Fabrication of a large-format blazed grating with radial grooves

Grayscale lithography & thermal reflow for 3D patterning

Jake A. McCoy

Dept. of Astronomy & Astrophysics
The Pennsylvania State University

14th International Workshop on Astromical X-ray Optics (AXRO) - December 5th 2023

Penn State Nanofabrication Laboratory

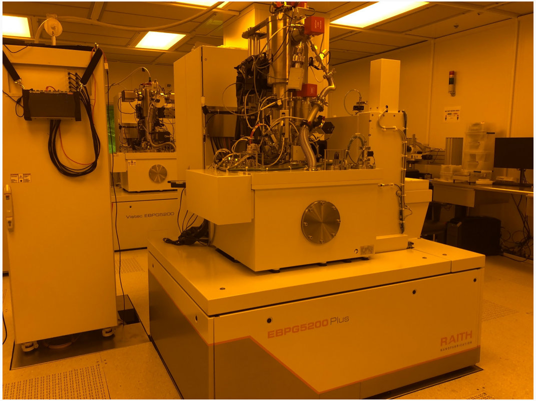


Figure: RAITH EBPG 5200 & 5200+ 100 keV EBL tools at PSU

Chad Eichfeld, Michael Labella, Fabien Grisé, The McEntaffer Group, *et al.*

- PSU Dept. of Astronomy & Astrophysics and PSU Materials Research Institute:
  - Custom diffraction gratings for soft x-ray and UV spectroscopy
- Focus on electron-beam lithography (EBL) for grating fabrication:
  - curved or fanned grooves with periods down to $\gtrapprox$ 100 nm
  - sawtooth surface reliefs (blazed gratings)
  - curved substrates [AXRO talk by Fabien Grisé]
- Gratings designed for a converging beam of a Wolter telescope?

Overview

1. Utility of gratings with radial grooves for x-ray optics
2. TASTE testing (nanofabrication development)
3. Prototyping & pattern functionalization strategies
4. Full-sized radial grating for *OGRE* development

Parallel grooves in a collimated beam

- For a plane wave illuminating grooves with $\hat{\mathbf{x}}$ as the dispersion direction, with $K \equiv 2\pi/d$:

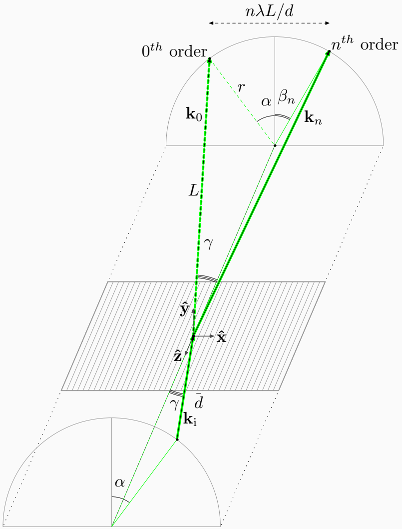
$$\begin{cases} \mathbf{k}_n \cdot \hat{\mathbf{x}} &= \mathbf{k}_i \cdot \hat{\mathbf{x}} + nK \\ \mathbf{k}_n \cdot \hat{\mathbf{z}} &= \mathbf{k}_i \cdot \hat{\mathbf{z}} \end{cases} \quad \text{for } n = 0, \pm 1, \pm 2 \ldots$$

- Assigning spherical coordinates to $\mathbf{k}_i$ and $\mathbf{k}_n$ yields the grating equation

$$\begin{cases} \sin(\beta_n)\sin(\gamma_n') + \sin(\alpha)\sin(\gamma) = \frac{n\lambda}{d} \\ \gamma_n' = \gamma \end{cases}$$

$$\sin(\alpha) + \sin(\beta_n) = \frac{n\lambda}{d\sin(\gamma)}$$

- But what happens in a converging beam?



Parallel grooves in a converging beam

- Each incident ray has a different direction depending on (x, z) on the grating:

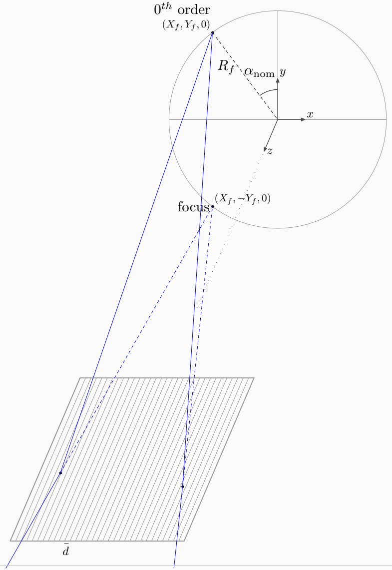
$$\mathbf{k}_\mathrm{i}(x,z) = \frac{2\pi}{\lambda L\left(x,z\right)}\left[\left(X_f - x\right)\hat{\mathbf{x}} - Y_f\hat{\mathbf{y}} - z\hat{\mathbf{z}}\right]$$

with $L\left(x,z\right) = \sqrt{\left(X_f - x\right)^2 + Y_f^2 + z^2}$

- This leads to α depending on grating position:

$$\sin\left(\alpha\right) = \frac{\sin\left(\alpha_\mathrm{nom}\right) + x/R_f}{\sqrt{1 + 2\left(x/R_f\right)\sin\left(\alpha_\mathrm{nom}\right) + \left(x/R_f\right)^2}},$$

which causes *aberrations in the dispersed spectrum* through β_n depending on α



Radial grooves in a converging beam

- Cash (1983) proposed a grating design with radial grooves that converge at the focal plane:

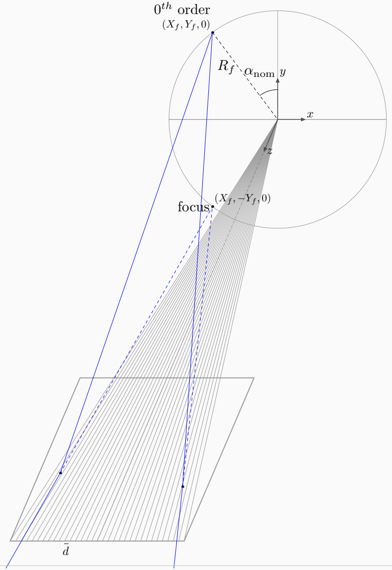
$$d(x,z) = \theta_d \sqrt{x^2 + z^2}$$

- *Diffraction occurs in the angular direction* while *specular reflection occurs radially*:

$$\begin{cases} \mathbf{k}_n \cdot \hat{\mathbf{d}} & = \mathbf{k}_\mathrm{i} \cdot \hat{\mathbf{d}} + nK \\ \mathbf{k}_n \cdot \hat{\mathbf{g}} & = \mathbf{k}_\mathrm{i} \cdot \hat{\mathbf{g}} \end{cases} \quad \text{for } n = 0, \pm 1, \pm 2 \ldots$$

- It can be shown that, for $|x| \ll z$,

$$\sin(\alpha) \approx \sin(\alpha_\mathrm{nom}) \left[1 - \frac{1}{2}\cos^2(\alpha_\mathrm{nom}) \frac{x^2}{z^2}\right]$$

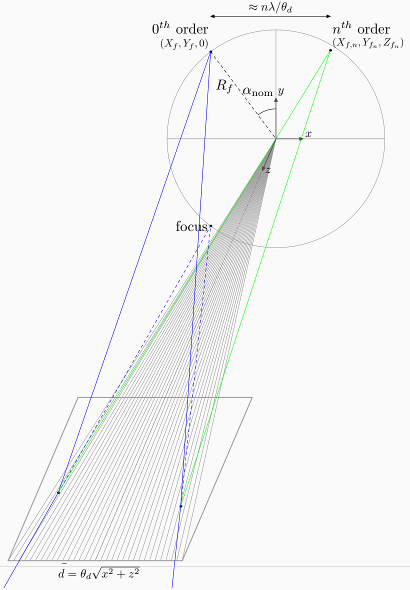


Radial grooves in a converging beam

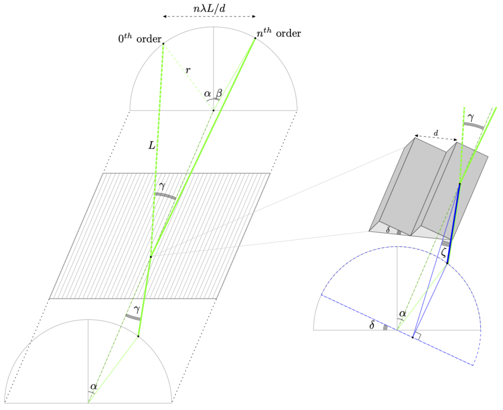
- The approximation $|x| \ll z$ is equivalent to stating that *the radial grooves do not converge too quickly*
- The condition of grazing incidence allows further approximation such that the X-position of the n^{th} diffracted order is

$$X_{n,f} \approx X_f + \frac{n\lambda}{\theta_d}\left[1 - \underbrace{\frac{1}{2}\frac{x^2}{z^2} + \frac{R_f^2}{2z^2} - \frac{X_f x}{z^2}}_{\text{small}}\right],$$

which provides utility as a dispersive element for spectroscopy

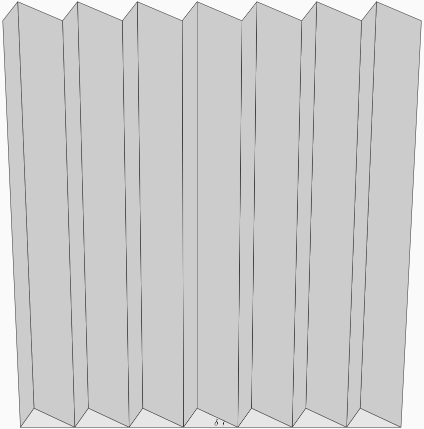


Blazed, radial grooves



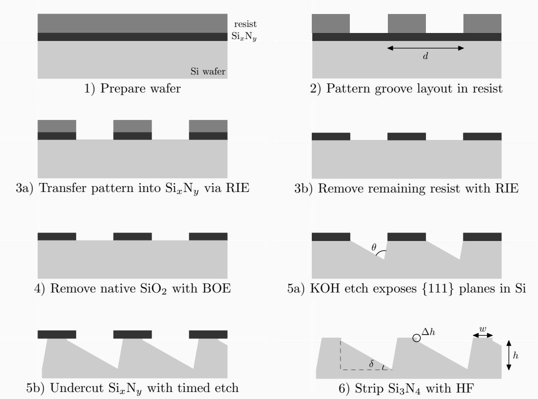
$$\lambda_b = \frac{d \sin(\gamma)}{n} [\sin(\alpha) + (2\delta - \alpha)]$$

Diffraction efficiency is maximized at the blaze wavelength, λ_b

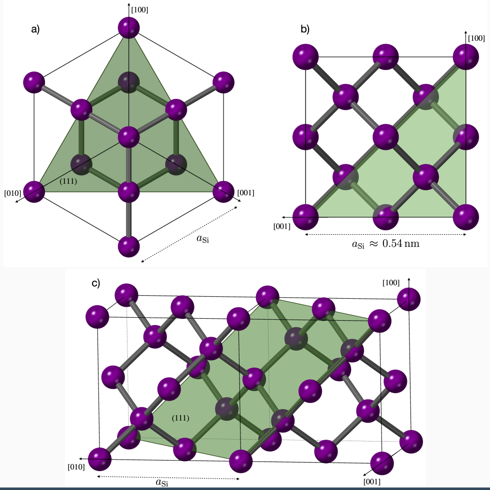


Radial grooves with blazed facets enable high spectral resolving power & spectral sensitivity simultaneously

Why not do KOH etching?



cf. Miles, McCoy, McEntaffer, et al. (2018)



The precision of a non-parallel groove layout is limited by cubic structure of crystalline Si

What is the TASTE process?

Thermally
Activated
Selective
Topography
Equilibration

coined by Schleunitz, et al. (2014)

- *In short,* TASTE describes how grayscale EBL can be combined with polymer thermal reflow to create 3D structures in resist
- But there are other variants of TASTE...

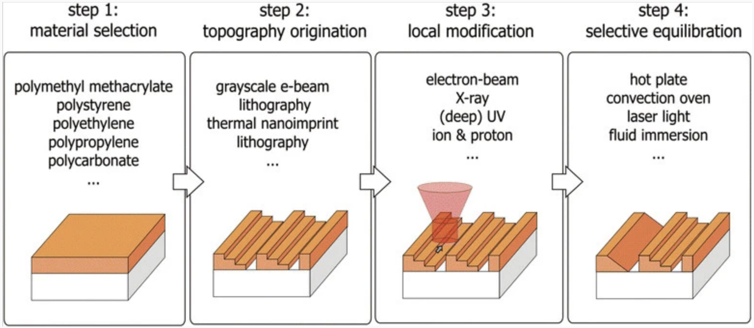


Figure: Fig. 3 from Schleunitz, et al. (2014)

Binary EBL and thermal reflow

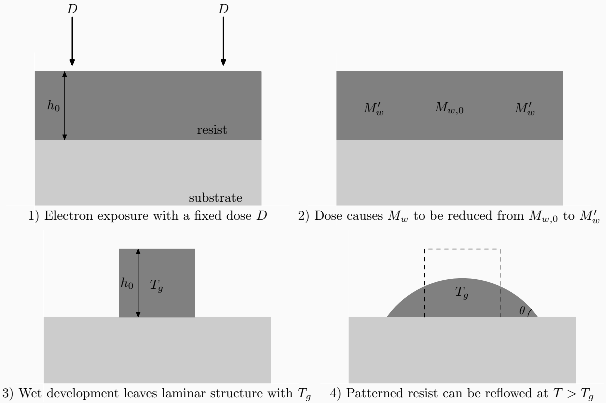


Figure: Traditional thermal reflow process

cf. Kirchner, et al. (2014)

- Electron exposure with dose D reduces molecular weight M_w via chain scission
- Exposed resist is soluble in developer; clear to substrate
- Remaining resist has a glass-liquid transition temperature T_g
  - 100 °C $\lessapprox T_g \lessapprox$ 130 °C for PMMA (depends on M_w)
- $T \gtrapprox T_g + 50$ °C treatment causes molten resist to equilibrate into a convex topography

Grayscale EBL (GEBL)

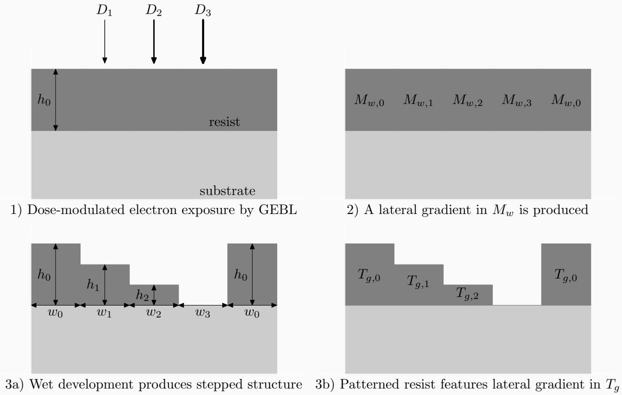


Figure: Properties of GEBL-processed resist processed that enable TASTE

- Dose-modulated electron exposure imparts lateral gradient in M_w
  - $\implies$ $D_1 < D_2 < D_3$ yields $M_{w,1} > M_{w,2} > M_{w,3}$
- M_w-dependent etch rates yields stepped structure
  - $\implies$ $M_{w,1} > M_{w,2} > M_{w,3}$ yields $h_1 > h_2 > h_3 = 0$ (timed wet development)
- Doses determined from 3DPEC algorithm in GenISys BEAMER using a resist-contrast curve

Selective thermal reflow

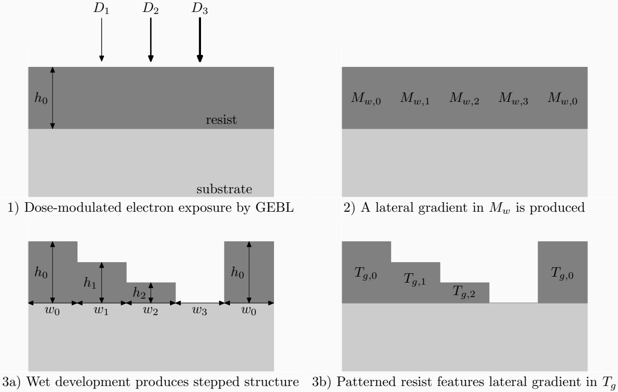
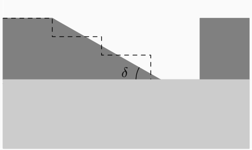


Figure: Properties of GEBL-processed resist processed that enable TASTE

- Developed GEBL structure exhibits lateral gradient in T_g

$\Longrightarrow$ $M_{w,0} > M_{w,1} > M_{w,2}$ yields $T_{g,0} > T_{g,1} > T_{g,2}$

$\Longrightarrow$ **Enables selective thermal reflow** at $T_{g,0} > T > T_{g,1}$



4) Patterned resist can be selectively reflowed at $T_{g,0} > T > T_{g,1}$

cf. Schleunitz, et al. (2014)

GEBL test patterns

GEBL process development

- Start with 950k PMMA A3 resist
- Spin coat to $\sim 130\,\text{nm}$ on silicon wafer

Test patterns (parallel gratings)

- *pattern A*: 6-level staircase where each step is 140 nm wide $\implies d = 840\,\text{nm}$ period
- *pattern B*: 4-level staircase where each step is 100 nm wide $\implies d = 400\,\text{nm}$ period
- *pattern C*: modified version of pattern B

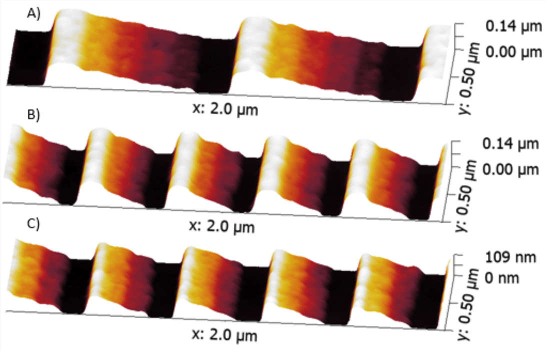
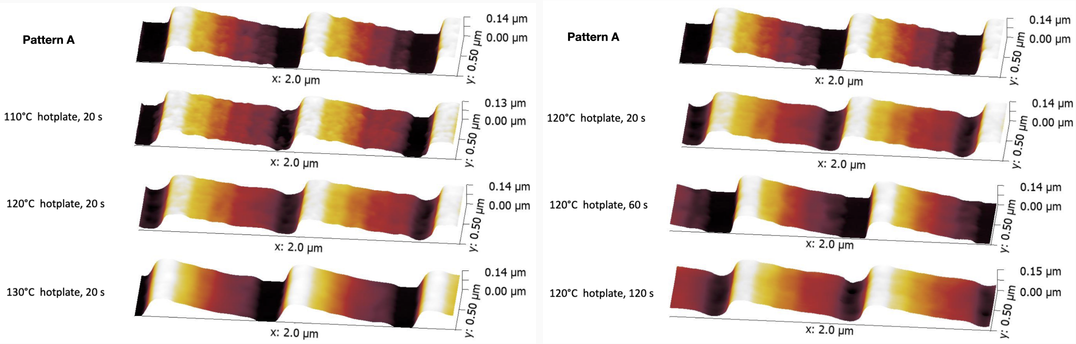


Figure: AFMs of GEBL test patterns in 130 nm-thick PMMA

McCoy, et al. (2018)

Thermal reflow

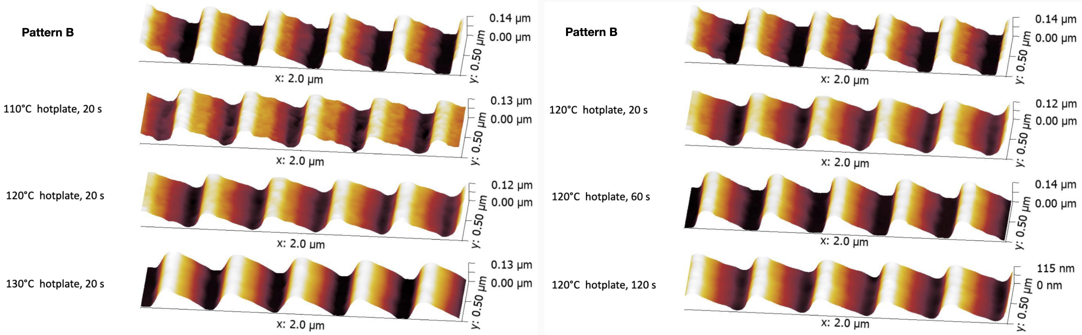
$d = 840\,\text{nm}$ period, $\sim 10°$ blaze angle



McCoy, et al. (2018)

Thermal reflow

$d = 400\,\text{nm period}, \sim 27°\ \text{blaze angle}$



McCoy, et al. (2018)

Pattern functionalization strategies

Ideas

1. Dry etch pattern into substrate
2. Cross-link resist with UV exposure
3. Aluminize PMMA with sequential infiltration synthesis

Plan

- Consider replication for final application
  - *nanoimprint lithography* (NIL)
  - *substrate conformal imprint lithography* (SCIL)
- Make single grating for prototype testing
  - Directly coat PMMA with thin metal layer for soft x-ray reflectivity
  - Test grazing-incidence diffraction efficiency at synchrotron facility

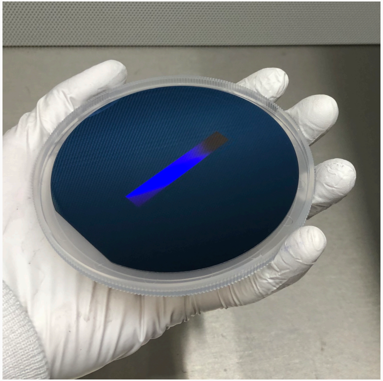


Figure: Surface-relief mold for grating prototype patterned in 130 nm-thick PMMA

McCoy, et al. (2020)

TASTE grating prototype

Grating prototype

- Start from pattern B with $d = 400$ nm
- Pattern 50 mm by 7.5 mm for synchrotron beam ($\sim$18 h exposure at 8 nA)

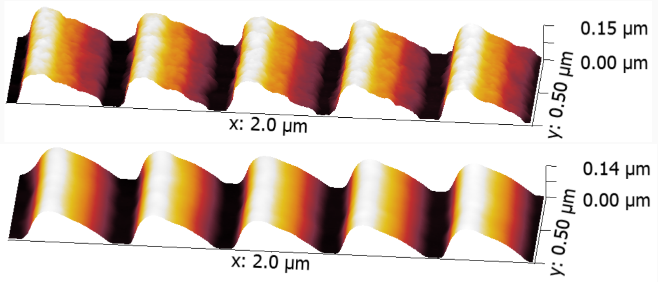


Figure: AFM of GEBL-processed resist (*top*), resist following thermal reflow at 116 °C for 30 min (*bottom*)

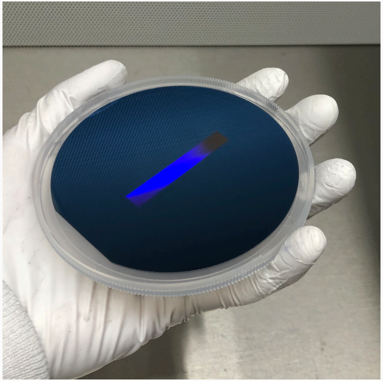
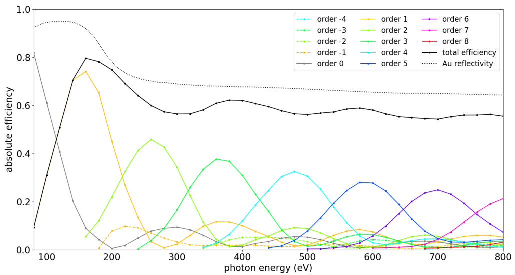


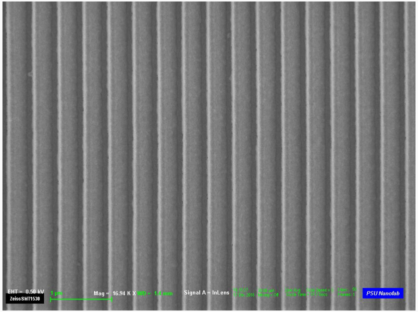
Figure: Surface-relief mold for grating prototype patterned in 130 nm-thick PMMA

McCoy, et al. (2020)

TASTE grating prototype

- Use electron-beam physical vapor deposition (EBPVD) for 15 nm of Au with 5 nm of Ti for adhesion
- Diminished, yet still significant, blaze response observed
  - Measured at Advanced Light Source (Lawrence-Berkeley Nat'l Lab, USA)





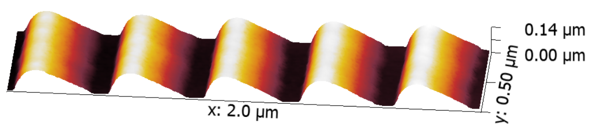


Figure: FESEM & AFM of coated grooves

McCoy, et al. (2020)

Scaling to very large areas

- System integration for OGRE development calls for:
  - Compatibility with JET-X optics 3500 mm focal length
  - Grating positioned $z_{\text{cen}} = 3000$ mm from focal plane
  - Patterning over 70 mm by 63 mm area on 6" optic with ~ 3 µm flatness
- Baseline $d(0, z_{\text{cen}}) = 315.15$ nm such that d ranges from 311.5 nm to 318.8 nm ($\theta_d \sim 20$ milliarcsec)
- SHAPEDETECTION fracture mode in BEAMER handles tilted lines

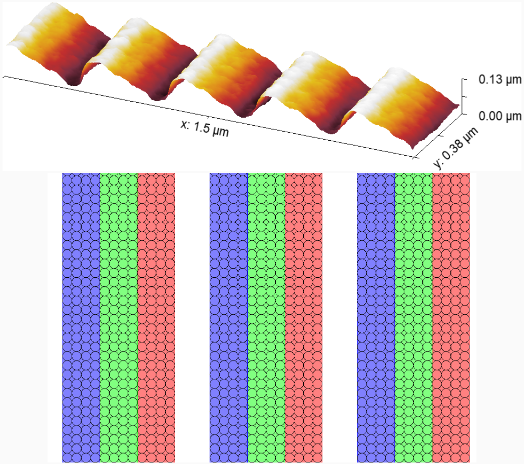


Figure: AFM of GEBL pattern: 315 nm period (*top*), fractured GPF (*bottom*)

Anticipation of the electron fogging effect

- Previous long writes (binary EBL) in some cases have been found to incur variation in critical dimension (CD) from over-exposure
- Attributed to the fogging effect

$$\mathrm{PSF}(r) \propto \frac{1}{\alpha^2} e^{-r^2/\alpha^2} + \frac{\eta}{\beta^2} e^{-r^2/\beta^2} + \frac{\Theta}{\gamma^2} e^{-r^2/\gamma^2}$$

- *forward scatter*: $\alpha \sim$ nm
- *backscatter/proximity*: $\beta \sim$ tens µm
- *fogging*: $\gamma \sim$ tens mm? value of Θ?

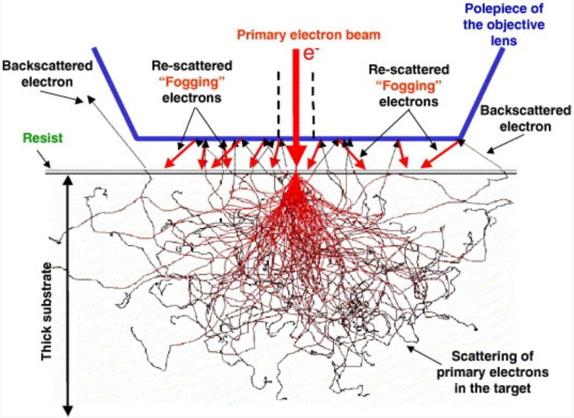
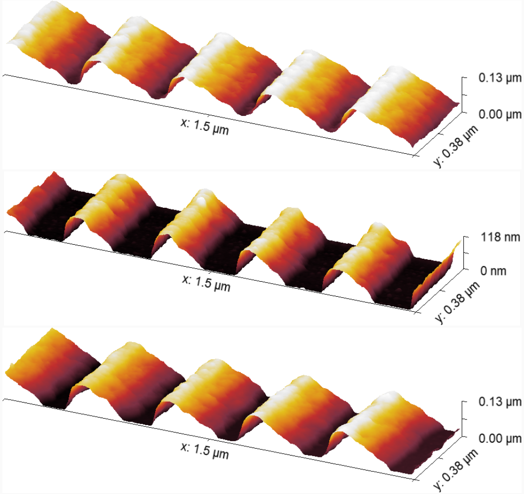


Figure: Fig. 1 from Hudek, et al. (2007)

tool-dependent rather than substrate-dependent

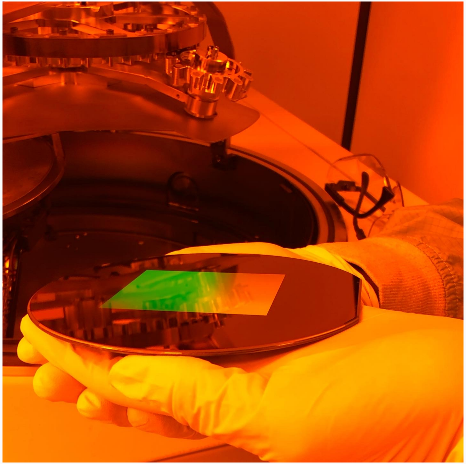
Impact of the fogging effect

- 25 mm by 25 mm area: patterns develop as expected with nominal doses
- Test pattern in center of background grating: patterns are over-dosed with nominal doses
- Test pattern in center of background grating: patterns are close to targeted from $\sim$ 86 % of nominal doses



Conclusions

- Master grating prototype; to be replicated by substrate conformal imprint lithography (SCIL) for OGRE
- EBL exposure for full 70 mm by 63 mm grating completed in $\sim$ 90 h at 30 nA ($>$ 600 GB of data)
- Coated via EBPVD with 15 nm of Au for reflectivity and 5 nm of Ti for adhesion post thermal reflow
- Tested for spectral resolving power at PANTER X-ray Test Facility [AXRO talk by Alex Higley]



References I

[1] W. C. Cash, Jr.
X-ray spectrographs using radial groove gratings.
Appl. Opt., 22:3971–3976, 1983.

[2] P. Hudek, U. Denker, D. Beyer, N. Belic, and H. Eisenmann.
Fogging effect correction method in high-resolution electron beam lithography.
Microelectronic Engineering, 84(5):814–817, 2007.
Proceedings of the 32nd International Conference on Micro- and Nano-Engineering.

[3] R. Kirchner and H. Schift.
Thermal reflow of polymers for innovative and smart 3d structures: A review.
Materials Science in Semiconductor Processing, 92:58–72, 2019.
Material processing of optical devices and their applications.

References II

[4] R. Kirchner, A. Schleunitz, and H. Schift.
Energy-based thermal reflow simulation for 3D polymer shape prediction using Surface Evolver.
Journal of Micromechanics and Microengineering, 24(5):055010, 2014.

[5] J. McCoy, R. McEntaffer, and C. Eichfeld.
Fabrication of astronomical x-ray reflection gratings using thermally activated selective topography equilibration.
Journal of Vacuum Science and Technology B: Nanotechnology and Microelectronics, 36(6):06JA01, 11 2018.

[6] J. A. McCoy, R. L. McEntaffer, and D. M. Miles.
Extreme ultraviolet and soft x-ray diffraction efficiency of a blazed reflection grating fabricated by thermally activated selective topography equilibration.
ApJ, 891(2):114, Mar 2020.

References III

[7] D. M. Miles, J. A. McCoy, R. L. McEntaffer, C. M. Eichfeld, G. Lavallee, M. Labella, W. Drawl, B. Liu, C. T. DeRoo, and T. Steiner.
Fabrication and diffraction efficiency of a large-format, replicated x-ray reflection grating.
ApJ, 869(2):95, Dec 2018.

[8] A. Schleunitz, V. A. Guzenko, M. Messerschmidt, H. Atasoy, R. Kirchner, and H. Schift.
Novel 3D micro- and nanofabrication method using thermally activated selective topography equilibration (TASTE) of polymers.
Nano Convergence, 1(7):1–8, 2014.

Flux license

Flux is licensed under GNU General Public License v3.

http://www.gnu.org/licenses

Inspired by **Metropolis** theme from Matthias Vogelgesang.
https://github.com/matze/mtheme