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Absolute, high-accuracy characterization of a variable line spacing grating for the European XFEL soft X-ray monochromator

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Abstract: The European XFEL is a free-electron laser (FEL) with a superconducting linear accelerator and three beamlines (SASE1, SASE2, and SASE3) covering the energy range from 250 eV to 24 keV. The SASE3 beamline is dedicated to the soft x-ray range (0.25-3 keV) and is designed to operate in both monochromatic and non-monochromatic mode. A variable line spacing – plane grating (VLS-PG) monochromator is placed along the beam transport system for the monochromatic mode. The VLS parameters of the grating profile are challenging from a manufacturing and measuring perspective, especially at the desired length of 530 mm. A shorter grating of 150 mm has been procured to allow early operation of the facility. We describe the characterization method that was used to assess the VLS parameters of the grating using Fizeau interferometry. The method is intrinsically absolute and limited only by the quality of the test grating and the noise level. Further measurements using a white-light-interferometry profilometer are also reported. We discuss the possibility of extending the method to the future 530 mm long grating.

OCIS codes: (050.1950) Diffraction gratings; (120.3940) Metrology; (120.3180) Interferometry; (340.0340) X-ray optics.

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1. Introduction

The European XFEL is a free-electron laser facility in Hamburg area, Germany. A 1700 m long, pulsed, superconducting linear accelerator produces electron bunches up to 17.5 GeV [1,2]. Three undulator chains (SASE1, SASE2, and SASE3) feed the corresponding beamlines, with different characteristics and energies. In particular, SASE3 is a soft X-ray beamline that covers a wide photon energy range in both monochromatic and non-monochromatic mode, from 270 to 3000 eV [3]. The monochromatic mode is provided by a reflective-grating-based monochromator in the beam transport optical setup. The optical setup is made up of a cylindrical pre-mirror that focuses the beam and a VLS grating. The result is a chromatic lateral dispersion of the beam over a slit, allowing an efficient filtering in energies with a high spectral efficiency. For this purpose, a 530 mm long grating was designed, but it turned out to be challenging to fabricate at this length. A shorter grating, 150 mm long with 120 mm clear aperture, was procured to allow early operation. The requirements for the short grating were also challenging because of the very precise VLS parameters that are required, but it was simpler to procure because of the shorter length. We report here on the metrological characterization of such a grating, with special focus on the absolute characterization of the VLS parameters that is crucial on this accuracy level.

2. VLS parameters and measurement setup

The grating has a certain number of design parameters that were measured. We report the specifications with tolerances in Table 1.

Table 1. VLS grating specifications

Substrate material	Monocrystalline silicon
Clear aperture	120 mm x 20 mm
Radius of curvature of the substrate	> 100 Km
Height error (substrate)	< 20 nm (P-V)
Groove profile depth	P–V (16 ± 2.4) nm
Duty cycle (valley to spacing ratio) c/d	(0.65 ± 0.1)
Groove Density Law (lines/mm) $b = b_0 + b_1 x + b_2 x^2$	$b_0 = (50 \pm 1.6)$ 1/mm $b_1 = (101 \pm 1) \times 10^{-5}$ 1/mm^2 $b_2 = (0 \pm 2) \times 10^{-7}$ 1/mm^3

Different parameters require different setups and instruments, which are described in the following sections.

2.1 Substrate characterization

For the substrate and the VLS characterization, Fizeau interferometry is used. The instrument is a 12 inches large-aperture Fizeau based on a Zygo Dynafiz [4], technical specifications reported in Table 2.

Table 2. Large-aperture Fizeau interferometer specifications

Measuring principle	Phase-shift interferometry
Diameter aperture	12 inches (304.8 mm)
Laser source	Stabilized He-Ne laser λ = 632.8 nm
Resolution	λ/12000 (high-resolution mode, double pass)
Camera size	1200 x 1200 pixels
Digitization	10 bit
Repeatability (nominal)	<0.25 nm (2σ)
Reference flats material	Fused silica
Reference flats quality (nominal)	λ/20
Reference flats clear aperture	12 in (304.8 mm)

The beam aperture has a 300 mm diameter, so the instrument can inspect the 150 mm long grating entirely, while this is not true for the future 530 mm long grating. (We discuss this issue and how we intend to approach it in Section 4.) For the substrate characterization, the grating is placed in normal incidence in front of the interferometer (Fig. 1(a)). The 2D map of

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the surface is measured and then corrected using the calibration map of the interferometer reference flat. The grating ruling etched on the surface does not disturb the measurement in this particular case because the first diffraction orders (± 1) are deflected at an angle $\vartheta = \sin^{-1}(\lambda . b)$ that, in our case, is going outside the acceptance angle of the interferometer. The measurement is not intrinsically absolute but becomes absolute when the map from the reference flat is removed from the measurement. The reference map is measured using a three-flat test–based procedure [5–10] by the producer and provided together with the flat. We did a verification of this reference map using a cavity measurement with our two flats [4] and the result was consistent with the calibration maps within the required precision of Table 1. For the VLS characterization, the required accuracy is much higher, so we could not trust this verification and, as a result, we used an intrinsically absolute method to carry out the measurement.

2.2 VLS parameters absolute characterization

The Littrow setup is used to measure the VLS parameters of the grating. The grating is rotated until the selected diffraction order goes back towards the interferometer (Fig. 1(b)).

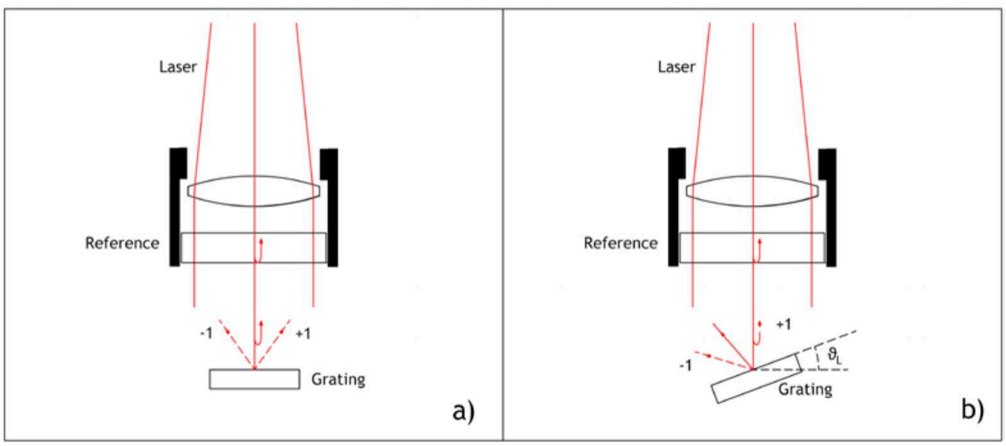

Fig. 1. Measurement setup for the grating substrate characterization in normal incidence (a) and for the VLS parameter measurement using the Littrow setup (b)

The angle of rotation ϑ_L is changing with the central ruling density b_0, the wavelength λ, and the chosen diffraction order m:

$$\vartheta_L = \sin^{-1}\left(\frac{m \cdot \lambda \cdot b_0}{2}\right). \tag{1}$$

The angle is measured in correspondence with different diffraction orders, internal or external, rotating the grating clockwise or counterclockwise with a high accuracy rotation table and aligning all the orders that have enough intensity to be detected. The rotation table is moved with a step motor, and the resolution of the stage is 0.002 degrees. In this way, we can measure the b_0 parameter.

For the other VLS parameters, the wavefront is diffracted back in the Fizeau interferometer in the Littrow setup and measured using phase-shift interferometry [5]. The angle errors of the diffracted beam along the grating are accounted by the formula:

$$\Delta\vartheta(x) \cong \sin^{-1}(m\lambda b). \tag{2}$$

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We do not measure angles with the Fizeau but rather a height profile that is given by an integration of the slope: $h(x) \cong \int \Delta\vartheta(x)dx$. The errors due to approximations in the formulas are in this case negligible because of the very small angle errors and tiny VLS parameters. A proper data analysis using the best fitting polynomial calculation gives a metrological measurement of the b_1 and b_2 VLS parameters. We used a least-square fitting algorithm provided with the Python "numpy" package.

As we show in Section 3.2, this method gives absolute measurements and is not sensitive to the reference flat contribution. The fundamental reason is that the measurements for corresponding internal and external orders can be combined, and the systematic error coming from the reference flat is wiped out. A similar setup is used in Ref [11]. in case of constant density gratings with the purpose to assess the quality of a ruling machine, while in our work the setup is fully developed to characterize a real VLS grating with a complete data analysis method.

2.3 Groove profile characterization

Groove profile depth and duty cycle are measured in several points of the sample using white light interferometry (WLI). The instrument is a Bruker Wyko NT9100 profilometer (Veeco Instruments Inc., www.veeco.com). Technical specifications of the profilometer that we used are reported in Table 3.

Table 3. Bruker Wyko NT9100 profilometer specifications

Measuring principle	White light phase-shift interferometry
Camera resolution	640 x 480 pixels
Vertical resolution	< 0.1 nm
rms repeatability	0.05 nm
Step height accuracy	0.8%
Magnification lens used: Mirau 50X	
Numerical aperture	0.55
Optical resolution	0.5 microns
Field of view	62 x 47 microns

The parameters are calculated with a statistical elaboration of the measured 2D maps as described in Section 3.3. The depth of the profile is calculated reporting the statistical occurrences of the heights and taking the distance between the two distributions corresponding to the peaks and the valleys. The valley-to-spacing ratio is calculated taking the percentage of points below the half of the height (50% in case of a perfectly laminar grating). These parameters are important for the calculation of the grating efficiency.

3. Description of the measurements and results

3.1 Substrate characterization

Using the setup described in Section 2.1, the substrate of the grating is measured. The resulting 2D map is reported together with the central profile (Fig. 2). The grating substrate was processed with classical polishing and ion-beam figuring and therefore is very flat. This was considered to be very important for the manufacturing of the grating that was done through holography and etching.

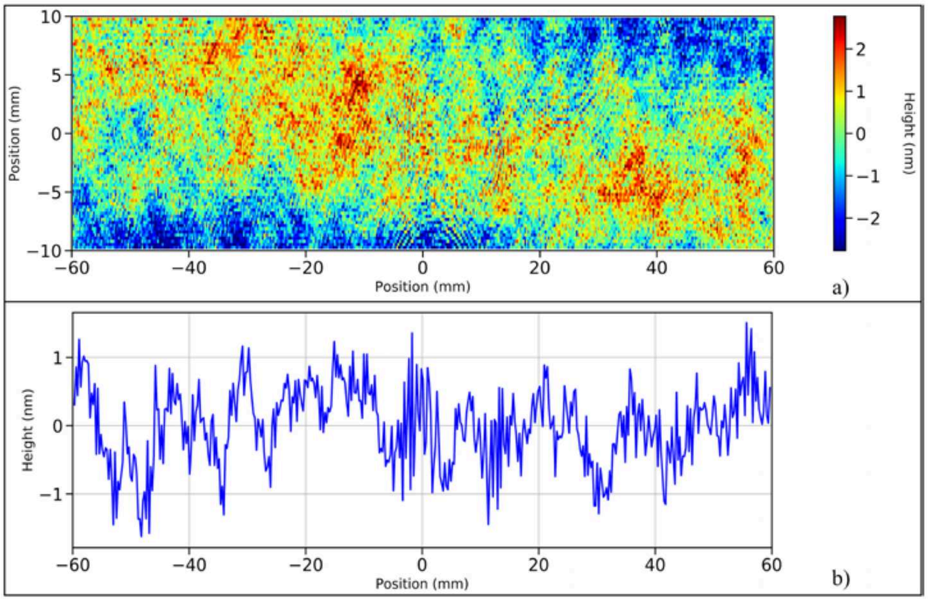

Fig. 2. Measurement of the grating surface, with 2D map (a) and central profile (b)

3.2 VLS parameters characterization

The VLS measurement is performed with the grating rotated to be aligned in the Littrow setup. We can see a typical measurement in Fig. 3, corresponding to diffraction order m = 1. The polynomial law for the grating density is $b = b_0 + b_1 x + b_2 x^2$. In case of very small angles, we can linearize Eq. (2) and, using the integration, we end up with a third-power law for the height profile: $h = m\lambda\left(b_0 x + b_1 \frac{x^2}{2} + b_2 \frac{x^3}{3}\right) + c$. In Fig. 3, the central profile and the 2D map are shown with the best parabola removed, to better display the residual wavefront aberrations and the influence of b_2.

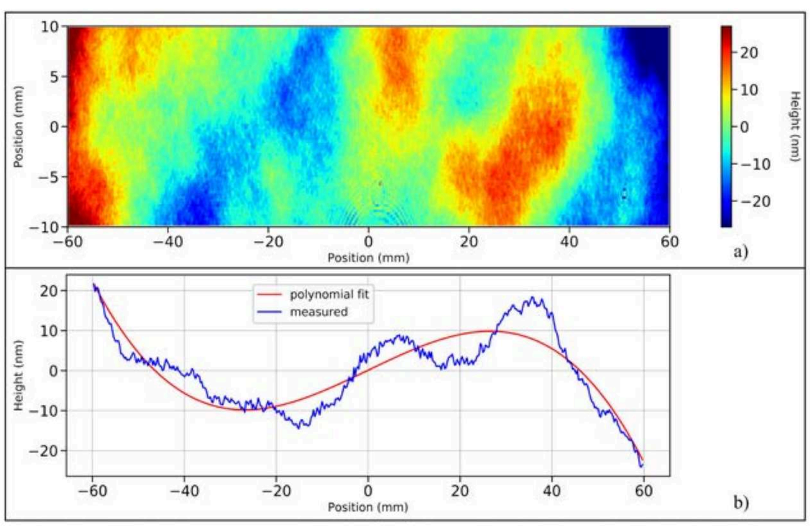

Fig. 3. Measurement of the diffracted wavefront from order 1, in the Littrow setup: 2D map (a) and central profile with the best third-order polynomial fit (b). The best fitting parabolic term is removed to show the small effect coming by the non-zero b_2 parameter.

The best third-order polynomial for the central profile is computed. The VLS parameters are then calculated. While in general the exact calibration of the lateral scale is not very important in an interferometric measurement of the height profile, in this case it is crucial because it is used in the integration and therefore directly affects the best-fitting VLS parameters. For this reason, we carefully calibrated the lateral resolution of the Fizeau interferometer using a mechanical mask with known dimensions and 0.1 mm tolerance placed inside a cavity measurement done with two flats. Additionally, we corrected also for the different tilt introduced because of the Littrow setup, even if it turned out to have a negligible effect on the first and second orders of diffraction because of the small angle. The final accuracy that we claim is the combination of the measurement reproducibility and the contribution on the uncertainty coming from all the variables in Eq. (2). We found that the uncertainty connected to the lateral resolution calibration is the most important one.

An important characteristic of this measurement method is that it is absolute after the different contributions coming from internal and external orders are combined, that is, when we rotate the grating clockwise or counterclockwise. The two measured wavefronts are in principle identical in magnitude but opposite in sign. This is confirmed by our measurements (Fig. 4).

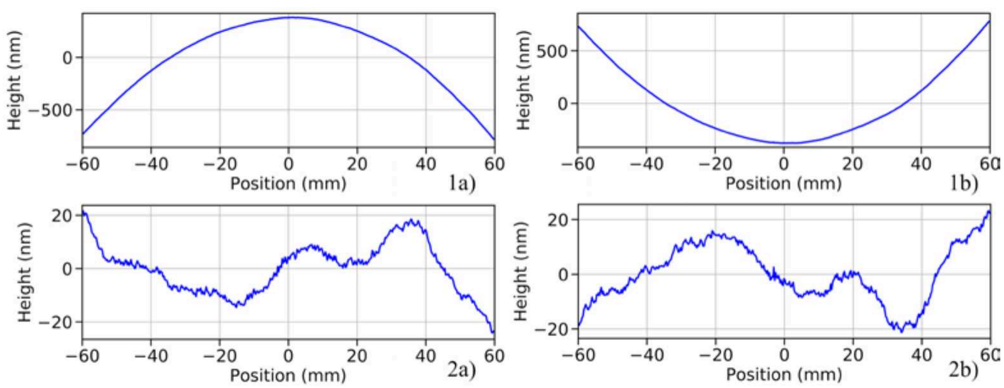

Fig. 4. Measurement of the diffracted wavefront from order 1, corresponding to internal (a) and external (b) order. Central profiles are including the parabolic term (1a and 1b) or not (2a and 2b).

Computing the difference between the internal and external orders and dividing by 2, we wipe out the residual contribution of the reference flat that is common to both measurements. In this way, the estimated VLS parameters are not influenced by any systematic error related to the reference flat. We were able to follow this procedure for diffraction orders 1 and 2.

To measure the b_0 parameter, the Littrow angle is measured according to Eq. (1) for several diffraction orders: it was possible to observe up to seven diffraction orders, internal and external.

We report the final results in Table 4, where the errors are calculated combining the reproducible errors and the ones propagated by the Eqs. (1)-(2). The errors are on the level of 1% to 3% and they are defining the accuracy on the b_1 and b_2 measurement in the current setup.

3.3 Groove profile characterization

It is very important to measure the details of the ruling structure for calculation of the efficiency of the grating. Using a white-light interferometer (Table 3), it is possible to directly inspect the groove profile in single points. The instrument is provided with a translation stage to measure the position of the point inspected. For the VLS gratings, it is generally very important to control this parameter because the characteristics of the grating are changing along its length. Here, this variation is desensitized by the very small VLS parameters: the difference in spacing is ± 24 nm from the central point, so, on the lateral resolution of the profilometer (0.5 microns), the spacing looks similar. We report an example of such a characterization. Calculating the statistical properties of the map, we measure the profile depth and duty cycle (Fig. 5). The measurement is repeated in several points of the grating to improve the statistics.

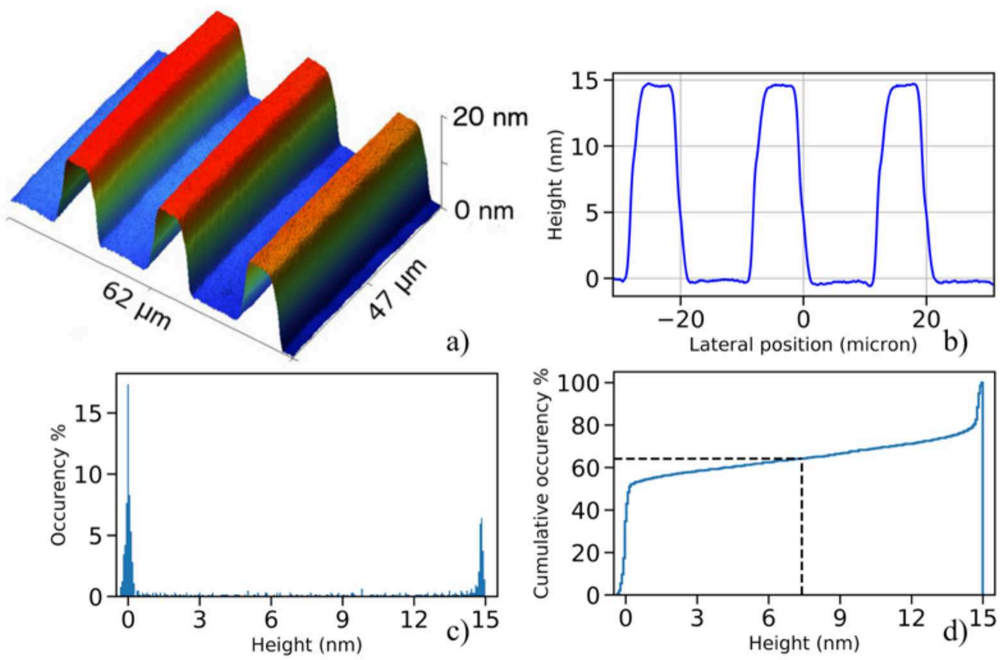

Fig. 5. Example of a WLI measurement: 2D map (a), central profile (b), statistical analysis to retrieve profile depth (c), and duty cycle (d).

Table 4. Measured parameters on the grating

Height error (substrate)	< 5 nm (P–V)
Groove profile depth	P–V 14.8 nm
Duty cycle c/d	0.64
Groove Density Law (lines/mm) $b = b_0 + b_1 x + b_2 x^2$	b_0 = (50.1 ± 0.3) 1/mm b_1 = (101 ± 1) x 10^{-5} 1/mm^2 b_2 = (12.3 ± 0.3) x 10^{-7} 1/mm^3

4. Proposed improvements of the measurement setup and conclusions

The future gratings designed for the European XFEL will be 530 mm long, and, therefore, it will not be possible to measure the entire surface in one shot with a 300 mm beam aperture Fizeau. An additional problem is that, despite the same specifications for the VLS parameters, the grating is much longer and the peak-to-valley of the diffracted wavefront as shown in Figs. 4(1a)-4(1b) will also scale accordingly. However, in our case, we would have a total of 20 microns over 500 mm, still small enough to be measured introducing a stitching strategy with limited angle correction [12].

A possible improvement of the setup would be a more accurate rotational stage to lower the uncertainty of b_0. This is in any case not considered very critical for the final usage of the grating. The other parameters, especially b_1 and b_2, are much more critical because they affect the focus position and the aberration of the beam, respectively. In case of b_2, we found that the grating was out of specifications, but the next step will be to use the measured parameters in a wavefront calculation code to analyze the real impact on the diffracted x-ray beam.

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5. Conclusion

We conclude by stating that we developed an extensive method to characterize the VLS grating designed for the European XFEL soft x-ray monochromator. The method is able to retrieve very small VLS parameters in an absolute way using Fizeau interferometry, and this is required when small tolerances are needed.