

Metrology and diffraction efficiency improvements for ultra-high aspect ratio, blazed soft x-ray transmission gratings

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ABSTRACT

Hot astrophysical plasmas are involved in dynamic processes across many orders of magnitude in length scales. They predominantly emit in the soft x-ray band, where high-resolution spectroscopy with resolving power $R = \lambda/\Delta\lambda \approx 5000$ is one of the high-priority science cases from the Astro2020 Decadal Survey. Grating-based spectrometer instruments - added to powerful x-ray telescopes - are the prime technology to address this priority. We have previously demonstrated a pair of co-aligned critical-angle transmission (CAT) gratings with record-high R up to 1.3×10^4 in 18th and 21st diffraction orders at Al-K (~ 1.5 keV). CAT gratings are blazed, freestanding soft x-ray transmission gratings fabricated from silicon-on-insulator wafers using advanced tools from the semiconductor and Micro-Electro-Mechanical Systems industries. Certain structural properties of the ultra-high aspect ratio CAT grating bars (such as bar tilt and bar profile) are initially defined in the deep reactive-ion etch (DRIE) step in the middle of the fabrication sequence. We have developed fast, non-destructive grating bar metrology using Mueller Matrix Spectroscopic Ellipsometry (MMSE) in combination with machine learning based on electromagnetic forward models that can provide essential feedback immediately after the DRIE step. Recently, we implemented an improved deep learning method that includes scanning-electron-microscope-derived bar profile data as ground truth and directly maps MMSE spectra to structural grating bar model parameters without the need for electromagnetic modeling. CAT gratings have been coated with thin layers of platinum to extend their bandpass and blaze angle range using atomic layer deposition. We present theoretical diffraction efficiency predictions for multilayer-coated CAT gratings that could extend the bandpass further without sharp efficiency dips from absorption edges.

Keywords: critical-angle transmission grating, x-ray spectroscopy, blazed transmission grating, soft x ray, grating spectrometer, high resolving power, machine learning, Mueller matrix spectroscopic ellipsometry

1. INTRODUCTION

Images have always inspired the human imagination. However, in order to understand the physics of many processes in the cosmos we have to gather high-resolution spectroscopic information from sources that are so far away that they simply appear as point sources in our telescopes. The soft x-ray band is dominated by emission and absorption lines from hot plasmas that carry highly ionized “metals” (O, C, Ne, Fe, N, Si, Mg, S). These plasmas carry information that describes the dynamics of many processes across up to fifteen orders of magnitude in size or distance, from dwarf star atmospheres to cosmic feedback on the scale of galaxy clusters. Grating spectrometers, fed by powerful x-ray telescopes, are the instruments of choice for collecting the spectra of point-source objects with high spectral resolving power $R = \lambda/\Delta\lambda$. The Astro2020 Decadal Review, “Pathways to Discovery in Astronomy and Astrophysics”¹ states: “Astronomy became astrophysics with the first spectrum. Spectroscopy determines compositions, magnetic field strength, space motion, rotation, multiplicity, planetary companions, surface structure, and other important physical traits...In the next decade, spectroscopy will be the

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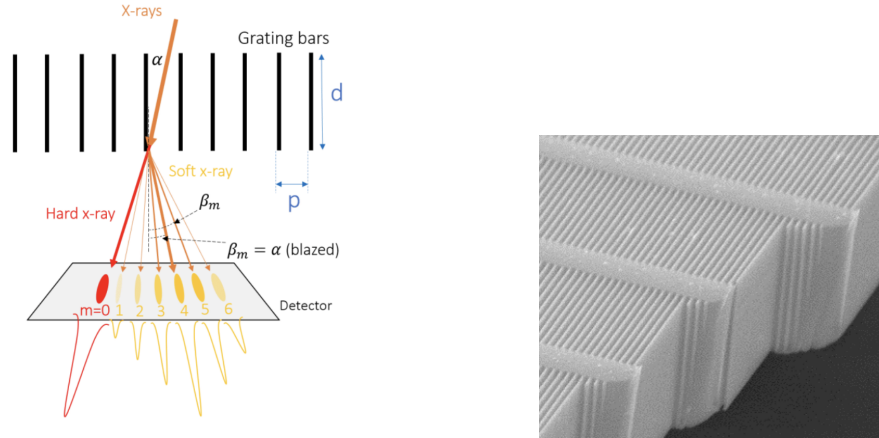


Figure 1. Left: Schematic cross-section through a CAT grating of period p . The m^{th} diffraction order occurs at an angle β_m where the path length difference between neighboring bars is $m\lambda$. When β_m is near the angle of specular reflection from the grating bar sidewalls ($|\beta_m| = |\alpha|$), the m^{th} order efficiency is enhanced. Right: Scanning electron micrograph (SEM) of a cleaved CAT grating membrane showing top, cross-section and sidewall views of the 200 nm-period silicon grating bars and their monolithically integrated 5 μm -period L1 cross supports (x rays enter from the top and leave out the bottom).

dominant discovery tool for astronomy.” For more specific examples of astrophysical subjects best studied with high-resolution soft x-ray spectroscopy, the reader can consult Refs. 2 and 3 and references therein.

We have developed blazed soft x-ray diffraction gratings - so-called critical-angle transmission (CAT) gratings - for over a decade, and steadily improved their performance.^{4,5} They combine the advantages of traditional transmission gratings (relaxed alignment and flatness tolerances, low mass, high transparency for harder x rays) with those from blazed reflection gratings (high diffraction efficiency, blazing into higher order leading to higher resolving power). More details about the different types of astrophysics-relevant diffraction gratings can be found in Ref. 6.

2. CAT GRATING PRINCIPLE

CAT gratings achieve blazing via reflection off their nm-smooth, ultra-high aspect ratio grating bar sidewalls at grazing angles α below the critical angle for total external reflection (see Fig. 1). This allows the efficient use of higher diffraction orders for higher spectral resolving power R on the order of 10^3 to 10^4 , depending on the spectrometer design. The grating bars are freestanding, held in place by a coarse cross support mesh. The lack of a support membrane avoids soft x-ray losses from membrane absorption, also improving efficiency.

The diffraction angle β_m for the m^{th} diffraction order is given by the grating equation

$$\frac{m\lambda}{p} = \sin \alpha - \sin \beta_m, \quad (1)$$

where p is the grating period. The small critical angles for soft x rays (typically on the order of 1-3 degrees) demand high-aspect ratio grating bars in order to intercept all incoming photons. Furthermore, we want the bars to be as thin as possible to minimize blockage and absorption.

Our recent work has focused on CAT grating designs optimized for MidEx, Probe, and Observatory class missions, using $p = 200$ nm.^{2,3,5,7} We have mostly produced gratings with $d = 4$ μm and $d = 5.7$ μm . Grating bar width b is typically 50-60 nm, but has been reduced down to ~ 45 nm for some gratings.⁸

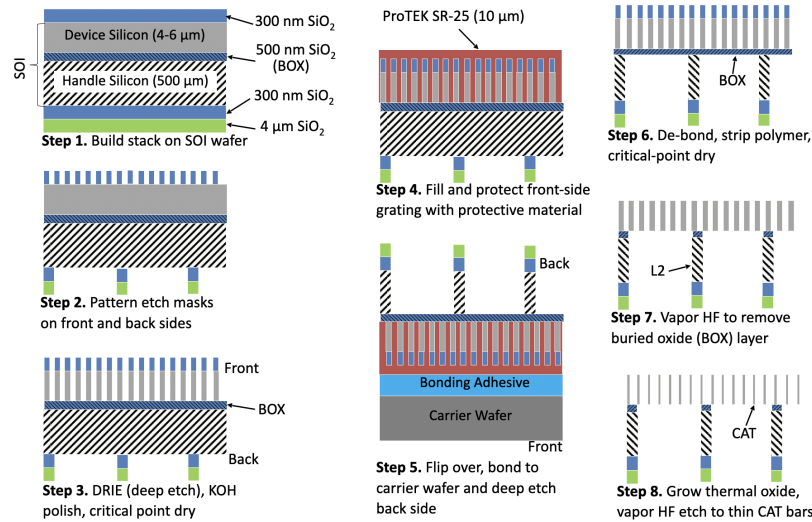


Figure 2. Simplified diagram of CAT grating fabrication steps: 1. Build layer stack. 2. Front side (device layer) patterning, etching the front side oxide mask (both at MIT LL), aligned back side patterning, etching back side oxide mask. 3. DRIE of the Si device layer, wet KOH sidewall polish. 4. Front side protection and mounting to carrier. 5. DRIE of Si handle layer. 6. Separation from carrier, front side clean, critical-point drying. 7. Buried oxide removal. 8. Grow oxide, vapor HF etch to remove oxide.^{8–11}

3. CAT GRATING FABRICATION

We have described CAT grating fabrication extensively in previous work.^{9–12} Here we summarize briefly (see also Fig. 2): Most recently, CAT gratings have been made from 200 mm silicon-on-insulator (SOI) wafers. The thin Si device (“front side”) layer of the SOI wafer is manufactured to thickness d (see Fig. 1). Using 193 nm 4X optical projection lithography (OPL) at MIT Lincoln Laboratory, patterns for CAT gratings, L1 line and L2 hexagonal support structures are simultaneously transferred into a silicon oxide layer that serves as a mask for the subsequent key step that creates the ultra-high aspect-ratio grating bars: the front side deep reactive-ion etch (DRIE). The ~ 0.6 mm thick handle (“back side”) layer of the SOI wafer is then DRIE’d with a hexagonal pattern that is aligned with the front side L2 hexagons. The grating bars are aligned parallel to the vertical $\{111\}$ planes of the (110) device layer. Since DRIE leaves rough sidewalls, this crystal orientation is used to “polish” the grating bar sidewalls post-DRIE through immersion in KOH solution.⁹ The gratings have to be dried in a critical-point dryer to prevent stiction due to liquid-vapor surface tension. Finally, the buried oxide layer separating device and handle layers is removed in the areas not covered by Si, resulting in a monolithic Si grating layer with freestanding CAT grating bars, integrated L1 and L2 supports, and a bulky L2 mesh. Once the freestanding structure has been made we can gently thin the bars further through repeated cycles of oxidation and oxide removal via HF vapor.⁸

CAT gratings can be fabricated in volume manufacturing mode from 200 mm SOI wafers,¹¹ in principle allowing for ~ 16 -20 gratings of roughly 30×30 mm² size to be produced from a single wafer. For the Rocket Experiment Demonstration of a Soft X-ray (REDSoX) Polarimeter¹³ we were able to extract up to 36 CAT gratings ($\sim 38 \times 11$ mm² in size) from a single wafer.¹⁴

4. BAR TILT AND PROFILE: YOU CAN ONLY IMPROVE WHAT YOU CAN MEASURE

The front side DRIE step determines some key features of the grating bars. Most important to us are the bar tilt and the bar profile. Bar tilt is strongly influenced by the electric field between the plasma sheath that contains the reactive ions and the substrate. If the field lines are not normal to the substrate, the deep etch will proceed at a finite angle relative to the substrate normal, creating non-zero bar tilt.^{15,16} This is especially pronounced near

discontinuities, such as edges of chips on carriers, or towards the edges of whole wafers. Similarly, as the etch progresses deeper and deeper, it is difficult to consistently maintain the same gap width between bars, leading to bar width variations, i.e., a non-rectangular bar profile. Since we are using the angle of incidence onto the sidewalls as our blaze angle, it is imperative that we control the sidewall angle to within $\sim \pm 0.2$ degrees. For a given average bar width, a rectangular cross section is expected to provide the highest diffraction efficiency. Therefore, we also need to control the bar profile.

Both bar tilt and profile are influenced by the detailed DRIE recipe, which can in principle be tuned to optimize the outcome of the etch. However, this requires feedback, i.e. measurement of bar tilt and profile.

We have previously characterized bar tilt over the surface of small chips and 100, 150, and 200 mm wafers, using small angle x-ray scattering (SAXS) with Cu K α x rays.¹⁷ Since the x rays get absorbed by the thick handle layer, we either have to at least thin down the handle layer to around 80 μm or produce a freestanding grating before we can perform relatively slow SAXS measurements. We also have to cleave the wafer into small chips to fit the samples into our SAXS tool. These processes are time consuming and potentially destructive. Similarly, we can obtain information about bar profile by cleaving a sample and looking at bar profiles with a high-resolution scanning electron microscope. Besides being destructive, this process is also slow and only gives very local information.

In contrast, Mueller Matrix Spectroscopic Ellipsometry (MMSE) is a fast, non-destructive technique that can be used immediately after the DRIE (or any other) step, without requiring any sample preparation. MMSE measures specular reflectivity off a substrate over a range of wavelengths and the polarization state of the reflected light for combinations of incident polarization states. As a result we can experimentally determine the 4×4 Mueller matrix, which describes how the Stokes vector for incident light transforms into the Stokes vector for outgoing light upon specular reflection off the sample. Each element of the matrix is a spectrum over the sampled wavelength range. The challenge in MMSE is how to relate the multitude of experimental spectra to structural parameters of the sample. For periodic structures rigorous coupled-wave analysis (RCWA) can be used to predict MMSE spectra, but traditional least-squares fitting of RCWA predictions through variation of model parameters is impractical due to the extensive computational requirements for a full model RCWA calculation across all measured wavelengths for every parameter change.

Instead, we have previously trained a neural network on MMSE spectra generated from RCWA predictions over a relevant set of model parameters. The frozen network was then used with fast (linear) gradient-based inversion to find best-fit model parameters without any additional costly RCWA calculations. This allowed us to map bar tilt on a 1 mm mesh of over 30,000 locations across a 200 mm wafer within about eight hours in very good agreement with sparse SAXS results from a few dozen locations across the width of the wafer.¹⁸

5. RECENT PROGRESS IN MMSE DATA ANALYSIS WITH DEEP LEARNING

The previously published work focused on a single grating bar parameter: bar tilt. MMSE data was taken in a conical geometry (plane of reflection parallel to the grating bars) at 65 degrees angle of incidence from the grating normal. The model only considered the grating bars and ignored the orthogonal coarse (5 μm -period) integrated support mesh.

5.1 Extracting Grating Bar Profile

To properly catch more complex features such as details of the grating bar profile we found that we had to extend the RCWA model into three dimensions, incorporate the cross support mesh, and take additional measurements with the plane of incidence perpendicular to the grating bars (the so-called “in-plane geometry” for CAT grating diffraction). For RCWA forward modeling we parametrized the bar profile with 4th order Legendre polynomials.

We again train a multilayer perceptron to emulate the RCWA model predictions, but this time over the parameter space that includes the bar profile Legendre polynomial coefficients and for both measurement orientations. We use this new frozen neural network to find best-fit model parameters for our experimental MMSE data for both orientations using iterative gradient descent.

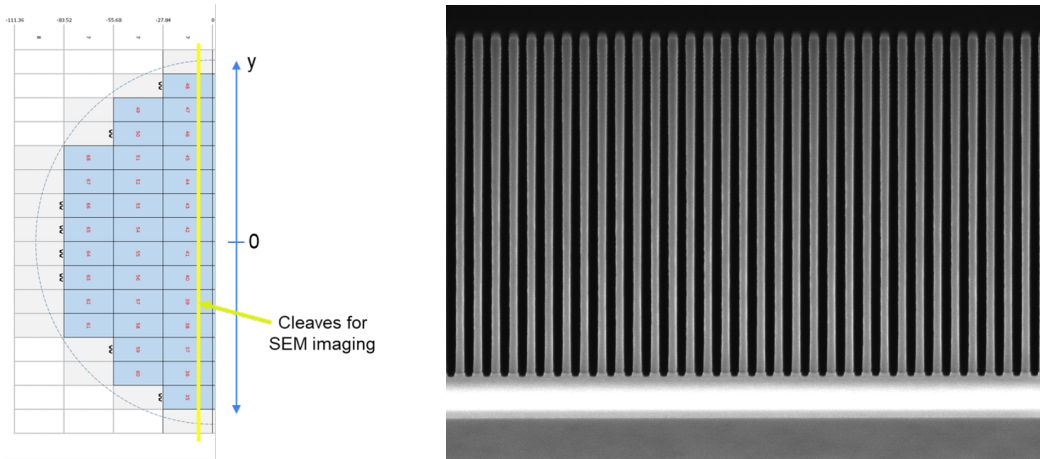


Figure 3. Left: Schematic showing exposure fields on half of a 200 mm wafer, and the line along which cross sections were obtained by cleaving. SEM cross section images were labeled based on their location along y . Right: Representative SEM cross section image used for bar profile extraction. The grating period is 200 nm, and the bar height is $\sim 4 \mu\text{m}$.

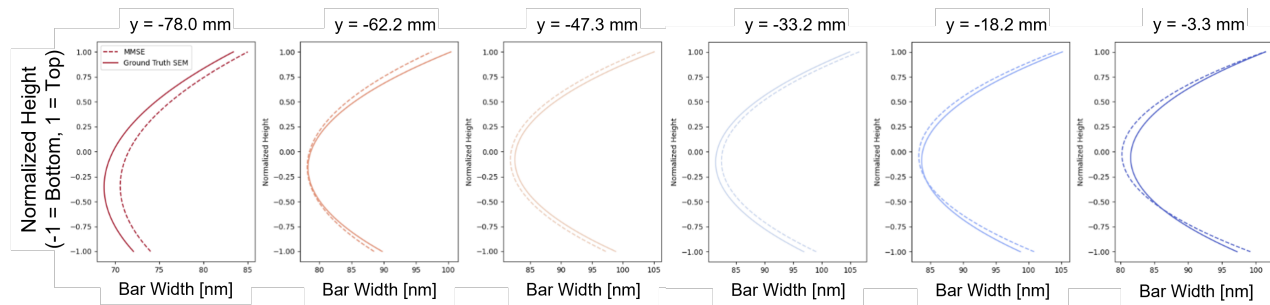


Figure 4. Comparison of Legendre polynomial fits to SEM-derived bar width profiles (solid lines) and MMSE derived profiles (dashed lines) for the same locations on the wafer. Bar shapes (with tilt subtracted) are assumed to be mirror symmetric around their centers.

We now have pairs of experimental spectra and best fit parameter sets. For new data we desire to predict parameters from a single forward pass without gradient descent iterations. We achieve this by training a convolutional neural network variational autoencoder (CNN-VAE) on the data-parameter pairs. Details are described in Ref. 19.

5.2 Extracting Bar Profiles from Scanning Electron Microscopy

MMSE is an indirect technique where the optical response of geometrical models of the examined structure is compared to measured spectra and model parameters are deduced from this comparison. It is important to validate the results by comparison with direct measurement methods, at least for a small number of representative samples.

For bar profiles, a straightforward and direct technique is scanning electron microscopy (SEM) of cleaved grating cross sections. We cleaved a 200 mm wafer in half after DRIE and MMSE and took SEM images at 79 locations along the wafer diameter (see Fig. 3). SEM images were analyzed and grating bar profiles were extracted using contrast normalization, adaptive binarization, bar detection via connected-component analysis, width detection, and scale calibration. Each image contains about 40 grating bars, and the average width along the bar height direction is used as an estimate of the typical bar shape. The standard error at each height estimates the measurement error, which is typically below 1 nm. Scale calibration was done using the grating pitch of 200 nm, defined by lithography. The average profile from each image is then fitted by Legendre

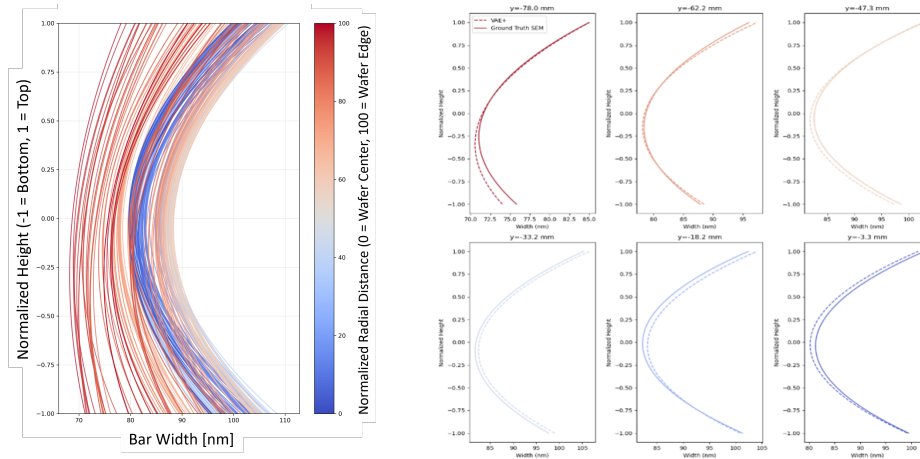


Figure 5. Left: Average bar width profiles derived from MMSE data using deep learning (and augmented by SEM profiles at selected locations) color coded as a function of distance from the wafer center. Right: Same as Fig. 4, but after augmenting training with SEM-derived profiles as ground truth.

polynomials up to 4th order, with root-mean-square errors in the 2-5 nm range. Out of 79 cross sectional SEM images, 71 were successfully processed with our profile extraction pipeline, with a mean bar width of 79.7 ± 5.3 nm. We use the bar profiles parametrized with Legendre polynomials for comparison with MMSE deep learning results at the locations where the SEM images were taken. As shown in Fig. 4, we can see that the agreement between SEM and MMSE-derived profiles (both parametrized using up to 4th order Legendre polynomials) is quite good.

5.3 Incorporating SEM Profiles as Ground Truth

We are using SEM profiles not only for comparison, but subsequently incorporated them to augment the training data for the CNN-VAE, thus supplying ground-truth parameters independent of the RCWA forward model. Fig. 5 shows average bar width profiles as a function of distance from the wafer center. Interestingly, the widest bars are not found in the wafer center, but about half way between center and wafer edge, before bar width rolls of noticeably towards the edge.

Not surprisingly, the agreement between SEM and MMSE-derived profiles improved after SEM data was included in the training data (see left of Fig. 5).

5.4 Comparing Etch Mask CD to MMSE/ML-derived Average Bar Width

Another way to check the validity of MMSE/ML-derived bar profiles is to look at direct measurements of the widths of the oxide mask lines from critical-dimension (CD) top-down SEM images before the DRIE step (provided by MIT Lincoln Lab). Variations in mask CD should lead to variations in average bar width. This expectation is confirmed in Fig. 6, showing that our deep learning approach to MMSE data analysis leads to results that are closely connected to physical reality. The average width of the deep-etched, high aspect ratio bars is approximately 10 nm less than the pre-DRIE mask width at each location, probably due to a combination of the reduced bar width in the middle of the bars, undercut, and mask erosion.

6. BAR TILT IS ETCH RECIPE DEPENDENT

All of this metrology would be of limited use if we cannot improve our processes. Fig. 7 compares bar tilt across different 200 mm wafers that were performed on different tools, but also with different DRIE recipes on the same tool. This demonstrates that etch recipes have an influence on bar tilt behavior, and deep learning analysis of MMSE data can provide immediate, non-destructive metrology for crucial fabrication feedback.

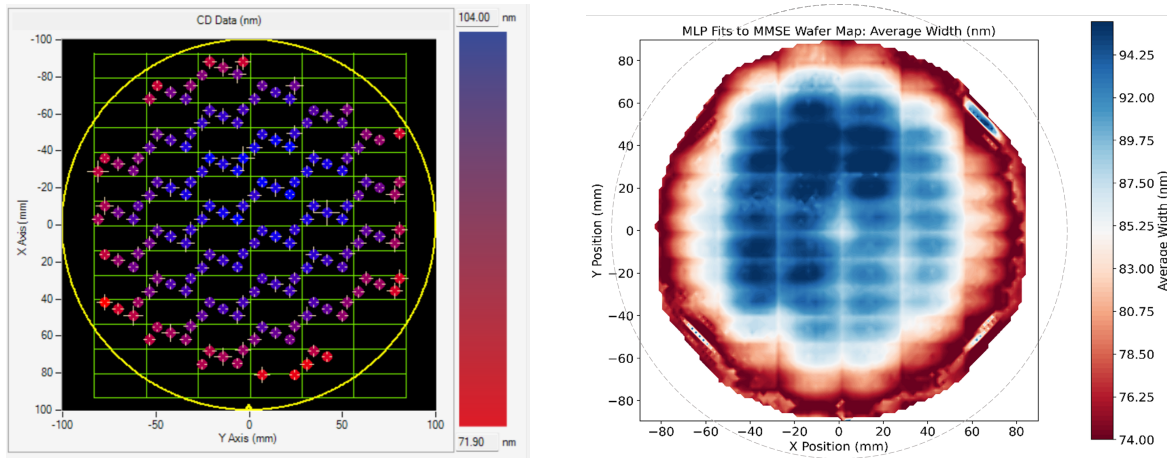


Figure 6. Left: Map of oxide mask width derived from top-down SEM images (five per exposure field) before the DRIE step. A clear roll-off in mask width can be seen towards the edge of the 200 mm wafer (yellow line). Right: Map of average bar width from our deep learning algorithm for MMSE data analysis. Each exposure field has a solid $10\ \mu\text{m}$ oxide line along its edges, and points adjacent to field boundaries are excluded. Towards the wafer edge (dashed line) MMSE spectra could not be fitted well, potentially because parameters were outside the training range.

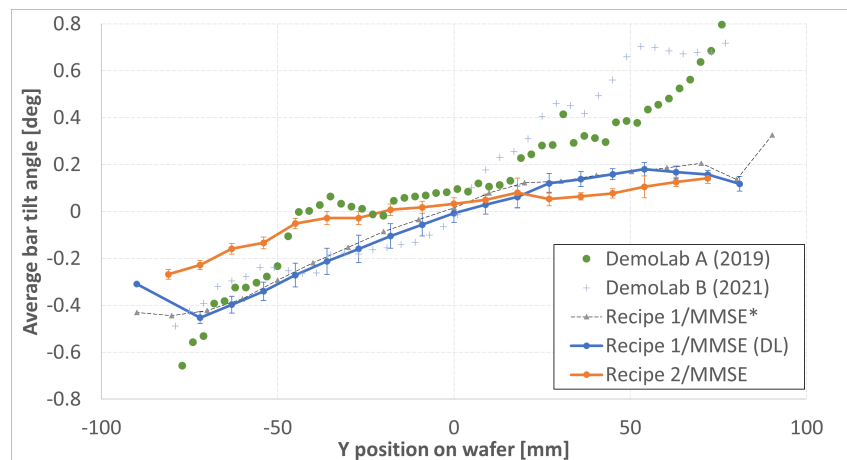


Figure 7. Average bar tilt angle as a function of y position on 200 mm wafers. “DemoLab A” and B are results from Ref. 17 measured with SAXS. “Recipe 1/MMSE*” is from Ref. 18 and aligns with SAXS data. “Recipe 1/MMSE (DL)” are results for the same wafer, but from an expanded data set and processed with our much faster deep learning (CNN-VAR) pipeline described above. “Recipe 2/MMSE” is from a wafer done on the same tool as Recipe 1, but with a different DRIE recipe, resulting in much reduced bar tilt variation, which increases the useful area for CAT grating fabrication.

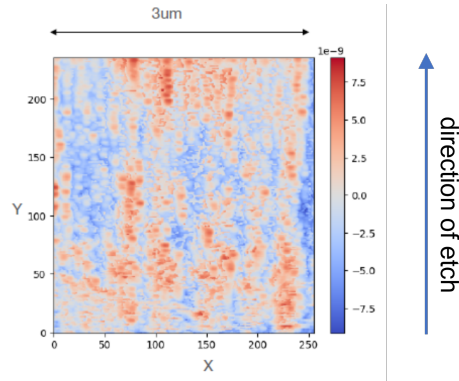


Figure 8. AFM surface height map of a grating bar sidewall from a cleaved sample. See text for details.

7. GRATING BAR SIDEWALL METROLOGY VIA ATOMIC FORCE MICROSCOPY

Apart from bar tilt and the micrometer-to-ten-nanometer-level bar profile, the few-nm to sub-nm roughness of the grating bar sidewalls has a strong influence on the diffraction efficiency of CAT gratings, since the sidewalls are used for blazing in the same geometry as grazing incidence reflection from x-ray mirrors. SEM imaging does not have sufficient spatial resolution to characterize roughness on the scale of 2-3 nm or below, and so far we have not been able to identify clear sensitivity of MMSE data (such as depolarization index, for example) to variations in sidewall roughness.

While experimentally challenging, we are looking at sidewall roughness using atomic force microscopy (AFM). This requires sample destruction (cleaving), and trying to find accessible sidewalls on grating samples stood up on edge.

There are clear morphologies visible in post-DRIE sidewalls that have not undergone wet KOH etching. There are clear signs of scalloping from the 2-step Bosch process, and striations that could be due to mask line edge roughness. There are also slow surface height variations on the spatial scale of microns. These could be related to the Legendre parametrized bar profiles, or they could be deformations due to reduced mechanical support for the grating bar sheets ($\sim 4 \times 4 \times 0.08 \mu\text{m}^3$) that are “dangling” from the cleaved edges, or other measurement artifacts.

Initial analysis from only a few samples shows reduced roughness due to KOH polishing, from ~ 2 nm to ~ 0.8 nm over the AFM-sensitive range of spatial frequencies. Post-KOH data shows what seems to be atomic steps. Such information will be useful in trying to find optimal parameters (time, concentration, temperature, surfactants) for the KOH etch, and potential sensitivity to misalignment relative to the $\{111\}$ silicon crystal planes.

8. EXTENDING HIGH-RESOLVING POWER SOFT X-RAY BANDPASS WITH MULTILAYER-COATED CAT GRATINGS

CAT gratings can be coated contiguously with thin metal films using atomic layer deposition (ALD). This changes the critical angle for total external reflection, extending the region of efficient blazing to larger angles (enabling higher resolving power) and higher energies, similar to the case of coatings for x-ray mirrors.^{20,21}

There is a tradeoff between higher reflectivity - which is usually achieved by going to smaller angles of grazing incidence - and higher resolving power, which is achieved by going to higher orders, which means blazing to larger angles.

Resolving power of an objective grating spectrometer to first order depends on the telescope point-spread function (PSF) and the diffraction angles of the utilized orders: $R \approx \beta_m / \text{PSF}$. We have demonstrated that CAT gratings are capable of delivering $R > 10^4$ in the proper setup with a 1 arsec focusing optic.²² If better optics are available, or if smaller R is sufficient, blazing to smaller angles becomes especially attractive. We have

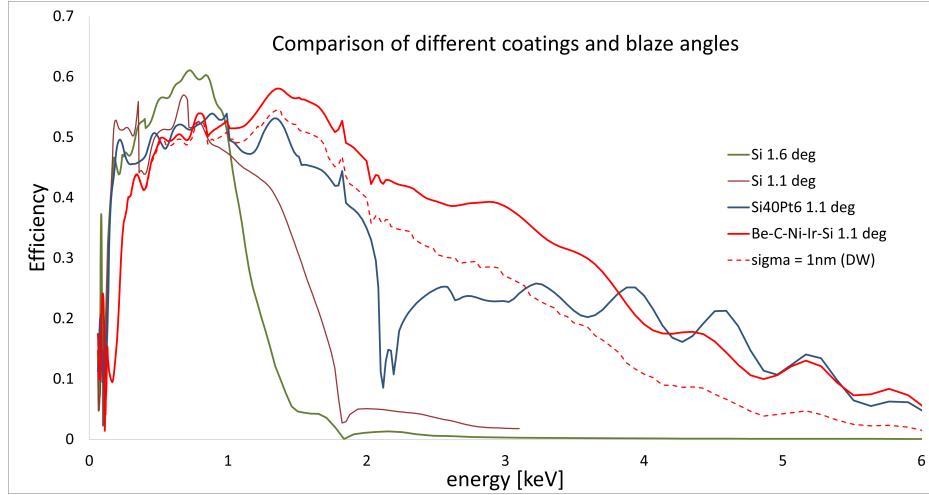


Figure 9. AFM surface height map of a grating bar sidewall from a cleaved sample. See text for details.

started to investigate smaller graze angles than proposed for Lynx (1.6°),⁷ and coating with multiple layers that could be deposited with ALD.

As an example, in Fig. 9 we show predicted efficiency for a 200 nm-period, $d = 5.7 \mu\text{m}$ CAT grating with 40 nm-wide Si bars that have been conformally coated with 6 nm each of a layer of Ir, Ni, C, and Be. The grating is illuminated at a graze angle of $\alpha = 1.1^\circ$. We show the sum of the efficiencies of the blazed orders (with $\beta_m \approx 2.2^\circ$) as a function of x-ray energy. Also shown is the same sum for a Si grating that is just coated by 6 nm of Pt, a bare Si grating, and a bare Si grating at $\alpha = 1.6^\circ$. We can see how going to smaller angles extends high efficiency to higher energies, and how covering high-Z metals such as Pt or Ir with Ni can smooth out the sharp dip at the Pt (or Ir) M absorption edges. Coating Ni in turn with C can smooth over the Ni-L absorption edge dip, and Be over the C-K edge. There are small losses at the softest energies, but very large gains at higher energies, all the way out to 4 or even 6 keV. However, these are RCWA predictions for perfect systems. If we try to estimate the effect of surface or interface roughness on the order of 1 nm with a simplified Debye-Waller-type approximation (similar to Ref. 11) we obtain the dashed line.

9. DISCUSSION AND OUTLOOK

CAT grating based soft x-ray spectrometers combined with narrow-PSF, large area x-ray telescopes can provide 1-2 orders of magnitude improvements in figures of merit for high-resolution spectroscopy,^{5,6} but fabrication is challenging. Improvements in fabrication require feedback provided by high-quality, fast and non-destructive metrology. We have developed a deep learning pipeline that can infer geometric model parameters for CAT grating bars (tilt, profile) quickly and reliably from MMSE data, which is relatively easy and fast to acquire without any specific sample preparation. Agreement with ground truth from SAXS and SEM imaging has been demonstrated. This opens a path towards faster process development, and better process control in the case of volume production for a large scale mission.

We intend to use both MMSE and AFM as diagnostic tools to develop optimized KOH polishing recipes for reduced sidewall roughness. We continue to explore means to make grating bars thinner after the BOX layer has been removed.⁸

We are also planning to study ALD coatings for CAT gratings further, both via simulation and experiment, and explore the parameter space they are offering.

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REFERENCES

- [1] National Academies of Sciences, Engineering, and Medicine, “Pathways to Discovery in Astronomy and Astrophysics for the 2020s,” (2021).
- [2] Smith, R. K., Bautz, M., Bregman, J., Brennenman, L., Brickhouse, N., Bulbul, E., Burwitz, V., Bushman, J., Canizares, C., Chakrabarty, D., Cheimets, P., Costantini, E., DeRoo, C., Falcone, A., Foster, A., Gallo, L., Grant, C., Günther, H. M., Heilmann, R. K., Heine, S., Hine, B., Huenemoerder, D., Jara, S., Kaastra, J., Kara, E., Kreykenbohm, I., Madsen, K., Marshall, H., McDonald, M., McEntaffer, R., Miller, J., Miller, E., Mushotzky, R., Nandra, K., Nowak, M., Paerels, F., Petre, R., Poppenhaeger, K., Ptak, A., Reid, P., Ronzano, K., Rozanska, A., Samra, J., Sanders, J., Schattenburg, M., Schonfeld, J., Schulz, N., Smale, A., Temi, P., Valencic, L., Walker, S., Wilms, J., and Wolk, S., “Arcus: exploring the formation and evolution of clusters, galaxies, and stars,” in [*Space Telescopes and Instrumentation 2022: Ultraviolet to Gamma Ray*], den Herder, J.-W. A., Nikzad, S., and Nakazawa, K., eds., **12181**, 1218121, International Society for Optics and Photonics, SPIE (2022).
- [3] Smith, R. K., “The Arcus Probe mission,” in [*UV, X-Ray, and Gamma-Ray Space Instrumentation for Astronomy XXIII*], **12678**, International Society for Optics and Photonics, SPIE (2023).
- [4] Flanagan, K., Ahn, M., Davis, J., Heilmann, R. K., Huenemoerder, D., Levine, A., Marshall, H., Prigozhin, G., Rasmussen, A., Ricker, G., Schattenburg, M., Schulz, N., and Zhao, Y., “Spectrometer concept and design for x-ray astronomy using a blazed transmission grating,” in [*OPTICS FOR EUV, X-RAY, AND GAMMA-RAY ASTRONOMY III*], O'Dell, S. and Pareschi, G., eds., *Proceedings of SPIE* **6688**, SPIE (2007). Conference on Optics for EUV, X-Ray and Gamma-Ray Astronomy III, San Diego, CA, AUG 29-30, 2007.
- [5] Heilmann, R. K., Bruccoleri, A. R., Gregory, J. A., Gullikson, E. M., Günther, H. M., Hertz, E. N., Lambert, R. D., Young, D. J., and Schattenburg, M. L., “Transmission grating arrays for the X-ray spectrometer on Arcus Probe,” *Journal of Astronomical Telescopes, Instruments, and Systems* **11**(1), 011004 (2024).
- [6] Heilmann, R. K., Huenemoerder, D. P., McCoy, J. A., and McEntaffer, R. L., “Diffraction gratings for x-ray spectroscopy,” (2024).
- [7] Günther, H. M. and Heilmann, R. K., “Lynx soft x-ray critical-angle transmission grating spectrometer,” *JOURNAL OF ASTRONOMICAL TELESCOPES INSTRUMENTS AND SYSTEMS* **5** (APR 2019).
- [8] Heilmann, R. K., Bruccoleri, A. R., Gullikson, E. M., Günther, H. M., Smith, R. K., and Schattenburg, M. L., “Improved efficiency critical-angle transmission gratings for high-resolution soft x-ray spectroscopy,” in [*Space Telescopes and Instrumentation 2024: Ultraviolet to Gamma Ray*], den Herder, J.-W. A., Nikzad, S., and Nakazawa, K., eds., **13093**, 1309313, International Society for Optics and Photonics, SPIE (2024).
- [9] Bruccoleri, A., Guan, D., Mukherjee, P., Heilmann, R. K., Schattenburg, M. L., and Vargo, S., “Potassium hydroxide polishing of nanoscale deep reactive-ion etched ultrahigh aspect ratio gratings,” *JOURNAL OF VACUUM SCIENCE & TECHNOLOGY B* **31** (NOV 2013).
- [10] Bruccoleri, A. R., Heilmann, R. K., and Schattenburg, M. L., “Fabrication process for 200 nm-pitch polished freestanding ultrahigh aspect ratio gratings,” *JOURNAL OF VACUUM SCIENCE & TECHNOLOGY B* **34** (NOV-DEC 2016).
- [11] Heilmann, R. K., Bruccoleri, A. R., Song, J., Cook, M. T., Gregory, J. A., Lambert, R. D., Shapiro, D. A., Young, D. J., Bradshaw, M., Burwitz, V., Hartner, G. D., Langmeier, A., Smith, R. K., and Schattenburg, M. L., “Toward volume manufacturing of high-performance soft x-ray critical-angle transmission gratings,” in [*SPACE TELESCOPES AND INSTRUMENTATION 2020: ULTRAVIOLET TO GAMMA RAY*], Den-Herder, J., Nikzad, S., and Nakazawa, K., eds., *Proceedings of SPIE* **11444**, SPIE (2021). Conference on Space Telescopes and Instrumentation - Ultraviolet to Gamma Ray / SPIE Astronomical Telescopes + Instrumentation Conference, DEC 14-18, 2020.

- [12] Bruccoleri, A. R., Guan, D., Heilmann, R. K., Vargo, S., DiPiazza, F., and Schattenburg, M. L., “Nanofabrication advances for high efficiency critical-angle transmission gratings,” in [*OPTICS FOR EUV, X-RAY, AND GAMMA-RAY ASTRONOMY VI*], O’Dell, S. and Pareschi, G., eds., *Proceedings of SPIE* **8861**, SPIE (2013). Conference on Optics for EUV, X-Ray, and Gamma-Ray Astronomy VI as part of the SPIE Optics + Photonics International Symposium on Optical Engineering + Applications, San Diego, CA, AUG 26-29, 2013.
- [13] Heine, S. N. T., Marshall, H. L., Garner, A., Angile, E., Baker, C. M., Bongiorno, S., Günther, H. M., Heilmann, R. K., LaMarr, B., McNeil, S., Juneau, J., and Ravi, S., “Mission design and development of the rocket experiment demonstration of a soft x-ray (REDSoX) polarimeter,” in [*UV, X-Ray, and Gamma-Ray Space Instrumentation for Astronomy XXIV*], Siegmund, O. H. and Hoadley, K., eds., **13625**, 1362510, International Society for Optics and Photonics, SPIE (2025).
- [14] Heilmann, R. K., Bruccoleri, A. R., Garner, A., Gullikson, E. M., Günther, H. M., Heine, S. N. T., Marshall, H. L., and Schattenburg, M. L., “Characterization of soft x-ray critical-angle transmission gratings for the REDSoX polarimetry sounding rocket,” in [*Optics for EUV, X-Ray, and Gamma-Ray Astronomy XII*], Gaskin, J. A. and Spiga, D., eds., **13626**, 136260I, International Society for Optics and Photonics, SPIE (2025).
- [15] Song, J., Heilmann, R. K., Bruccoleri, A. R., Hertz, E., and Schattenburg, M. L., “Metrology for quality control and alignment of CAT grating spectrometers,” in [*SPACE TELESCOPES AND INSTRUMENTATION 2018: ULTRAVIOLET TO GAMMA RAY*], DenHerder, J., Nikzad, S., and Nakazawa, K., eds., *Proceedings of SPIE* **10699**, SPIE (2018). Conference on Space Telescopes and Instrumentation - Ultraviolet to Gamma Ray, Austin, TX, JUN 10-15, 2018.
- [16] Song, J., Heilmann, R. K., Bruccoleri, A. R., and Schattenburg, M. L., “Characterizing profile tilt of nanoscale deep-etched gratings via x-ray diffraction,” *JOURNAL OF VACUUM SCIENCE & TECHNOLOGY B* **37** (NOV 2019).
- [17] Heilmann, R. K., Bruccoleri, A. R., Gullikson, E. M., Smith, R. K., and Schattenburg, M. L., “Soft x-ray performance and fabrication of flight-like blazed transmission gratings for the x-ray spectrometer on Arcus Probe,” in [*Optics for EUV, X-Ray, and Gamma-Ray Astronomy XI*], O’Dell, S. L., Gaskin, J. A., Pareschi, G., and Spiga, D., eds., **12679**, 126790L, International Society for Optics and Photonics, SPIE (2023).
- [18] Mudide, S., Keller, N., Andrew Antonelli, G., Cruz, G., Hart, J., Bruccoleri, A. R., Heilmann, R. K., and Schattenburg, M. L., “Machine learning driven measurement of high-aspect-ratio nanostructures using mueller matrix spectroscopic ellipsometry,” *Journal of Vacuum Science Technology B* **43**, 012801 (01 2025).
- [19] Mudide, S., *Metrology of High-Aspect Ratio Nanostructures: From Physics-Based Measurement to Physics-Informed Machine Learning*, master’s thesis, Massachusetts Institute of Technology, Cambridge, MA (2026).
- [20] Heilmann, R. K., Bruccoleri, A. R., Kolodziejczak, J., Gaskin, J. A., O’Dell, S. L., Bhatia, R., and Schattenburg, M. L., “Critical-angle x-ray transmission grating spectrometer with extended bandpass and resolving power $> 10,000$,” in [*SPACE TELESCOPES AND INSTRUMENTATION 2016: ULTRAVIOLET TO GAMMA RAY*], DenHerder, J., Takahashi, T., and Bautz, M., eds., *Proceedings of SPIE* **9905**, SPIE (2016). Conference on Space Telescopes and Instrumentation - Ultraviolet to Gamma Ray, Edinburgh, SCOTLAND, JUN 26-JUL 01, 2016.
- [21] Heilmann, R. K., Kolodziejczak, J., Bruccoleri, A. R., Gaskin, J. A., and Schattenburg, M. L., “Demonstration of resolving power $\lambda/\Delta\lambda > 10,000$ for a space-based x-ray transmission grating spectrometer,” *APPLIED OPTICS* **58**, 1223–1238 (FEB 10 2019).
- [22] Heilmann, R. K., Bruccoleri, A. R., Burwitz, V., deRoo, C., Garner, A., Günther, H. M., Gullikson, E. M., Hartner, G., Hertz, E., Langmeier, A., Müller, T., Rukdee, S., Schmidt, T., Smith, R. K., and Schattenburg, M. L., “X-ray performance of critical-angle transmission grating prototypes for the Arcus mission,” *ASTROPHYSICAL JOURNAL* **934** (2022).