Near-normal-incidence extreme-ultraviolet efficiency of a flat crystalline anisotropically etched blazed grating

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We have measured the extreme-ultraviolet (EUV) efficiency at an angle of incidence of 10° of a flat crystalline anisotropically etched blazed grating. The measured efficiencies are high for uncoated gratings and agree well with a calculated model derived from a reasonable estimate of the groove profile. The highest groove efficiencies derived from the measurements are 48.8% at 19.07 nm and 64.1% at 16.53 nm for the -2 and -3 orders, respectively, which are comparable to the best values obtained yet from a holographic ion-etched blazed grating. This presents opportunities to instrument designs for high-resolution EUV spectroscopy in astrophysics where high efficiency in high orders is desirable.

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1. Introduction

At extreme-ultraviolet (EUV) and soft-x-ray wavelengths there is a constant struggle to improve the efficiency of diffraction gratings, especially in normal-incidence applications. Groove efficiency is defined to be the measured efficiency in a specific order divided by the reflectance of the ungrooved surface, whether the surface is bare or coated with a single layer or a multilayer. Because ideal blazed gratings have maximum groove efficiencies approaching 100% in the order of choice,[1] they are generally preferred to laminar gratings, which have theoretical maxima of only 40.5% in either first order.[2,3] Two major factors adversely affect efficiency. First, efficiency decreases as roughness increases, because rougher surfaces produce more scattered light. Second, deviations in the groove profile from an ideal shape cause power to be diffracted into orders other than the desired one and introduce stray light.

Ion-etched holographic gratings have been shown to be both smoother and more efficient than ruled replica gratings. Multilayer-coated holographic ion-etched gratings have achieved near-normal-incidence measured efficiencies in the range of 5%–16% at selected EUV wavelengths,[4–8] in contrast to values of 2% or less for multilayer-coated ruled replicas.[9–12] The range in groove efficiency was 4%–10% for ruled replica gratings and 21%–35% for holographic ion-etched gratings. Ruled replica gratings tend to have roughness greater than ~1 nm rms, whereas holographic ion-etched gratings have values typically less than ~0.5 nm rms. Until recently blazed gratings had not achieved the same measure of success in groove efficiency as their laminar cousins because of the difficulty in fabricating ideal groove profiles at the small blaze angles required for operation at EUV wavelengths. Using a polymer-overcoat technique, groove efficiencies as high as 53% (near-normal incidence) have now been obtained at EUV wavelengths from holographic ion-etched blazed gratings,[13,14] but their groove profiles show sag or curvature of the blazed facet that is difficult to eliminate completely.

A fabrication technique developed at the Space Nanotechnology Laboratory (SNL) of MIT now promises to produce gratings with EUV efficiency comparable to or higher than that of holographic ion-etched gratings. The technique involves holographic recording of a grating pattern on the surface of a crystalline silicon wafer, where the wafer has its surface normal rotated from the [111] direction along the [110] axis.[15,16] These wafers are etched anisotropically with potassium hydroxide, and the offcut (111) planes

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form a blazed groove profile of low roughness and flat blaze facets. Such fabrication can lead to nubs at the groove peaks, which reduce efficiency, but further processing steps can remove the nubs. Alternatively, replicas can be made using a nanoimprint lithography procedure,[15] and the inverted nubs produce troughs in the replicas, in which the loss in efficiency is not as great as in a master grating. Such gratings have been produced successfully as part of the development for the Constellation-X mission,[16] and studies at grazing incidence have produced measured efficiencies of >30% and groove efficiencies of 40%–50% over the range of 1–7 nm.[17,18] When such gratings are used at near-normal incidence, the blaze angles shift the efficiency maxima of certain orders to EUV wavelengths. Here we report results of the first EUV efficiency measurements made at near-normal incidence for a crystalline silicon anisotropically etched blazed grating.

2. Sample and Measurements

The grating was fabricated at SNL using an anisotropic etch procedure on a crystalline silicon wafer. The grating had a density of 5000 grooves/mm and the nominal blaze angle was 7.5°. The grating was a master and so groove peaks included nubs. The original substrate was flat and 25 mm × 18 mm in size, but this was cleaved parallel to the short side into two pieces of roughly equal area, one of which is to be coated with a multilayer. Efficiency measurements of the other grating piece were made at beamline X24C, National Synchrotron Light Source. The grating was mounted in a reflectometer[19] and oriented so that outside orders would be emphasized over inside orders.

The X24C monochromator[20] had a grating with 600 grooves/mm and provided a spectral resolving power of ~600. The wavelength scale was calibrated by observing the absorption edges of thin-film filters, which yielded an uncertainty of 0.03 nm.[21] A silicon filter of thickness 500 nm was used to suppress high-order radiation from the monochromator. The grating was measured in *p* polarization, with the electric vector parallel to the plane of incidence (perpendicular to the grating grooves), and polarization was ~90%. Measurements were made at only one angle of incidence, 10° from normal. The beam footprint was ~1 mm square, and measurements were made near the sample center. Beam intensity was measured with a photodiode equipped with a 1 mm wide slit to provide sufficient spatial resolution to resolve grating orders.

With the monochromator set at a selected wavelength, the detector was scanned through the diffracted orders. However, time constraints limited the number of detector scans to three wavelengths, and only two orders were scanned at two of these wavelengths. Measurements of the incident beam intensity as a function of wavelength were used to derive the efficiency from measurements of the diffracted beam intensity.

The signals from diffracted orders of uncoated gratings are small at EUV wavelengths, usually in the picoampere range. Figure 1 shows the results for the three wavelengths measured: 16.53, 19.07, and 22.54 nm. At 16.53 nm the separation between dispersed orders is ~5°. The detector occults the incident beam at diffraction angles less than 0°, and thus inside orders higher than +2 could not be observed. Also, the detector background is enhanced at diffraction angles less than 15°. The reason for this enhancement is not known, but the shape of the background in Fig. 1 is typical. The enhancement of outside orders (negative) compared with the inside orders (positive) in Fig. 1 verifies the orientation of the blazed grooves relative to the incident beam. An estimate of the background was obtained ±0.5° from the order peak, and the average of these two values was subtracted from the peak current. The resulting measured efficiency of four outside orders at the three wavelengths investigated is shown in Fig. 2.

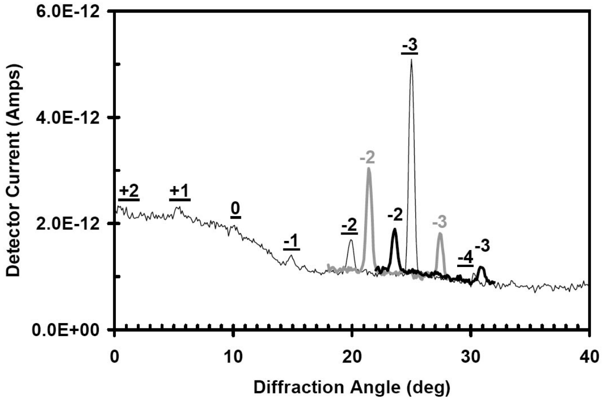

Fig. 1. Measured detector current made at an angle of incidence of 10° for wavelengths of 16.53 nm (thin black curve), 19.07 nm (thick gray curve), and 22.54 nm (thick black curve). Diffraction order labels are for 16.53 nm (black underlined), 19.07 nm (gray), and 22.54 nm (black).

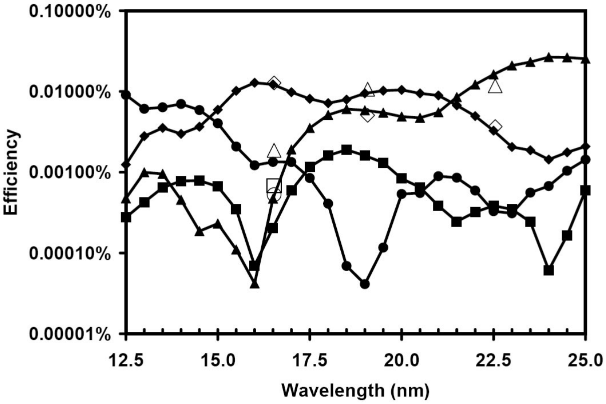

Fig. 2. Measured efficiency in the −4 (open circles), −3 (open diamonds), −2 (open triangles), and −1 (open squares) orders. The solid curves are the results of model calculations for the −4 (filled circles), −3 (filled diamonds), −2 (filled triangles), and −1 (filled squares) orders.

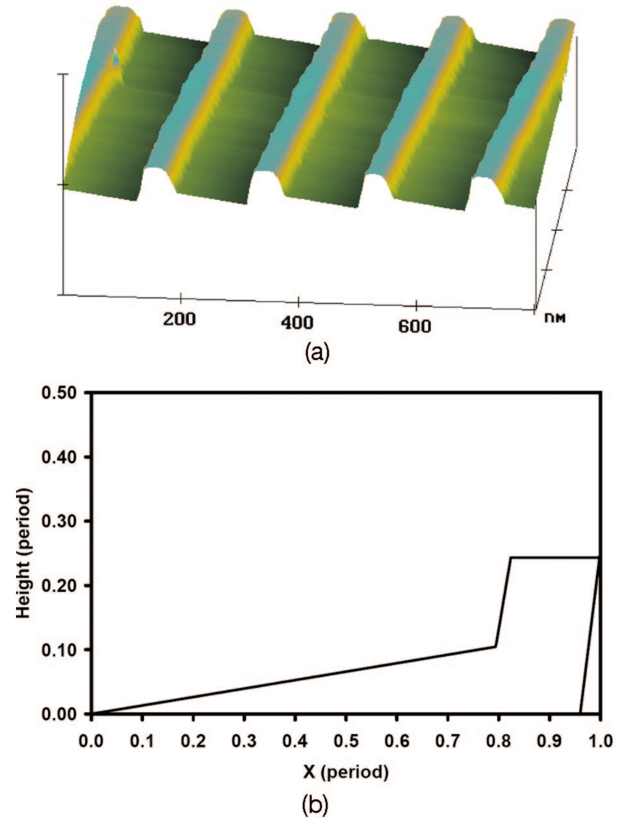

Fig. 3. (a) Atomic force micrograph of a similar crystalline anisotropically etched blazed grating (with nubs). (b) Simulated grating groove profile used to model the measured efficiency. The profile has been normalized to the groove period (200 nm) in both the *X* and height directions.

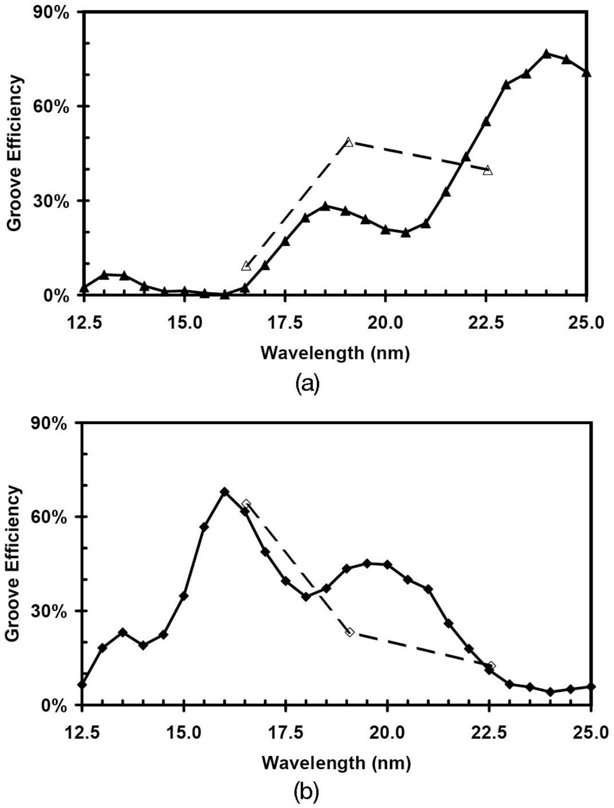

Fig. 4. Groove efficiency in the (a) -2 and (b) -3 orders. The open symbols and dashed curves were calculated using the measured efficiency, and the filled symbols and solid curves were calculated from the model.

3. Models

No topographical measurement was available for this grating, but in Fig. 3(a) we show a three-dimensional plot made with an atomic force microscope (AFM) for a similar grating, which shows nubs at the groove peak. Nub height and width were approximately equal with a value of 35 nm (approximately one sixth of the period). In Fig. 3(b) we show a simulated groove profile with a period of 200 nm, a blaze angle of 7.5°, and a nub height and width of 35 nm. Using PC Grate MLX 2000 and this groove profile, we modeled the measured efficiency of the grating. A layer of SiO_2 was assumed to coat the surface, and we calculated the models for three different thicknesses of this layer: 0.0, 1.0, and 2.0 nm. Roughness was not included in this model because it has a negligible effect for values of <0.2 nm, as expected for this grating. The best agreement with the measured efficiency was found with the model that had a SiO_2 thickness of 1.0 nm, and the calculated results are shown in Fig. 2. The calculated -2 order peaks at 24.0 nm, the -3 order peaks at 16.0 nm, and the crossover wavelength between the two orders is $\sim$21.3 nm. There is good agreement between the calculated and measured efficiencies.

Groove efficiency was calculated by dividing calculated or measured efficiency, respectively, by the calculated reflectance of 1 nm of SiO_2 on Si. The results are shown in Fig. 4. The maximum groove efficiencies derived from the measurements are 48.8% at 19.07 nm and 64.1% at 16.53 nm for the -2 and -3 orders, respectively. Model groove efficiencies at the closest calculated wavelengths are 26.8% at 19.00 nm and 61.7% at 16.50 nm for the -2 and -3 orders, respectively, in rough agreement. The highest calculated groove efficiencies are 76.8% at 24.00 nm and 68.0% at 16.00 nm for the -2 and -3 orders, respectively.

4. Conclusions

The crystalline anisotropically etched blazed grating is of high quality. The measured efficiencies are high for uncoated gratings and agree well with a calculated model derived from a reasonable estimate of the groove profile. We could only make measurements at three wavelengths, none of which was near the calculated maxima in the -2 and -3 orders. However, the highest groove efficiencies derived from the measurements are 48.8% at 19.07 nm and 64.1% at 16.53 nm for the -2 and -3 orders, respectively, which are comparable to the best values (53.0% at 15.79 nm in the -2 order)[13,14] obtained to date from a holographic ion-etched blazed grating. These results present opportunities for high-resolution EUV spectroscopy in astrophysics where high efficiency in high orders is desirable.[22–24] By serendipity the parameters for this particular grating are close to those of straw-man designs for such instruments. In a future paper we will report the results of topographical measurements with an AFM and of efficiency measurements made after coating the grating with a multilayer.

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