SAND2017-13066C

Precision Alignment of Analyzer Gratings for High Energy X-Ray Phase Contrast Imaging

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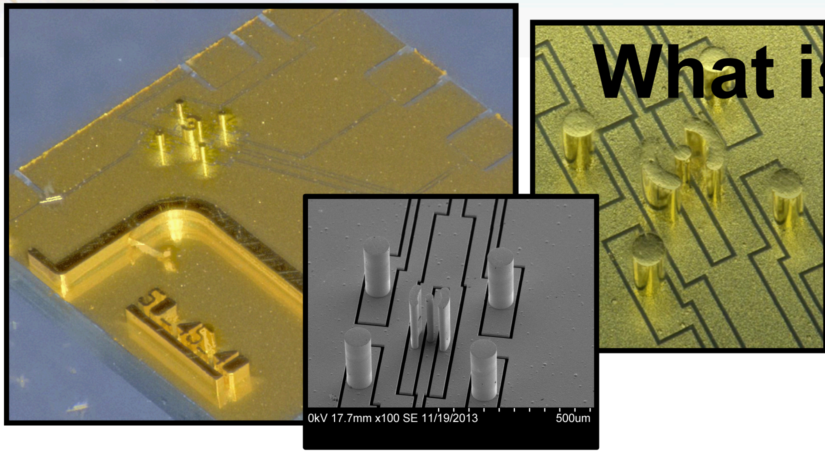
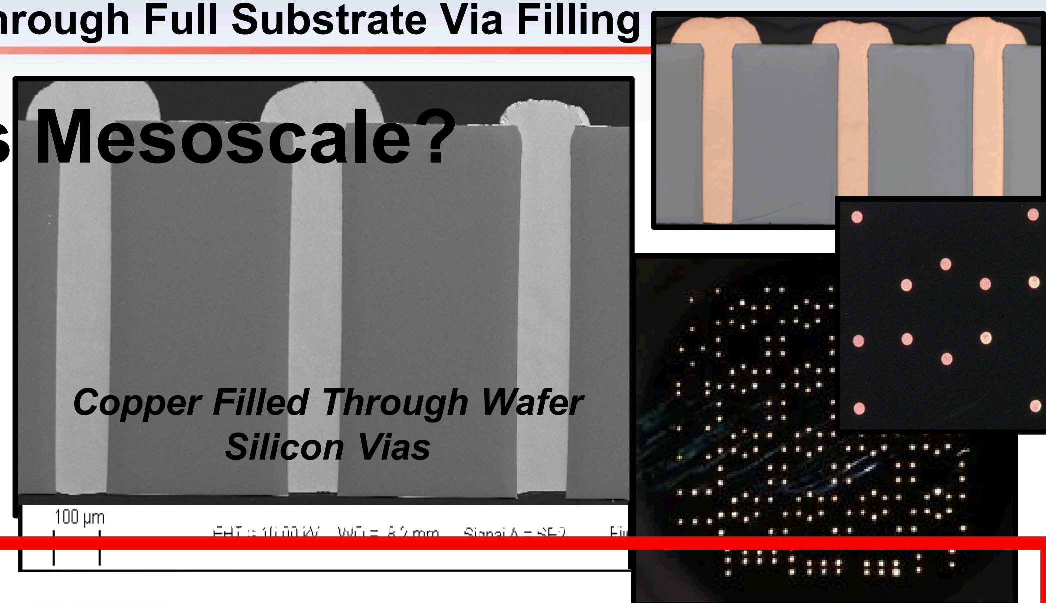
Mesoscale Fabrication
Electroplating for Forming, Filling, and Coating

What is Mesoscale?

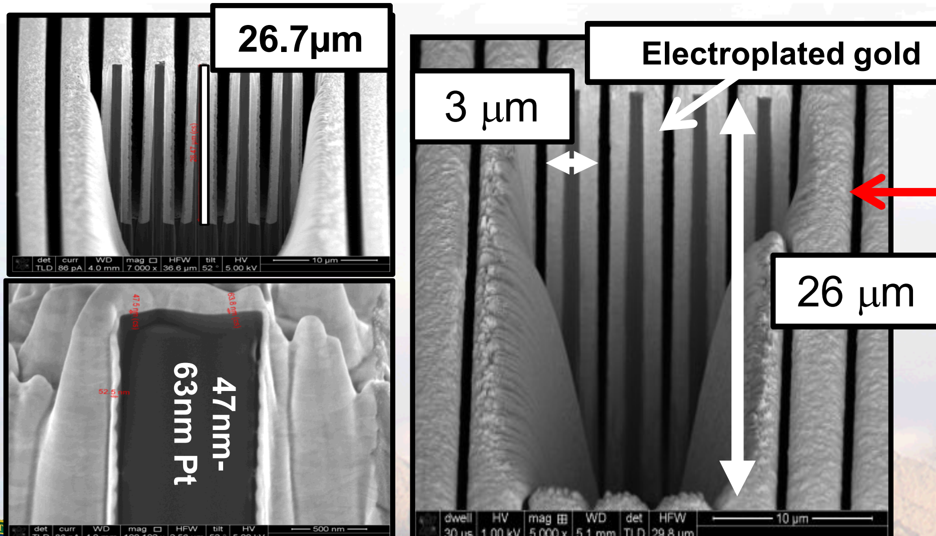
Electroformed 3D Au Stylus Ion Trap

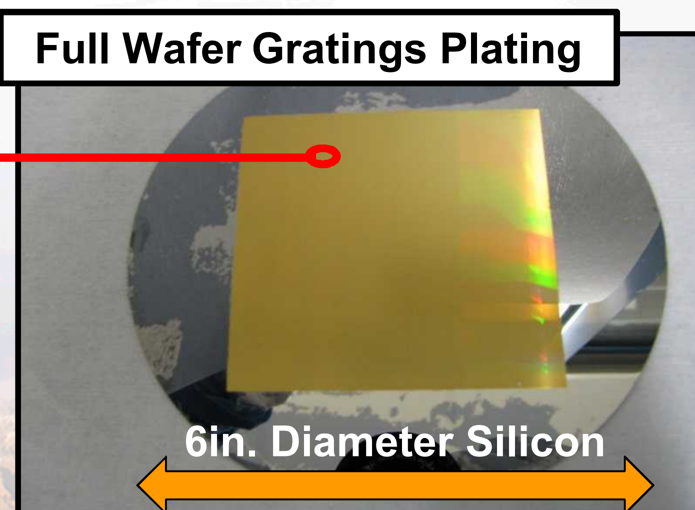


Through Full Substrate Via Filling



Precision Electro-coating



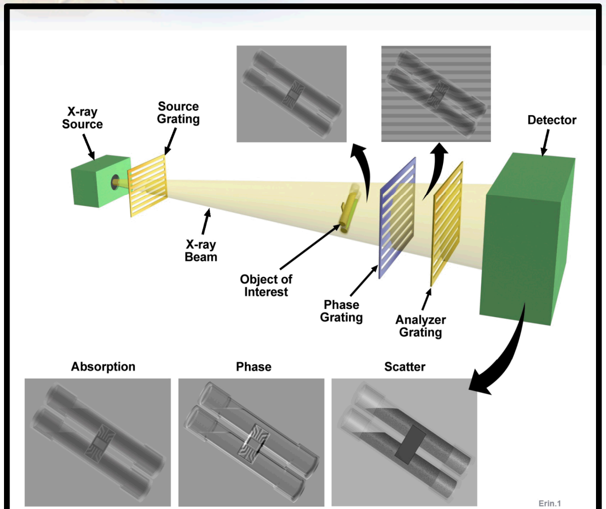


$$n = (1 - \delta) + i \cdot \beta$$

Sandia's Three Grating XPCI System

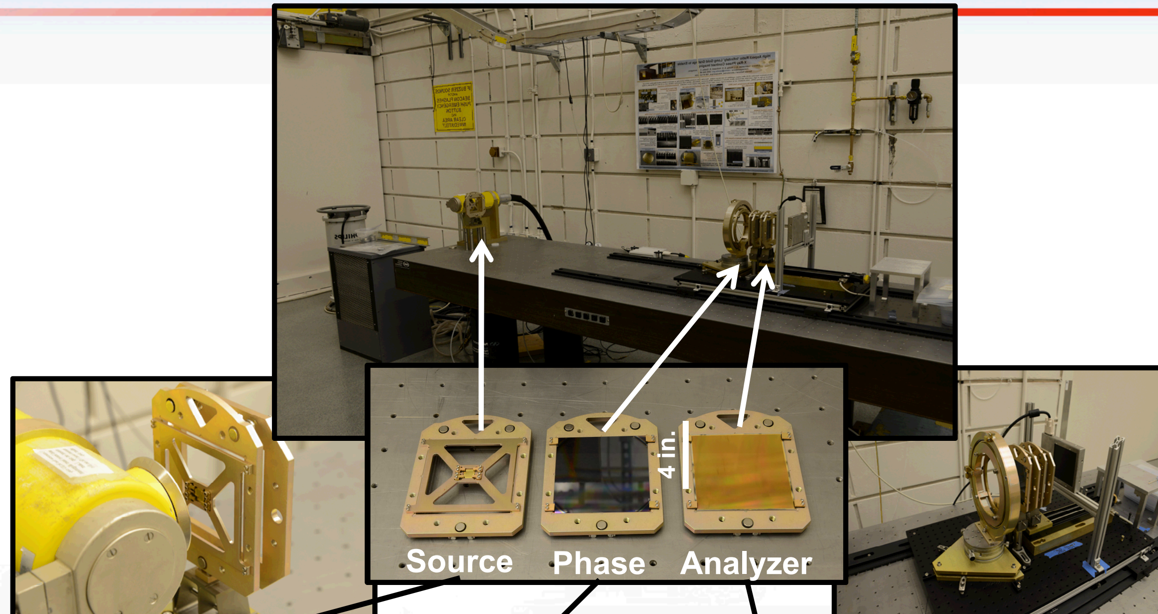
$\beta \rightarrow$ Absorption $\quad \delta \rightarrow$ Phase

δ / β = ~10^3 for low density materials

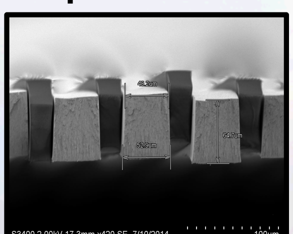


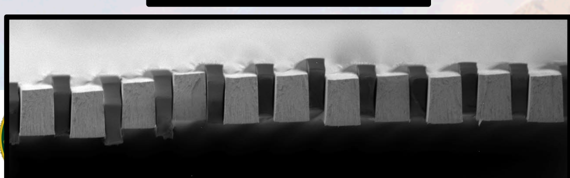
Images courtesy of Erin Miller - PNNL

28keV System



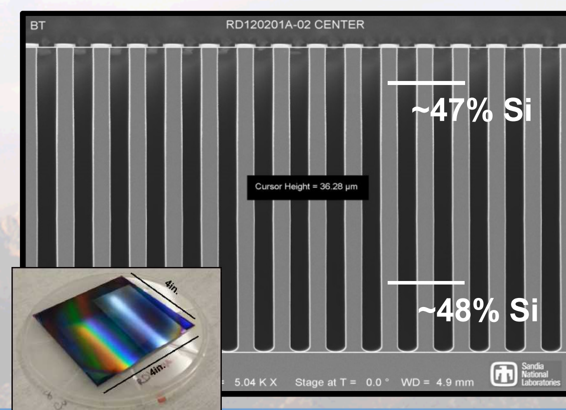
G0 Source Grating
65 µm Thick Au



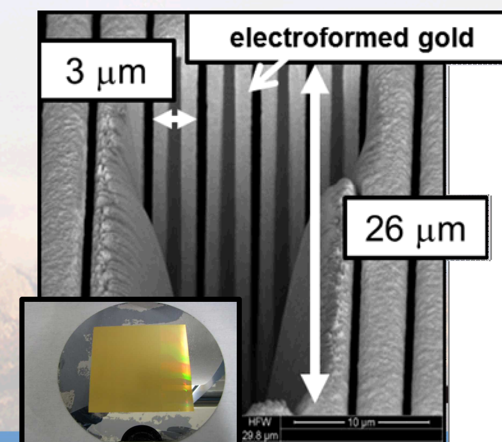


Delamination result of cross-section

G1 Phase Grating
40 µm deep DRIE Si



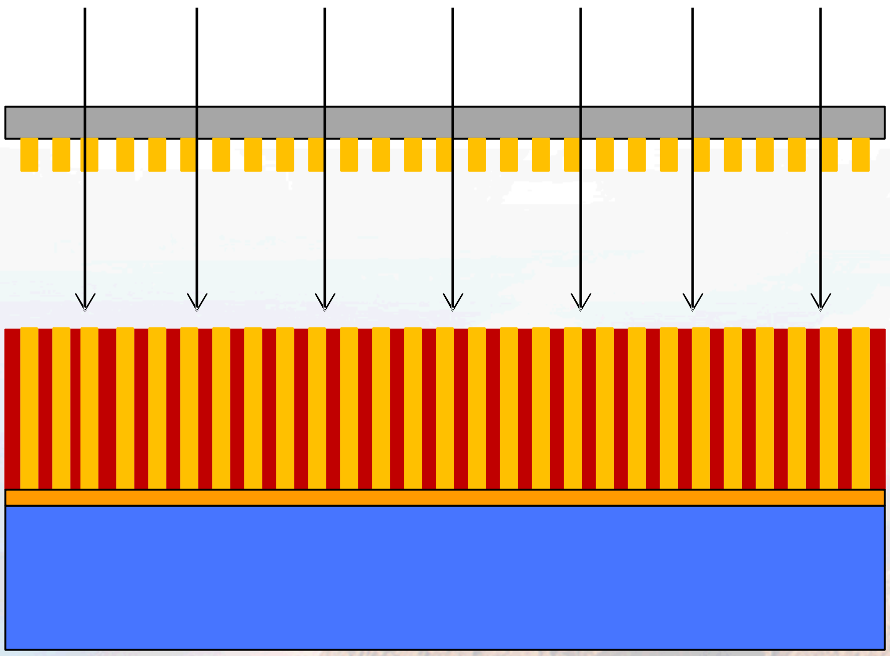
G2 Analyzer Grating
26 µm Thick Electrocoated Si

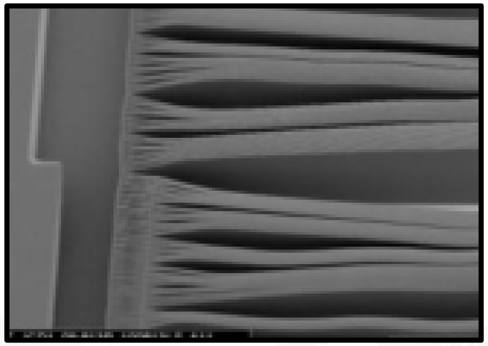


Analyzer Grating Fabrication Through Electrofilling

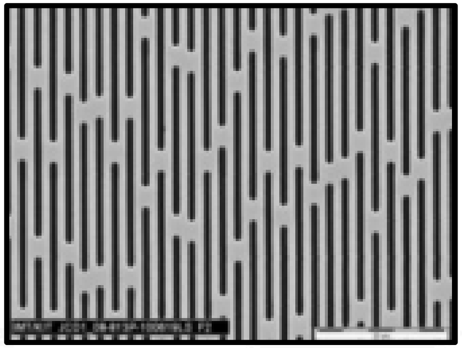
Aspect Ratios > 20:1 are needed to improve contrast
1µm Wide High Z Absorber
50% Grating Duty Cycle

- **Resist swelling occurs**
- **Structural stability of resist is low**





Infinite grating length[1]



25um Length, 3um wide support bars[1]

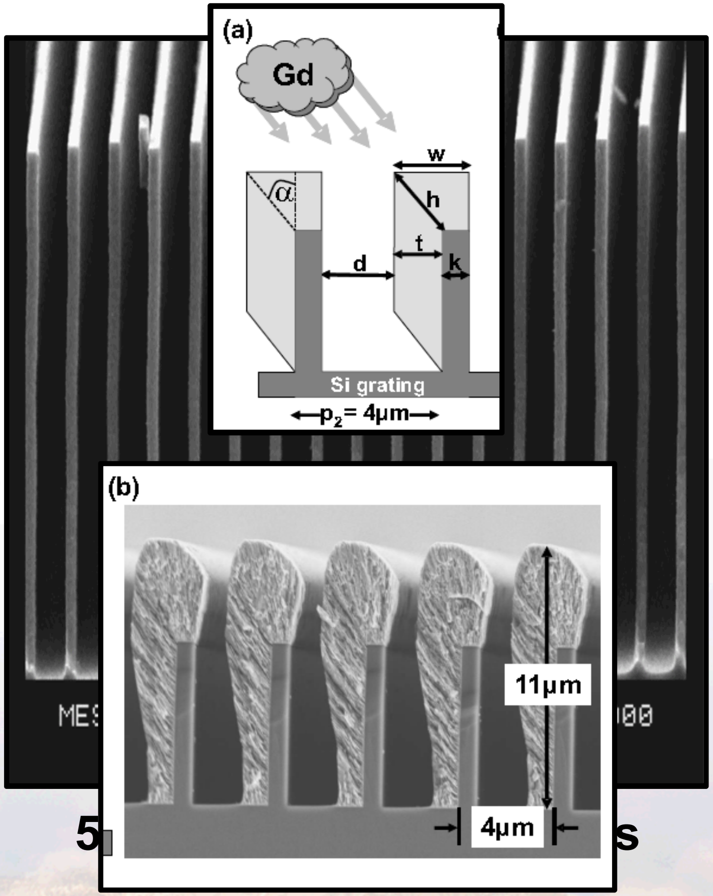
Mohr et al. has been able to achieve very impressive aspect ratios as high as 100:1, however this approach is limited through the necessity of support structures or tiling gratings to achieve large area systems.

1. Mohr, J., et al., *High Aspect Ratio Gratings for X-Ray Phase Contrast Imaging.* International Workshop on X-Ray and Neutron Phase Imaging with Gratings, 2012. **1466**: p. 41-50.

Coating Instead of Filling

- Use DRIE Si as the grating template
- Capable of depositing a wide variety of materials
- Better structural stability

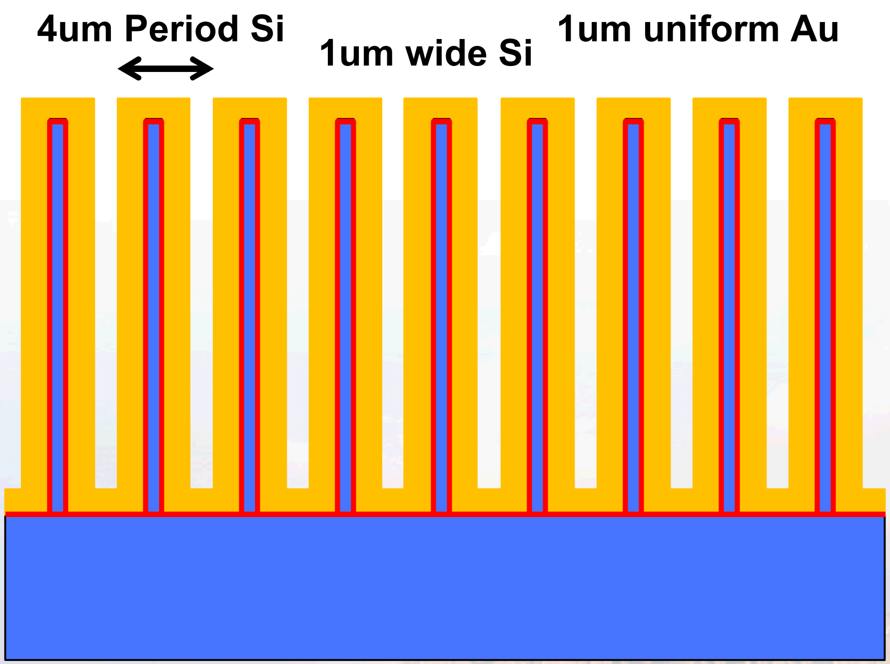
- Angled Physical Vapor Deposition
- Highly anisotropic
- High aspect ratios possible (50:1)
- Non-uniform deposition

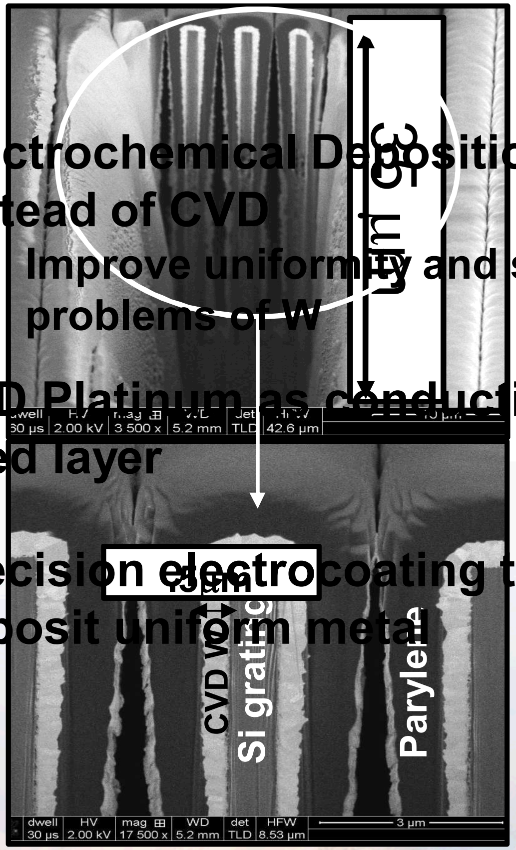


Grunzweig et al., Rev. Sci. Instrum. 79, 053703 (2008)

Electro-coating Instead of Filling

- CVD Tungsten (W) is deposited with very high tensile stress (GPa)
- Non-uniform deposition
- Parylene required for added rigidity

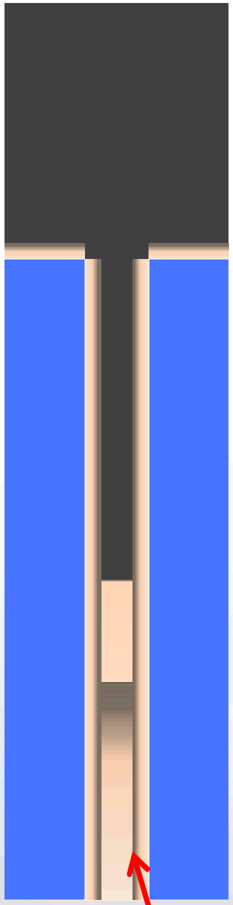




- Electrochemical Deposition instead of CVD
  - Improve uniformity and stress problems of W
- ALD Platinum as conductive seed layer
- Precision electrocoating to deposit uniform metal

Challenges in Electro-coating vs Filling

Max (bulk) Ion Concentration



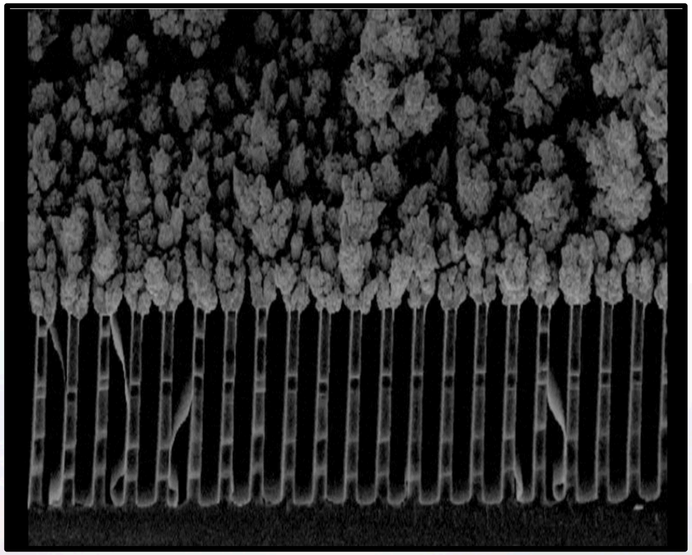
Severely depleted region

- Very different than bottom-up filling of a mold
- Diffusion limitations are compounded with ionic depletion due to reduction
- Conventional applied bias leads to non-uniform plating

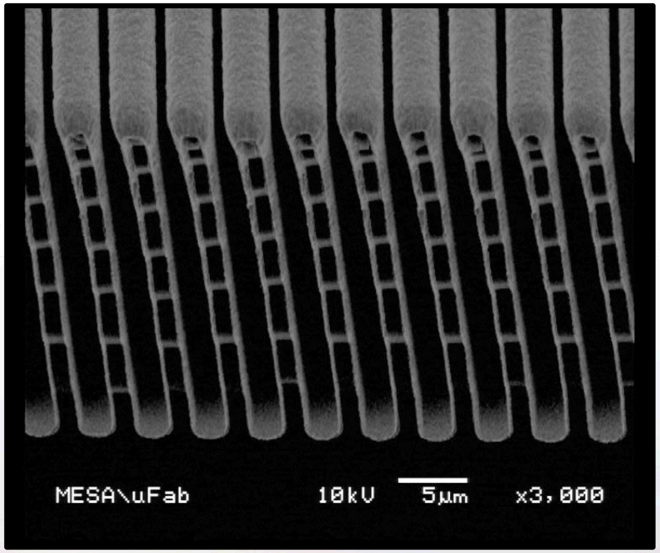
Needs to Improve Plating

- **Incorporate more time for ionic replenishment**
- **Equilibrate concentrations over plating surfaces**
- **Increase period of time between deposition pulses**

DC



AC



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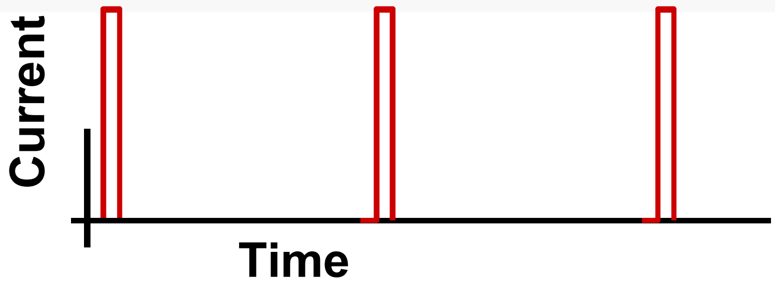
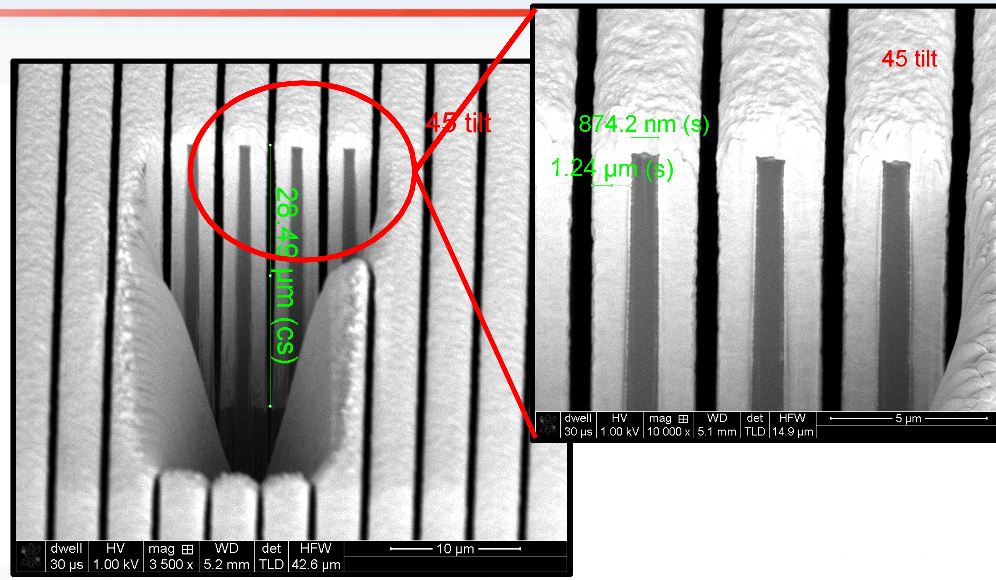
Optimized Pulse Plating Conditions

~3% duty

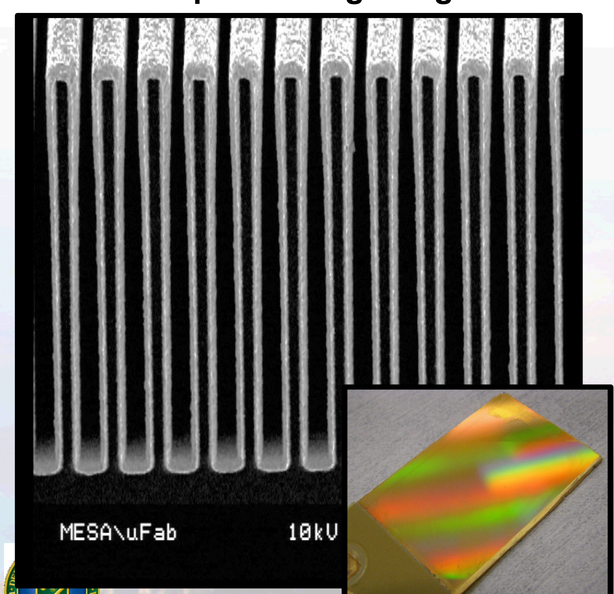


Drastically reduced kinetic and plating rate

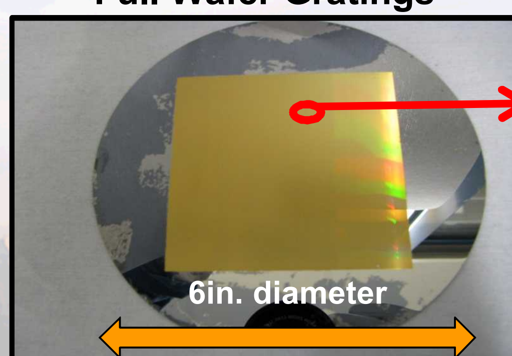


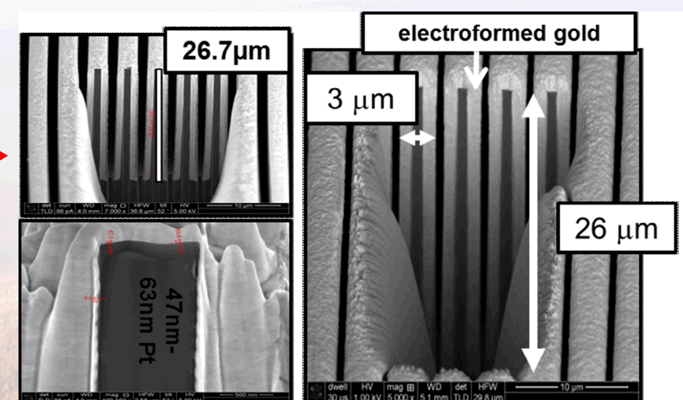
Uniform Plating in 30:1 aspect ratio gratings

50:1 aspect ratio gratings



Full Wafer Gratings





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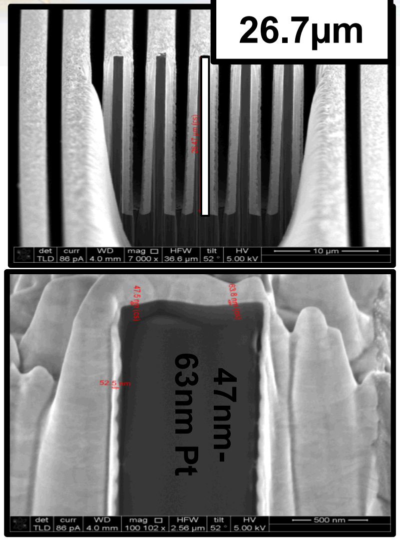
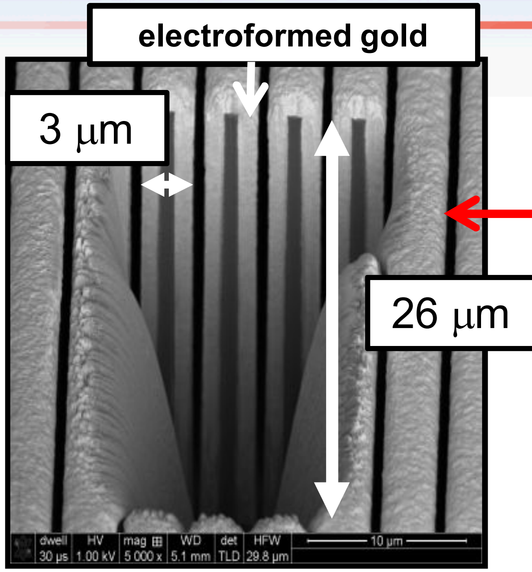
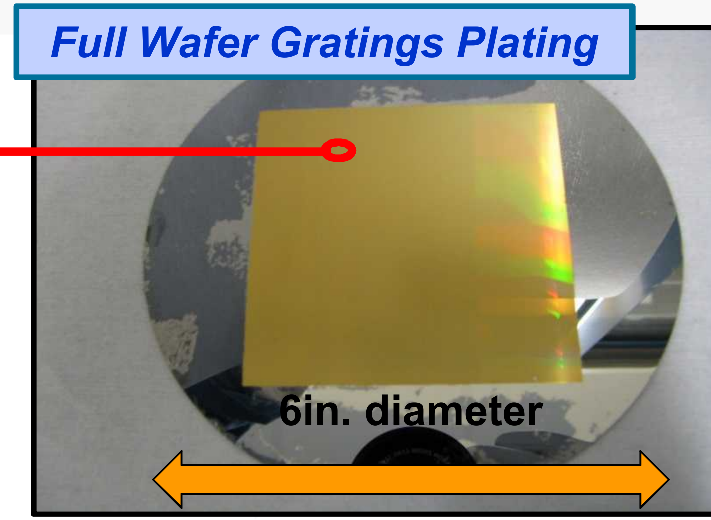
Precision Electro-coated Si Gratings







Bottom-Up Approach (UV or LIGA)

- High aspect ratios possible
- Resist is not structurally stable
- Resist swelling occurs
- FOV is limited

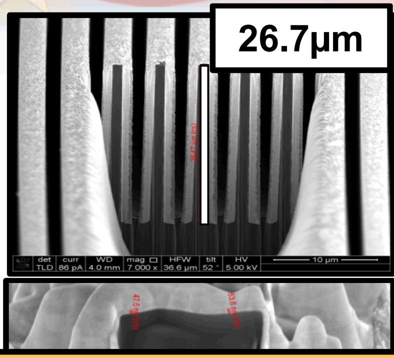
Tungsten CVD of Si DRIE Gratings

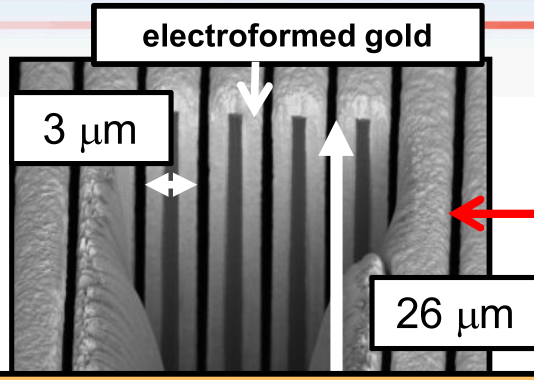
- Non-uniform deposition in high aspect ratios
- Highly stressful
- Parylene required for added rigidty

Electroplating Approach

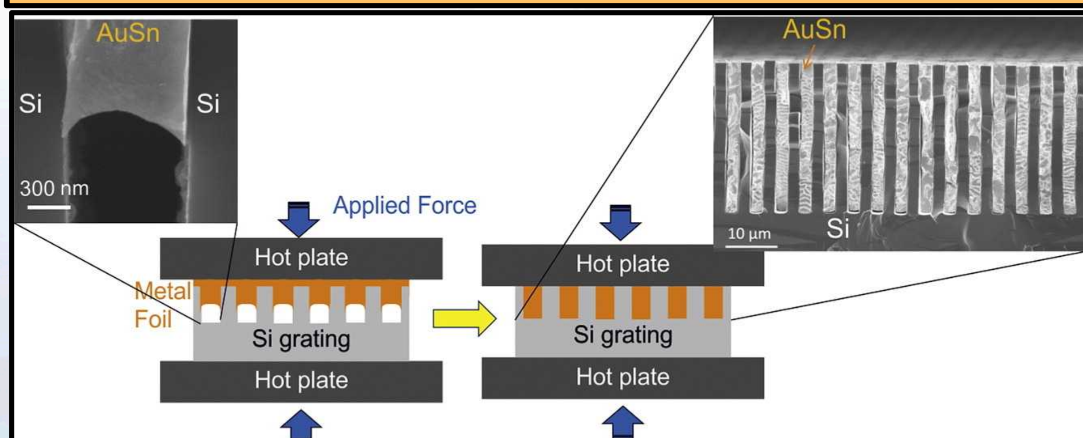
- High absorber material (Au) is possible
- Uniformity can be tailored to desired aspect ratio
- ALD Pt provides a conformal seed layer
- Low stress Au
- Lateral support structure are not necessary
- 50um tall Si gratings have been plated
- 4in. X 4in. area achieved with 26um tall Si gratings
- Uniform plating process is complex and geometry dependent
- Increasing to larger areas becomes cost inhibited

Precision Electro-coated Si Gratings





Increasing the aspect ratio of our Si template will allow high energy XPCI systems or alternatively allow cheaper materials to be used on lower energy systems



AuSn Embossing into Si template

- **Cost effective material**
- **Less sensitive filling mechanism than electro-coating**
- **High uniformity is feasible with full wafer bonding tools**
- **Less dense material requires thicker template**
- **Higher energy XPCI systems requires thicker template**

L. Romano et. al. Microelectronic Engineering 176(2017) 6-10

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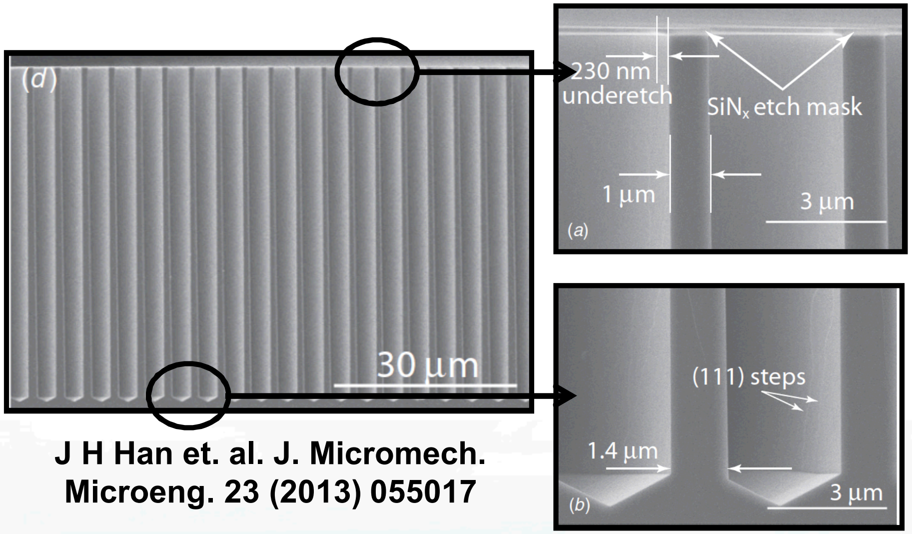
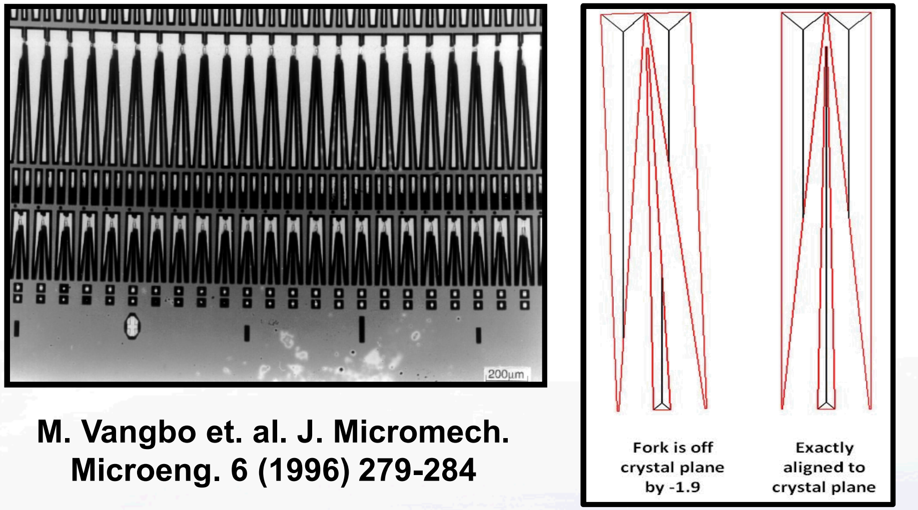
Alternative Etch for Increasing Aspect Ratio Crystallographic Etching in KOH

Etch selectivities of 600:1 have been reported between [110] and [111] crystal planes



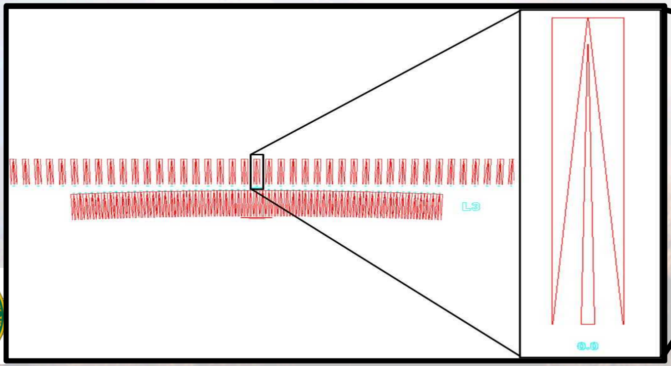
J H Han et. al. J. Micromech. Microeng. 23 (2013) 055017

Wafer flats are aligned within 2º of a crystal plane → Need a method for accurate alignment within 0.1º

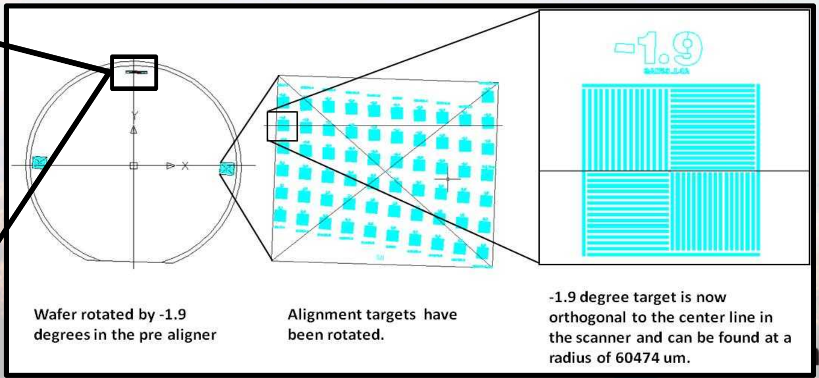


M. Vangbo et. al. J. Micromech. Microeng. 6 (1996) 279-284

Arrayed Comb Structures at 0.1º increments from -3º to +3º

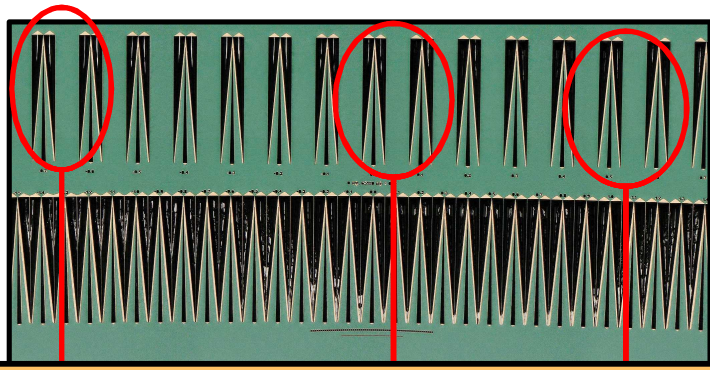


Layer 0 Etch and Alignment Scheme



Alternative Etch for Increasing Aspect Ratio Crystallographic Etching in KOH

25 minute layer 0 etch in 6 M KOH



Replace this with an image of a grating etched after performing this alignment

Stop by Patrick Finnegan's Poster for more information on our KOH etching results.

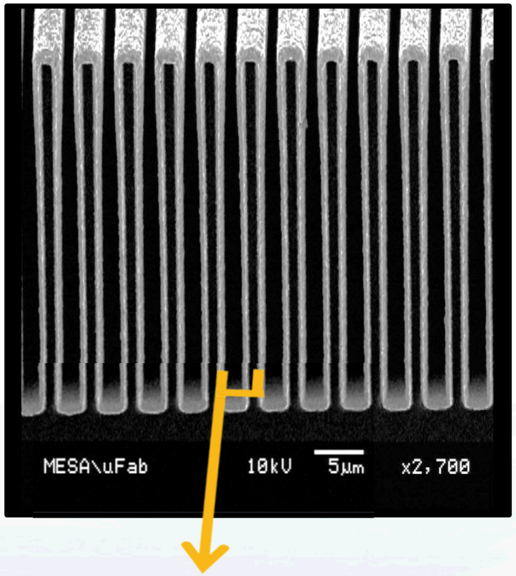
Poster #37 Wednesday 13 September 12:30-14:30

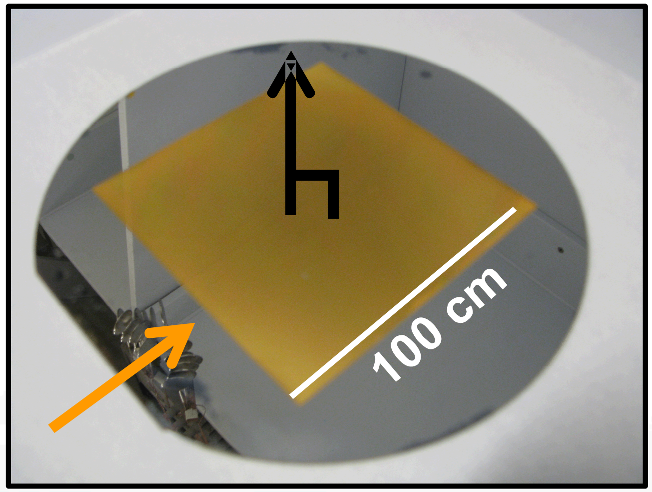
"Extending the Aspect Ratio of Etched Silicon for Precision Electro-coating with Gold to Fabricate XPCI Analyzer Gratings"

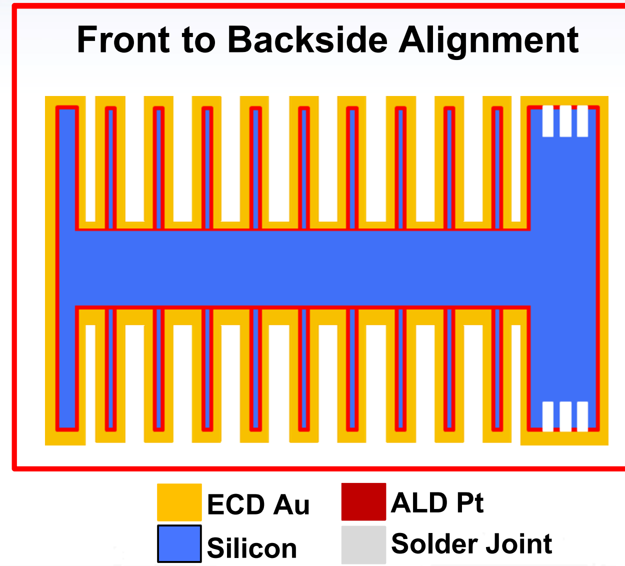
Increase Effective Aspect Ratio of Gratings

Standard X-ray propagation direction

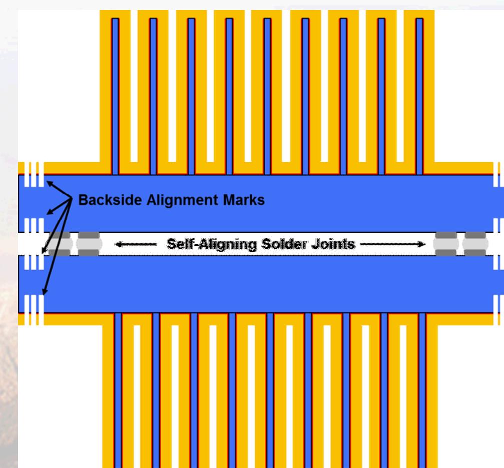
Alternative X-ray propagation direction







Align and Stack Multiple Gratings



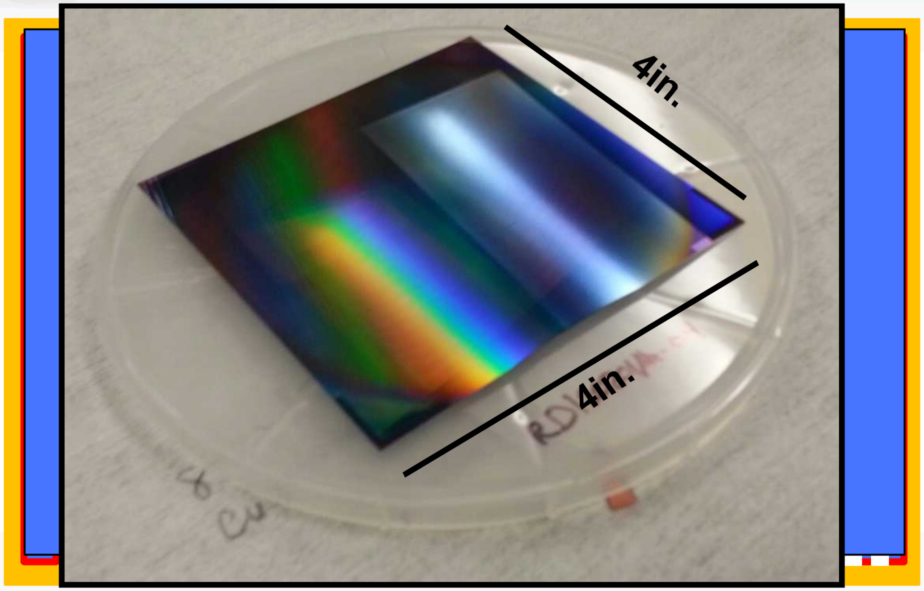
Orient x-ray k-vector down the length of the grating as opposed to perpendicular to the grating

- **Effective aspect ratio increases to 10,000:1**
- **Decreases field of view from 100 cm x 100 cm down to 30 µm x 100 cm**

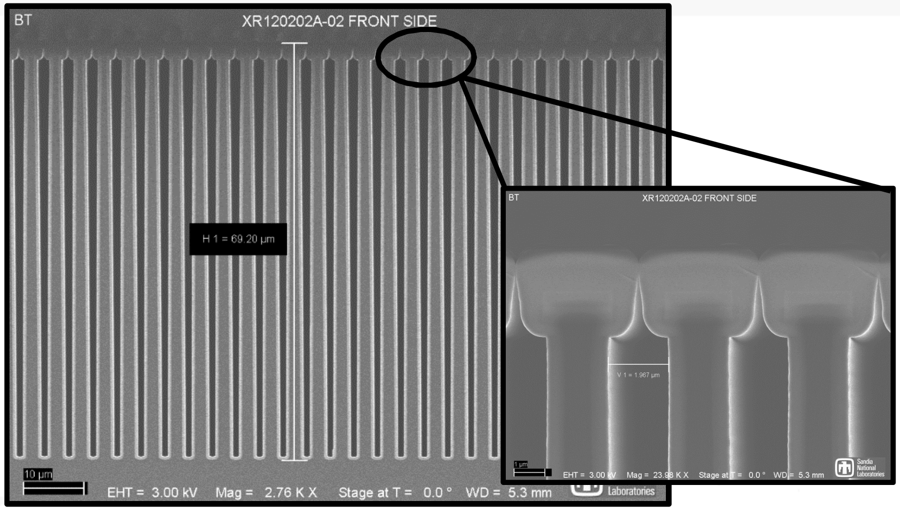
Front to Backside Aligned Gratings

Silicon ALD Pt ECD Au

Large Fields of View Possible

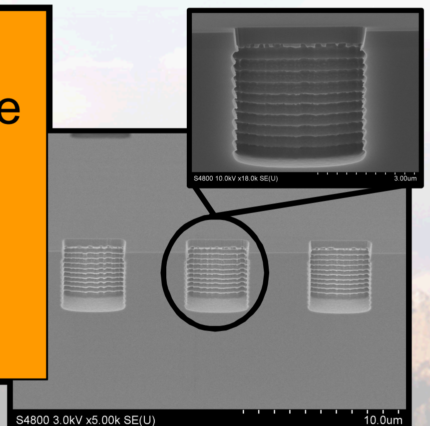


Oxide Coated Front Side Gratings

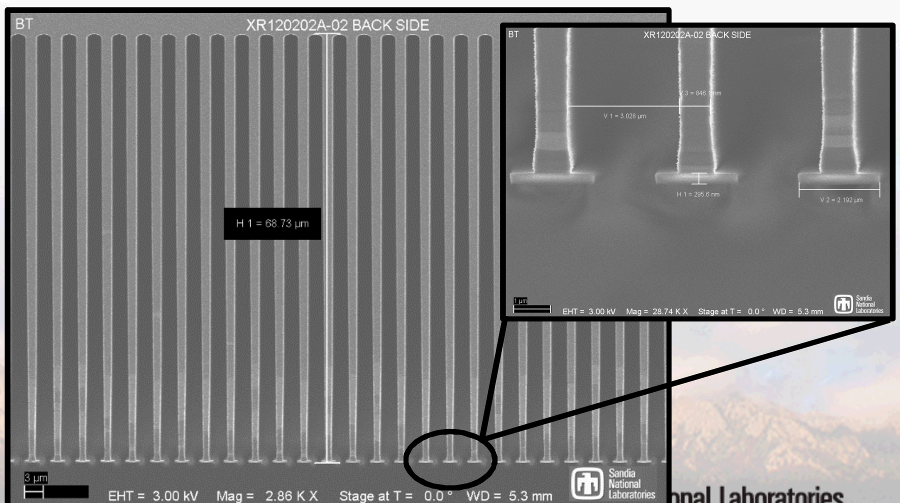


Backside Alignment (BSA) Marks

This image will be replaced by a similar one provided by and approved by Nikon

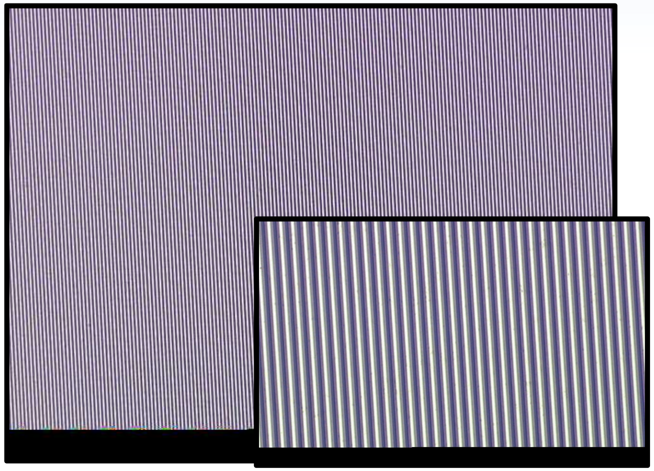


Backside Gratings

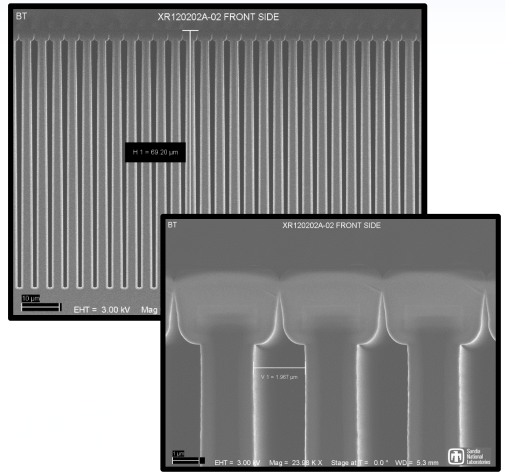


Frontside Grating Oxide Removal

Before Oxide Removal

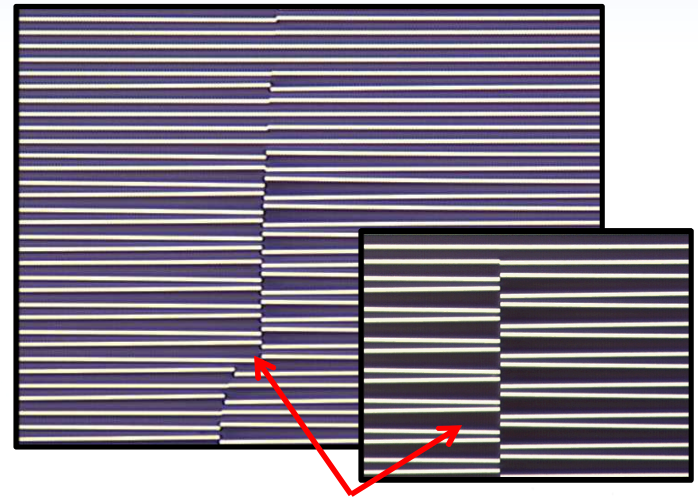


Top Down Optical Image



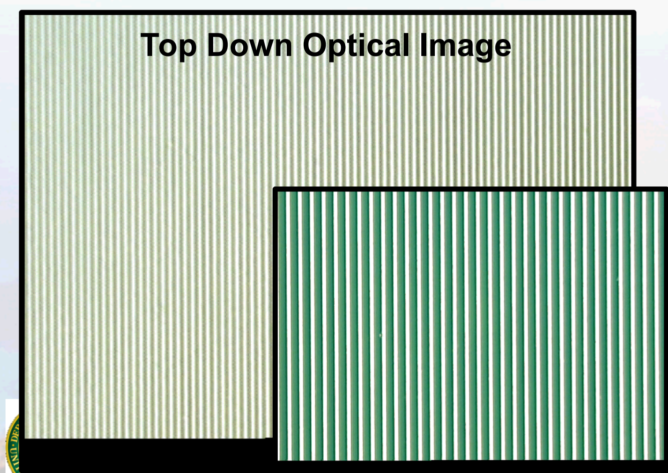
Cross-section SEM

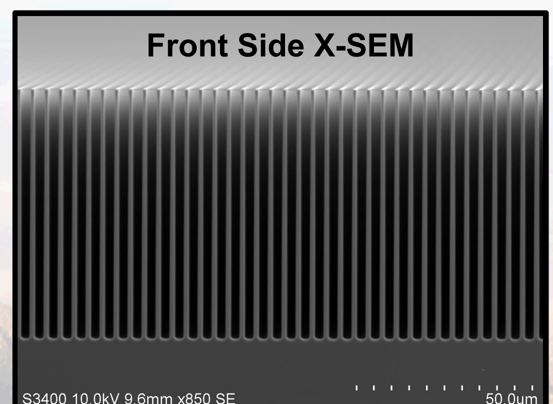
Buffer Oxide Etch (BOE) w/o Critical Point Dryer

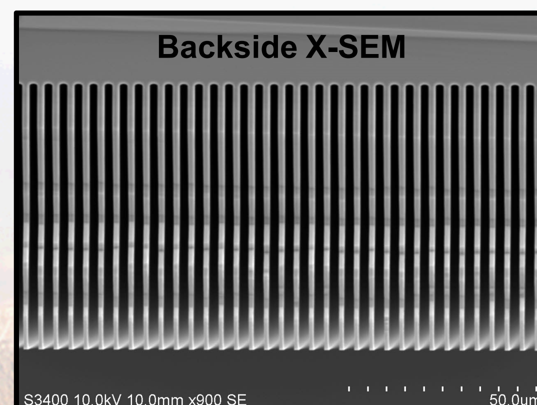


Si grating breaks while chemistry is drying

BOE with Critical Point Dryer







Critical point dryer reduces surface tension while chemistry dries protecting the Si grating

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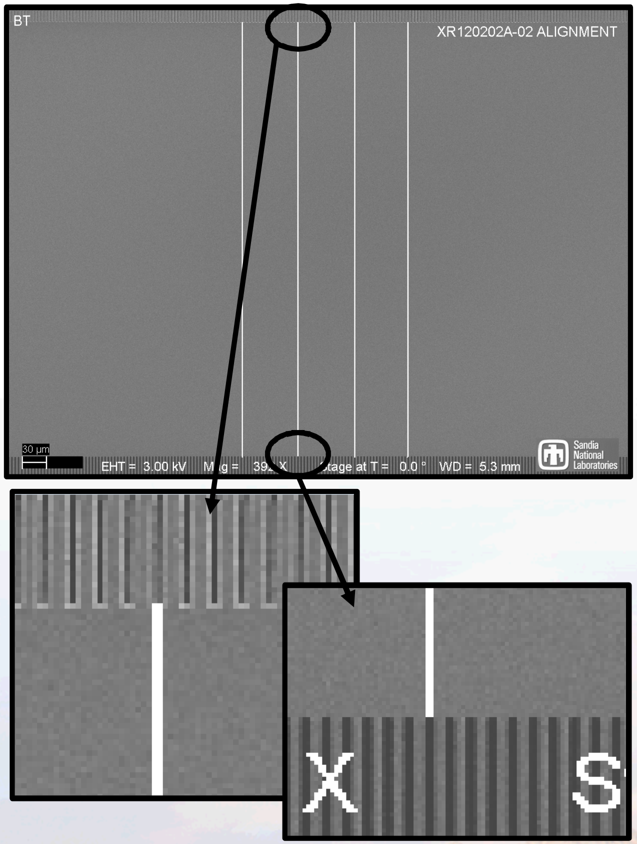
Measuring Front to Back Alignment Accuracy

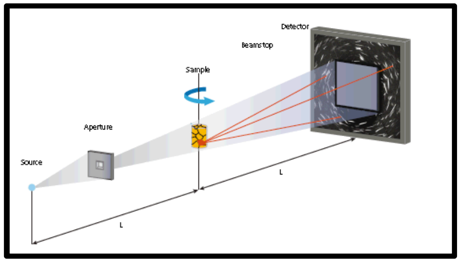
Large separation distance and small feature sizes make it difficult to obtain quantitative alignment measurements

Cross-section SEM Image

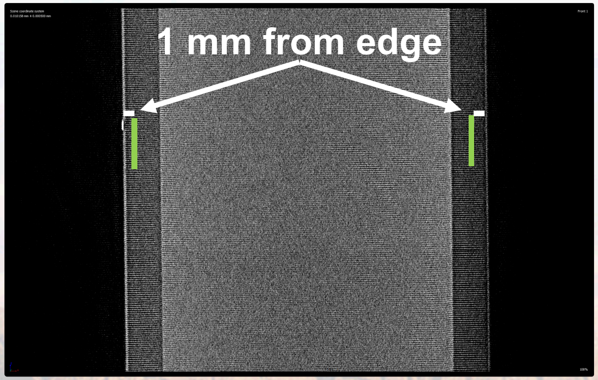


SEM imaging is not capable of quantifying alignment

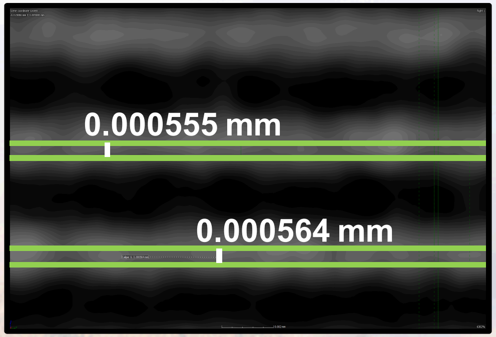
Zeiss Xradia 520 Versa NanoCT System



Side view CT scan of BSA Gratings



Superimposed Pitch Measurements



Qualitatively our alignment is within 0.5 µm as each pixel from the CT scan is 0.5 µm

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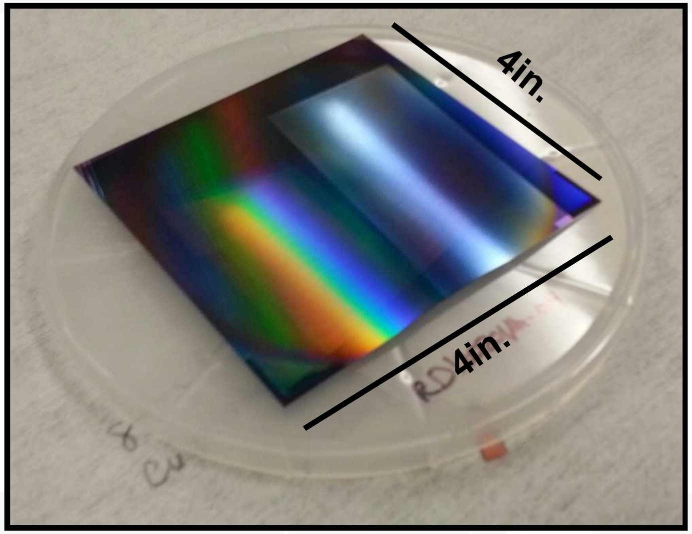
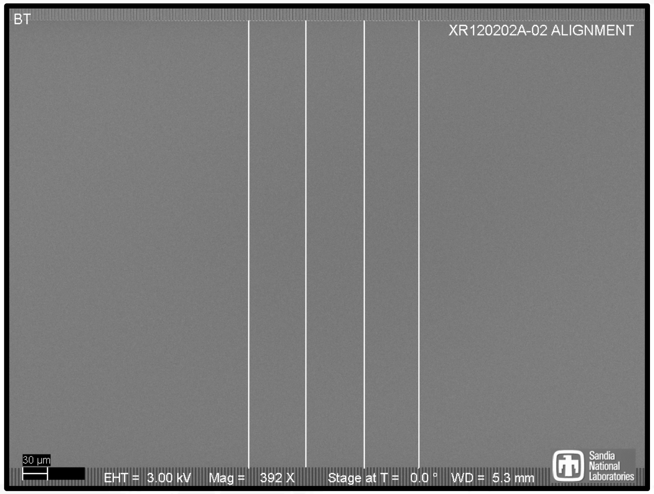
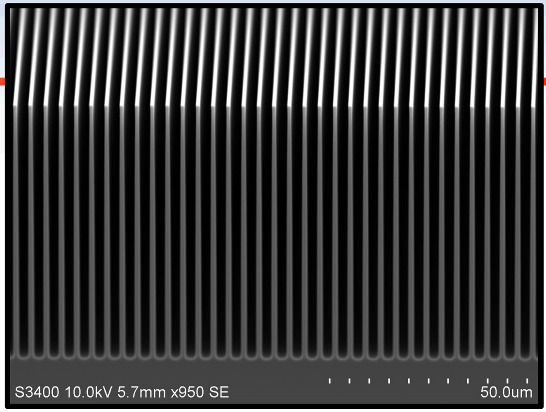
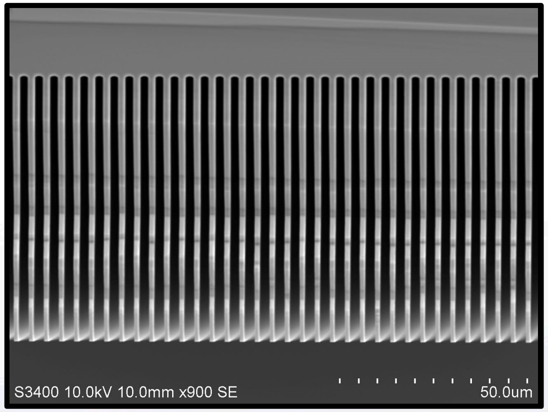
Summary









Grating Fabrication with Front-to-Back Alignment

- Doubles the effective aspect ratio
- Cost effective material can be used to fill this template at current x-ray energies
- Higher x-ray energies possible with precision Au electroplating
- Large area fields of view possible

Future Work

- Develop double sided Au electro-coating process
- Investigate precision alignment of multiple stacked gratings

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Acknowledgements

Andrew Hollowell, Christian Arrington, Kalin Baca, Jonathan J. Coleman, Patrick Finnegan, Ryan Goodner, Steven Grover, Jeffrey Hunker, Jaime McClain, Kate Musick, Collin Smith, Kyle Thompson, Amber Dagel

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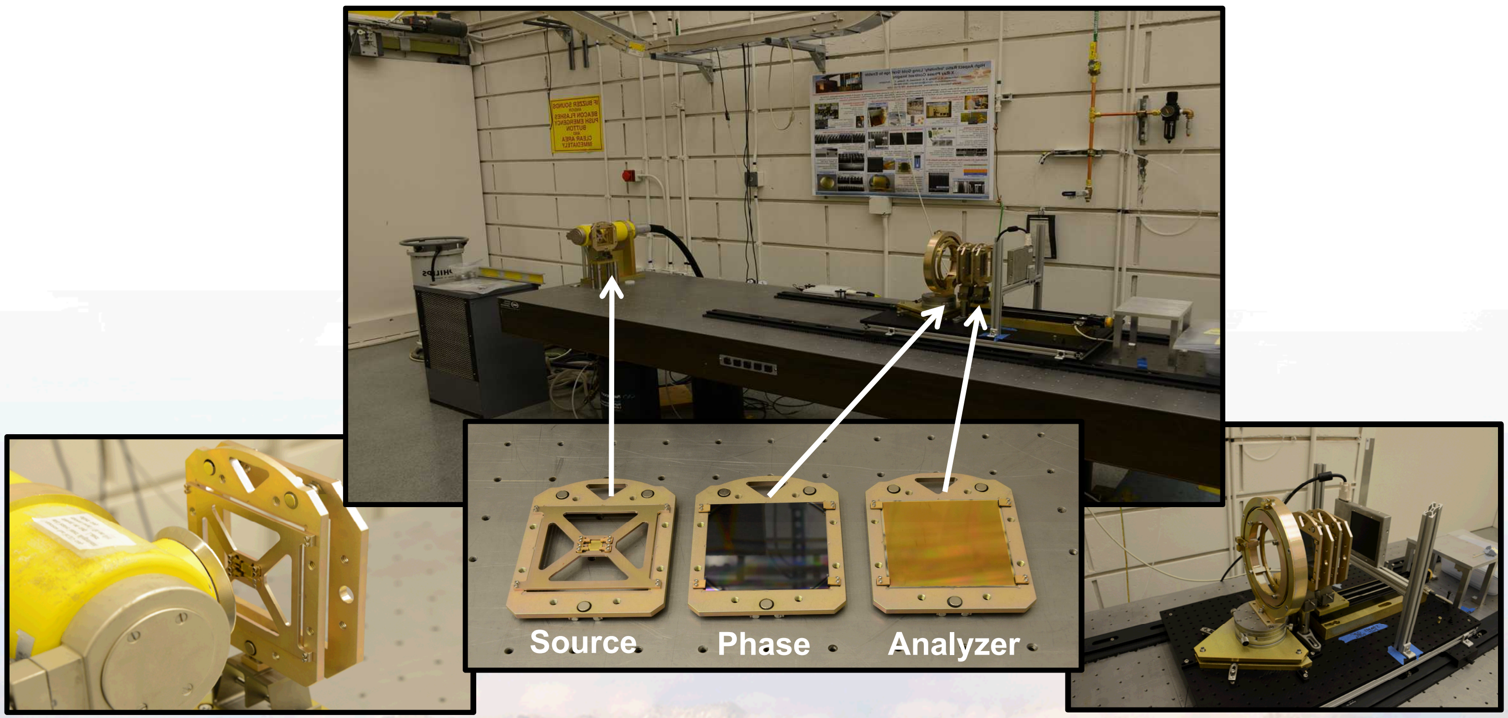
aehollo@sandia.gov – 505 844 8301



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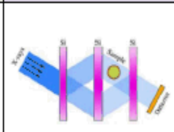
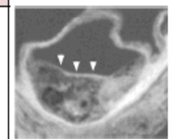
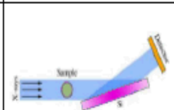
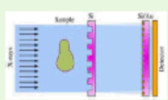
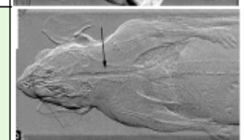
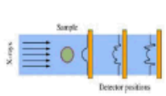
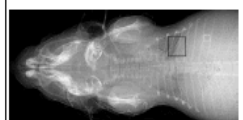
3 Grating System w/ Conventional X-Ray Tube

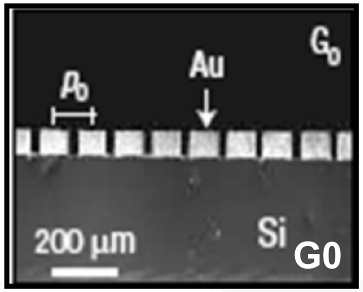
Method	working principles	setup scheme	measured phase quantity	PROS	CONS	
Crystal interferometer	beam splitting and recombining crystals (Si)		phase ϕ	• highest phase sensitivity	• mechanical instability • monochromatic (synchrotron) • limited FOV (crystals) • low efficiency	
Analyzer based interferometer	bragg reflection		phase gradient $\nabla\phi$	• high phase sensitivity	• monochromatic (synchrotron) • low efficiency	
Grating based interferometer	grating interference		phase gradient $\nabla\phi$	• good phase sensitivity • polychromatic • large FOV • high efficiency	• requires gratings	
Propagation based interferometer	Free space Fresnel propagation		Laplace of phase $\Delta\phi$	• simple design • polychromatic	• requires high-res detector • low phase sensitivity • low efficiency	

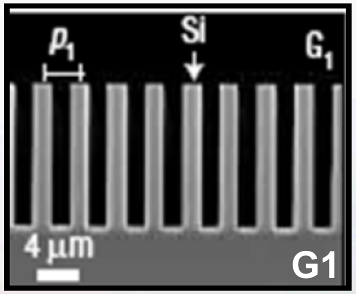
$$n = (1 - \delta) + i \bullet \beta$$

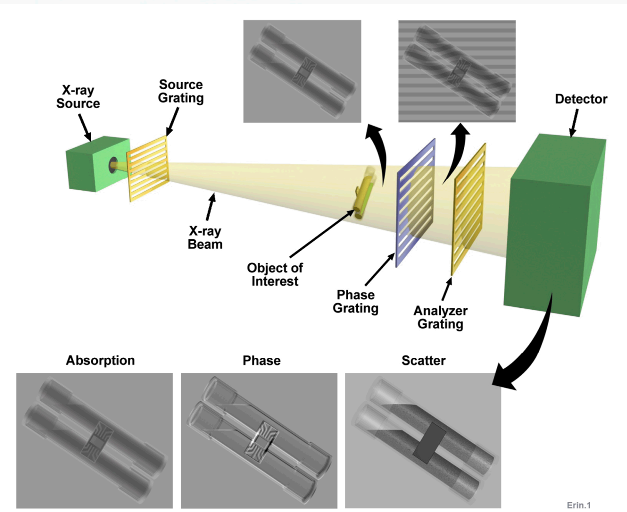
β → Absorption δ → Phase

δ/ β = ~10^3

Phase imaging is ~10^3 times higher Sensitivity than absorption imaging



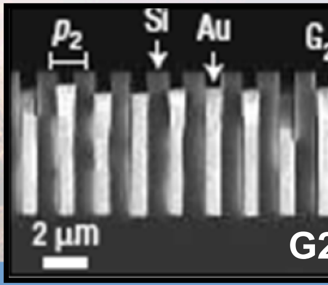




G0 – Source Absorption Grating
Creates an array of individually coherent but mutually incoherent sources

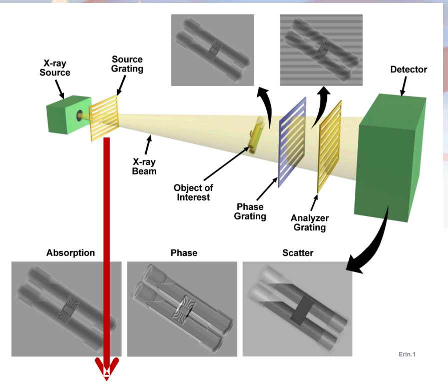
G1 – Phase Grating
Creates zero and π phase shifts to form an interference pattern at G2 plane

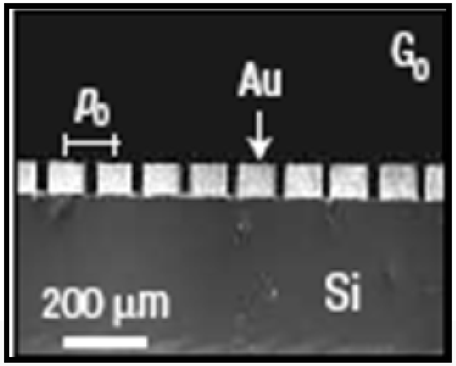
G2 – Analyzer Absorption Grating
Modulate the interference signal on detector (local fringe position → single intensity variation recorded by detector)



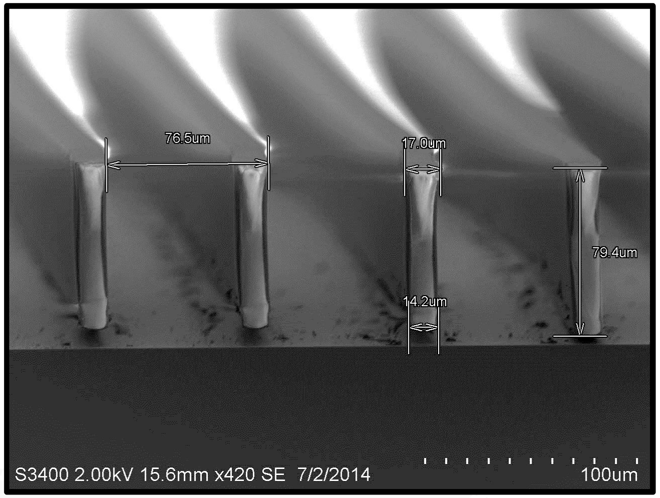
Images courtesy of Erin Miller - PNNL

G0 Source Grating – UV Photoresist

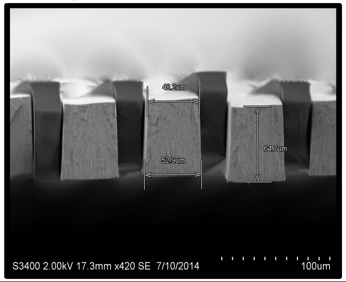


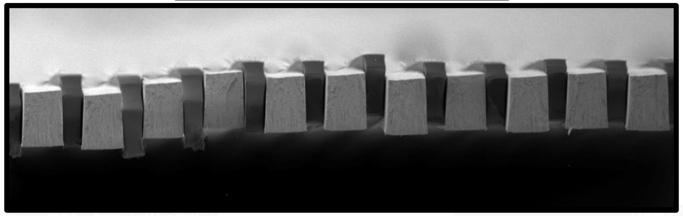


KMPR Negative Photoresist

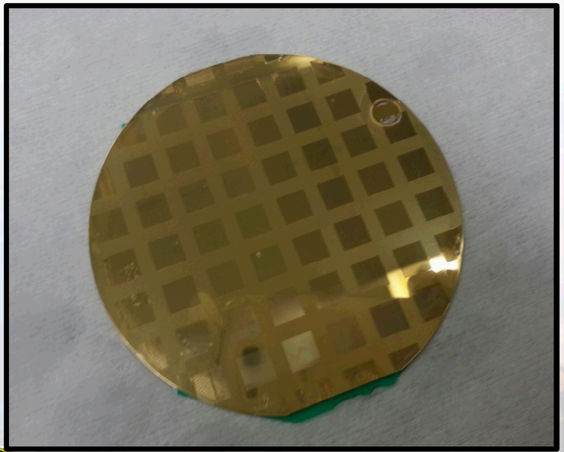


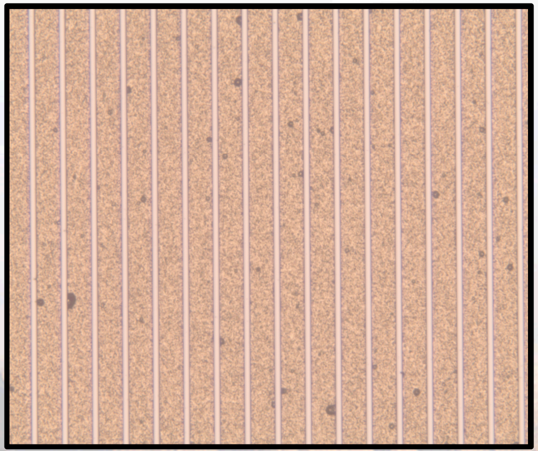
65µm Thick Au

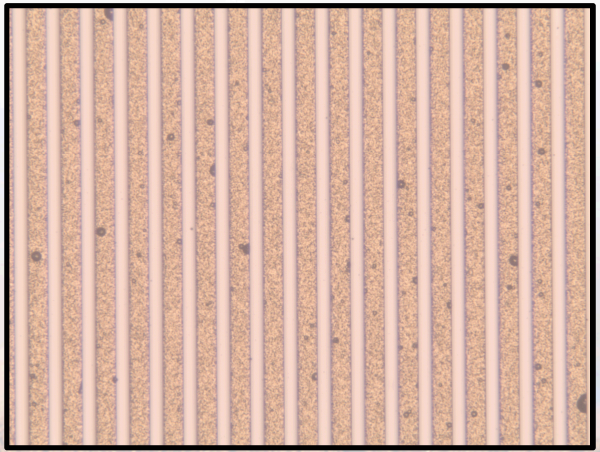




Delamination result of cross-section

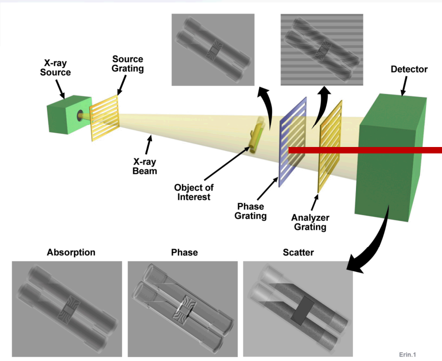


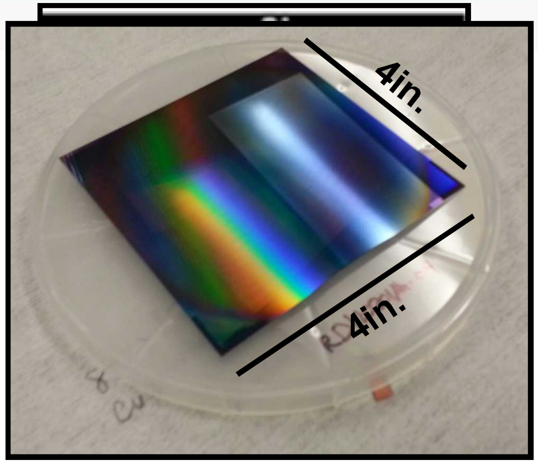




Pitch: 76, 77, 78, 79, 80µm - Linewidth: 13, 18, 23, 28µm
Thickness: 65µm, Highest Aspect Ratio of 5:1

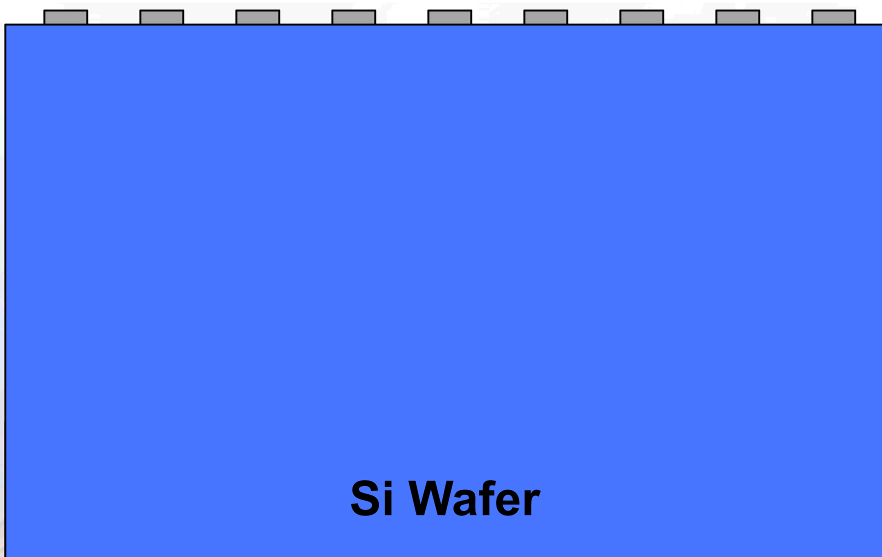
G1 Phase Grating – Si Etching



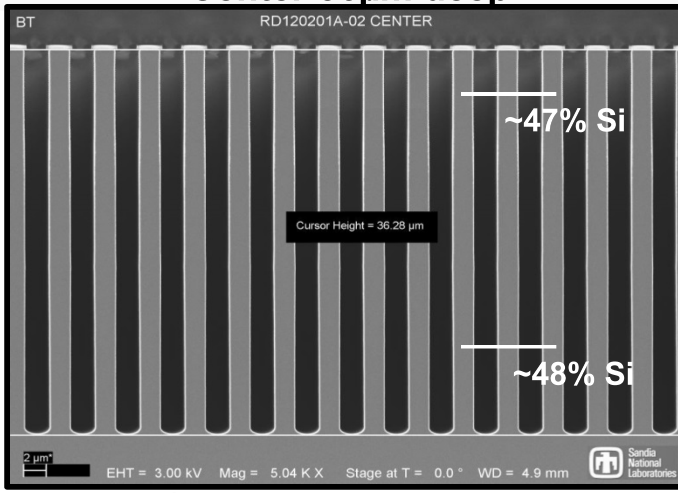


4um Period Si

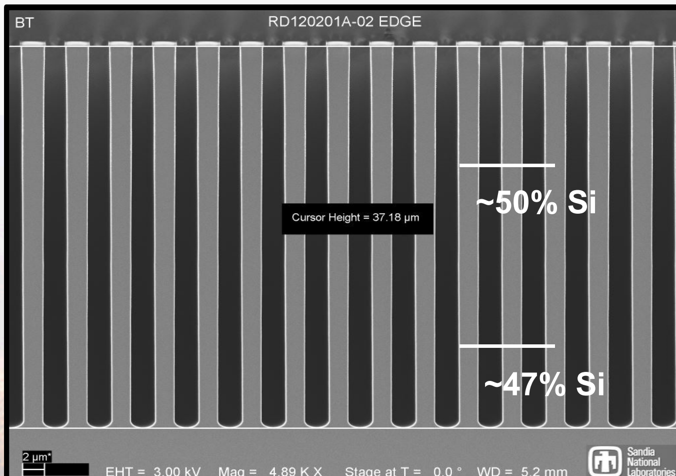
2µm wide Si



Center 36µm deep



Edge 37µm deep



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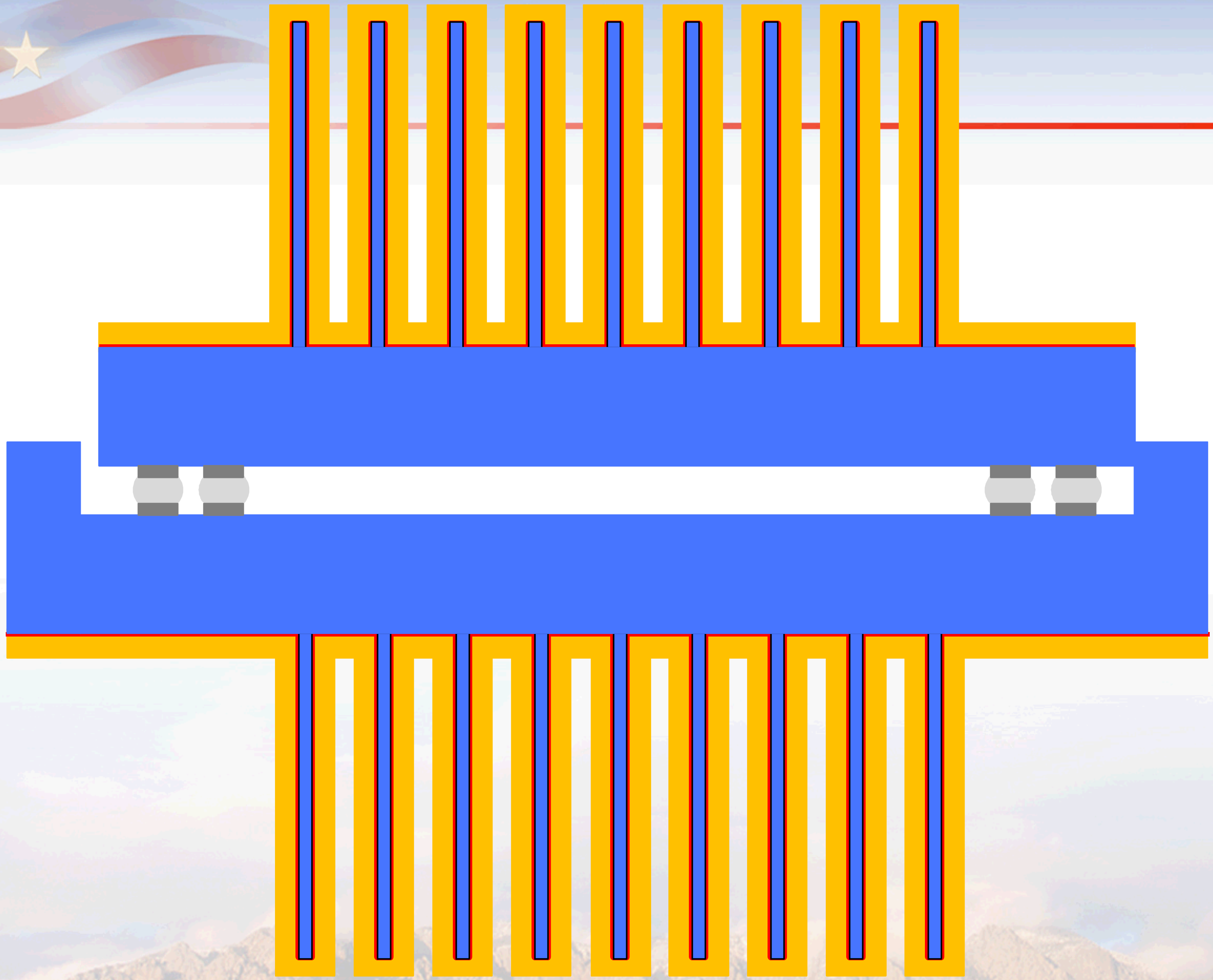
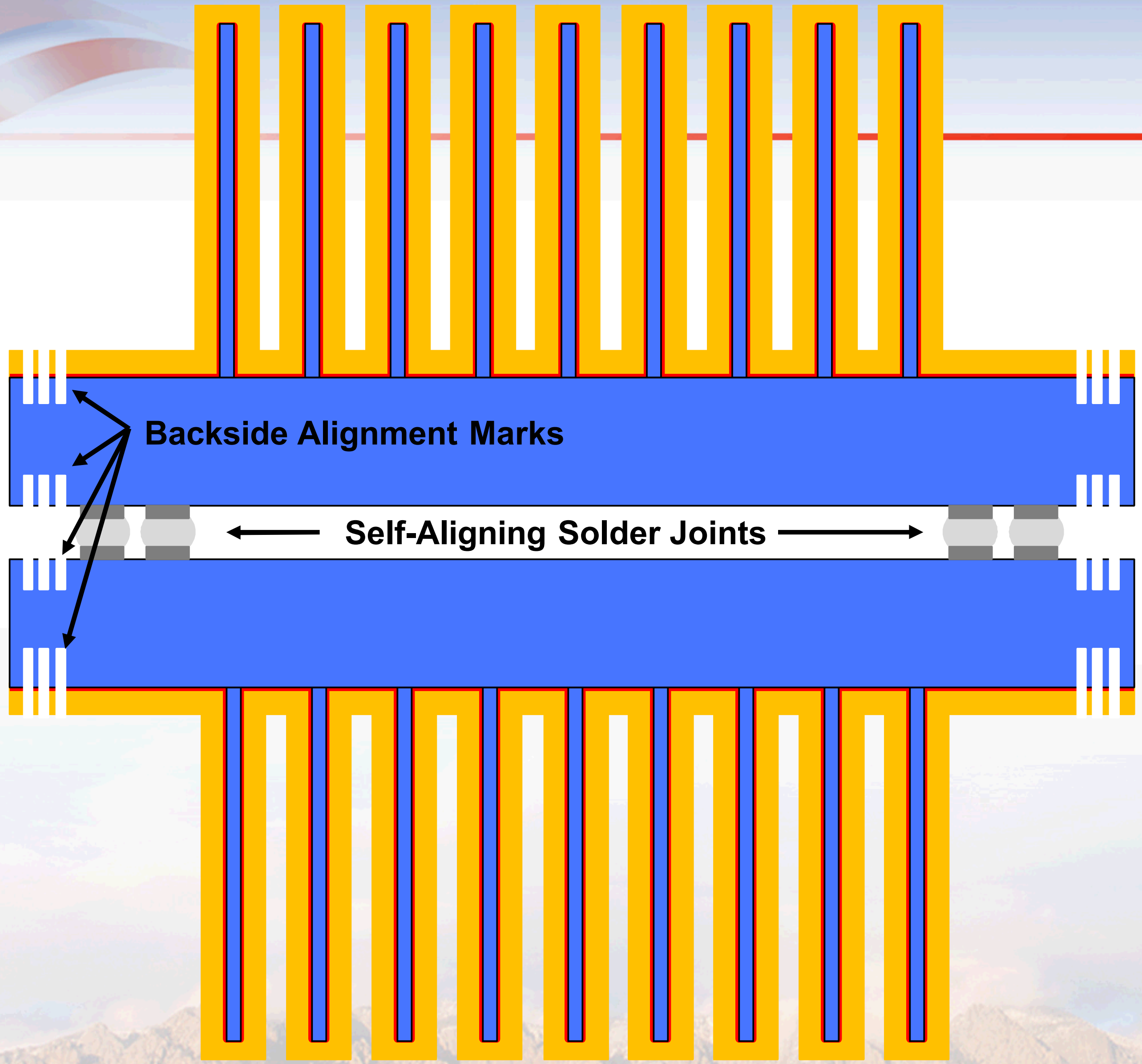
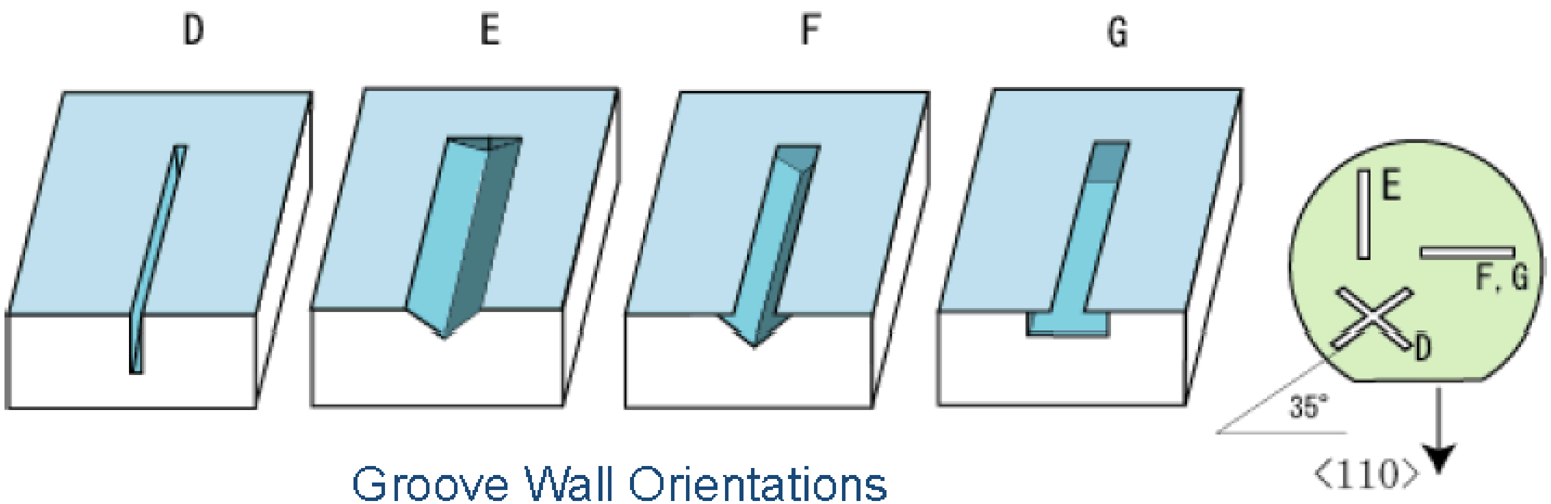



Backside Alignment Marks
Self-Aligning Solder Joints

Variation in etching profile on (110) silicon wafer

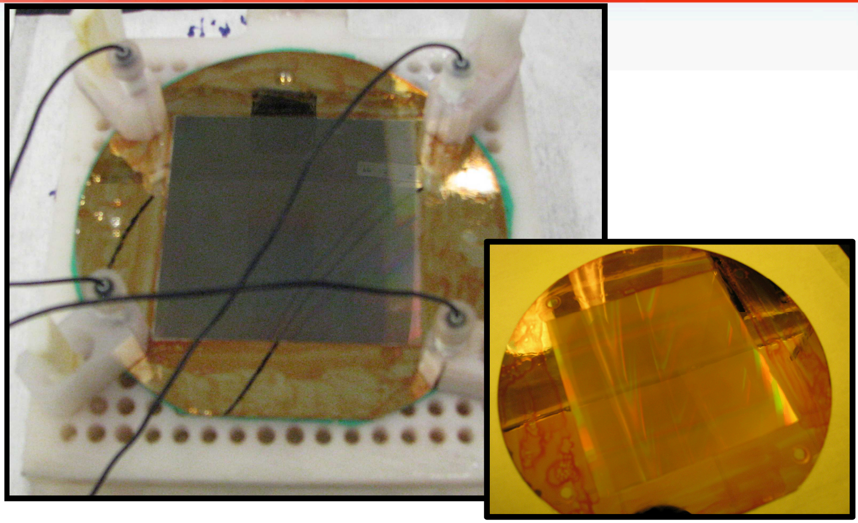


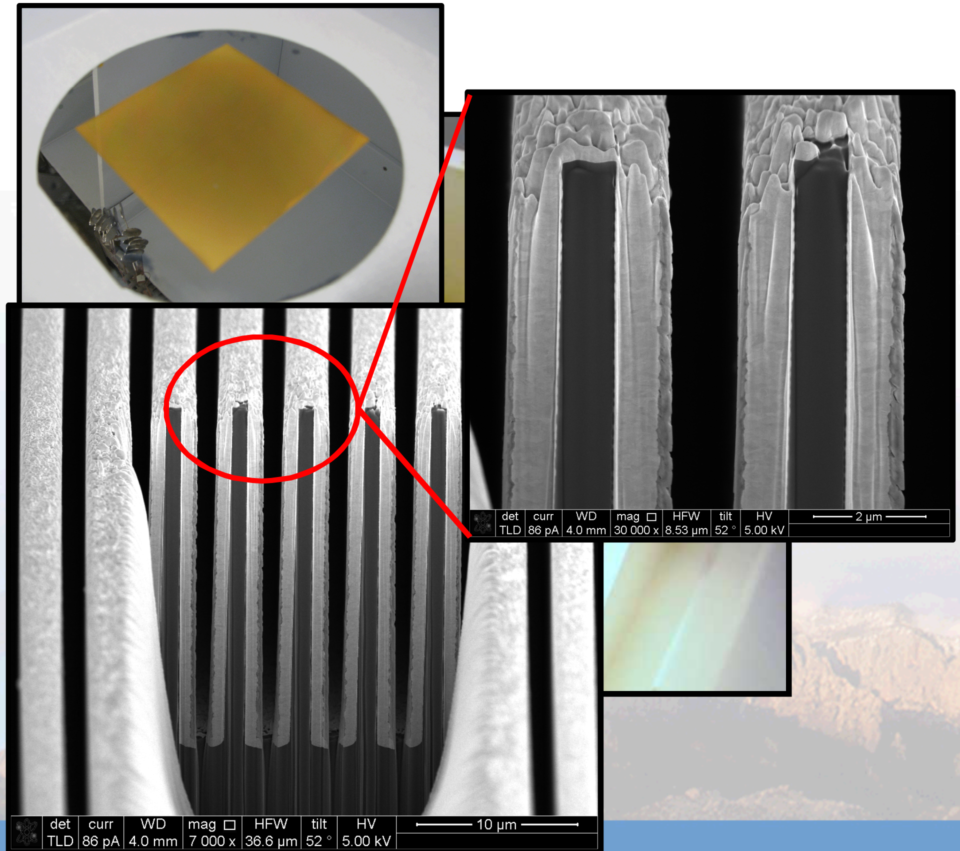
Groove Wall Orientations

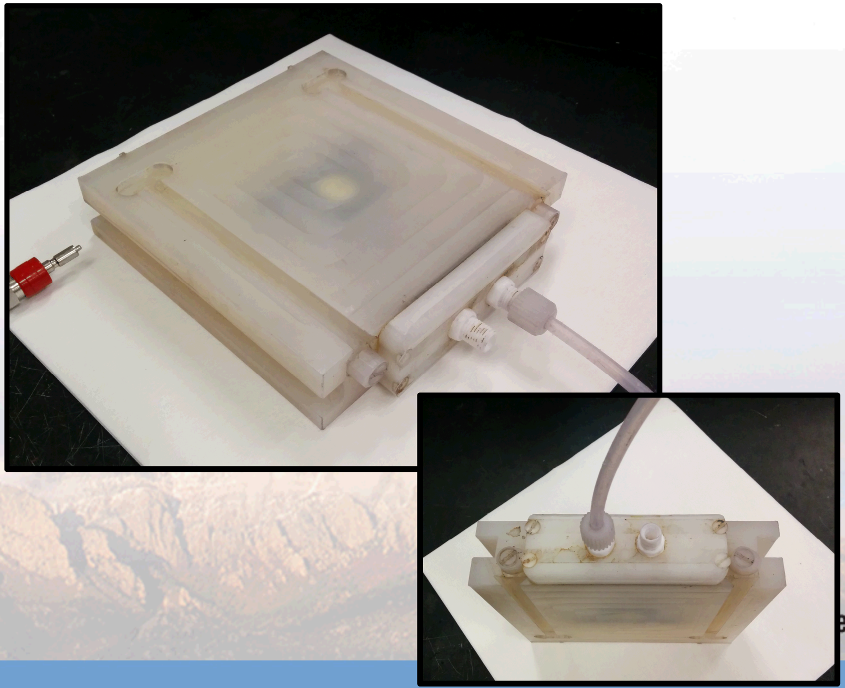
(111)	(111)	(100)	(110)
KOH TMAH EDP	KOH TMAH EDP	KOH TMAH	KOH/IPA TMAH/surfactant EDP

Chemistry Flow and Fixture Modifications to Realize 16in.^{2} Uniformity

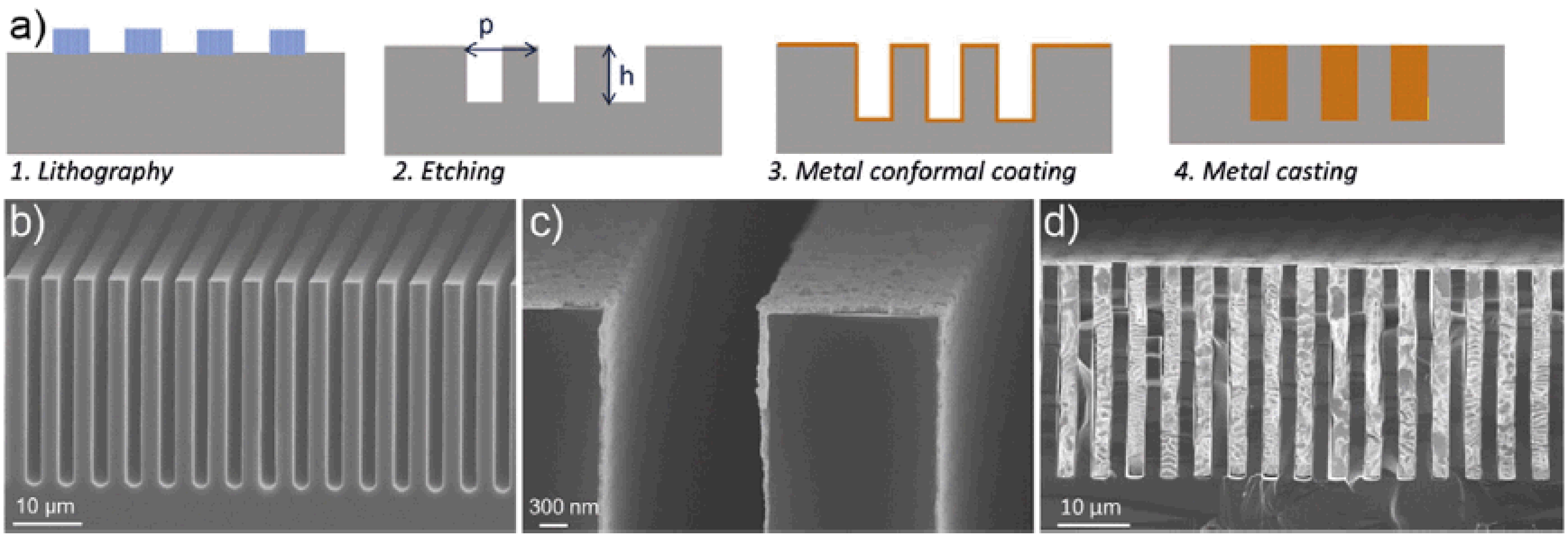
- Chemistry flow dictates ionic replenishment
- Multiple contact points evenly distributes conductivity
- Vibrating fixture enables even solution replenishment and ionic distribution







A1



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A1 Andrew , 8/30/2017

Vapor Hydrofluoric Acid (VHF)