Title
Ultra-high Resolution Optics for EUV and Soft X-ray Inelastic Scattering

Author
Voronov, Dmitry L.

Publication Date
2010-03-01

Ultra-high Resolution Optics for EUV and Soft X-ray Inelastic Scattering

Dmitry L. Voronov,[a] Rossana Cambie,[a] Minseung Ahn,[b] Erik H. Anderson,[a]
Chih-Hao Chang,[b] Eric M. Gullikson,[a] Ralf K. Heilmann,[b] Farhad Salmassi,[a]
Mark L. Schattenburg,[b] Valeriy V. Yashchuk,[a] and Howard A. Padmore[a]

[a]*Lawrence Berkeley National Laboratory, CA, United States*
[b]*Massachusetts Institute of Technology, Cambridge, MA, United States*

Abstract. We describe a revolutionary new approach to high spectral resolution soft x-ray optics. Conventionally in the soft x-ray energy range, high spectral resolution is obtained by use of a relatively low line density grating operated in 1st order with small slits. This severely limits throughput. This limitation can be removed by use of a grating either in very high order, or with very high line density, if one can maintain high diffraction efficiency. We have developed a new technology for achieving both of these goals which should allow high throughput spectroscopy, at resolving powers of up to 10^6 at 1 keV. Such optics should provide a revolutionary advance for high resolution lifetime free spectroscopy, such as RIXS, and for pulse compression of chirped beams. We report recent developmental fabrication and characterization of a prototype grating optimized for 14.2 nm EUV light. The prototype grating with a 200 nm period of the blazed grating substrate coated with 20 Mo/Si bilayers with a period of 7.1 nm demonstrates good dispersion in the third order (effective groove density of 15,000 lines per mm) with a diffraction efficiency of more than 33%.

Keywords: blazed gratings, multilayer EUV, soft x-rays, lithography, anisotropic etch
PACS: 07.85.Fv, 07.85.Qe

INTRODUCTION

Recent accomplishments in the development of ultra-high resolution diffraction gratings for EUV and soft x-ray applications are discussed below. The work started at the Advanced Light Source (ALS), LBNL in 2007 as a Laboratory Directed Research and Development (LDRD) project. The goal is to establish and demonstrate the technology required for ultra-high resolution Resonant Inelastic soft X-ray Scattering (RIXS) [1-4]. RIXS is a relatively new probe of matter, which can directly measure the energies of the soft excitations that are thought to be at the root of the complex properties of correlated electronic systems such as high Tc superconductors. Its main feature is that it is a spectroscopic probe that avoids limitations imposed by core hole lifetime energy broadening of conventional spectroscopies.

Although many interesting experiments have been performed, the power of the RIXS technique has not been fully exploited because conventional grating spectrometers have not been capable of achieving the extreme resolving powers that RIXS can utilize. State of the art spectrometers in the soft x-ray energy range achieve 0.25 eV resolution, compared to the energy scales of soft excitations and superconducting gap openings that are only a few meV. Achieving such an improvement cannot be made with conventional grating optics. First, the improvement of the resolution would dictate use of slit sizes that are extremely small and, therefore, difficult to fabricate. Second, with such small slit sizes, almost all flux would be lost. Third, extremely large spectrometers would be required. And fourth, because of the large size of an emission spectrometer and strongly collimated beam, unrealistically small optical slope errors would be required in the optics of the spectrometer.

The present investigation is directed to develop a technology for fabrication of small-sized diffraction gratings with ultra-high spectral resolution and high efficiency capable to overcome the deadlocks listed above.

There are three principle ways to achieve the necessary specifications for RIXS at meV energy resolution in the soft x-ray energy range – Fig. 1.

One way is to use a reasonably low density grating in high order - Fig. 1a. For this approach, simulations [5] have shown that it is possible to design a multilayer (ML) grating that effectively diffracts almost all the energy into a defined order. The key to this is to reduce the land to period ratio to the point, where there is no overlap between orders, and to design the land with a width to period ratio to match the location of the first maximum in the normal slit response function. However, the issue is that higher orders would require impractically small land to period ratios.

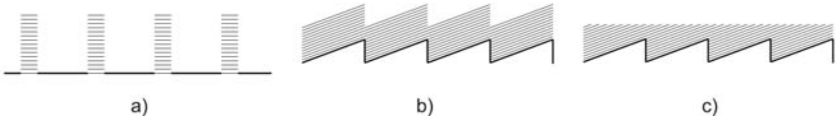

FIGURE 1. Different approaches to obtain high resolution in the soft x-ray energy range: a) grating operating in high order [5]; b) blazed multilayer grating optimized for high order [6-14], c) extremely high line density sliced grating working in the first order [5,12-14].

Another approach that has been under development in different labs for the last two decades is based on blazed multilayer gratings optimized for high order [6-13] – Fig. 1b. The performance of the blazed grating is determined by the surface quality and dimensional tolerances of the echellette substrate, as well as by the quality of the deposited multilayer. The developed fabrication methods based on interference lithography combined with ion-beam etching [7,10] and gray-scale e-beam lithography [8] allow currently to achieve a diffraction efficiency of 30-40% at the EUV wavelengths of 12-16 nm with a grating with the groove density up to 3000 grooves/mm optimized for diffraction in the first or the second order [7,8,11]. In the present work (see the next section), we report on fabrication details and experimental characterization of a blazed multilayer grating with 5000 grooves/mm optimized for third order diffraction of 14.2 nm EUV light [14]. Note that so far high diffraction efficiency has been achieved only in the EUV energy range. In order to extend the use of the blazed multilayer gratings to soft x-ray energies, one needs to further increase the density of the grooves and/or optimize the grating efficiency for diffraction in significantly higher orders. In this case, the requirements for the grating fabrication tolerances would be extremely hard to achieve.

NEW METHOD FOR FABRICATION OF ULTRA-HIGH RESOLUTION GRATINGS

In order to get a resolving power *in first order* diffraction of 10^6 in a reasonably compact x-ray spectrometer, a grating with 10^6 grooves and with extremely high line density, ~50,000 l/mm, would be desired. For high energy x-ray applications, there is a unique opportunity to use an asymmetrically cut crystal as an ultra-high density grating [15-17]. Unfortunately, it is difficult, if not impossible, to find a crystal with a lattice constant, large enough for soft x-ray applications [18].

For EUV and soft x-ray applications, such a grating can be fabricated by slicing and polishing the ML structure at some angle to the plane of the ML. This reveals the periodic structure with the period determined with the ML bilayer spacing and slice angle values. A high dispersive power of a slicing grating with a $MoSi_2$/Si ML at an angle of 10 degrees was experimentally demonstrated in the EUV wavelength range [19-20]. However, the number of grooves of such a grating is limited by the number of bilayers. The existing deposition techniques do not allow exceeding a total number of layers of ~10^3 (see analysis in Ref. [5]).

Figure 1c illustrates a revolutionary new grating design suggested in Ref. [5] that can potentially provide 10^5-10^6 grooves with a reasonably small grating with length of 2-10 cm. Such gratings can be fabricated by polishing blazed ML gratings, transforming the structure in Fig. 1b to one in Fig. 1c. The resulting sliced grating has a short-scale periodicity of lines (bilayers), which is defined by the multilayer period and the oblique-cut angle.

Compared to a blaze grating, a sliced grating (Fig. 1c) should possess higher efficiency because of a decreased shadowing effect. Moreover, a sliced grating should provide a more regular diffraction pattern without strong mixing of orders that would be characteristic for a blazed multilayer grating diffracting in very high order. Indeed, the higher the diffracted order, the smaller order separations are. However, on the top of an echellette substrate, it is practically impossible to fabricate an ideal multilayer structure, as one schematically shown in Fig. 1b. Due to angular dispersion of the depositing beam and due to the dependence of deposition rate on the beam incidence angle, there appears a significant perturbation of the multilayer structure in the vicinity if the echellette groove's edges. By polishing, a significant part of the perturbed area of a groove would be removed, and the diffraction quality of the grating should be improved.

A first prototype of a multilayer sliced grating, based on the described technique, has been shown in Ref. [13]. The grating had a Sc/Si multilayer with bilayer period of 25 nm deposited by dc-magnetron spattering on an echellette substrate with period of 10 µm and groove angle of 6 degrees (Fig. 2a). The Sc/Si multilayer was mechanically polished using a number of diamond pastes. The resulting sliced grating had a diffraction efficiency of ~7% for the optimized 38th order diffraction of EUV light with the wavelength range of 41-49 nm (Fig. 2b).

The prototype sliced ML grating appears to be far from an ideal sliced grating, which would be expected to diffract solely in the first order determined by the period of the sliced ML lines. In the contrary, it diffracts in multiple orders corresponding to the residual 10 µm periodicity of the grating structure (Fig. 2c). The major distortion factor is the gradient of periodicity of the sliced multilayer lines inside one echellette groove that also repeats with the 10-µm period of the echellette substrate. The gradient of periodicity of the lines is due to the curvature of the echellette facets and the mentioned perturbation of the multilayer coating near the groove's edges. The last problem was redoubled by the presence of the residual nubs, seen in Fig. 2a, which are the unetched artifacts shielded by the nitride mask.

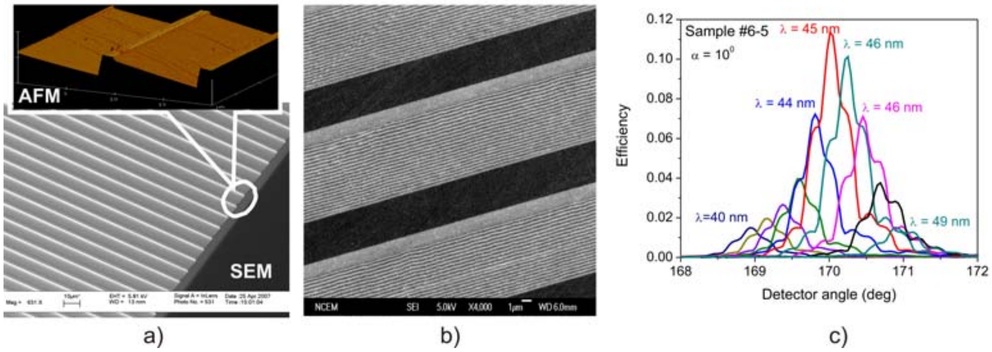

FIGURE 2. A first prototype of a multilayer sliced grating: a) structure of the echellette substrate, anisotropically etched in a Si wafer [13]; b) Scanning Electron Microscope (SEM) image of the sliced Sc/Si prototype grating; c) angular dependence of the diffraction efficiency of the prototype grating measured at different light wavelengths. The incidence angle was 10 degrees. The low resolution of the used spectrometer was not optimized for resolving the diffraction orders.

Significant efforts were directed to improve the facet profile. This became possible due to a careful optimization of the fabrication processes, removing the nubs, and using an echellette substrate with a smaller period [14]. Additionally, the ML deposition process was optimized in order to produce a more uniform coating with minimum deposition on the anti-blazed facets.

Using the improved technology, a blazed Mo/Si ML grating with 20 bilayers deposited on a nub-free echellette substrate with a period of 200 nm [9] was fabricated. The efficiency of the grating was measured at ALS beamline 6.3.2 [21,22] to be 33%for the optimized third order diffraction. The grating profile and the results of the diffraction measurements are presented in Fig. 3.

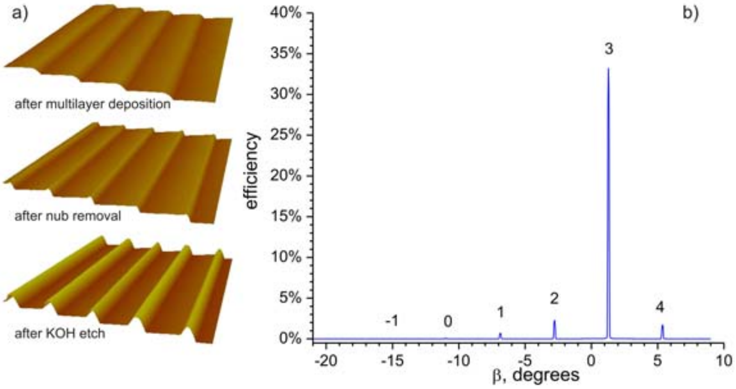

FIGURE 3. Blazed grating fabricated with the improved processes: a) structure of the echellette substrate anisotropically etched in a Si wafer, after the nubs were removed, and afrter the Mo/Si ML coating deposition. b) Angular dependence of the diffraction efficiency of the prototype grating measured at the light wavelength of 14.2 nm.. The incidence angle was 11 degrees. Diffraction efficiency in the optimized third order is about 33%.

CONCLUSIONS

The crucial point for both blazed and sliced ML grating is the quality of the echellette substrate. Surface roughness of the working facet is probably the major factor that decreases the efficiency of the discussed ML gratings in comparison with the efficiency of the ML reflector itself (see also [6]). The level of the groove surface smoothness of 0.3-04 nm achieved in the present work is acceptable for EUV and soft x-ray applications with $\lambda \geq 1$nm. We also believe that the geometry of the grating facets would be improved by using a collimated ion-beam source for ML deposition. This work is in progress.

ACKNOWLEDGMENTS

The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, Material Science Division, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231 at Lawrence Berkeley National Laboratory.

Disclaimer

Certain commercial equipment, instruments, or materials are identified in this document. Such identification does not imply recommendation or endorsement by the US Department of Energy, LBNL or ALS, nor does it imply that the products identified are necessarily the best available for the purpose.

REFERENCES

1. Workshop on "Soft X-Ray Science in the Next Millennium: The Future of Photon-In/Photon-Out Experiments. (Pikeville, Tennessee March 15–18, 2000), http://www.phys.utk.edu/WPWebSite/ewp_workshop_XRay_ Report.pdf.
2. S. Eisebitt, Eberhardt, W., J. El. Spec. Rel. Phen. **110-111**(1-3), 335-358 (2000).
3. A. Kotani, S. Shin, Rev. Mod. Phys. **73**(1), 203-246 (2001).
4. M. Altarelli, "Resonant X-ray scattering: a theoretical introduction," in: *Magnetism: A Synchrotron Radiation Approach*, Springer-Verlag, 2006, pp. 201-242.
5. D. L. Voronov, R. Cambie, R. M. Feshchenko, E. Gullikson, H. A. Padmore, A. V. Vinogradov, V. V. Yashchuk, Proc. SPIE **6705**, 67050E/1-12 (2007).
6. J. H. Underwood, C. Kh. Malek, E. M. Gullikson, M. Krumrey, Rev. Sci. Instrum. **66**(2), 2147-2150 (1995).
7. M. P. Kowalski, R. G. Cruddace, K. F. Heidemann, R. Lenke, H. Kierey, Jr., T. W. Barbee, W. R. Hunter, Opt. Lett. **29**(24), 2914-2916 (2004).
8. P. P. Naulleau, J. A. Liddle, E. H. Anderson, E. M. Gullikson, P. Mirkarimi, F. Salmassi, E. Spiller, Opt. Comm. **229**, 109–116 (2004).
9. M. Ahn, R. K. Heilmann, and M. L. Schattenburg, J. Vac. Sci. Technol. **B 26**(6), 2179-2182 (2008).
10. H. Lin, and L. Li, Appl. Opt. **47**(33), 6212-6218 (2008).
11. Lin, H., Zhang, L., Li, L., Jin, Ch., Zhou, H. and Huo, T., *High-efficiency multilayer-coated ion-beam-etched blazed grating in the extreme-ultraviolet wavelength region,* Opt. Lett. **33**(5), 485-487 (2008).
12. D. Voronov, R. Cambie, R. Feshchenko, E. Gullikson, H. Padmore, F. Salmassy, A. Vinogradov, V. V. Yashchuk, "High resolution sliced multilayer grating for soft x-rays," in: Proceedings of 9th International Conference on the Physics of X-Ray Multilayer Structures (Montana February 3-7, 2008).
13. D. L. Voronov, R. Cambie, E. M. Gullikson, V.V. Yashchuk, H. A. Padmore, Yu. P. Pershin, A.G. Ponomarenko, V. V. Kondratenko, Proc. SPIE **7077**, 7077-7-1-12 (2008).
14. D. L. Voronov, E. H. Anderson, R. Cambie, J. Meyer-Ilse, E. M. Gullikson, V. V. Yashchuk, H. A. Padmore, M. Ahn, Ch.-H. Chang, R. K. Heilmann, M. L. Schattenburg, Proc. SPIE **7448**, 7448-18/1-11 (2009).
15. R. W. James, *The Optical Principles of the Diffraction of X-rays,* London: Bell, 1948.
16. Yu. Shvyd'ko, *X-Ray Optics,* Berlin: Springer-Verlag, 2004.
17. Yu. V., Shvyd'ko, M. Lerche, U. Küetgens, H. D., Rüter, A. Alatas, J. Zhao, Phys. Rev. Lett. **97**, 235502/1-4 (2006).
18. H. Petersen, C. Jung, C. Hellwig, W. B. Peatman, W. Gudat, Rev. Sci. Instrum., **66**(1), p. 1-14 (1995).
19. V. E. Levashov, E. N. Zubarev, A. I. Fedorenko, V. V. Kondratenko, O. V. Poltseva, S. A. Yulin, I. I. Struk, A. V. Vinogradov, Opt. Comm. **109**, 1-4 (1994).
20. R. M. Fechtchenko, A. V. Vinogradov, D. L. Voronov, Opt. Comm. **210**, 179-186 (2002).
21. J. H. Underwood, E. M. Gullikson, M. Koike, P. J. Batson, Proc. SPIE **3113**, 214-221 (1997).
22. J. H. Underwood, E. M. Gullikson, M. Koike, P. J. Batson, P. E. Denham, K. D. Franck, R. E. Tackaberry, W. F. Steele, Rev. Sci. Instrum. **67**(9), 3372-3375 (1996).