a material of low contraction (C=1), which maintains high mobility even when all or almost all solvent has evaporated.

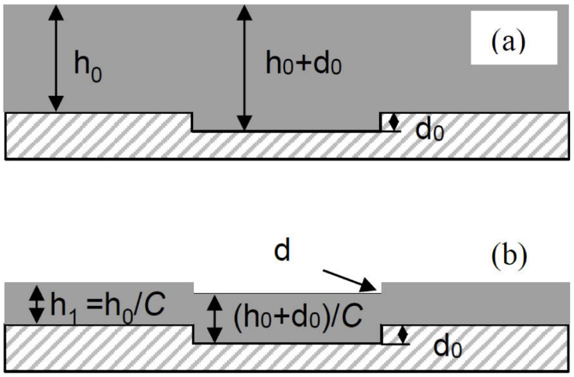

Fig. 3. Impact of resist contraction/shrinkage on planarization. The surface of the resist is ideally plane due to surface tension for a low-viscosity liquid resist. At some point the resist becomes solid and immobile. Further solvent evaporation results in shrinkage/contraction of the material which results in a residual depression on the resist surface.

We surveyed a wide range of the materials and found that mr-NIL6000 nanoimprint resist (Microresist Technology GmbH) gives the best results in this case [13]. The resist has a low glass transition temperature of 1°C and remains semi-liquid by the end of spin-coating when almost all the solvent has evaporated. It exhibits great planarization properties reducing the groove depth of the coarse grating down to a few tens of nanometers. An optimized baking procedure provides evaporation of the residual solvent and additional relaxation of the surface of the melted material. This results in reduction of the residual surface undulations down to 7 nm peak-to-valley measured after curing with UV exposure [13].

3. Single step reduction process for low blaze angle gratings for ALS-U

The mr-NIL6000 resist was spun on the coarse 100 lines/mm grating at a speed of 1000 rpm for 90 s and then baked at 100°C for 1 min, and then cured by UV exposure. The cured resist was baked again, and then another layer of the resist was applied under the same conditions. The double-layer coating provides almost 100% planarization of the coarse saw-tooth grating with residual surface undulations below 1 nm p/v (Fig. 4(b)).

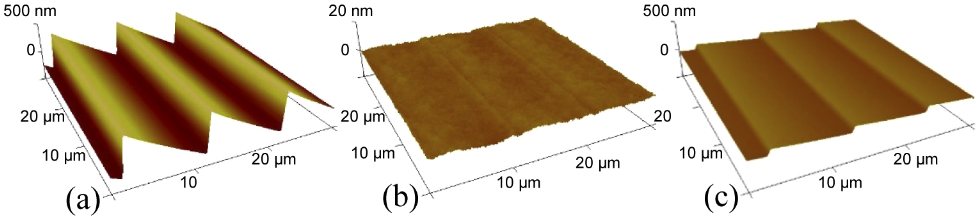

Fig. 4. AFM images of the coarse grating after wet anisotropic etching (a), planarization (b), and plasma etch (c).

To meet the goal of angles of 0.3°–0.5° for ALS-U gratings, the blaze angle of the coarse grating has to be reduced by a factor of 10 or so. Reduction of the blaze angle is achieved by plasma etching which provides removal of both resist and Si with etch rates V_R and V_{Si} respectively. The reduction factor, R, is defined by the etch rate ratio as $R = 1/(1- V_{Si}/V_R)$. To achieve a reduction factor of up to 10, the plasma process should provide an etch rate of Si almost as high as the one for the resist, $V_{Si}/V_R \approx 0.9$. At the same time the plasma etch process should not compromise the smooth surface of the resist during the etching, develop surface roughness, generate etching defects etc. Since the etch rate ratio is close to 1 any imperfections of the resist surface such as roughness, pits, grass etc. would be transferred to the facets of the Si grating at almost the same scale.

A Viper etcher (Oxford Instruments Inc.) was used to perform plasma etching for the planarized coarse grating. This ICP (Inductively Coupled Plasma)-type tool provides flexible control over a wide range of plasma parameters via gas mixture composition, power, bias etc. We developed a set of etching recipes which provide reduction factors from 1.1 to 14. Some of the recipes are listed in the Table 1. An AFM image of the grating after plasma etch reduction by a factor of 8.7 is shown in Fig. 4(c). The etching was performed using a ($CFH_3 + CH_4 +$ Ar) gas mixture at a pressure of 10 mTorr and a flux of 10 sccm for all the gases, ICP power of 100 W, and RF power of 40 W. The obtained blaze angle of 0.42° meets the requirements for the x-ray gratings for ALS-U. The low blaze angle grating has an almost perfect triangular groove profile with highly flat blazed facets and short anti-blazed facets tilted by the anti-blaze angle of 11° (Fig. 5(a)). Gratings with such good groove profile were shown to have a diffraction efficiency close to the theoretical one in the soft-x-ray range [12]. The reduction process not only preserves the saw-tooth grooves of the anisotropically etched Si grating but results in improvement of the groove shape since groove imperfections being topographic features scale down by the same factor as the groove depth. For example, the facets of the coarse grating are slightly curved with a sag of about 20 nm which reduces down to 3 nm p/v for the low blaze angle grating (Fig. 6). Similarly the roughness of 0.88 nm rms of the facet surface after the KOH etching performed at the elevated temperature of 40°C, scaled down to 0.38 nm after the reduction (Fig. 7).

Table 1. Plasma etch recipes for blaze angle reduction

Pressure, mTorr	Gas flux, sccm				Power, W		Etch Rate Ratio	Reduction Factor
	CHF_3	CF_4	Ar	O_2	RF	ICP	V_{Si} / V_{resist}	$1/(1-V_{Si}/V_{resist})$
10	10	10	10	0	20	60	0.93	14
10	5	10	5	0	30	100	0.9	9.7
10	10	10	10	0	30	100	0.88	8.7
10	5	5	5	0	30	100	0.86	7.2
10	5	5	5	0	40	100	0.83	5.9
10	0	15	0	0	50	100	0.69	3.2
10	0	15	0	12	40	100	0.58	2.4
10	0	15	0	13.5	30	100	0.43	1.7
10	0	10	0	15	40	100	0.29	1.4
10	0	10	10	10	40	100	0.15	1.2
10	7	0	10	20	40	100	0.12	1.1

Summarizing this part, we developed a single step reduction process for the low blaze angle gratings for ALS-U.

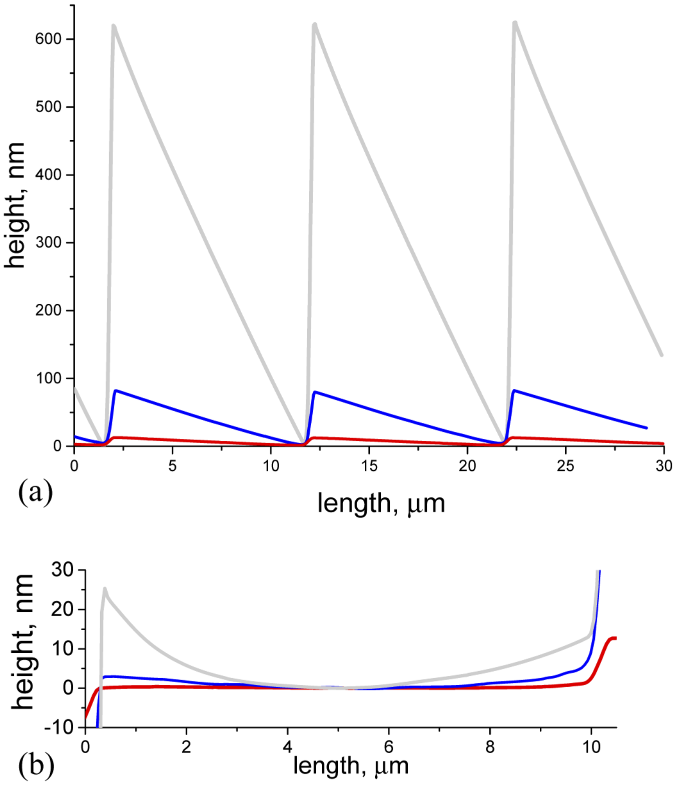

Fig. 5. Groove profile (a) and the blazed facet curvature (b) of the coarse grating before (grey) and after 1st (blue) and 2nd (red) reduction

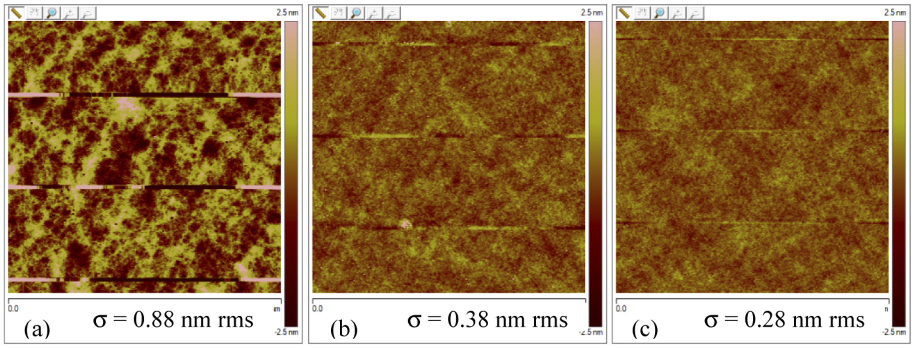

Fig. 6. Surface roughness of the coarse grating before (a) and after 1st (b) and 2nd (c) reduction. AFM scan size is 30 µm by 30 µm.

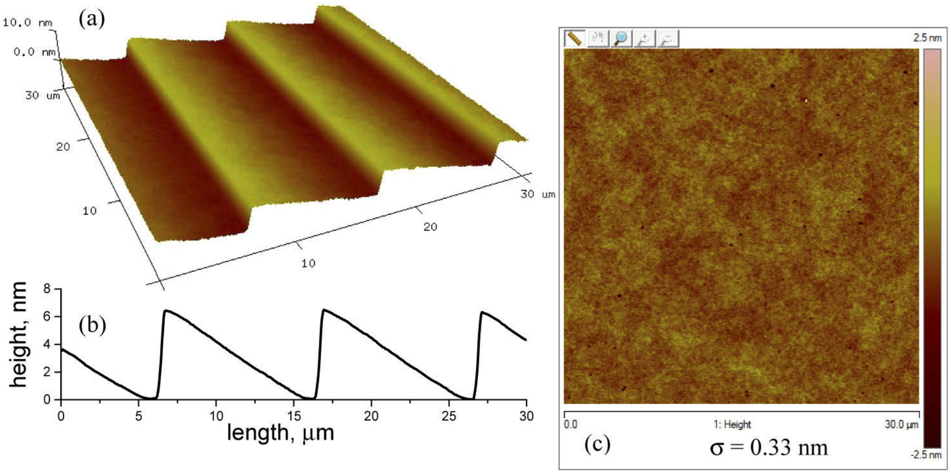

Fig. 7. A 3D image (a), a profile (b), and a top-view AFM image (c) of the ultra-low blaze angle grating. The groove topography was removed from the top-view image for a purpose of surface roughness measurements.

4. Achievement of ultra-low blaze angles for a Mo/Si multilayer blazed grating

In this Section we investigate the possibility of making multilayer blazed gratings with extremely low blaze angles for EUV lithography as well as for tender and even hard x-ray applications. To achieve a high diffraction efficiency for a multilayer blazed grating, the parameters of the grating substrate and the multilayer should be coupled accordingly. Although precise optimization of multilayer blazed gratings is performed via numerical simulations [14,15] a simple rule of thumb can be used for the low asymmetry case of near normal incidence diffraction as used for EUV applications [16], namely that the depth of the grating grooves should be equal to an integer number of multilayer bi-layers. This condition provides perfect stitching of the interfaces of the multilayer stacks to the adjacent grooves, resulting in the highest possible diffraction efficiency. A multilayer blazed grating optimized for operation in the 1st diffraction order should have an extremely low blaze angle of 0.04° to provide the depth of the 10-micron wide grooves equal to the multilayer d-spacing of 6.95 nm of the Mo/Si multilayers used for EUV lithography. To avoid reduction of diffraction efficiency, the stitching errors should not exceed 10% of the groove depth [15]. This puts a very tight tolerance of ±0.004° on the ultra-low blaze angle. Fabrication of such a low aspect ratio structure with nanometer precision is a great technological challenge. Any groove imperfections such as surface roughness and facet curvature should be much smaller than the groove depth otherwise the blazing properties of the grating will degrade and the efficiency will be compromised. For example, in previous work, facet curvature of a Sc/Si multilayer blazed grating resulted in diffraction into several orders and significant reduction of diffraction efficiency of the blazed order was observed [17].

To achieve the ultra-low blaze angle, the planarization/reduction process described in Sec. 3 and 4 was repeated. (This time the planarization was achieved by a single-layer coating of the grating with a reduced groove depth of 70 nm). The 2nd reduction cycle resulted in a groove depth of 12.7 nm and a blaze angle of 0.07°. The facet curvature was reduced below 1 nm p/v and the roughness reduced down to 0.28 nm approaching to the roughness of the resist surface transferred to the Si (Figs. 5 and 6).

To precisely tune the blaze angle to the goal of 0.04°, a 3rd planarization/reduction cycle was applied. The plasma etch recipe was modified to increase the etch rate of the resist and provide an etch rate ratio of 0.43 and reduction factor of 1.74. The etching was performed using a CH_4

and O_2 gases with a flux of 15 sccm and 13.5 sccm respectively, at a pressure of 10 mTorr, ICP power of 100 W, and RF power of 40 W.

AFM images and the profile of the final grating are shown in Fig. 7. The blazed facets have minor s-shape curvature well below 0.5 nm p/v, which is the residual non-planarity of the resist surface transferred to the Si grooves. The groove depth of 7.2 nm measured as a distance between the parallel facets is close to the goal of 6.95 nm, as well as the blaze angle of 0.041°. The anti-blaze facets are very short since the anti-blaze angle of 1° is larger than the blaze angle by a factor of 24. Roughness of the facet surface of 0.33 nm rms was measured after line flattening of the AFM image (Fig. 7(c)).

A Mo/Si multilayer composed of 40 bi-layers was deposited on the ultra-low blazed grating by dc-magnetron sputtering. The diffraction efficiency was measured in a diffractometer on beamline 6.3.2 at the ALS. The reflectivity of the witness multilayer deposited on a plane substrate was measured to be 67% for the resonance wavelength of 13.36 nm at an incidence angle of 5° from the normal (Fig. 8(a)). The multilayer d-spacing of 6.88 nm was found by simulation of the reflectivity curve. Integral diffraction efficiency of 60.8% was measured for the multilayer grating using a 10 mm wide detector slit by scanning the monochromator over the wavelength range of 12.5-14.5 nm, in the same way as for the witness multilayer (Fig. 8(a)).

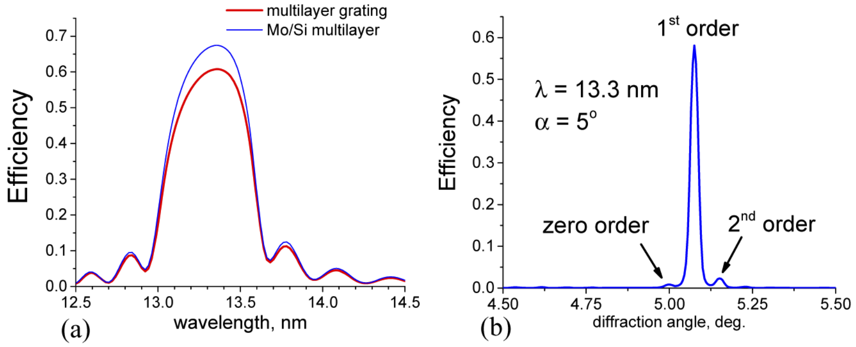

Fig. 8. Diffraction efficiency of the low blaze angle Mo/Si multilayer grating measured by monochromator (a) and detector (b) scans.

The diffraction efficiency of separate diffraction orders was measured by a detector scan using a 0.1 mm narrow slit at the wavelength of 13.3 nm and an incidence angle of 5° (Fig. 8(b)). The efficiency of the 1st diffraction order was found to be 58.1%, while the efficiency of the zero and the 2nd orders is about 2.3% and 0.8% respectively. The high efficiency of the blazed order confirms that the parameters of the grating and the multilayer are well matched, while the minor asymmetry of the zero and the 2nd orders is caused by the residual mismatch of the groove depth of 7.2 nm and the d-spacing of 6.88 nm.

Although the multilayer blazed grating exhibits a record diffraction efficiency of 58%, it is still lower than the multilayer reflectance of 67%. This can be caused by many reasons; here we will list the most obvious ones. The efficiency of an ideal grating is somewhat lower than the multilayer (or single layer coating) reflectance as part of the diffracted energy is distributed over non-blazed orders. For an ideal grating with a perfect groove profile and exact matching the grating and multilayer parameters this has a minimum effect, but efficiency reduces further with any deviation from the ideal case. The other possible reason of efficiency reduction is process defects generated during the spin-coating and plasma etch. For example, some tiny nano-pits observed on the AFM images of the grating (Fig. 7(c)) result in some increase of the roughness compared to the 0.28 nm for the previous reduction cycle (Fig. 6(c)). Usually the total area of such defects is negligibly small, however, the number of defects increases with the number of the reduction cycles. That is why it was so important to develop the single step reduction process for ALS-U gratings, described in Sec. 3.

Most of the efficiency reduction is probably caused by the anti-blazed facets. Although our fabrication method provides anti-blazed facets much narrower than any other technique such as diamond ruling, they are still as wide as 4% of the grating pitch. The part of the multilayer stack resting on top of the anti-blazed facets does not contribute to the diffraction efficiency since the Bragg condition is compromised. Although zero anti-blazed facets are not possible even for anti-blaze angle of 90° due to smoothening of the steep steps by the multilayer, reduction of the anti-blazed facet width by a factor of 10 by further process improvements seems realistic. Interestingly, the anti-blaze facet problem is not so severe for multilayer gratings for the tender x-rays. Even gratings with substantially wider anti-blaze facets can exhibit good diffraction efficiency due to low absorption and high transparency of the off-Bragg portion of the multilayer stack in the tender x-ray range [14].

The fabricated ultra-low blaze angle grating is suitable for FEL applications since rather wide anti-blazed facets and large apex angle of about 179° of the grating grooves reduce a risk of damage during the laser exposure.

5. Summary

We developed a single-step blaze angle reduction process providing a highly accurate blaze angle of 0.4° required for diffraction gratings for ALS-U beamlines. The optimized planarization procedure provides almost 100% planarization of a coarse Si grating made by anisotropic wet etching. The developed plasma etching recipes provide blaze angle reduction by a factor as high as 9 and allows for tuning the blaze angle down to an optimal value with high precision. The reduction process preserves the saw-tooth shape of the grating grooves and improves smoothness of the blaze facets required for high diffraction efficiency. The technique allows achievement of extremely shallow grooves required for EUV lithography and tender x-ray application. We demonstrated fabrication of a 100 lines/mm Mo/Si multilayer blazed grating with 7.2 nm deep grooves and the blaze angle of only 0.041°. The grating exhibited record diffraction efficiency of 58% of the 1$^{\text{st}}$ diffraction order at a wavelength of 13.3 nm.

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Disclosures. The authors declare that there are no conflicts of interest related to this article.

Data availability. No data were generated or analyzed in the presented research.

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