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Variable line spacing diffraction grating fabricated by direct write lithography for synchrotron beamline applications

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ABSTRACT

A Variable Line Spacing (VLS) diffraction grating has been fabricated using an optical direct write technique. This grating is now in use at the Advanced Light Source, in beamline 12.0.1, delivering light for EUV lithography. Direct Write Lithography (DWL) with focused light at $\lambda = 442$ nm was used for the first time to record a VLS grating pattern on a substrate coated with a photoresist. The pattern was transferred to the Si substrate surface using reactive plasma etch. Precision of groove placement was verified by wavefront measurements of a witness grating recorded simultaneously with the VLS pattern. Atomic force microscope measurements confirmed near ideal groove shape and high smoothness of the grating grooves. The grating coated with a Ru coating demonstrated diffraction efficiency of 39.5% in the negative first diffraction order which corresponds to theoretical efficiency at the wavelength of 13.5 nm. This work validates the DWL approach as a promising technique for advanced grating fabrication.

Key words: diffraction grating, x-rays, direct write lithography, wavefront measurements, AFM, plasma etch.

1. INTRODUCTION

The synchrotron community is hungry for new types of x-ray diffraction gratings because they are the key optical component for EUV and soft x-ray instrumentation. Requirements for x-ray gratings are constantly growing to provide higher throughput and higher spectral resolution. Although traditional manufacturing methods are capable of meeting some of the needs, new optical designs are often limited by the availability of gratings. For example: advanced spectroscopy techniques such as Resonant Inelastic X-ray Scattering (RIXS) [1] require gratings with extreme resolving power and efficiency [2,3]. In lower resolution RIXS, with application in energy sciences, the need is for gratings with extremely large aperture, driving the development of gratings with highly curved grooves for aberration correction. The variation of the line spacing is often exploited to focus and to correct aberrations. As optical deigns become more sophisticated the fidelity required of the line-space pattern is beyond what interference lithography can provide and a focused beam pattern writing tool is required.

Optical direct write lithography (DWL) is a very promising technique for making x-ray diffraction optics including Variable Line Spacing (VLS) diffraction gratings. A grating pattern is recorded by a scanned, focused laser beam on the grating blank surface coated with a resist. Exposure is followed by nanofabrication processing to mill the pattern to the Si substrate and form lamellar or blazed grooves [4,5]. The required pattern is achieved using an interferometrically controlled stage to carry the substrate and by varying the illumination intensity of the focused beam during the writing process.

The DWL approach is flexible and is capable of providing virtually any groove density variation that is required for high order aberration correction. In terms of writing principles, the DWL method is similar to classical diamond ruling. However DWL is much faster and hence less sensitive to environmental drift issues and can provide more precise gratings in a cost effective manner. We investigated capabilities of several DWL tools for grating manufacture earlier [4]. Here we demonstrate the fabrication and characterization of a VLS diffraction grating for the beamline 12.0.1 at the Advance Light Source (ALS) by the DWL method.

2. GRATING FABRICATION AND TESTING

The constant included angle monochromator of the ALS EUV lithography beamline 12.0.1 requires a VLS plane grating operating in converging light provided by a spherical premirror [6]. Variation of groove density is designed to provide an erect focal plane and to correct the spherical aberration of the premirror. The groove density is $g_0 = 200 lines/mm$ in the center of the grating and varies along the grating length according to the formula:

$$g = g_0(1 + g_1 w + g_2 w^2 + g_3 w^3), \qquad (1)$$

where w is a coordinate along the grating surface, and the polynomial coefficients are $g_1 = -2.4771 \times 10^{-3}\mathrm{mm}^{-1}$, $g_2 = -3.882 \times 10^{-6}\mathrm{mm}^{-2}$, $g_3 = -1.721 \times 10^{-8}\mathrm{mm}^{-3}$.

Due to the need to improve the overall transmission of the beamline and to optimize it for EUV lithography, the grating was recently replaced with one written by the DWL technique. Presently, we have fabricated and installed a lamellar groove grating to validate the DWL approach. We are in the process now of producing a blazed grating using the anisotropic etching techniques developed in our lab [7].

We used a DWL66 writer from Heidelberg Gmbh [8] equipped with a HeGd laser of wavelength of 442 nm, available at the Biomolecular Nanotechnology Center [9], UC Berkeley, for the grating pattern recording. The DWL 66 is an early version of this class of machine, and current Heidelberg machines have a writing speed that is 10-50 times faster and have absolute errors a factor of 5 smaller. However it is an adequate tool for making low groove density gratings for low and medium resolution monochromators.

High throughput of the BL12.0.1 monochromator is paramount for EUV lithography purposes while spectral resolution requirements are modest. The requirements for surface slope errors are relaxed (1-2 µrad) and can be satisfied with relatively inexpensive substrates. We used a Si substrate of 100 mm in diameter and 5 mm in thickness from Gooch & Housego with surface flatness specified as better than $\lambda/10$ ($\lambda = 633$ nm) for 80 % of the clear aperture of the substrate. The thickness of the substrate was limited by the dimensions of the stage of the DWL66 tool used in this work, which cannot accommodate thicker substrates without reconfiguration. Because of this limitation there was some concern regarding flatness of the grating surface which was a subject of thorough monitoring through the whole fabrication process.

The surface of the substrate was characterized with a Zygo GPI Fizeau interferometer in order to locate the best region for the grating pattern. It was found that despite substantial global curvature of the substrate its central part is very flat (Fig. 1a). The substrate was marked and placed into the DWL66 stage in an appropriate way to write the VLS grating in that best region (Fig. 1b). The substrate was spin-coated with BARLi antireflective coating and then S1805 photoresist with thickness of 250 nm for each layer.

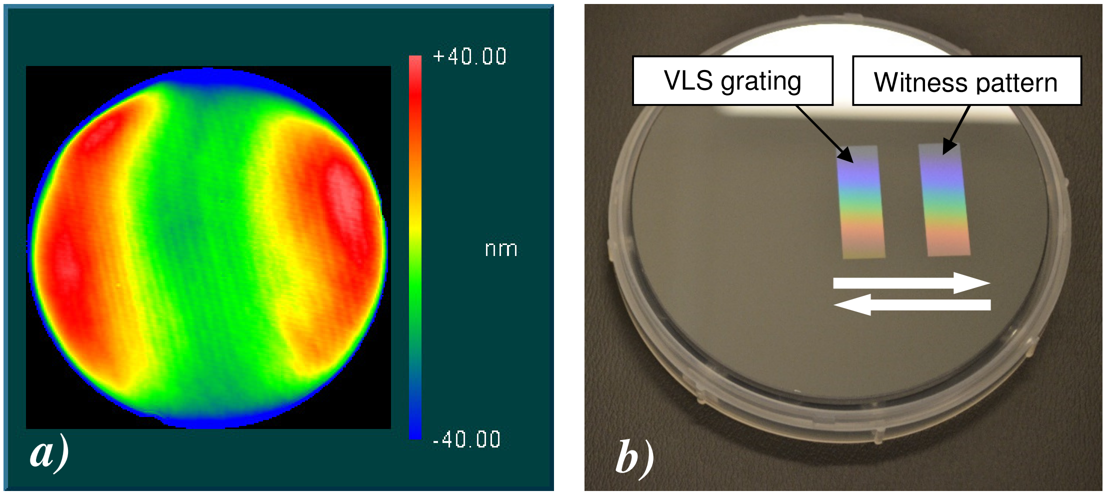

Figure 1. Wavefront measurements of a 5 mm thick Si substrate before pattern recording (a). Photograph of the VLS grating and the witness constant groove density pattern on the silicon substrate (b). The VLS pattern was located in the central part of the substrate with the best flatness of the surface. White arrows depict the direction of fast scanning during the recording process.

After exposure and resist development the Zygo interferometer was used to characterize groove position accuracy [10] with a differential wavefront measurement technique that we have previously developed [4]. Briefly a grating is set in the Littrow geometry for an n^{th} diffraction order and the diffraction wavefront recorded. For an ideal constant groove density plane grating the wavefront should be flat while low frequency errors in position of grating grooves result in perturbed fronts. Waviness of the grating surface as well as imperfection of the instrument optics also contribute to the wavefront curvature. To separate these last contributions, the grating can be flipped to the Littrow geometry for the negative -nth diffraction order. Subtraction of the two wavefront measurements cancels the wavefront errors related to the surface of the grating and instrument optics while the wavefront distortions caused by groove displacement double. The groove placement errors can be easily calculated by the formula [10]:

$$\sigma=2\varepsilon \sin\beta , \qquad (2)$$

where σ is wavefront errors, ε is an absolute displacement grooves from their ideal positions, β is a diffraction angle in the Littrow geometry.

These differential wavefront measurements work very well for plane constant groove density gratings which produce equally spaced interference fringes over whole the grating area (Fig. 2b) and are easy to convert into groove displacements. Characterization of spherical or VLS gratings is complicated due to their focusing properties which result in curved wavefronts. Interference fringes can be resolved only in a narrow area of the VLS pattern (Fig. 2b). Wavefront measurements of focusing optics require additional collimating optics which can contribute to wavefront error due to its own imperfections and aberrations. To avoid these difficulties the VLS grating was characterized indirectly. For this purpose an additional witness pattern was recoded alongside the VLS pattern (Fig. 1b). The witness pattern was a 200 lines/mm constant groove density grating of the same size with grooves parallel to the ones of the VLS grating. The patterns were written simultaneously by scanning along the grooves across both the patterns as depicted by arrows in Fig. 1b. As the first line of the VLS pattern was exposed the writing head moved to the witness pattern to write a first line of the witness pattern then went back to the VLS pattern to expose the 2nd line etc. Low frequency variations in groove density caused by stability issues of any kinds are assumed to be the same for both the gratings. In this way wavefront measurements of the constant groove density pattern provide information on groove placement accuracy for the VLS pattern.

A differential wavefront error map obtained by subtraction of wavefronts for the 1st positive and negative orders of the witness grating is shown in Fig. 2c. The error surface is very uniform along the direction parallel to grooves, i.e. along the fast scan direction, while substantial wavefront waviness is observed along the grating length (slow scan direction). This indicates that the writing process was affected by slow frequency variation of environmental conditions such as air temperature, pressure, humidity etc. An average wavefront error profile was calculated by averaging all the lines of the wavefront error map shown in Fig. 2c, and errors in groove position along the grating length were calculated using formula (2). The results are shown in Fig. 3a. The groove displacement does not exceed +/-100 nm peak-to-valley which is less than 4% of the grating period. Groove displacement results in variation of groove density of the grating of 0.004 mm^{-1} rms as shown in Fig. 3b with a red curve. The errors in groove density can be compared to variation of groove density of the VLS grating due to the polynomial terms. A contribution of the second order term which minimizes defocus for a range of energies and compensates aberrations of the spherical premirror is much bigger than the errors (compare blue and red curves in Fig. 3b). Variation of local groove density can affect resolution of a monochromator since they result in variation of diffraction angle. However, the diffraction angle variation caused by such small groove density errors is below 0.5 µrads (Fig. 3c) which is well within the tolerance range. One can conclude that low frequency errors in groove position should not affect performance of the VLS grating monochromator.

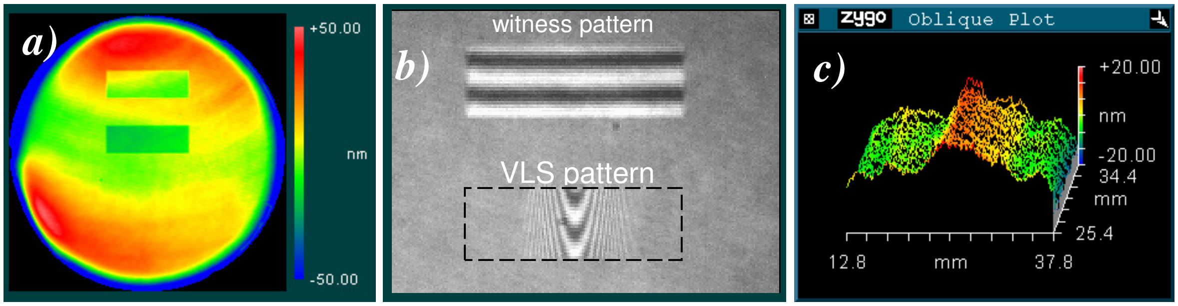

Figure 2. Wavefront measurements of the DWL patterns: (a) the Si substrate with the VLS and witness patterns in zero order: (b) interference fringes for the VLS and the witness patterns in the Littrow geometry for the 1st diffraction order: (c) differential wavefront error map of the witness pattern.

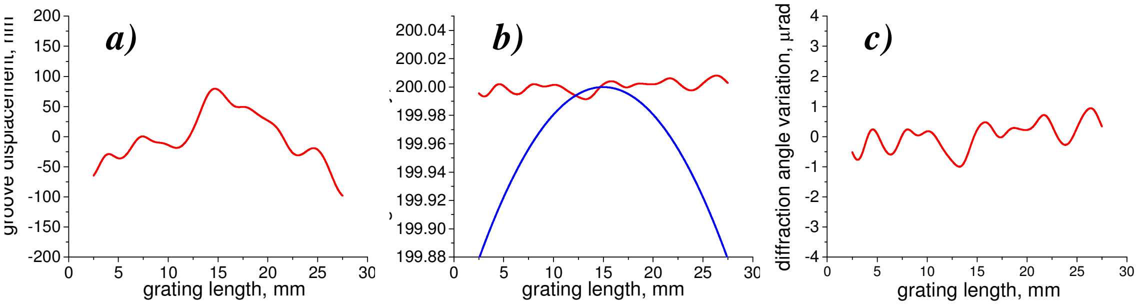

Figure 3. Groove displacement (a), groove density variation (b), and diffraction angle variation (c) for the 200 lines/mm witness grating. Calculated variation of the groove density due to the second order polynomial term of the VLS grating is shown with a blue line for comparison.

The patterned substrate was processed using nanofabrication capabilities of the Molecular Foundry, LBNL, to transfer the resist patterns to the Si surface. Reactive CHF_3 plasma etch was applied to make lamellar grooves of optimal depth (Fig. 4). The goal for the groove depth of 28 nm provides maximum diffraction efficiency according to results of simulations (Fig. 5) performed using GSolver electromagnetic code [11]. AFM measurements confirmed the high quality of the rectangular grooves with a surface roughness of 0.1 nm rms measured over a 20×20 μm^2 scan area (Fig. 4). The measurements yield a groove depth of 29 nm which meets the goal for the depth value within the accuracy of the measurements.

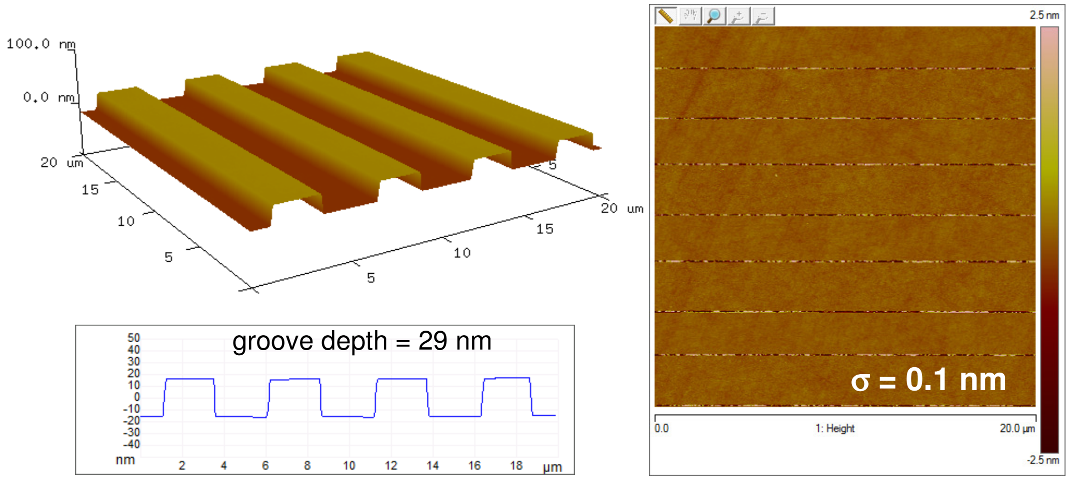

Figure 4. AFM image, profile, and surface roughness measurements of the silicon VLS grating.

The Si gratings were coated with a 30 nm thick Ru coating which is the best material for high reflectivity at the wavelength of 13.5 nm. The coating was deposited at the CXRO facility, LBNL. Diffraction efficiency of the VLS grating was measured at beamline 6.3.2, at the ALS [12]. The measurements were performed at a wavelength of 13.5 nm in the monochromator geometry, i.e. by rotation of the grating at a constant included angle of 166º. The grating demonstrates diffraction efficiency of 35.9% of the negative 1st diffraction order which is almost as high as the theoretical value of 36.98% calculated with the PCGrate electromagnetic code [13] using the groove profile measured by AFM (Fig. 4). At the same time the zero order is almost completely suppressed confirming the optimal depth of the lamellar grooves was achieved.

After grating fabrication the substrate was cut down to dimensions of 30×20 mm^2 to fit the grating mounting in the monochromator. Additional wavefront measurements were performed afterwards to characterize possible changes of the shape of the grating surface. It was found that after cut the grating surface relaxed into a cylindrical shape (Fig. 7b) with a radius of curvature of 8 km (Fig. 7c). The residual slope variations after subtraction of the best fit do not exceed 0.15 µrad rms (Fig. 7d). The average radius of curvature of 8 km is a typical tolerance for x-ray optics. The curvature can affect focal distance slightly but the resulting defocusing can be compensated if necessary by tiny included angle corrections or slit position adjustment.

The lamellar grating was recently installed in the monochromator of beamline 12.0.1 at ALS. Tests of the monochromator performances are currently underway and their results will be reported soon.

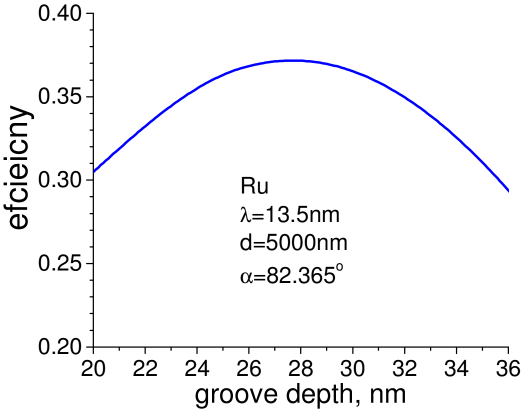

Figure 5. Calculated dependence of the diffraction efficiency of a Ru-coated lamellar diffraction grating on groove depth for an incidence angle of 82.365º and the wavelength of 13.5 nm.

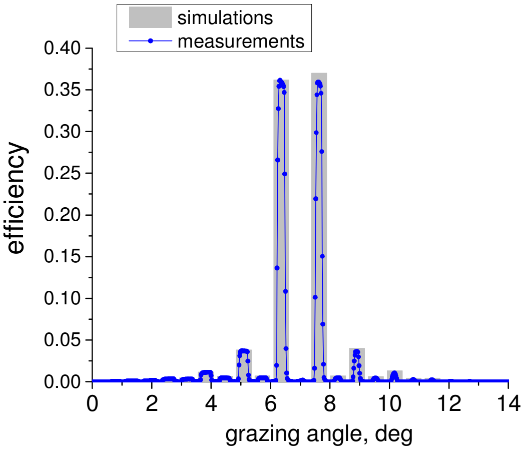

Figure 6. Calculated (bars) and measured (line with symbols) diffraction efficiency of the Ru-coated VLS grating at a constant included angle of 166º versus the grazing angle of incidence.

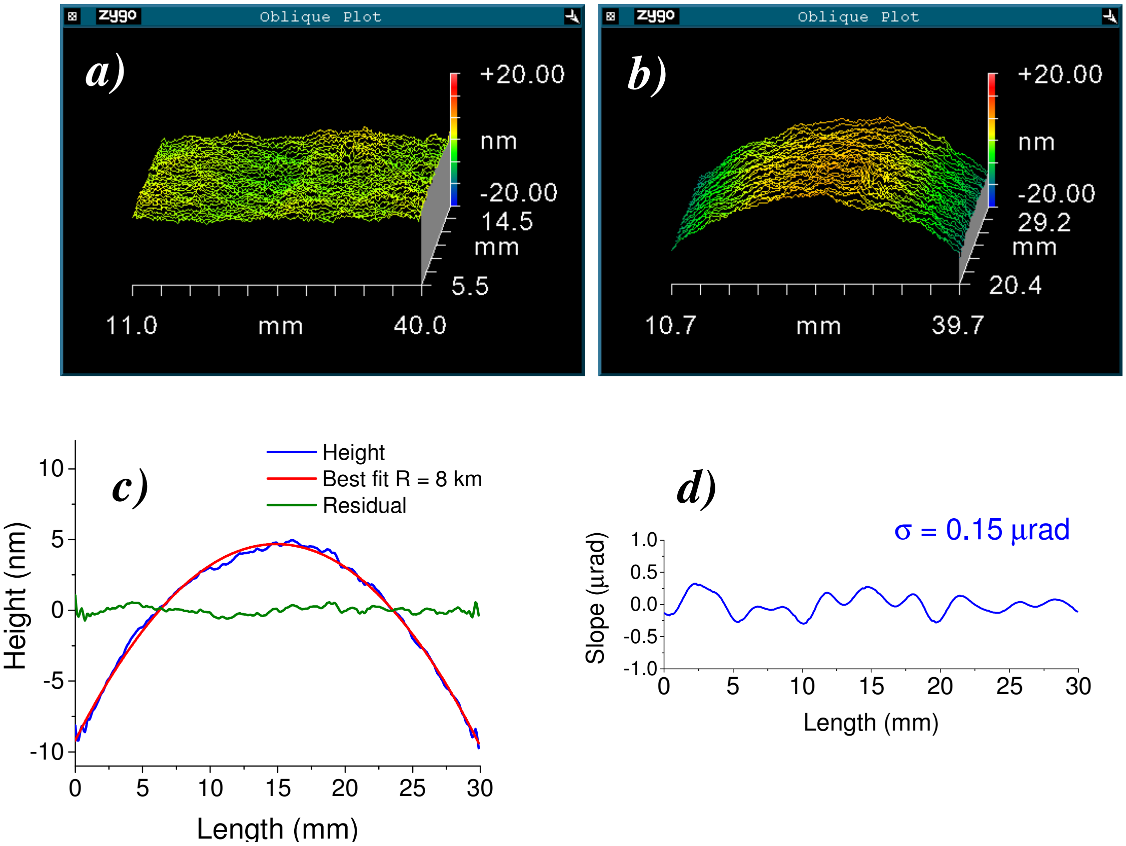

Figure 7. Wavefront measurements of the VLS patterned area before (a) and after (b) substrate cutting. Average profile of the substrate with a radius of curvature of 8 km (c). Residual slope variation of the rating after subtracting best fit (d).

SUMMARY

Fabrication of a VLS diffraction grating for EUV/soft x-ray applications by optical direct write lithography was demonstrated for the first time. All the fabrication steps including pattern generation, pattern recording, plasma etch, coating, and characterization were performed using in-house capabilities of LBNL and UC Berkeley. The grating was comprehensively characterized in terms of surface shape, groove placement accuracy, groove shape, surface roughness, and diffraction efficiency. The lamellar grating coated with a Ru coating demonstrated diffraction efficiency as high as the theoretical value confirming the high quality of the grating. The results of this work validate the DWL approach as a potentially leading technique for the fabrication of diffraction gratings for advanced x-ray instrumentation.

ACKNOWLEDGEMENTS

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DISCLAIMER

REFERENCES

1. A. Kotani, S. Shin, “Resonant inelastic x-ray scattering spectra for electrons in solids,” Rev. Mod. Phys. 73 (2001) 203-246.
2. V.N. Strocov, “Concept of a spectrometer for resonant inelastic X-ray scattering with parallel detection in incoming and outgoing photon energies”, J. Synchrotron Rad. 17, (2010) 103–106
3. T. Warwick, Y.-D. Chuang, D. L. Voronov and H. A. Padmore, “A multiplexed high-resolution imaging spectrometer for resonant inelastic soft X-ray scattering spectroscopy”, J. Synchrotron Rad. 21 (2014) 736-743
4. D.L. Voronov, S. Diez, P. Lum, S.A. Hidalgo, T. Warwick, N. Artemiev, and H.A. Padmore, “Fabrication of x-ray gratings by direct write mask-less lithography”, Proc. of SPIE 8848, (2013) 88480Q.
5. D.L. Voronov, E.M. Gullikson, F. Salmassi, T.Warwick, and H.A. Padmore, “Enhancement of diffraction efficiency via higher order operation of a multilayer blazed grating”, Opt. Lett. 39(11) 3157-3160 (2014)
6. http://www.cxro.lbl.gov/MET
7. D.L. Voronov, E.H. Anderson, M. Ahn, R. Cambie, Ch.-H. Chang, E.M. Gullikson, R.K. Heilmann, F. Salmassi, M.L. Schattenburg, T. Warwick, V.V. Yashchuk, L. Zipp, H.A. Padmore, “High-efficiency 5000 lines/mm multilayer-coated blazed grating for EUV wavelength range”, Opt.Lett. 35(15), 2010, 2615-2617.
8. http://www.himt.de
9. Biomolecular Nanotechnology Center, QB-3, UCB http://qb3.berkeley.edu/qb3/bnc/index.cfm
10. M.C. Hutley, Diffraction gratings, Academic press, 1982, 330 p
11. http://www.gsolver.com

12. http://www.cxro.lbl.gov/reflectometer
13. http://www.pcgrate.com