Low line density blazed gratings with low blaze angles

S Lemke, S Alimov, J Knedel, O Kutz, I Rudolph, T Seliger and A Sokolov

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Precision gratings

stephanie.lemke@helmholtz-berlin.de

Abstract. Gratings installed in synchrotron beamlines and instruments have standard line densities ranging from ~300 L/mm up to 2400 L/mm with blaze angles from 0.3° to 2°. For FEL beamlines lower line densities and lower angles are required. To manufacture these in the required high quality poses some challenges with regard to the ruling process and the ion etching process. Our investigation and process results will be described here. Additionally, first results of the process on a grating substrate are demonstrated.

1. Introduction

Since synchrotron grating fabrication was established at HZB in the early 2010s, eight beamlines were equipped and nine spectrometer gratings were supplied for BESSY II. In total, we supplied over 100 gratings to synchrotron sources around the globe. A first low line density grating is already installed at MAX IV in Lund, Sweden [1]. During the fabrication of this 92 L/mm grating, different adaptations to the standard manufacturing processes were necessary. To achieve even lower line density blaze profiles, down to 50 L/mm, and a shallow blaze angle below 0.3° more investigations on the processes were performed.

2. Process adaptation

Ruling of low blaze angle gratings at the GTM6 [2] is a process of deforming gold with a diamond tool to create the profile as a mask. Usually, the gold layers evaporated on the substrates have thicknesses up to approximately 500 nm. For low line density (*LD*) gratings the ruling layer must be up to five times thicker, depending on the blaze angle of the ruled profile and the *LD*. For example, for 50 L/mm and 8° the gold layer needs a minimum height of 2.8 µm, for 4° it is still 1.4 µm. To heighten the layer thickness causes growing micro roughness.

The ruling process at the GTM6 is a non-cutting machining, a pure deformation of the gold surface to form the blaze profiles. For standard rulings low forces, below 0.3 N, are applied. In case of the standard diamond tool a force of more than 5 N was necessary to achieve a well-formed groove for 50 L/mm. We observed that in that case, the gold deformation process is no longer strictly non-cutting (figure 1). Also the air bearings, guiding the ruling diamond tool, are reaching their maximum load capacity. The risk for the ruling machine is too high to use as a standard grating manufacturing process.

Rulings were performed utilizing our standard and a non-standard diamond tool. Similar ruled blaze angles were aimed at with both tools. The profile form shows differences in linearity (figure 2). These will influence the micro roughness since waviness and micro roughness cannot be totally distinguished in fitting. The non-standard tool gave the highest facet roughness, which is not directly proportional to the reduced ruling force, and can be best explained by the second deformation of the gold and the smoothing of the gold grains by the diamond tool (figure 1). The results are compared in table 1.

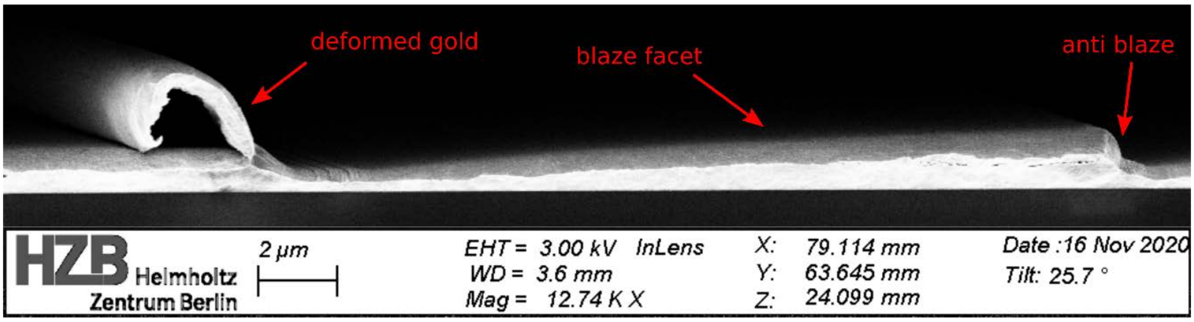

Figure 1. Ruled line and deformed gold, which will be compressed when the next line is ruled on a test sample.

Table 1. Ruling and etching results of three test samples.

Ruling tool	standard		non-standard
Ruling force (N)	5.3	7.6	0.9
Ruled			
Blaze angle (°)	8.01 ± 0.07	2.81 ± 0.03	2.78 ± 0.08
Anti blaze angle (°)	62 ± 6	28 ± 5	70 ± 2
Facet roughness (nm rms)	1.8 ± 0.4	4.7 ± 0.7	6.6 ± 0.8
Etched			
Blaze angle (°)	0.24 ± 0.01	0.146 ± 0.005	0.145 ± 0.003
Anti blaze angle (°)	7.6 ± 2.0	1.3 ± 0.1	8.7 ± 0.5
Facet roughness (nm rms)	0.84 ± 0.15	0.49 ± 0.07	1.2 ± 0.2

The different etching rates of gold and silicon lead to the reduction of the blaze angle by ion etching [2]. Standard gratings are etched with a selectivity of 3 to 12. To achieve a high selectivity of 19, we used a different main etching gas. The results after ion etching are also listed in table 1. By additionally adjusting the ion energies, a further increase of selectivity above 25 is feasible to reach blaze angles of approximately 0.1°.

The sample ruled with the standard tool and high ruling force was further analyzed by ex- and in-situ metrology, the sample with the non-standard tool and the gratings by ex-situ metrology.

3. Measurement results

The samples were measured with ex-situ methods and in-situ at wavelength. Both together allow to analyze the obtained profile and its performance in the beamline.

3.1. Ex-situ metrology

After ruling the blaze profile, the line structure is measured by different techniques to evaluate the grating quality. The line placement is measured by an optical setup, while the blaze profile is tested by atomic force microscopy (AFM).

3.1.1. Line placement To obtain the line density, measurements of the diffraction angle were performed in Littrow configuration using a He-Ne laser source with 633 nm. The grating is rotated through the diffraction orders, at any given coordinate on its optical surface. While the sample is rotated, the intensity of the reflected light is measured. The LD is given by

$$LD = \frac{2 \cdot \sin(\alpha - \alpha_0)}{m \cdot \lambda} \qquad (3.1)$$

were *m* is the measured diffraction order, λ the measurement wavelength, α the angle of the diffraction order and α_0 is the rotation offset of the zeroth order.

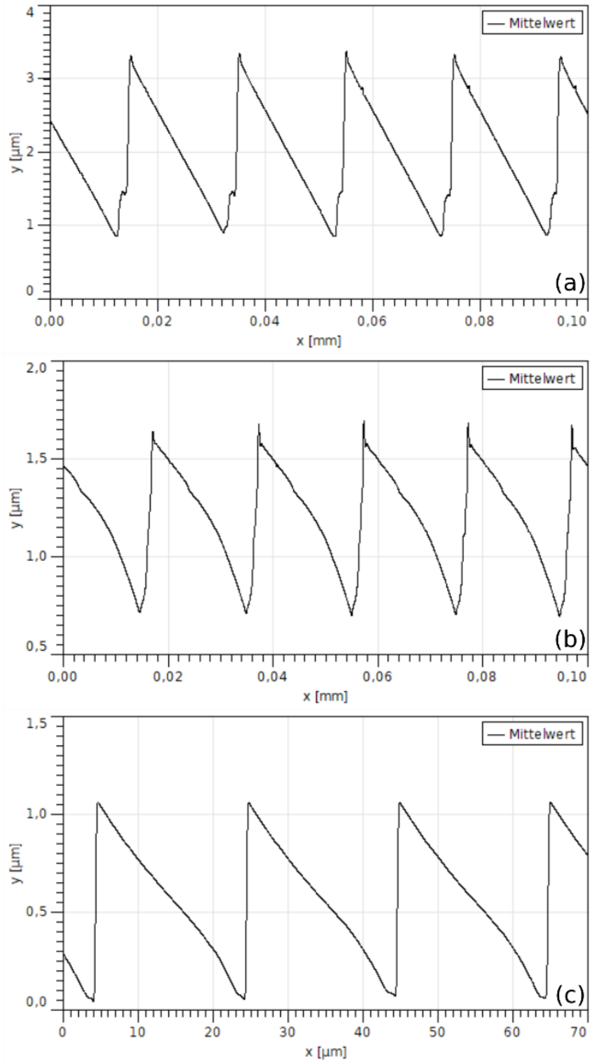

Figure 2. Ruled profiles with 8° and 2.8° blaze angle on test samples: (a) standard tool 8°, (b) standard tool 2.8° and (c) non-standard tool 2.8°.

For each position the diffraction orders are scanned, the intensity peak fitted with a gaussian function and finally the *LD* is fitted using eq. 3.1.

When the measurement points are placed along the grating length, it is possible to measure the uniformity of the *LD* or in case of variable line spacing (vls) gratings the polynomial. The polynomial can be given as

$$LD = a_0 + a_1 \cdot x + a_2 \cdot x^2 + a_3 \cdot x^3 \qquad (3.2)$$

for a given coordinate *x* along the grating length and a_i as vls coefficients.

While the test samples were constant line gratings ($a_{i>0} = 0$), the ruling on the grating substrate followed a polynomial to embed a focussing function in the grating. The specified value and its tolerance is compared to the achieved value fitted from over 50 measurements of the line density along the length of the grating (table 2).

Table 2. Requested and achieved vls coefficients of the grating.

	a_0	a_1
specified	50 ± 0.1	$-1.5\cdot10^{-3} \pm 2.25\cdot10^{-5}$
measured	49.9980 ± 0.0003	$-1.517\cdot10^{-3} \pm 8\cdot10^{-6}$

Additionally, stray light measurements were performed on grating samples. For these, the grating is rotated and the reflected intensity of the laser is measured by a photo diode. The typical high stray light background for ruled blaze gratings is clearly visible between the diffraction orders (figure 3a). Also the blazing effect can be seen: the background rises around the ruled blaze angle of approximately 3°. For comparison a laminar grating consisting of a photo resist mask with 150 L/mm is shown (figure 3b).

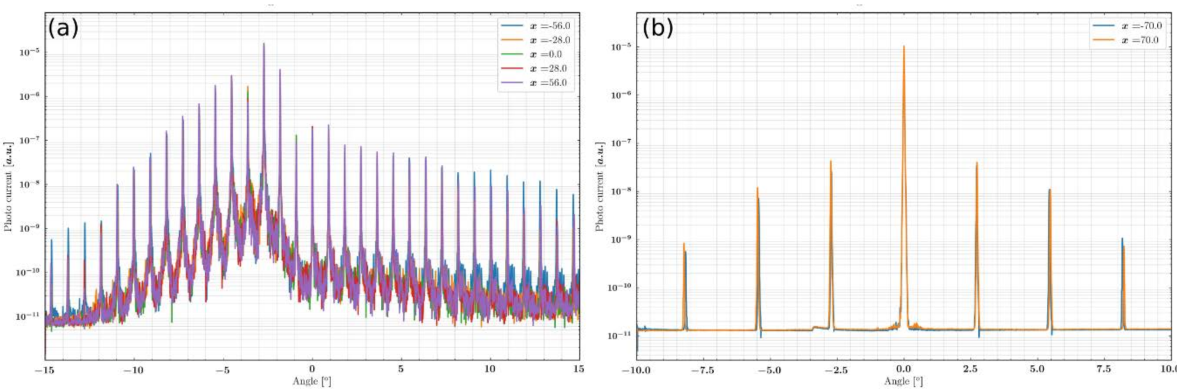

Figure 3. Stray light measurement of the 50 L/mm ruled blaze grating (a) in comparison to a 150 L/mm laminar grating as a photoresist mask (b). Both masks are on grating substrates.

3.1.2. Profile form To check the profile of a grating the standard method is measuring it by AFM. The AFM measurements are used to get information about the obtained blaze angle, the roughness of the blaze facets and the profile shape. The profile form is checked before and after the ion etching process. And again, if a reflection coating is applied to the grating. If the material and the structure size allows it, white light interferometry (WLI) can also be used.

In the beginning of the process development, the low line density profiles made it difficult to obtain the blaze angle due to non-linearity of the AFM scanner[1]. Therefore, the samples were additionally measured by WLI (figure 4). While the WLI measurements had no non-linearity, not all artefacts of the etched profiles could be measured. For example spikes at the apex were out of range of the PSI measurement mode and the pixels in the images were left blank. We tested another AFM scanner[2] and it did not show the problematic non-linearities. The new AFM scanner was available for the grating measurement.

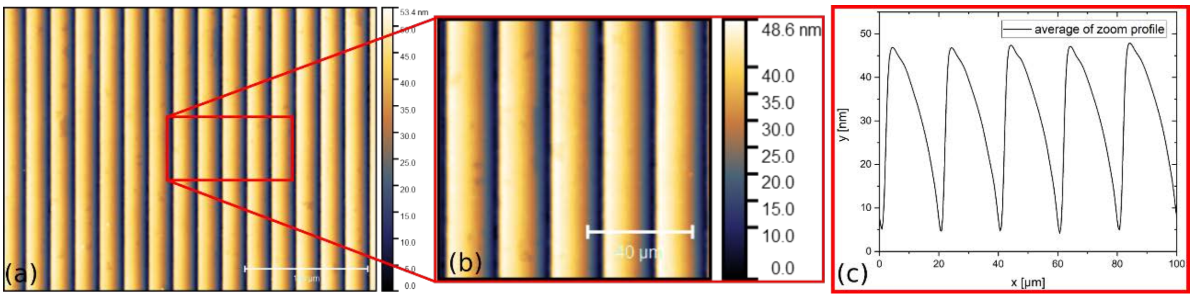

Figure 4. Average height profile derived from WLI measurements x20 (a), zoom (b) and according line profile of a test sample on a silicon wafer (c).

The profile parameters of the ruled, the etched and gold coated test sample are listed in table 3. The grating substrate was measured by AFM only.

[1] Nanosurf NaniteAFM
[2] Nanosurf FlexAFM

Table 3: Overview of profile parameters of test sample and of the grating.

Test sample	Ruled [a]	Etched [b]	Coated [b]
Blaze angle (°)	2.81 ± 0.04	0.146 ± 0.005	0.142 ± 0.005
Antiblaze angle (°)	28 ± 5	1.32 ± 0.12	1.35 ± 0.17
Facet micro roughness (nm rms)	4.7 ± 0.7	0.49 ± 0.07	0.52 ± 0.06
Grating [c]	Ruled	Etched	
Blaze angle (°)	2.91 ± 0.08	0.150 ± 0.004	
Antiblaze angle (°)	49 ± 2	7.0 ± 1.4	
Facet micro roughness (nm rms)	11.2 ± 1.1	0.99 ± 0.09	

[a] Nanosurf NaniteAFM
[b] Veeco WYKO NT1100
[c] Nanosurf FlexAFM

3.2. *In-situ metrology*
The test sample was measured at the reflectometer at the Optics Beamline at BESSY II [3]. For the measurements a gold layer was evaporated on the test sample on a silicon wafer (figure 5).

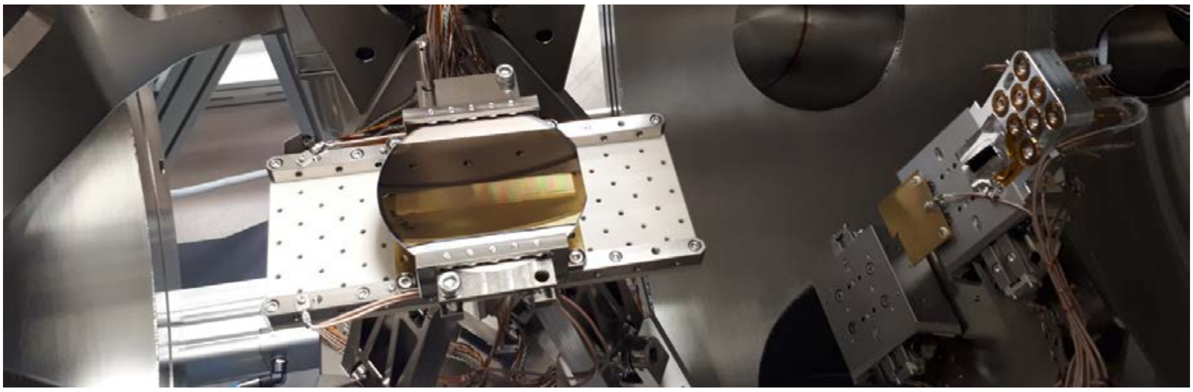

Figure 5. Test sample mounted in reflectometer at BESSY II Optics beamline.

The measured efficiency was compared to the efficiency expected of a simulated profile (see figure 6). For the simulation Reflec[3] was used, which assumes ideal triangular profiles. For example, the rounded shape of the blaze facets is not taken into account in the simulation. The agreement of simulation and measurement is better than 80%, which is normal and acceptable.

4. Summary and outlook
The process to manufacture gratings of low line density down to 50 L/mm and low blaze angles is now established at HZB. Ruling layers with the required thicknesses were evaporated onto the samples. The ruling process was adapted using a non-standard tool to minimize the necessary force during ruling. A high selectivity ion etching process was used to achieve the low blaze angle of 0.15°. The measurement equipment was upgraded to ensure the exact measurement of these profiles.

The first results of the commissioning of the delivered grating after installation in a grazing incidence variable line spacing spherical grating monochromator for SwissFEL (ATHOS beamline [4]) are expected by the end of 2022.

The focus of the ongoing improvements will be to commission the ruling engine GTM24. The GTM24 will be able to rule gratings with optical areas longer than 150 mm. And the ion etching process has to be transferred to another machine that can etch longer substrates. Additional ion beam etching to further decrease the facet roughness was already tested for a 92 L/mm test sample at the new machine and could be established as an after treatment process.

[3] Version 23.14 of APR/05/2016

5. Acknowledgments
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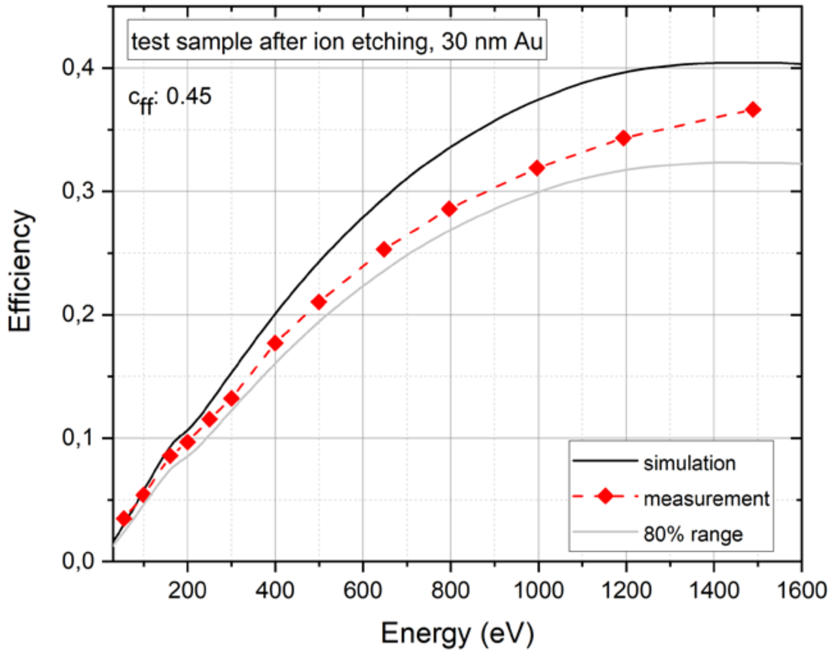

Figure 6. Measured (red) and simulated (black) efficiency for c_{ff} = 0.45 mount.

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