Lawrence Berkeley National Laboratory

Lawrence Berkeley National Laboratory

Title

Development of near atomically perfect diffraction gratings for EUV and soft x-rays with very high efficiency and resolving power

Permalink

https://escholarship.org/uc/item/59j6s6vj

Author

Anderson, Erik

Publication Date

2012-08-10

Development of near atomically perfect diffraction gratings for EUV and soft x-rays with very high efficiency and resolving power

D L Voronov,[1,]* E H Anderson,[1] R Cambie,[1] P Gawlitza,[2] L I Goray,[3]
E M Gullikson,[1] F Salmassi,[1] T Warwick,[1] V V Yashchuk[1] and H A Padmore[1]

[1]Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720, USA
[2]The Fraunhofer Institute for Material and Beam Technology, Winterbergstraße 28 01277 Dresden, Germany
[3]Saint Petersburg Academic University, 8/3 Khlopina St, St. Petersburg, 194021, Russian Federation

*email: dlvoronov@lbl.gov

Abstract. Multilayer-coated Blazed Gratings (MBG) can offer high diffraction efficiency in a very high diffraction order and are therefore of great interest for high-resolution EUV and soft x-ray spectroscopy techniques such as Resonance Inelastic X-ray Scattering. However, realization of the MBG concept requires nano-scale precision in fabrication of a saw-tooth substrate with atomically smooth facets, and reproduction of the blazed groove profile in the course of conformal growth of a multilayer coating. We report on recent progress achieved in the development, fabrication, and characterization of ultra-dense MBGs for EUV and soft x-rays. As a result of thorough optimization of all steps of the fabrication process, an absolute diffraction efficiency as high as 44% and 12.7% was achieved for a 5250 l/mm grating in the EUV and soft x-ray regions respectively. This work now shows a direct route to achieving high diffraction efficiency in high order at wavelengths throughout the soft x-ray energy range with revolutionary applications in synchrotron science.

1. Introduction

A variety of monochromators and spectrographs currently used at synchrotron radiation facilities around the world utilize Grazing Incidence diffraction Gratings (GIG). The GIGs take advantage of a grazing incidence geometry which provides high reflectance. However, a grazing incidence geometry imposes strict limitations on a grating design and eventually on grating performance. Both incidence and diffraction angles should be close to 90º to keep the incident and diffracted rays within a critical angle range. This leads to the use of 1st order for relatively low groove density GIGs being possible, much lower than can be fabricated. The size of GIGs is typically rather large simply to collect the required optical aperture at grazing incidence. The use of grazing incidence also significantly increases optical aberrations and leads to limitations in resolving power. Finally, the diffraction efficiency of a GIG can be severely limited by grazing incidence due to a significant area of a coarse groove being shadowed at the oblique illumination.

Many of the limitations of GIGs can be overcome by a grating coated with a multilayer.

Multilayers can provide high normal incidence reflectance in the EUV and high reflectivity over small spectral ranges in the soft x-ray range at relatively large grazing angles or even at the normal incidence. The most promising grating design is a multilayer coated blazed grating (MBG) which can combine high groove density, high diffraction efficiency, and can operate in a high diffraction order. Such a grating can be the basis of a new generation of compact high resolution EUV/soft x-rays spectrometers, and can have a great impact on high resolution x-ray spectrometry techniques like RIXS [1].

A concept of a soft x-ray MBG was suggested more than two decades ago [2], and proof of principle experiments demonstrated the validity of the approach [3]. However, the first attempts showed significant technological difficulties which obstructed realization of the advantages of MBGs. The measured diffraction efficiency was much lower than theoretical expectations.

To provide high absolute efficiency of an MBG, the fabrication process should address a few crucial requirements. The blazed facets should ideally be flat and extremely smooth and the triangular shape of grooves of the saw-tooth substrate should be conformally replicated by all the interfaces of a ML coating. The surface smoothness requirements for a MBG are much tighter than those of GIGs because a grazing incidence geometry gives a significant forgiveness for high frequency surface imperfections. At relatively large grazing angles the high frequency roughness becomes a major limiting factor for ML reflectivity. A ML can provide high reflectance only if the roughness of ML interfaces does not exceed typically one tenth of the ML d-spacing. Since a d-spacing of the ML is within the range of 2-7 nm, the surface of the blaze facets should be almost atomically smooth.

The flatness of the blazed facets is another crucial parameter since it directly affects the blazing ability of a MBG. Moreover, it is crucial to keep the facet flatness and groove shape in the course of ML deposition, which is very challenging because the growth of a ML on a highly corrugated saw-tooth surface is a complex process. In this paper we report on progress addressing these challenges in the development of high quality EUV/soft x-ray MBGs at the Advanced Light Source.

2. Fabrication of saw-tooth substrates with anisotropic etch of silicon

To fabricate saw-tooth substrates of high quality, we use a process based on anisotropic etching of silicon single crystals [4,5]. The process includes a lithography step to create a periodic pattern on a crystal surface, plasma etch or lift-off process to transfer the pattern to a hard mask, an anisotropic etch of the Si in aqueous KOH or NH_4F solution to create slanted blazed facets, followed by an isotropic etch to finalize shaping of triangular grooves. The details of the process can be found elsewhere [6]. The process takes advantages of the high crystallographic selectivity of the anisotropic etch which

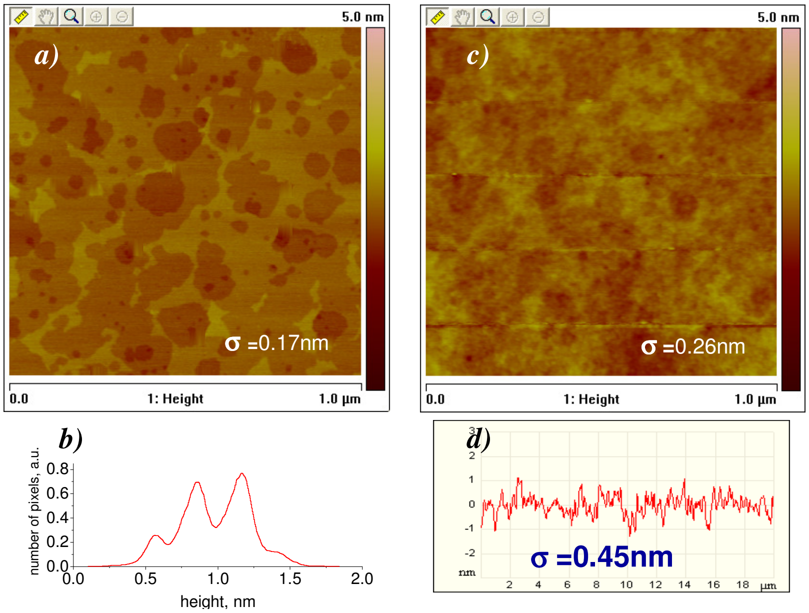

Figure 1. An AFM image of a 1×1 µm^2 area of anisotropically etched (111) surface of Si (a) and a height distribution in the same area (b); an AFM image of a 1×1 µm area of a saw-tooth substrate with an average groove profile subtracted shows residual high frequency roughness (c), and a 20 µm long AFM scan along a groove of a saw-tooth substrate shows mid-frequency roughness (d).

provides both a triangular-like groove profile and high smoothness of the (111) surface. The typical morphology of a (111) surface of a silicon single crystal after KOH etch is shown in Fig. 1. The surface consists of atomically smooth (111) terraces and atomic steps. Although four terraces are observed on the 1×1 um^2 AFM scan, most of the are a is covered only by two of them. The surface roughness approaches the lowest possible limit for KOH etching, which is half the step height equal to the d-spacing of the (111) planes, 0.314 nm.

The saw-tooth substrate fabrication process includes many technological steps, and each of them contributes into a net roughness budget. One of the best saw-tooth substrates is shown in Fig 1c,d. After subtraction of the saw-tooth profile the residual roughness of 0.26 nm was measured over a 1×1 um^2 area. A 20 µm long AFM scan along the grating grooves reveals is a middle frequency roughness of the grating facets which is below 0.5 nm rms. Such smoothness is good enough for EUV applications. For x-ray application it would be desirable to reduce the middle frequency roughness down to 0.3 nm. Nevertheless, achieved quality of the substrates allows soft x-ray operation at relatively small Bragg angles.

3. Structure and performance of MBGs

The saw-tooth substrates were coated by highly reflective multilayers deposited by different sputtering techniques. A TEM cross-section image in Fig. 2 shows structure of a 5000 lines/mm MBG coated with 30 Mo/Si bi-layers with a d-spacing of 7 nm using dc-magnetron sputtering. The MBG with a blaze angle of 6° was designed for near-normal incidence operation in the EUV range [6]. It demonstrated absolute diffraction efficiency of 37.6% in the 3rd diffraction order (Fig. 2, right). The super flat surface of the blazed facets is replicated ideally with the interfaces of the ML stack. However, the layers are significantly deformed in the vicinity of anti-blazed facets. Simulations of the diffraction efficiency using rigorous approaches [7] show that degradation of the saw-tooth profile in the course of deposition is one of the main reasons for reduction of diffraction efficiency. The perturbations of the ML stack are caused by a smoothening processes as well as self-shadowing effects for the highly corrugated surface of a saw-tooth substrate. The shadowing is a major issue for a magnetron sputtering geometry, when the large dimensions of a magnetron source causes a high divergence of the deposition flux which significantly increases the shadowing effects. Use of an ion-beam sputtering technique which provides a highly collimated deposition flux mitigates greatly the shadowing problem [8]. However, the relatively high energy of the deposited particles, which is intrinsic for the ion-beam sputtering process, enhances the mobility of surface atoms and results in more pronounced smoothening of saw-tooth grooves. The impact of the smoothening increases with the groove density of the substrate and results in dramatic degradation of the groove profile during the growth of MLs composed of low melting point materials like Al/Zr [9]. Optimal choice of a sputtering gas and energy of the ions was found to be an affective tool to control the smoothening processes, reduce groove degradation, and improve diffraction efficiency [8].

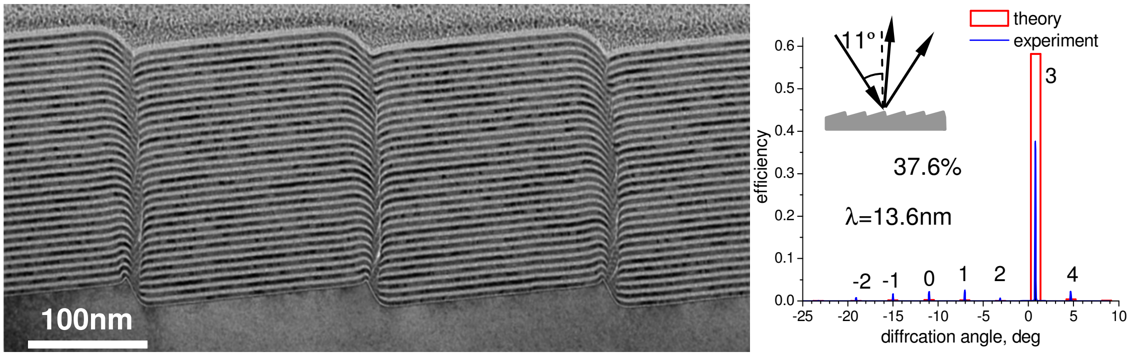

Figure 2. Cross-section TEM image of a MBG with groove density of 5000 lines/mm and the blaze angle of 6°, coated with a Mo/Si multilayer (left). Diffraction from the grating at the wavelength of 13.6 nm and the incident angle of 11° (right).

The best replication of a saw-tooth profile by a Mo/Si multilayer was achieved for a 5250 lines/mm MBG with the blaze angle of 2° [10]. Very low steps of the saw-tooth substrate produce much less shadowing even for a magnetron sputtering geometry (Fig. 3, left). This makes it possible to avoid the collapse of the layers at the foot of each step. Moreover, the deposition condition can now be optimized to minimize smoothening effects. It was done by optimization of the sputtering gas pressure, which affects the energy of deposited particles and ultimately the surface atom mobility. In this way almost perfect propagation of the substrate groove profile through the ML stack was achieved, which resulted in a record diffraction efficiency of 44% in the EUV range (Fig. 3, center). The same MBG demonstrated high performance in the soft x-ray range. Although Mo/Si MLs are not the best soft x-ray reflector, the efficiency of the MBG was measured at 12.7% at a wavelength of 1.34 nm (Fig. 3 right). This corresponds to a groove efficiency of 60% with respect to the reflectivity of 21.3% of a witness Mo/Si multilayer deposited on a flat substrate under the same conditions.

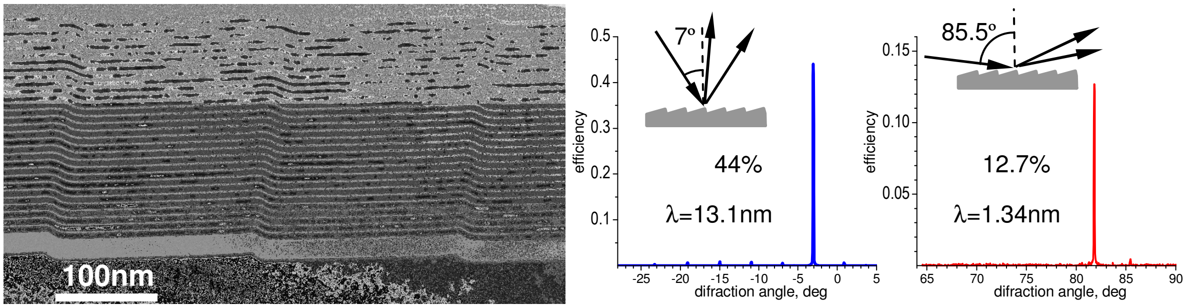

Figure 3. Cross-sectional TEM image of a MBG with groove density of 5000 lines/mm and a blaze angle of 2°, coated with a Mo/Si multilayer (left). Diffraction from the grating at the wavelength of 13.1 nm and an incident angle of 7° (center), and 1.34 nm and 85.5° (right).

Summary

High quality diffraction gratings for EUV and soft x-rays were developed. The unique ability of MBGs to combine high groove density, high order operation, and high diffraction efficiency was demonstrated. The gratings will be the basis for a new generation of compact high resolution x-ray spectrometers. The work was supported by the US Department of Energy under contract number DE-AC02-05CH11231.

References

[1] Kotani Sh Sh *Rev.* 2001 *Mod. Phys.* **73** 203
[2] Rife J C, Barbee Jr T W, Hunter W R and Cruddace R G 1990 *Physica Scripta* **41** 418
[3] Underwood J H, Malek C Kh, Gullikson E M and Krumrey M 1995 *Rev. Sci. Instrum.* **66** 2147
[4] Fujui Y, Aoyama K I and Minowa J I 1980 *IEEE J. Quantum Electron.* **16** 165
[5] Philippe P, Valette S, Mata Mendez O and Maystre D 1985 *Appl. Opt.* **24** 1006
[6] Voronov D L, Ahn M, Anderson E H, Cambie R, Chang Ch-H, Gullikson E M, Heilmann R K, Salmassi F, Schattenburg M L, Warwick T, Yashchuk V V, Zipp L and Padmore H A 2010 *Opt. Lett.* **35,** 2615
[7] http://www.pcgrate.com
[8] Voronov D L, Gawlitza P, Cambie R, Dhuey S, Gullikson E M, Warwick T, Braun S, Yashchuk V V and H.A. Padmore 2012 *JAP* **111** 093521
[9] Voronov D L, Anderson E H, Cambie R, Cabrini S, Dhuey S D, Goray L I, Gullikson E M, Salmassi F, Warwick T, Yashchuk V V and Padmore H A 2011 *Opt. Express* **19** 6320
[10] Voronov D L, Anderson E H, Gullikson E M, Salmassi F, Warwick T, Yashchuk V V and Padmore H A 2012 *Opt. Lett.* **37** 1628

Acknowledgements

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