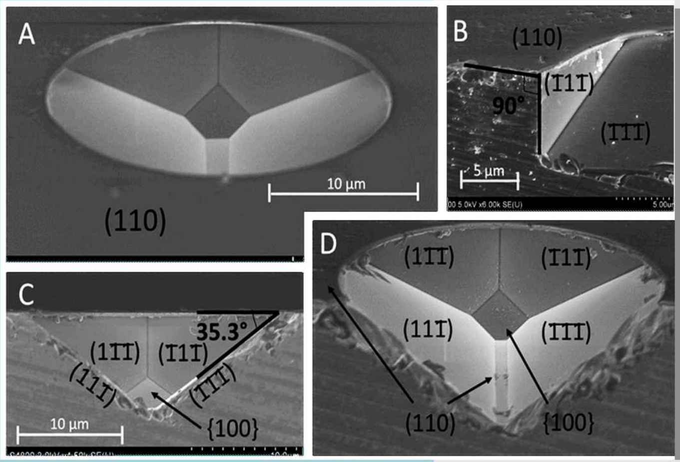
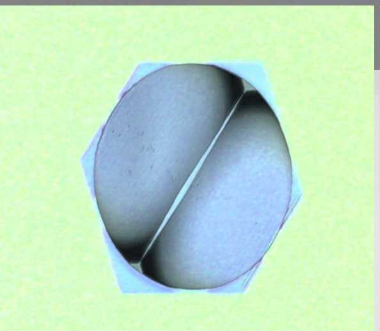
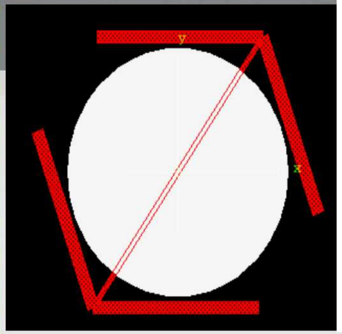
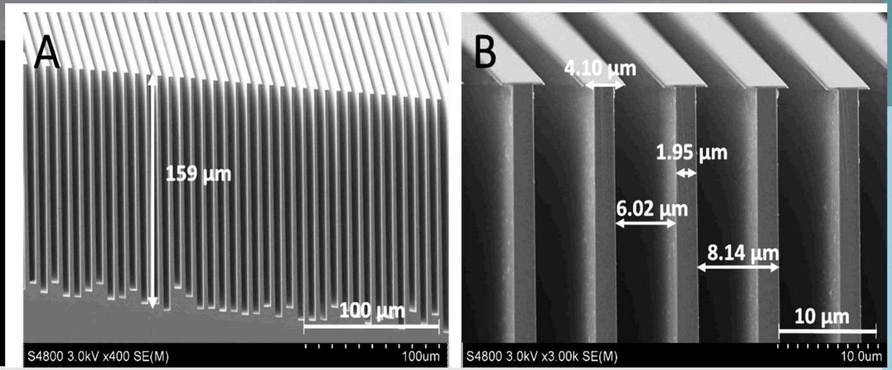
SAND2019-5951PE

Fabrication of Extremely High Aspect Ratio Diffraction Gratings for X-ray Phase Contrast Imaging









PRESENTED BY

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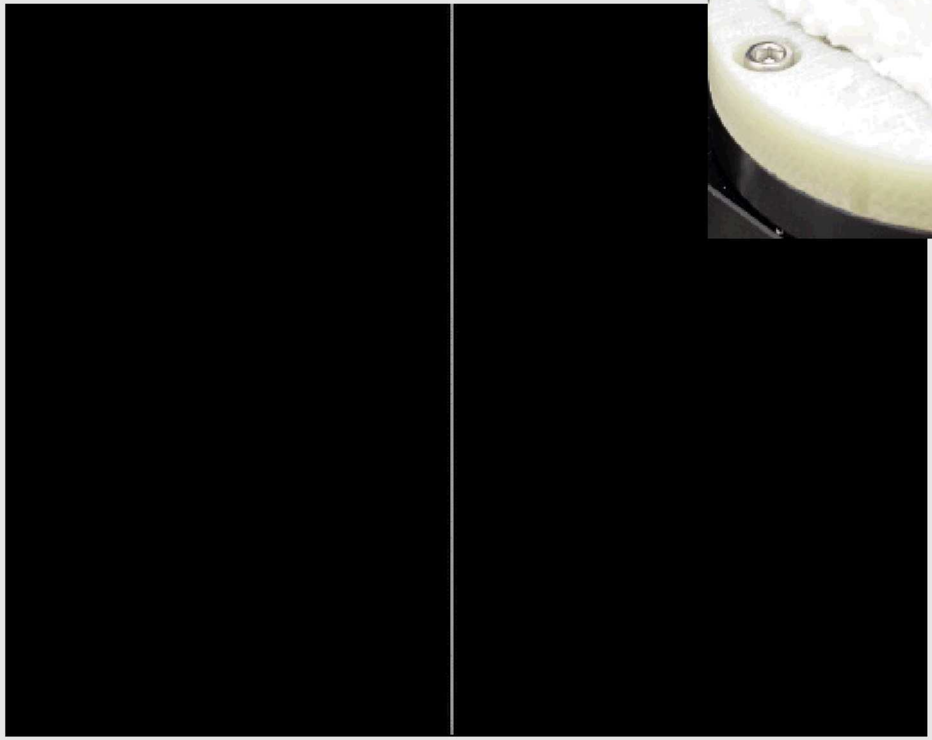
Sandia National Laboratories

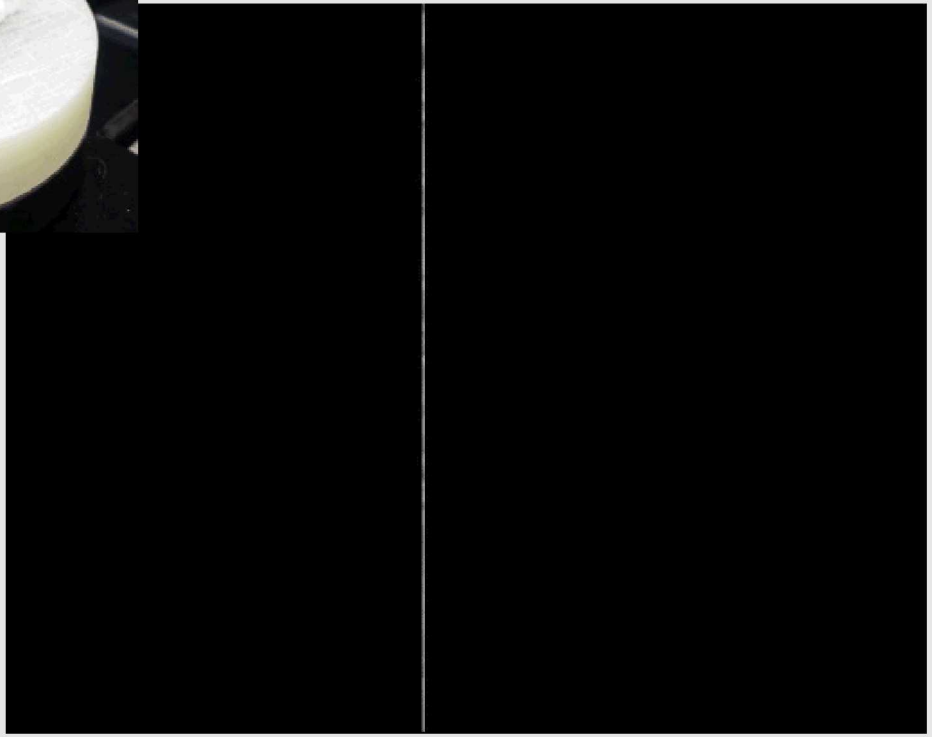
Albuquerque, New Mexico

Supported by the Laboratory Directed Research and Development program at Sandia National Laboratories, a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Conventional X-ray vs XPCI



Transmission



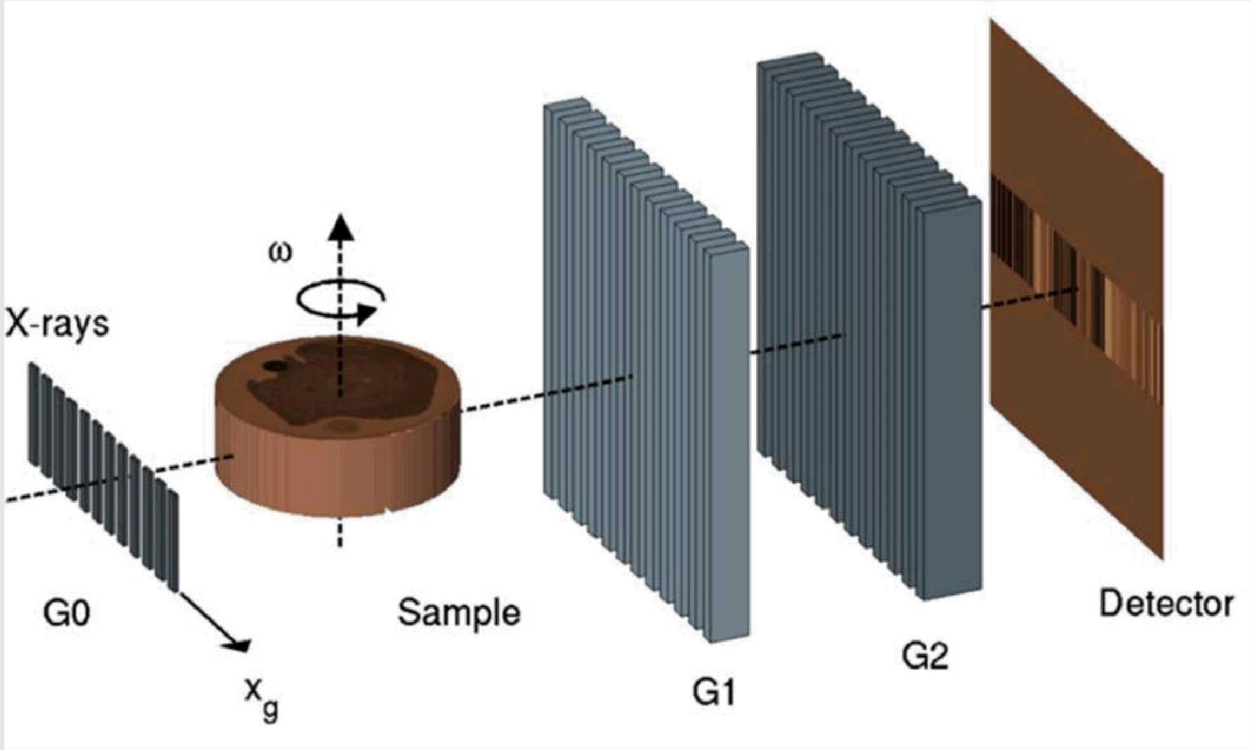
Dark field

Conventional X-Ray Imaging

Many failure mechanisms of low density materials are difficult to detect without destructive post mortem analysis

X-ray Phase Contrast Imaging

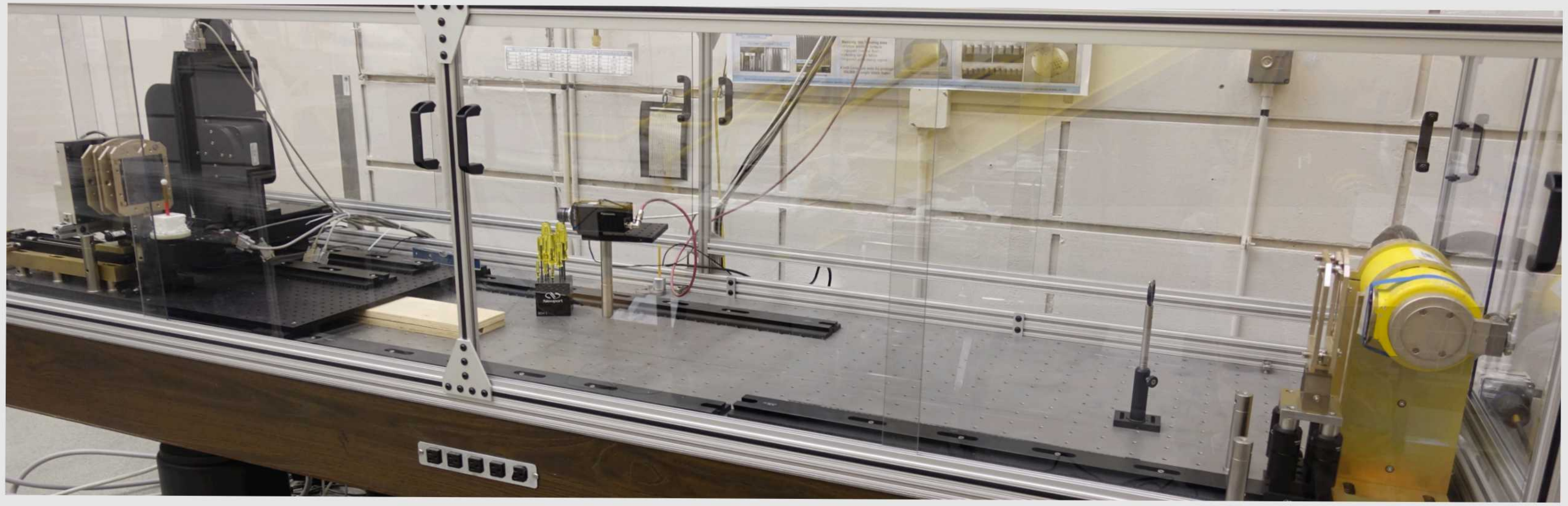
- High contrast in low-density materials
- Talbot-Lau interferometer makes it possible in the lab *without a synchrotron*

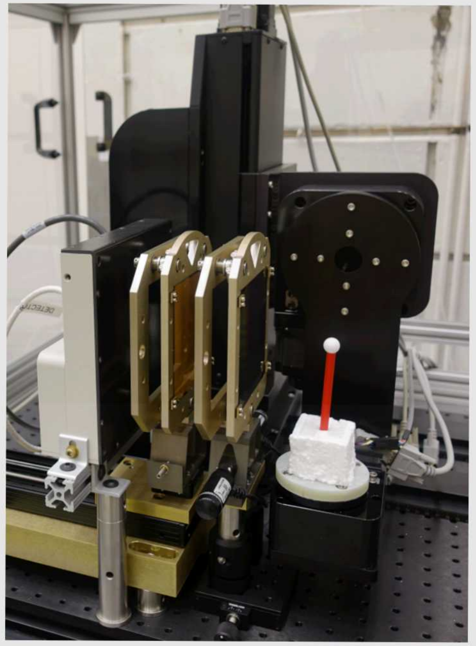


Talbot-Lau Interferometer

M. Nielsen, et. al., Phys Med Biol, **57**, 5971, 2012.

28 keV grating-based XPCI system at Sandia National Labs





G0
87 μm period
18 μm slit 70 μm thick

G1
4 μm period
2 μm slit 40 μm thick

G2
2 μm period
1 μm slit 30 μm thick

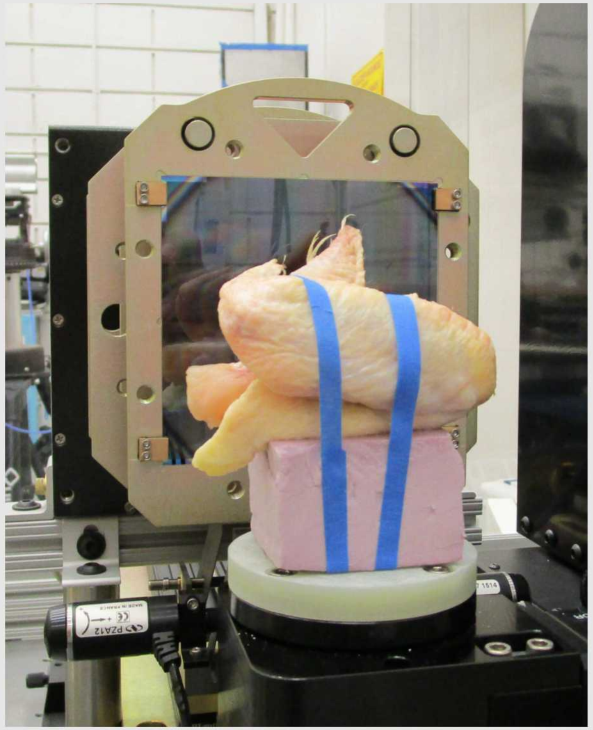
Chicken Wing

Absorption

Phase contrast

Dark field

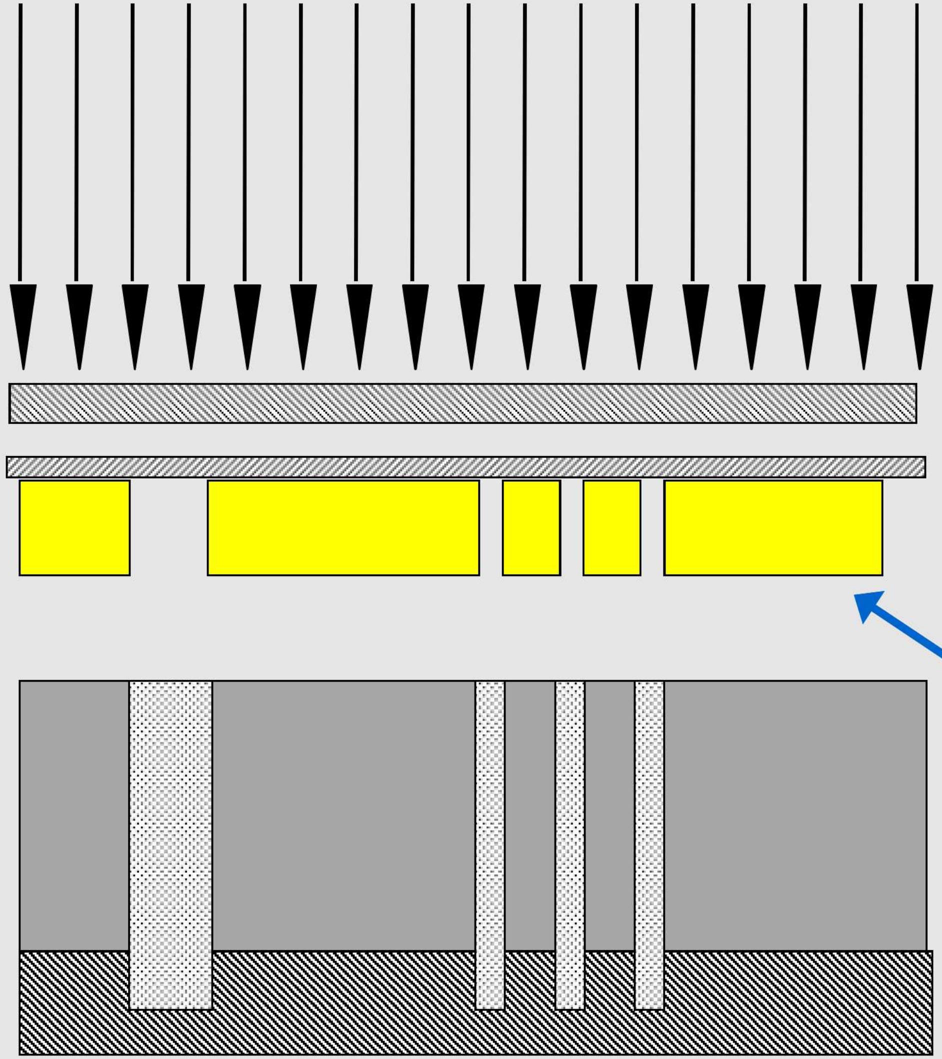
Combined



Evaluation of grating fabrication techniques

- LIGA (***Li**thographie, **G**alvanoformung, **A**bformung*)
  - Samples prepared by x-ray exposure of thick photoresist
  - Lose structural integrity and fall onto one another as aspect ratio increases
- Anisotropic wet etching silicon
  - Alignment to crystalline planes is difficult
- Bosch etching
  - Limited depth due to ion scattering

"LIGA": LIthographie, Galvanoformung, Abformung
(Lithography) (Electroplating) (Molding)



x-rays

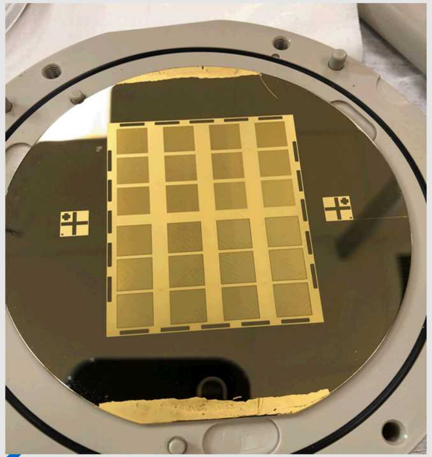
filter(s)

X-Ray MASK
Substrate

Gold Absorber

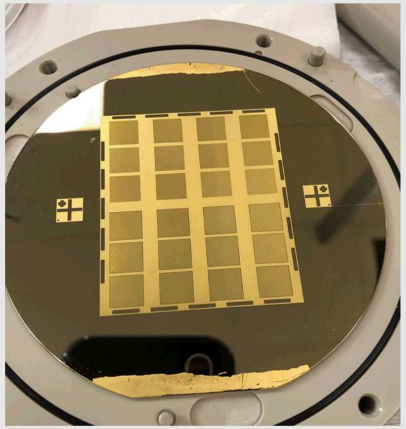
Metallized
Substrate

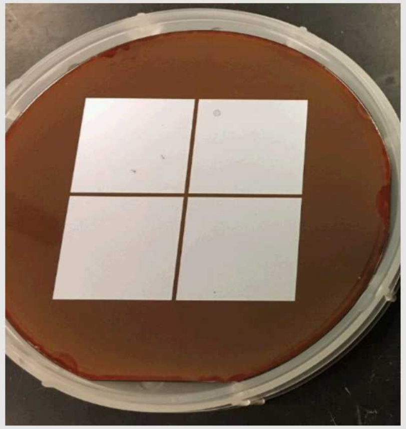
PMMA

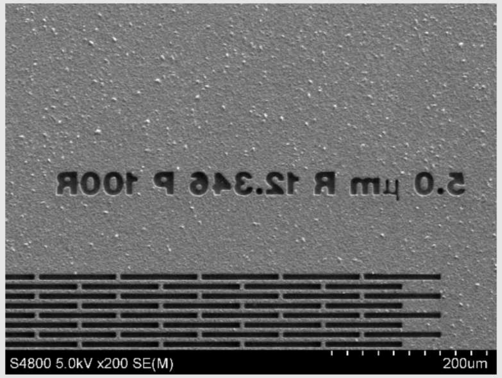


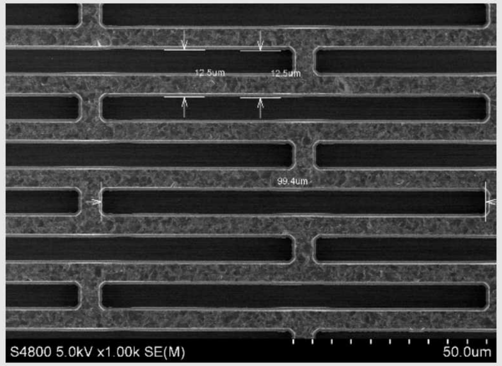
LIGA X-ray mask fabrication

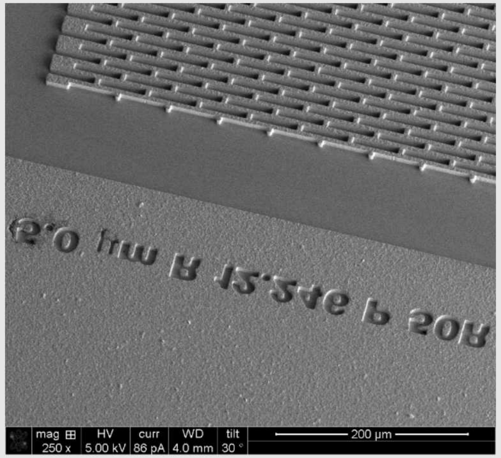
- Microfabricated x-ray mask patterned and gold plated
- Backside etched nitride to reveal 1 micron thin SiNi membrane for x-rays to pass through

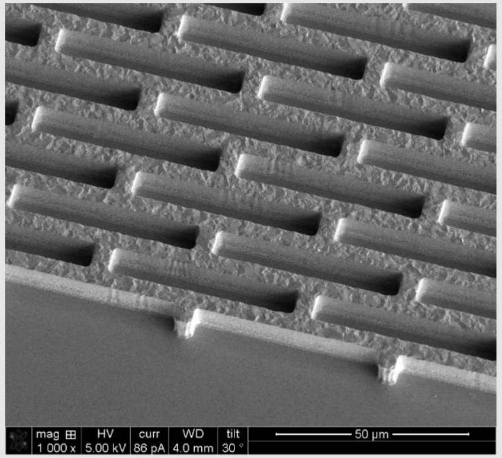








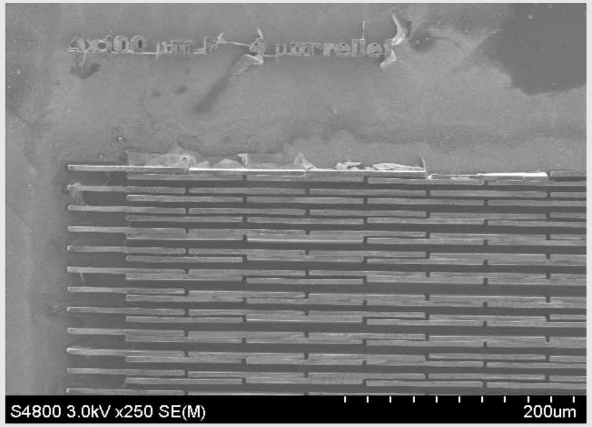


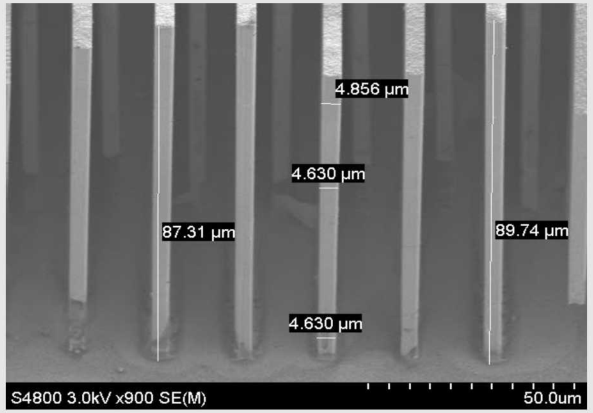


Nickel and gold plated XPCI LIGA features

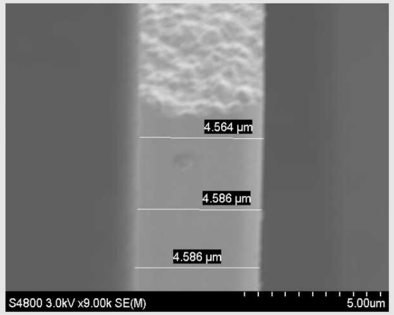
~87 μm nickel height, 4 μm features

~4.0 micron features

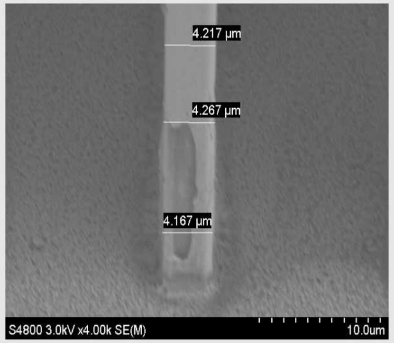




~4.586 micron feature at top

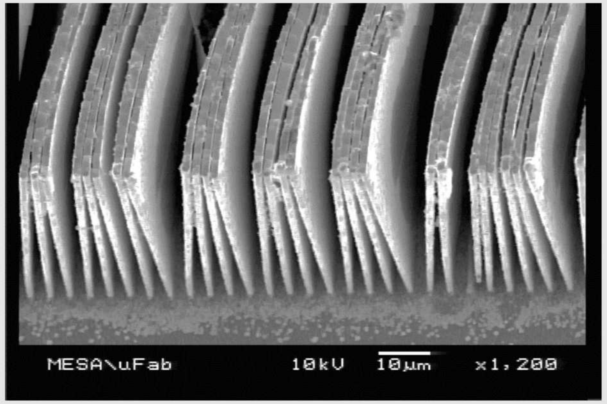


~4.267 micron feature at bottom

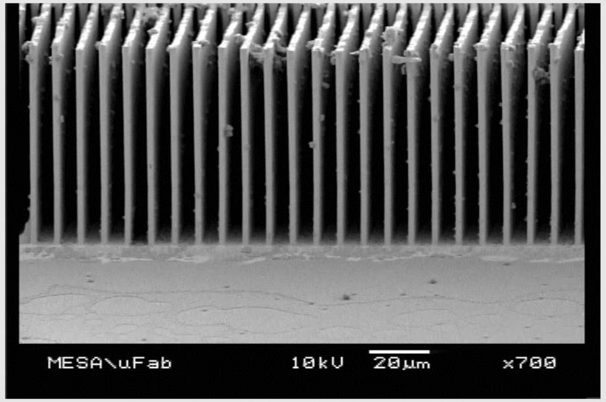


~ 73 μm gold height, 1.2 and 2 μm features

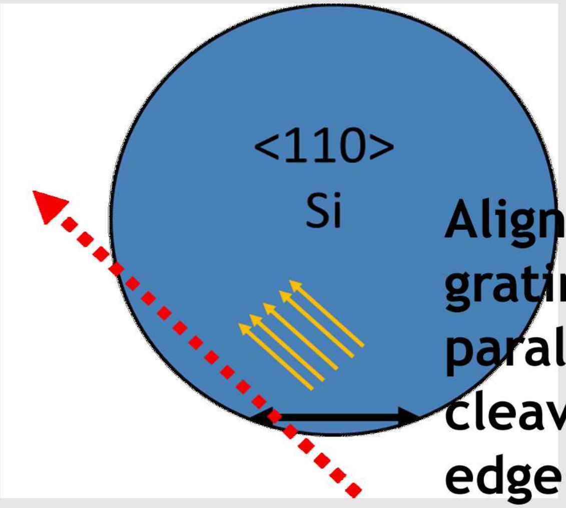
~1.25 micron features



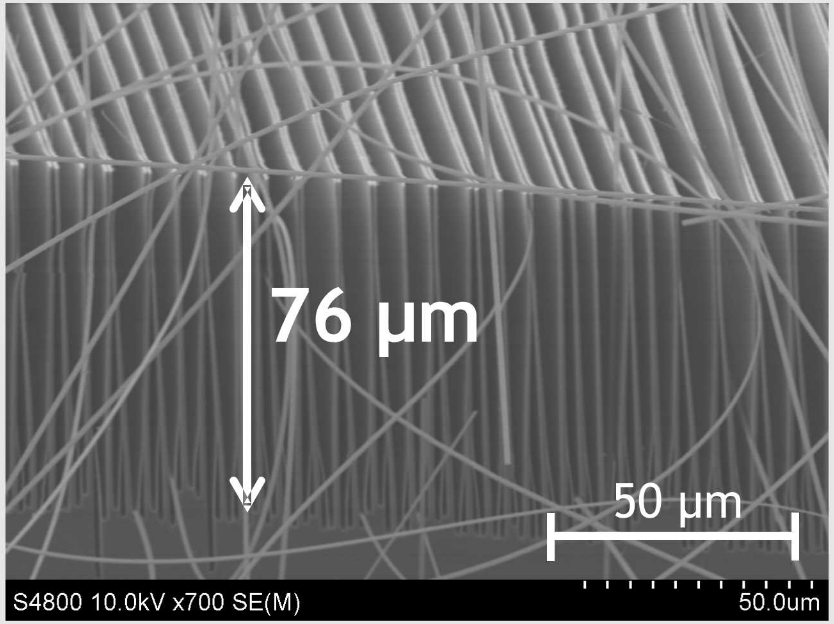
~2 micron features

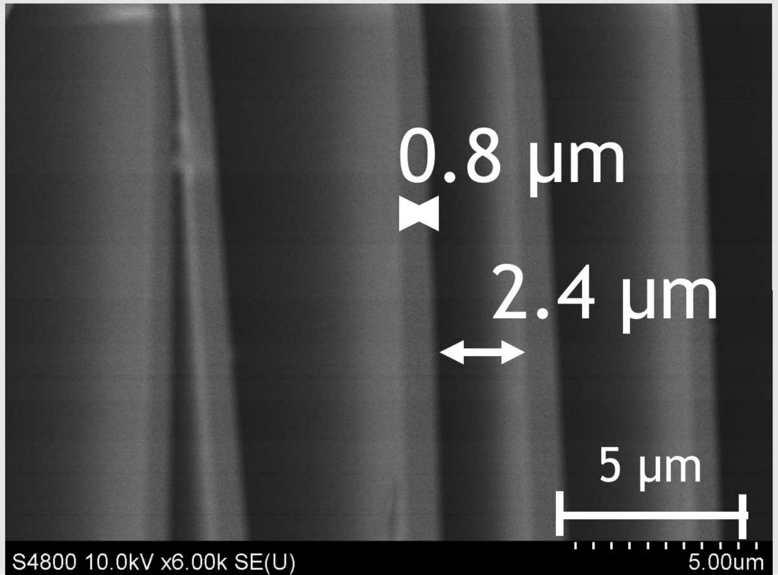


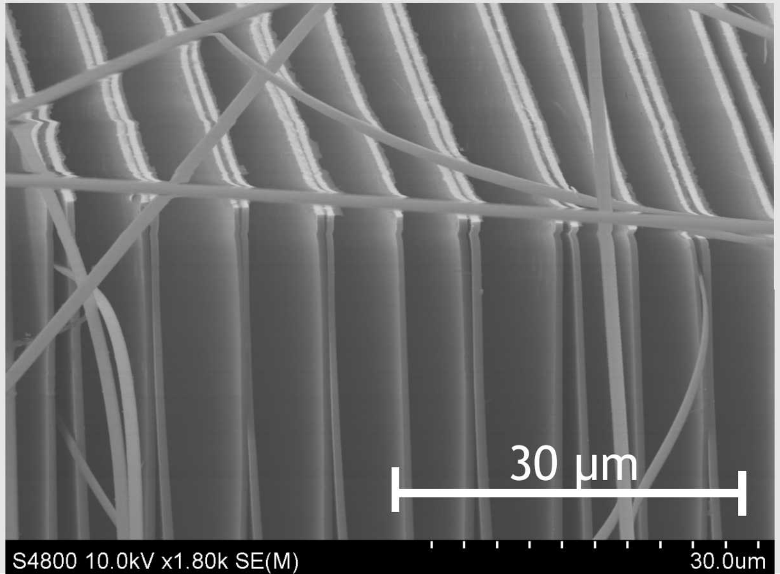
Simple alignment technique for <111> crystalline alignment



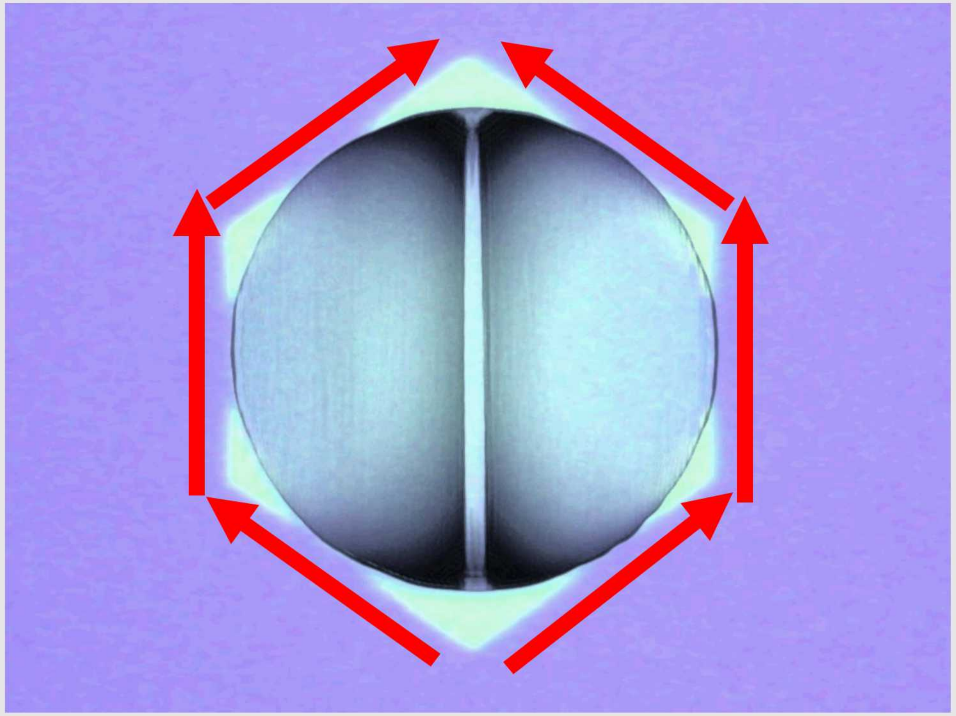
Cleave wafer 35.3° to flat





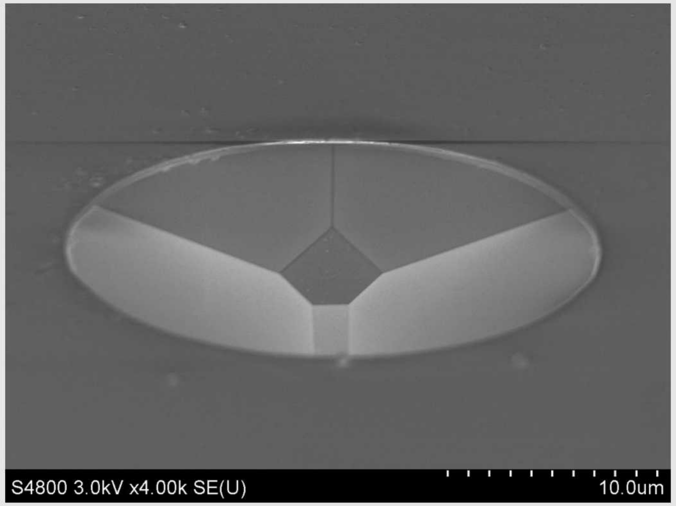


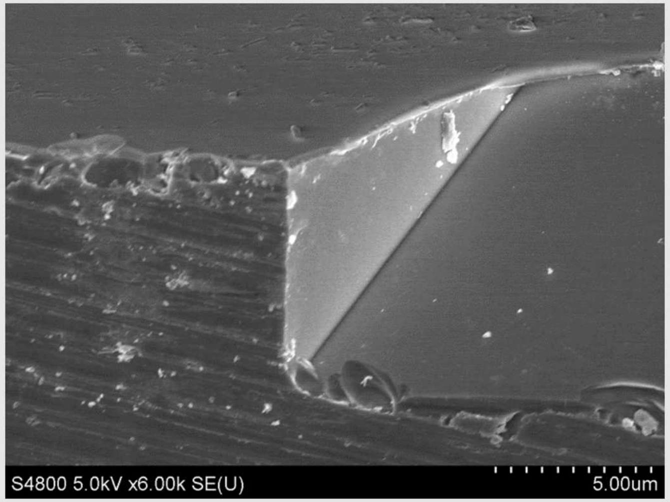
Anisotropic wet etching silicon in potassium hydroxide (KOH) Hexahedron sidewall profiles in (110) silicon

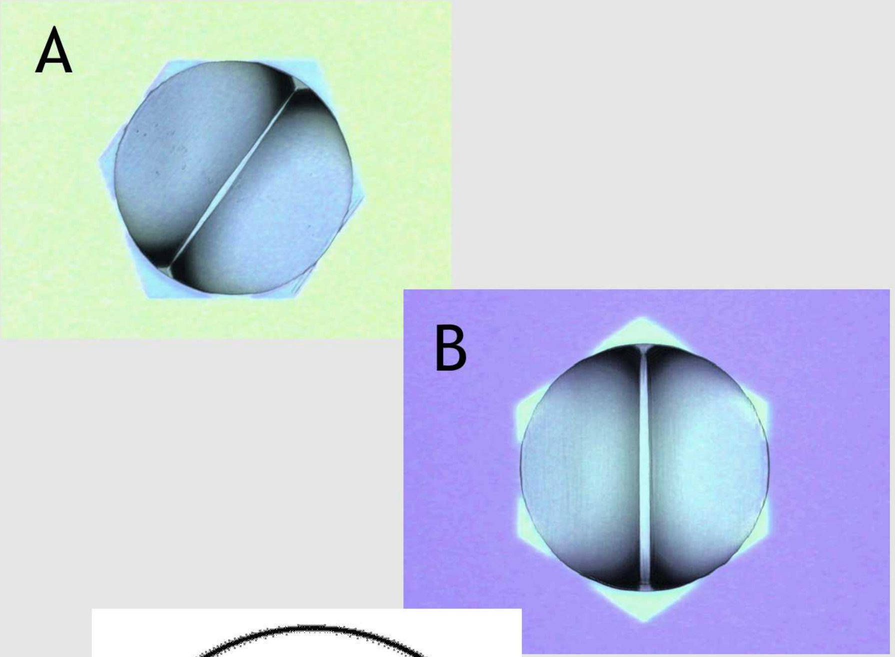


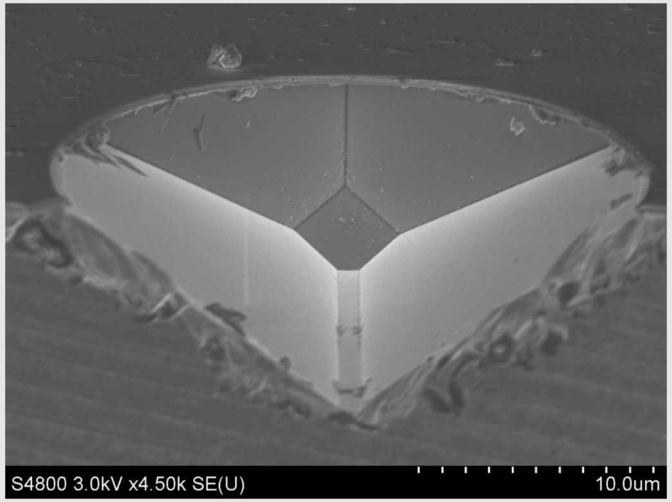
A hexahedron is formed by KOH etching a cavity through a round hole on (110) Si wafer

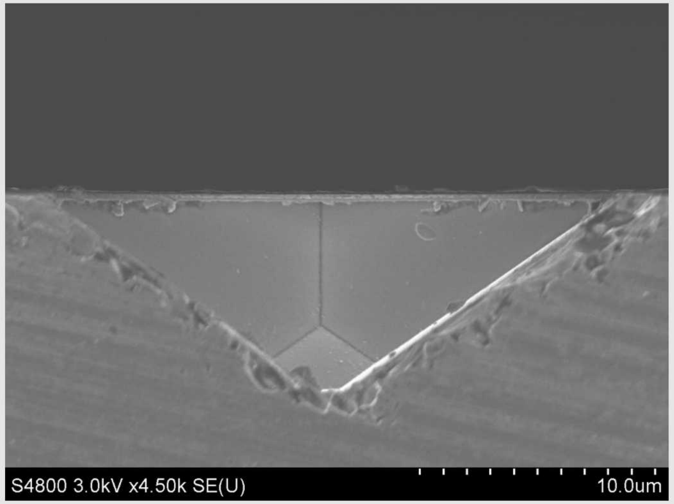
Anisotropic wet etching silicon in potassium hydroxide (KOH) Determining crystalline orientation

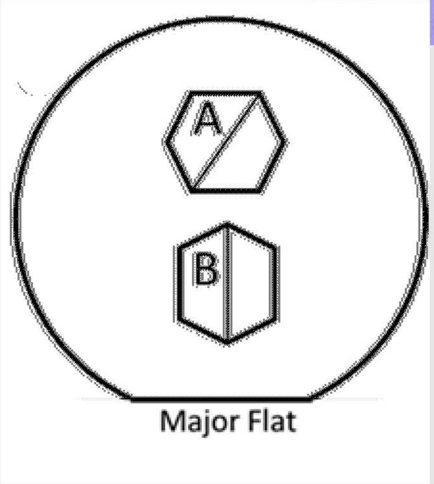




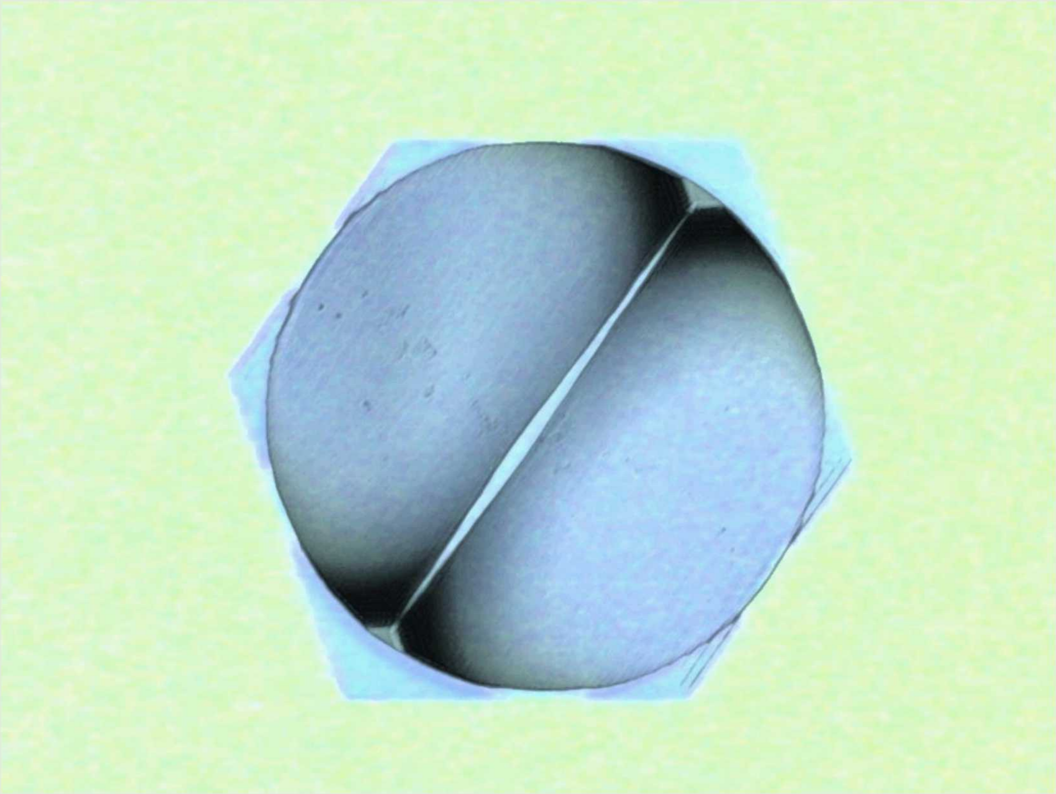


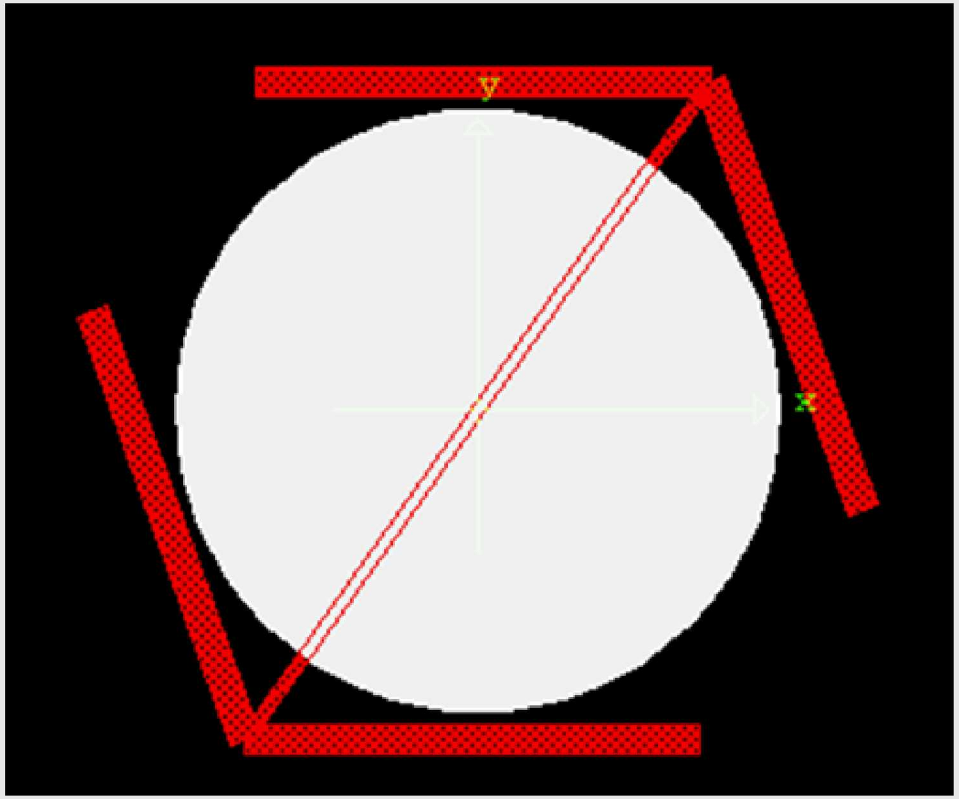




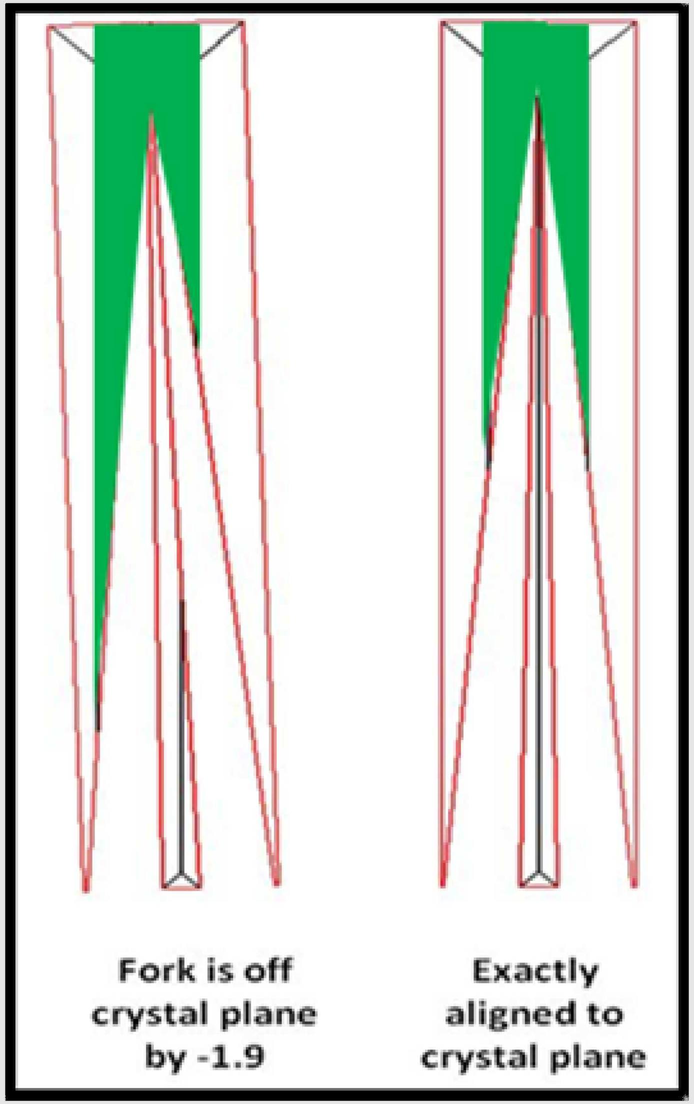


Hexahedron alignment

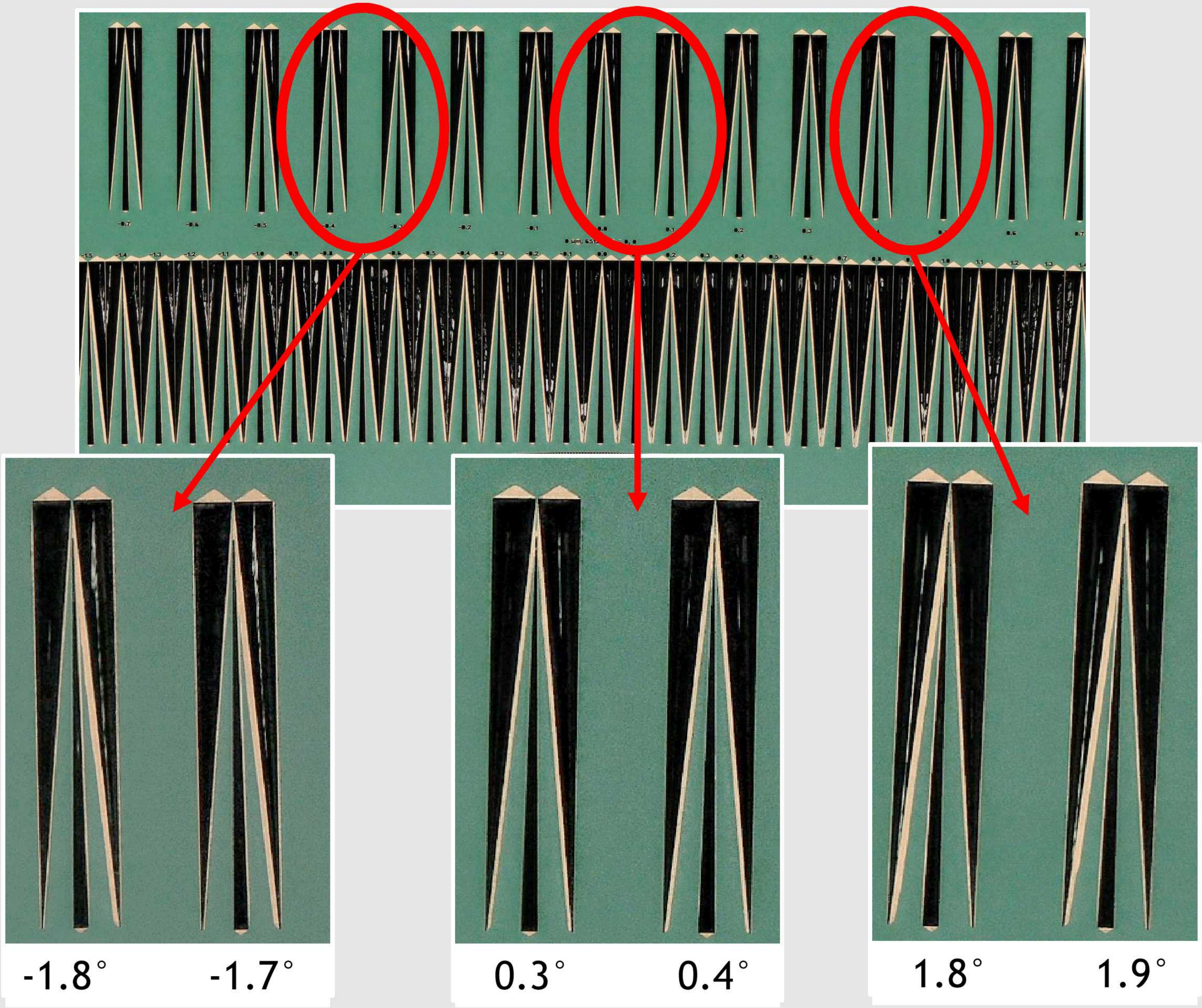




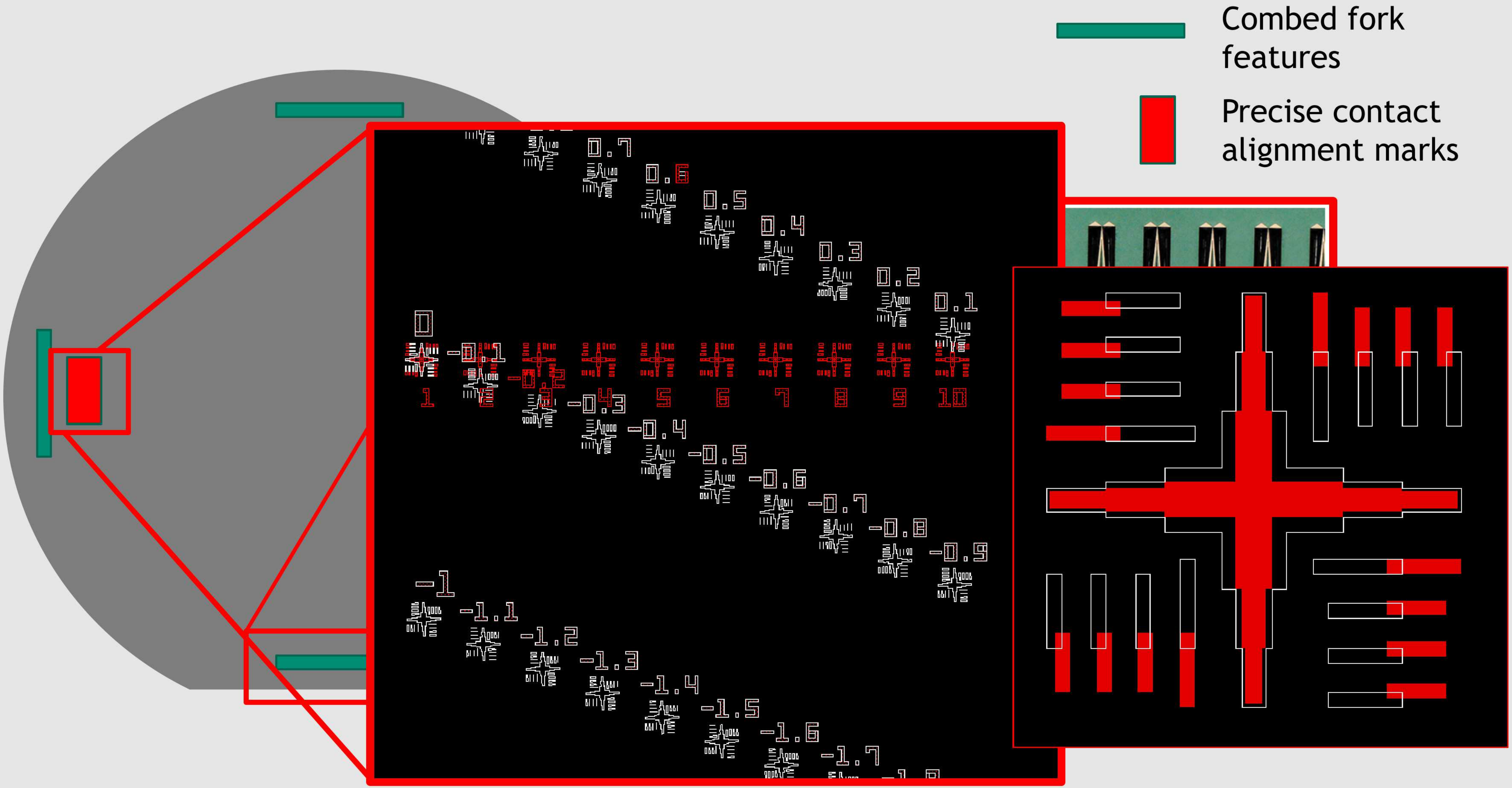
Determining accurate crystallographic alignment using combed fork



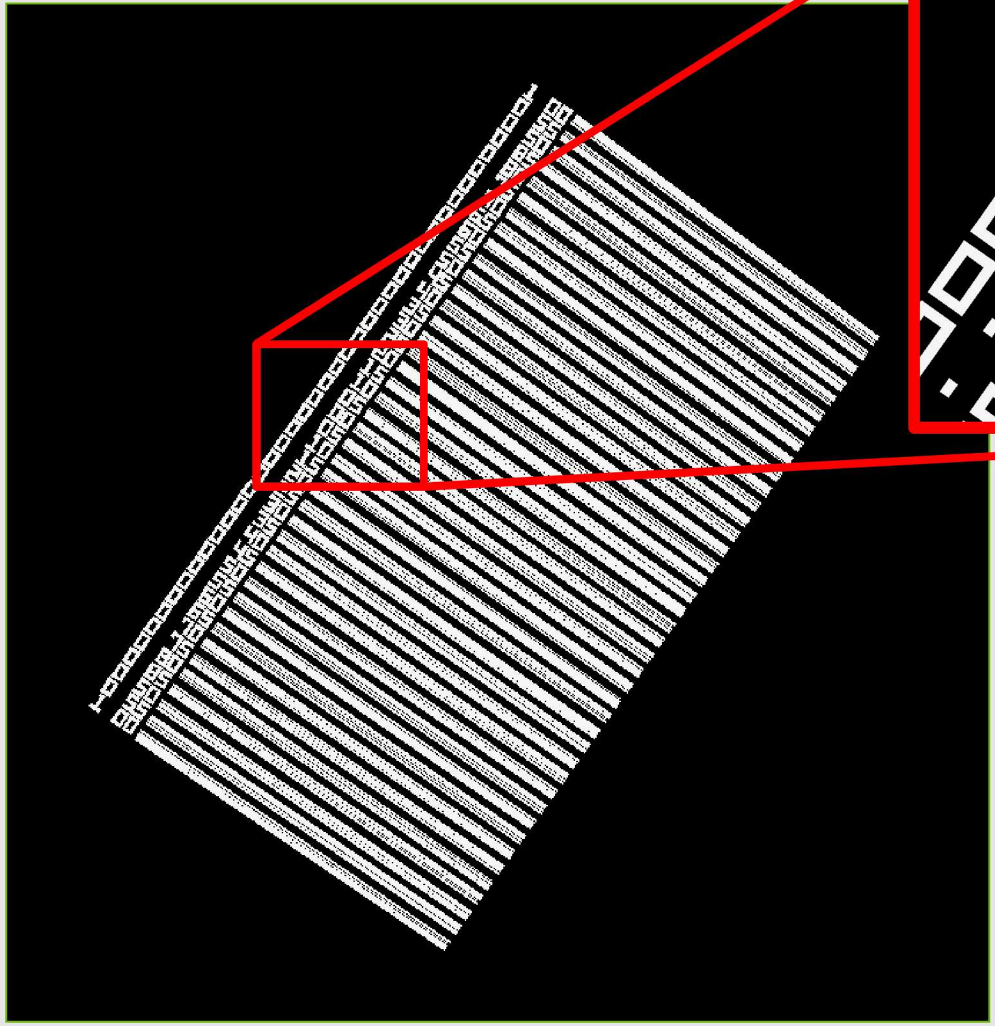
M. Vangbo et. al. J. Micromech. Microeng. 6 (1996) 279-284

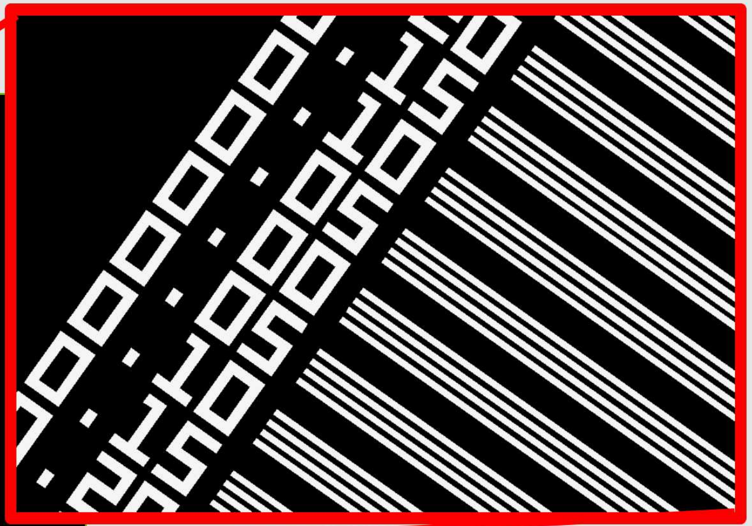


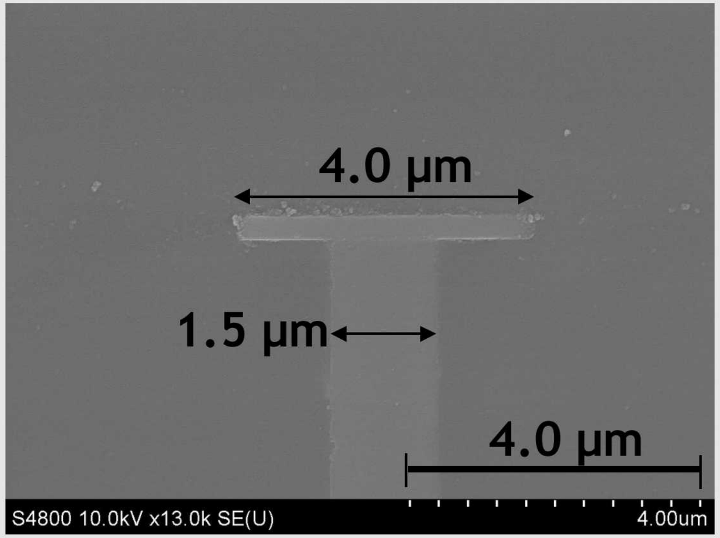
New path forward for accurate crystallographic alignmnet

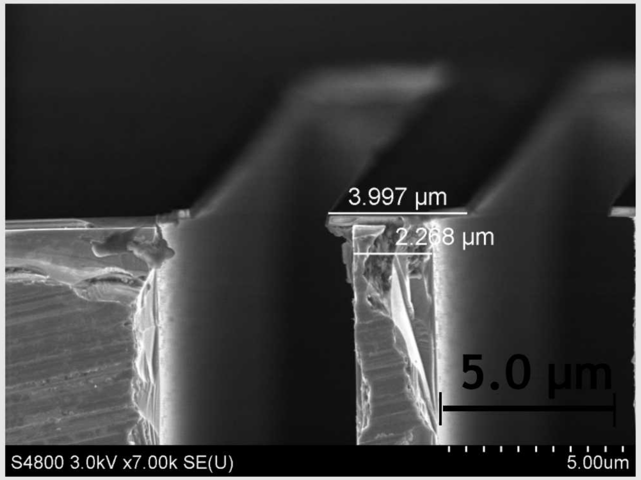


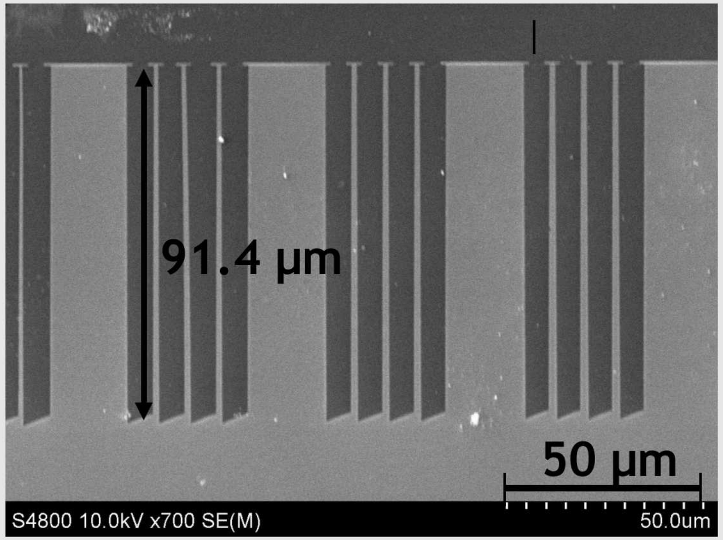
Small gratings each arrayed at 0.05°



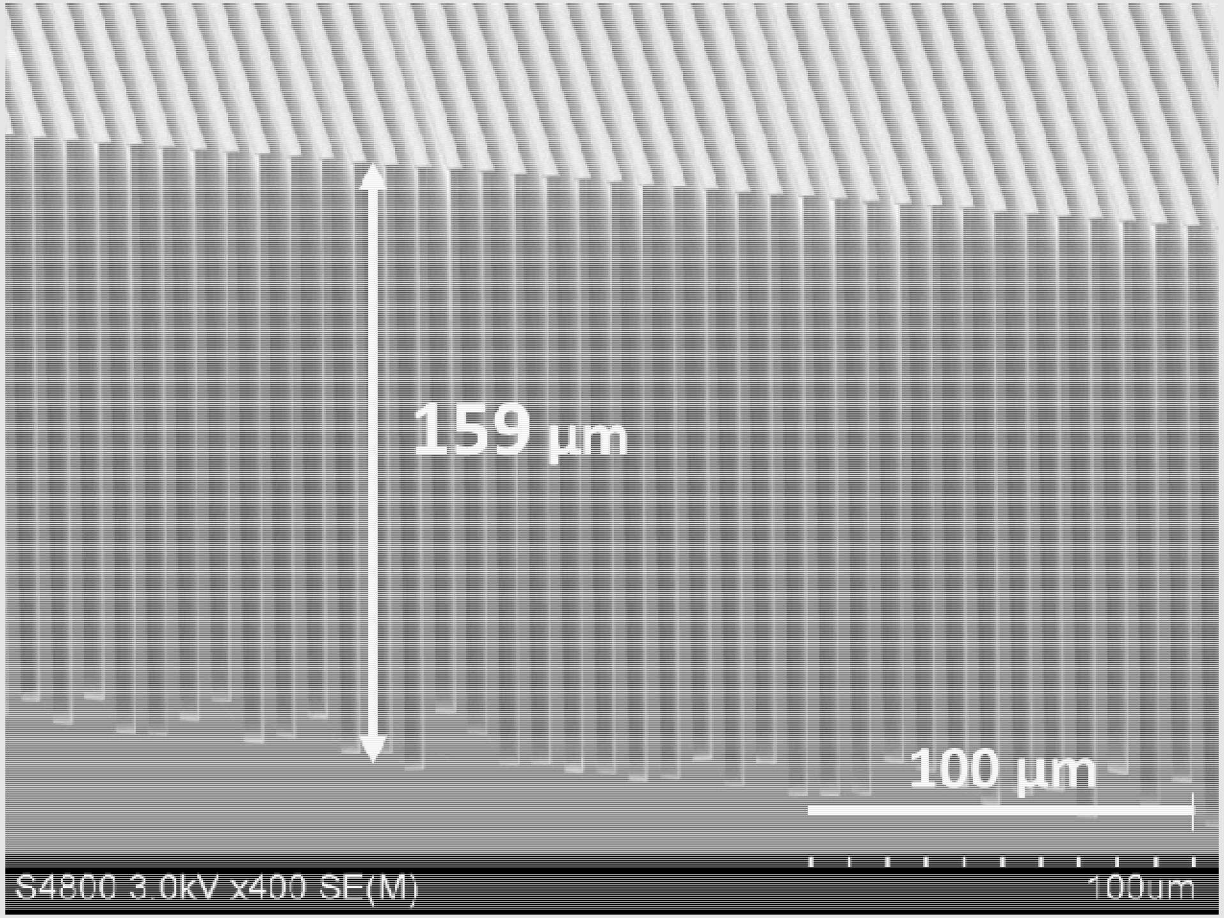


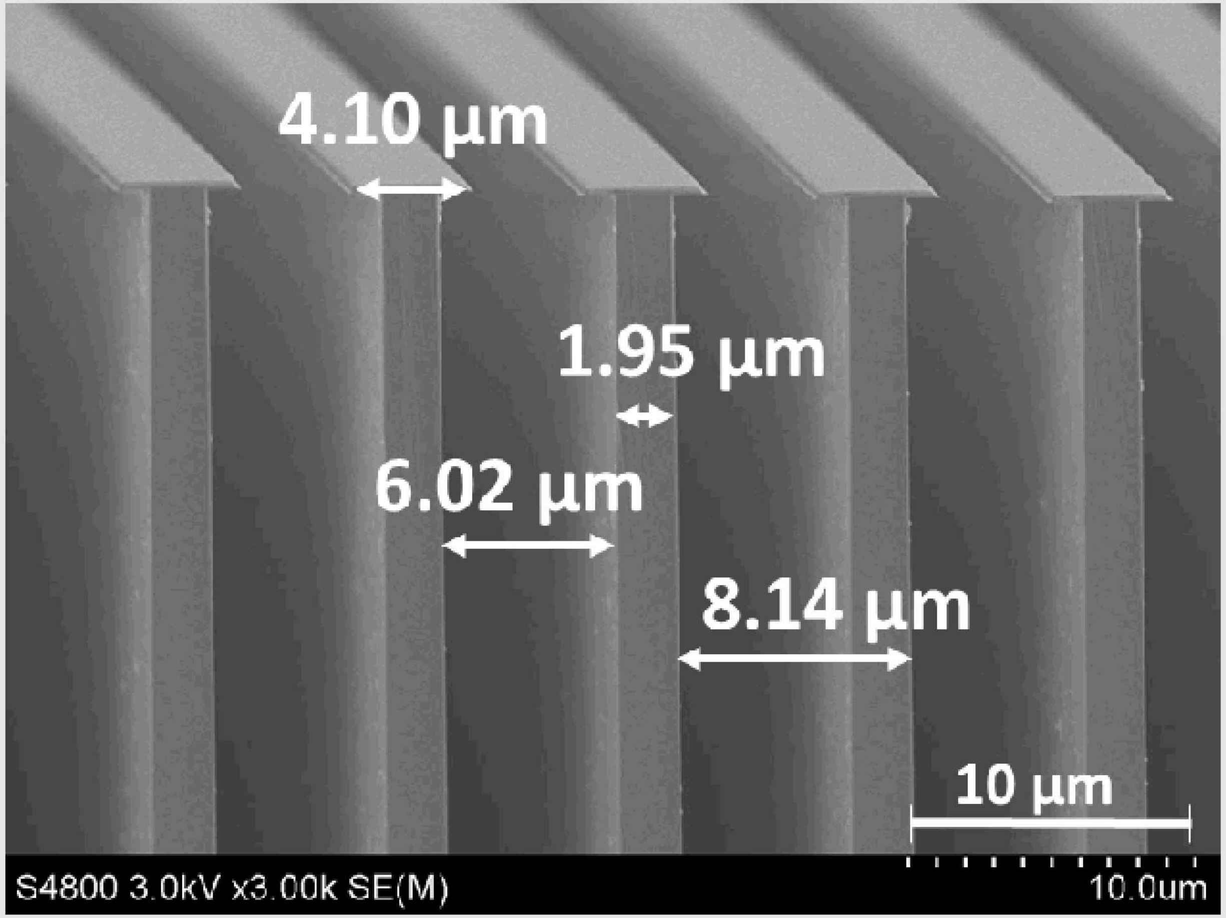






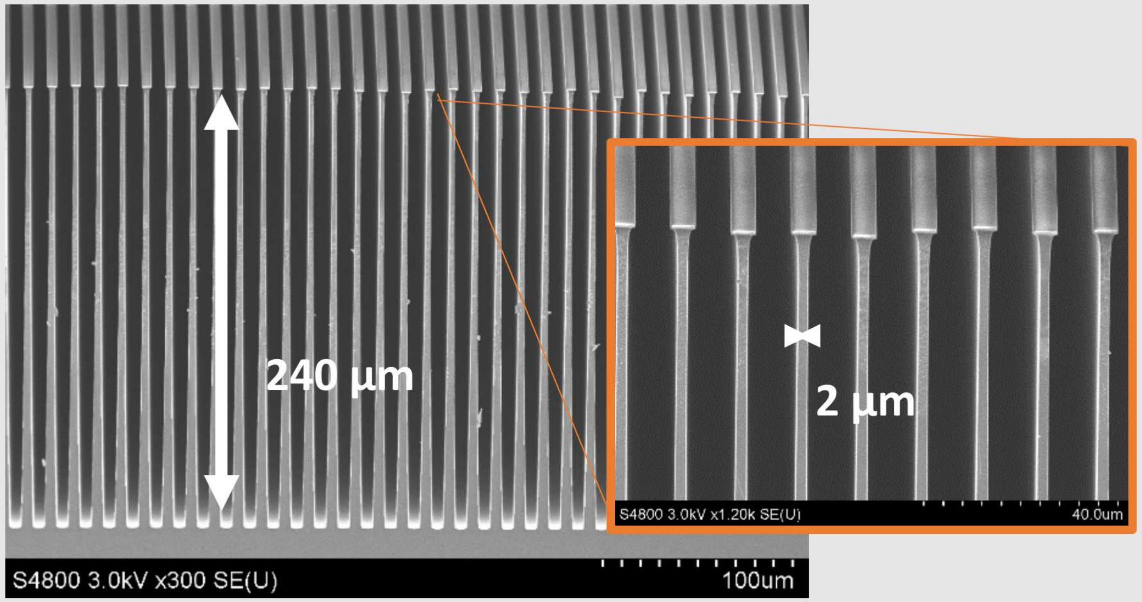
80:1 aspect ratio Anisotropic KOH etch with accurate alignment





Lateral etch rate of the <111> Si plane cpompared to the <110> was approximately 160:1

Bosch etched gratings using SF_6 and C_4F_8

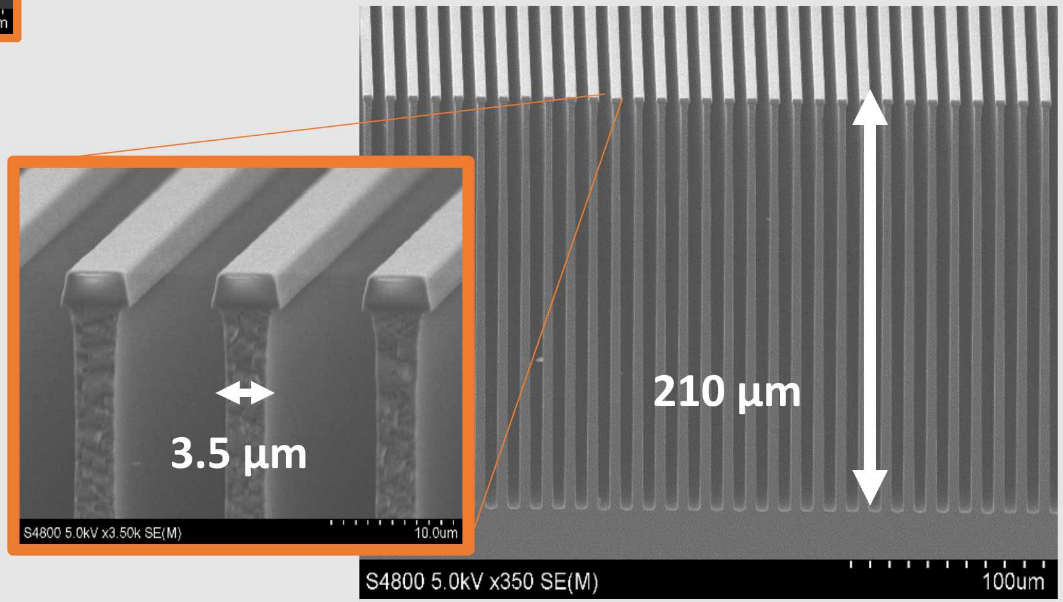


Dep - 1.8 sec, 150sccm C4F8, 10V bias
Etch A - 2.1 sec, 75sccm SF6, 600V bias
Etch B - 2.0 sec, 75sccm SF6, 10V bias

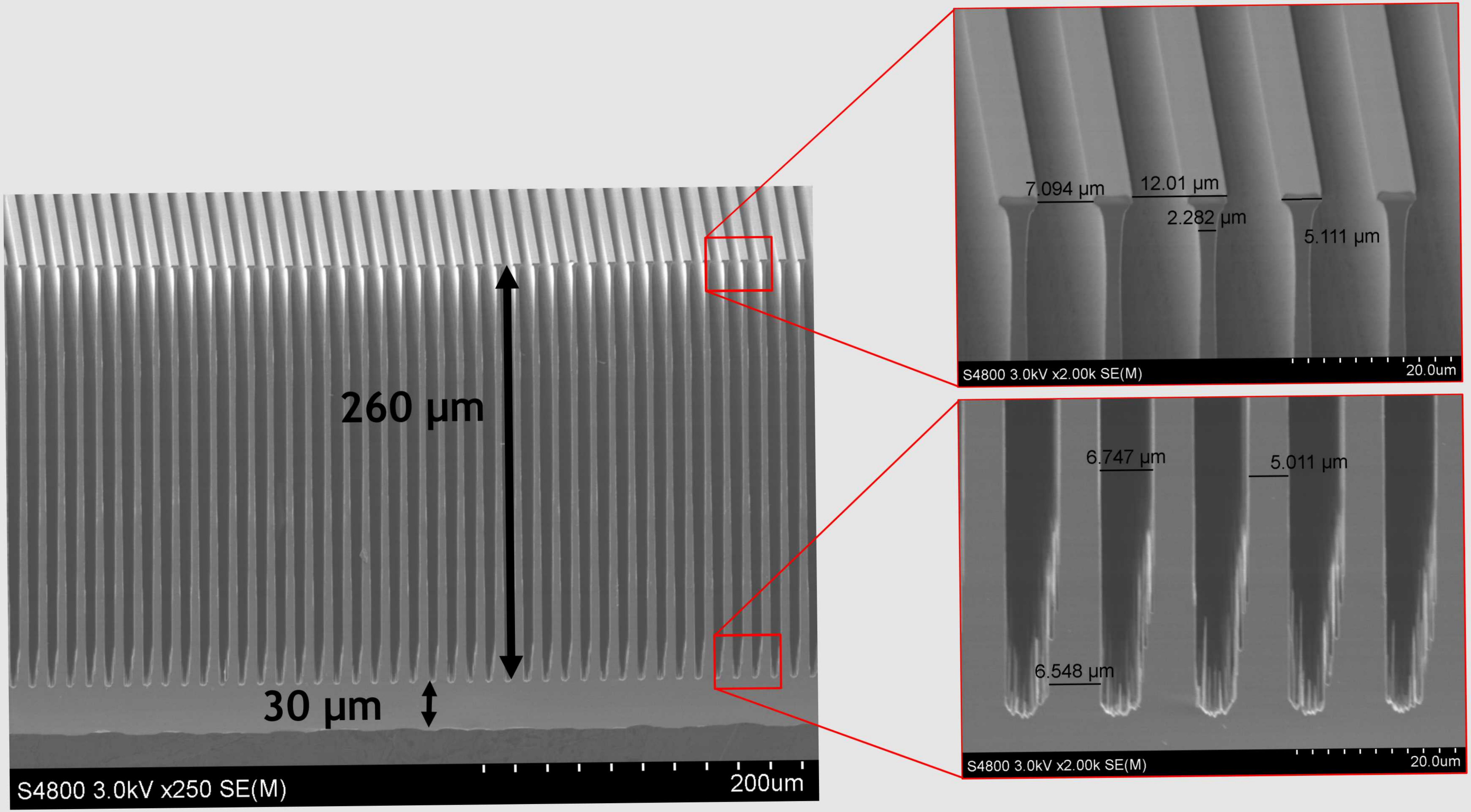
By increasing the C_4F_8 dep time and the SF_6 etch cycle bias and gas flow, uniformity improved

Bosch etches use alternating gasses SF_6 is the etchant phase and C_4F_8 passivates sidewalls, by depositing a polymer, to achieve straight vertical etches

Dep - 4.0 sec, 150sccm C4F8, 10V bias
Etch A - 2.1 sec, 250sccm SF6, 700V bias
Etch B - 2.0 sec, 450sccm SF6, 10V bias

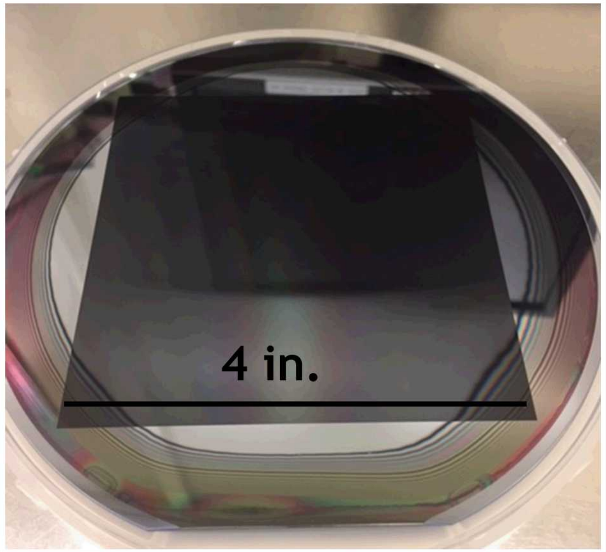


Bosch etch depth limitations

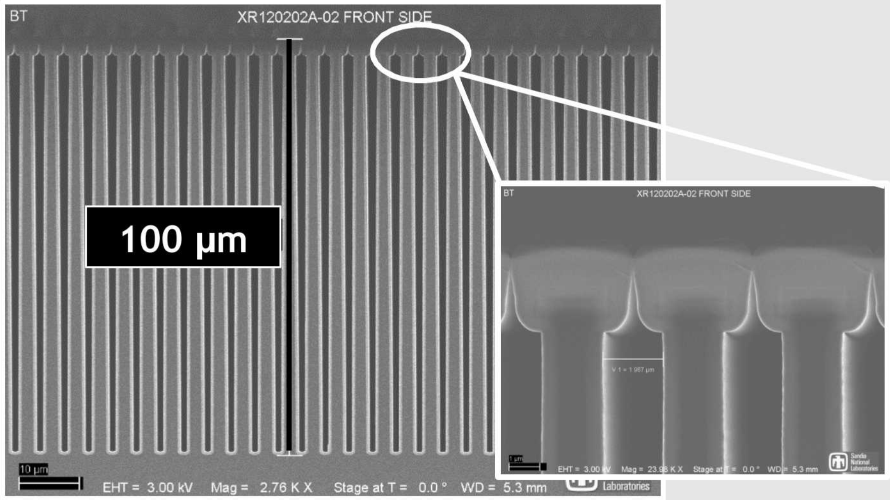


Front to Backside Aligned Gratings

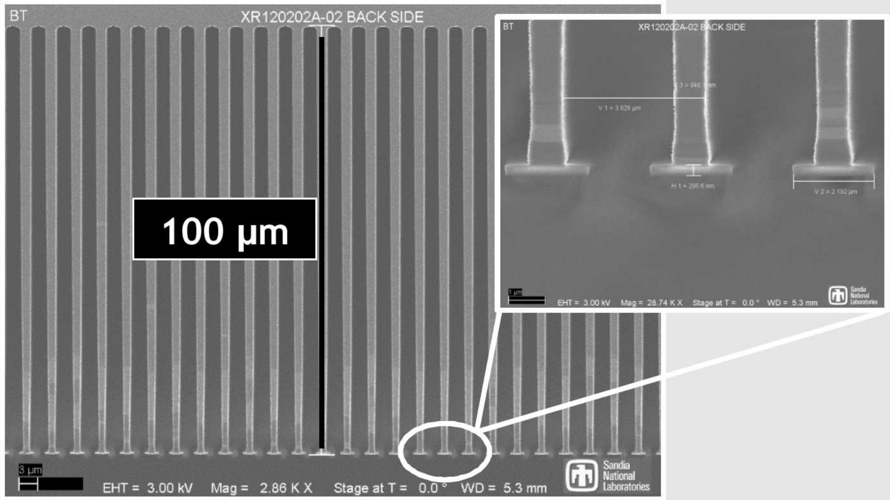
Large Fields of View Possible



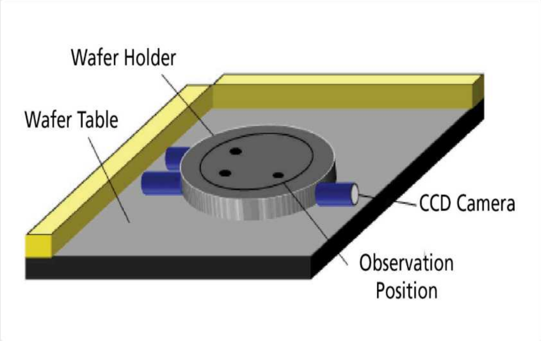
Oxide Coated Front Side Gratings

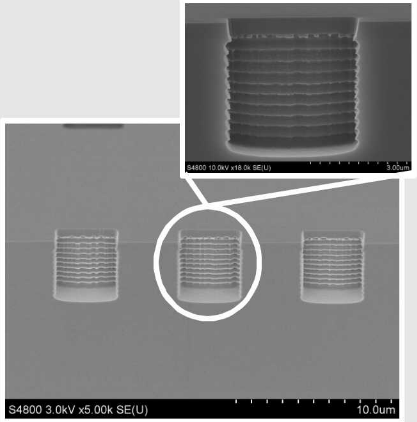


Backside Gratings

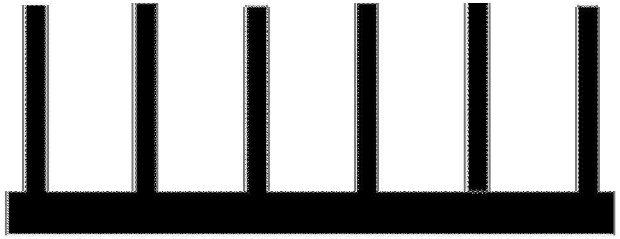


Nikon Backside Field Image Alignment (BS-FIA)

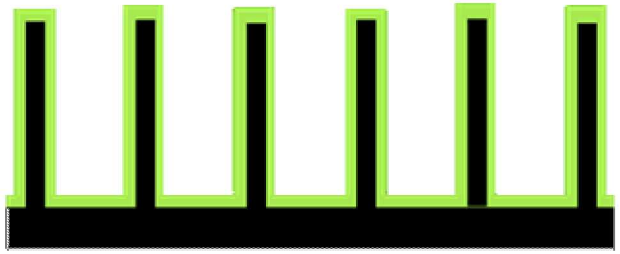




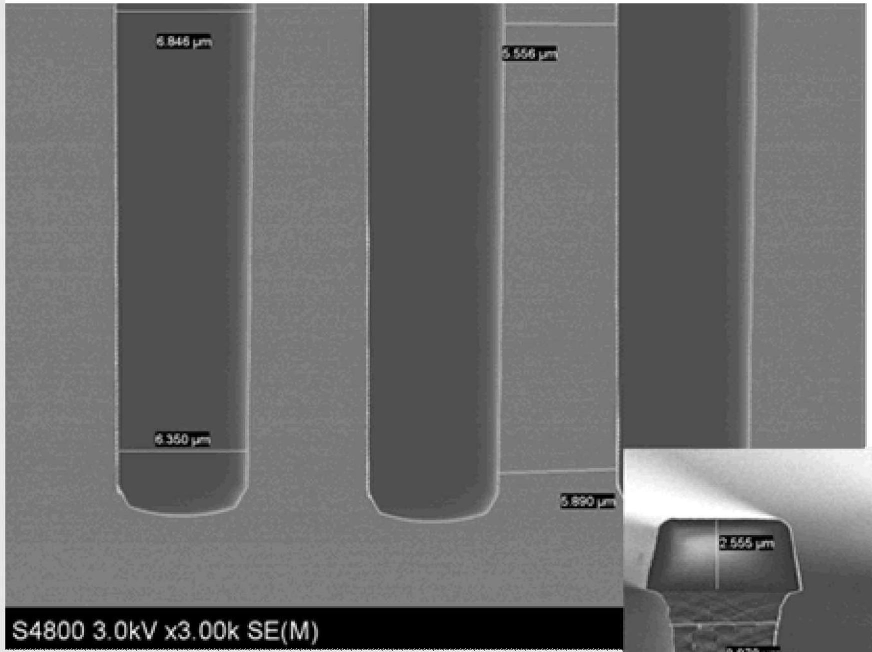
Platinum Atomic Layer Deposition (ALD) conformal coating



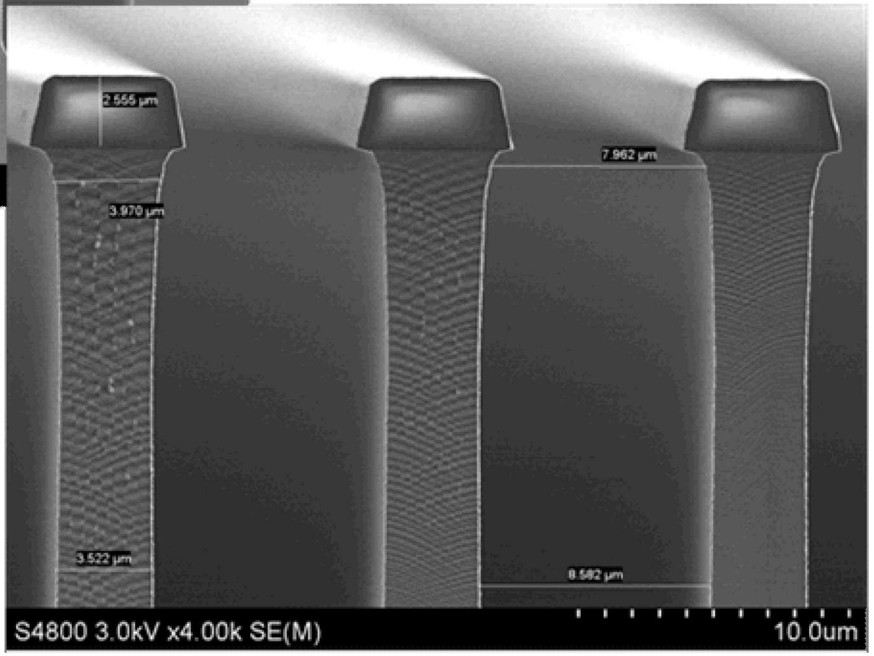
Etched grating in Si



ALD Pt conformal deposition



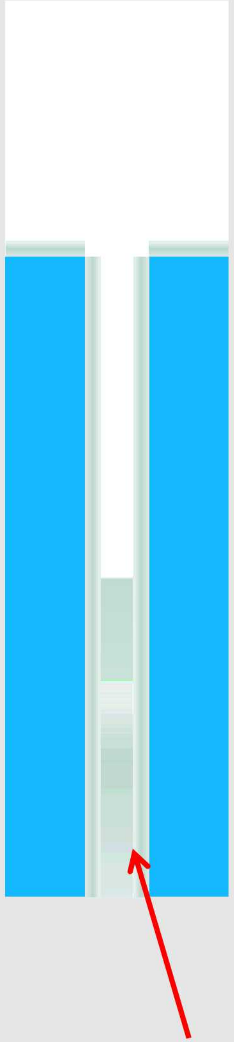
SEM images of ALD Pt conformally coating 240 µm deep HAR gratings



Using ALD Pt provides many different options for filling HAR diffraction gratings

Challenges in Precision Electro-coating

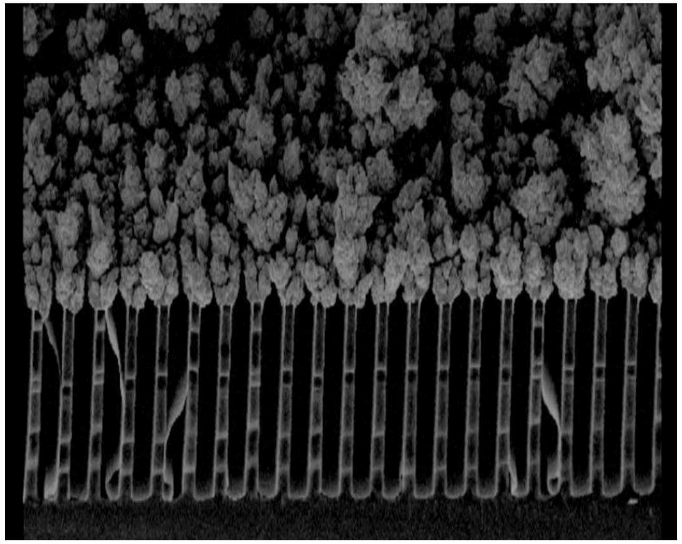
Max (bulk) Ion Concentration

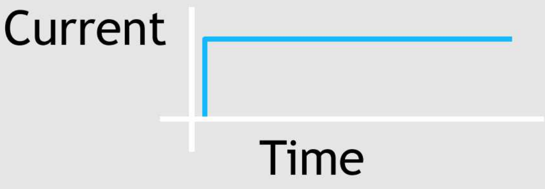


Severely depleted region

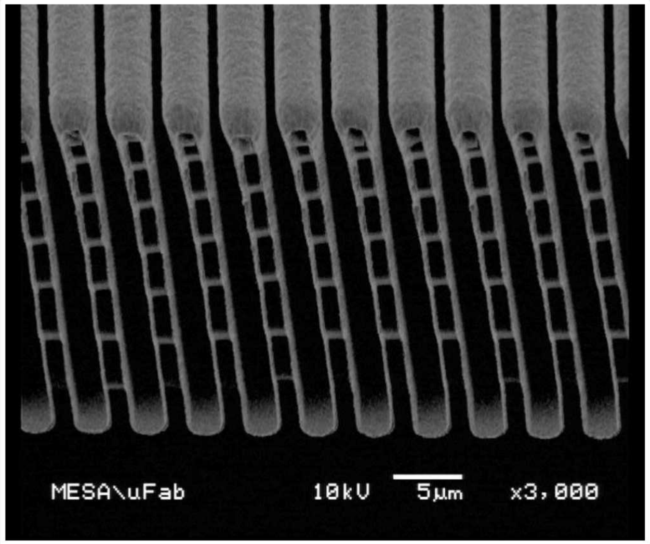
Modifications to Improve Plating
- Incorporate more time for ionic replenishment
- Equilibrate concentrations around plating surfaces
- Increase period of time between deposition pulses

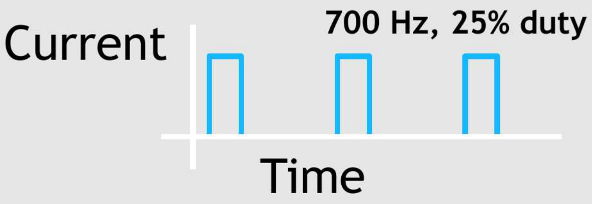
DC Plating





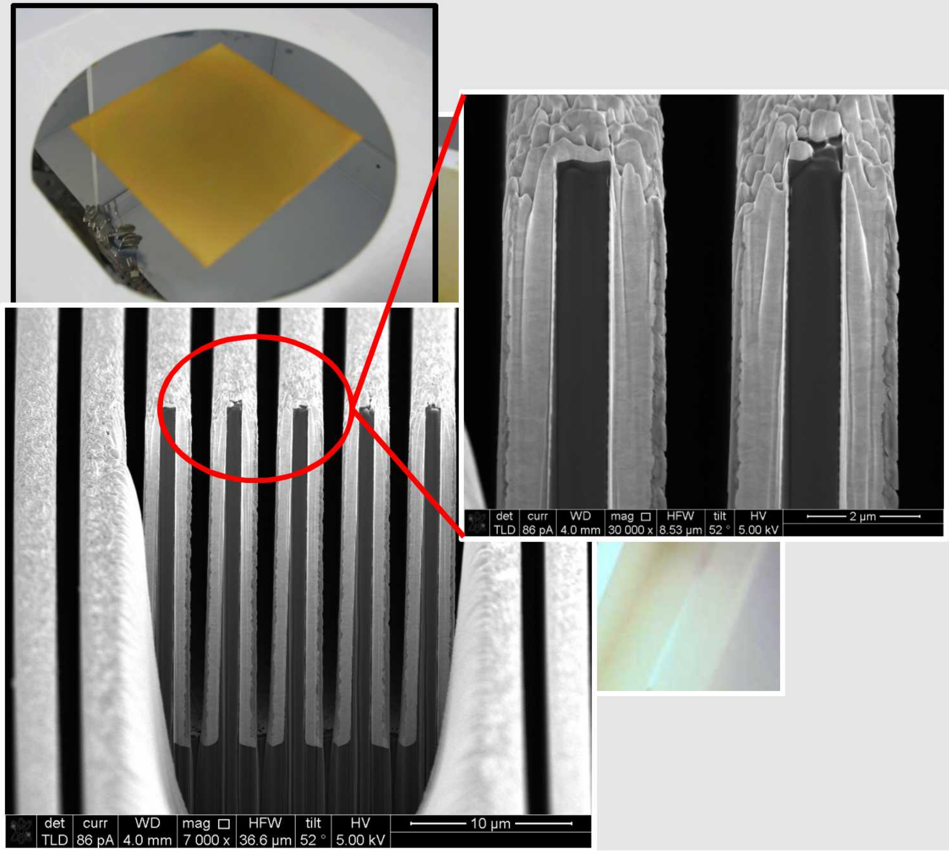
AC Plating

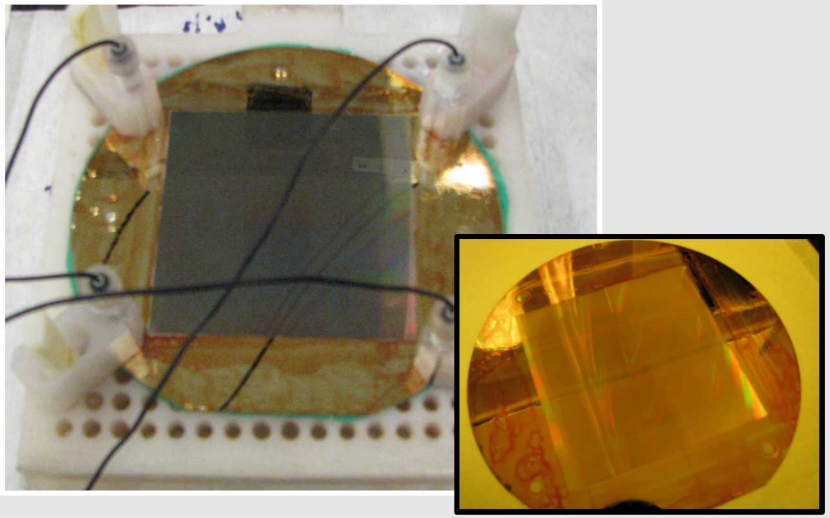


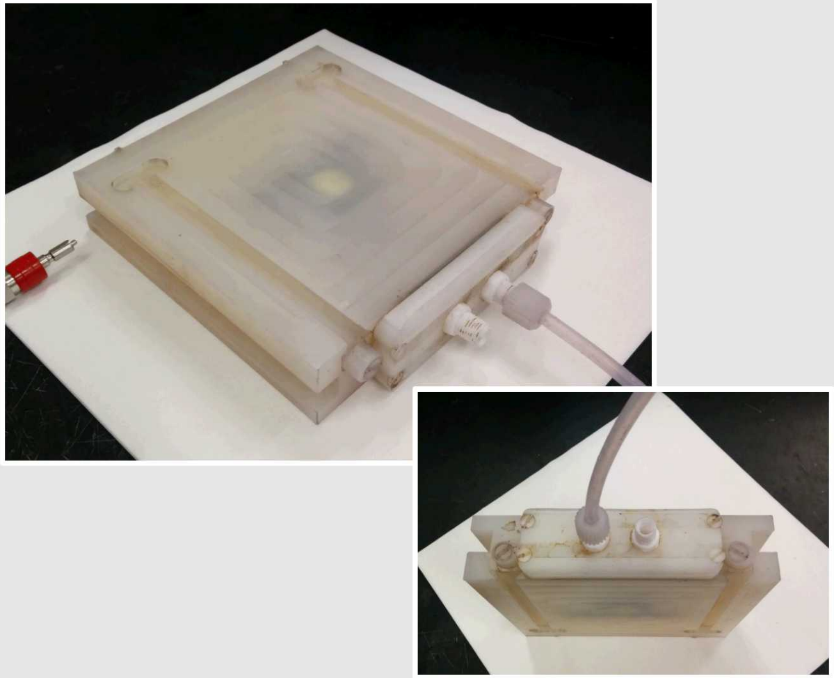


Chemistry Flow and Fixture Modifications to Realize 16in.^{2} Uniformity

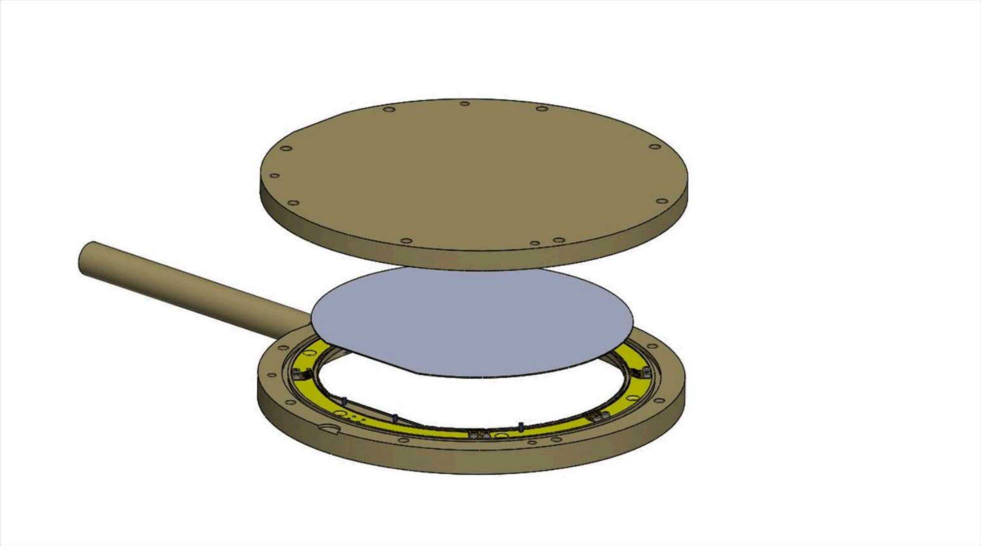
- Chemistry flow dictates ionic replenishment
- Multiple contact points evenly distributes conductivity
- Vibrating fixture enables even solution replenishment and ionic distribution

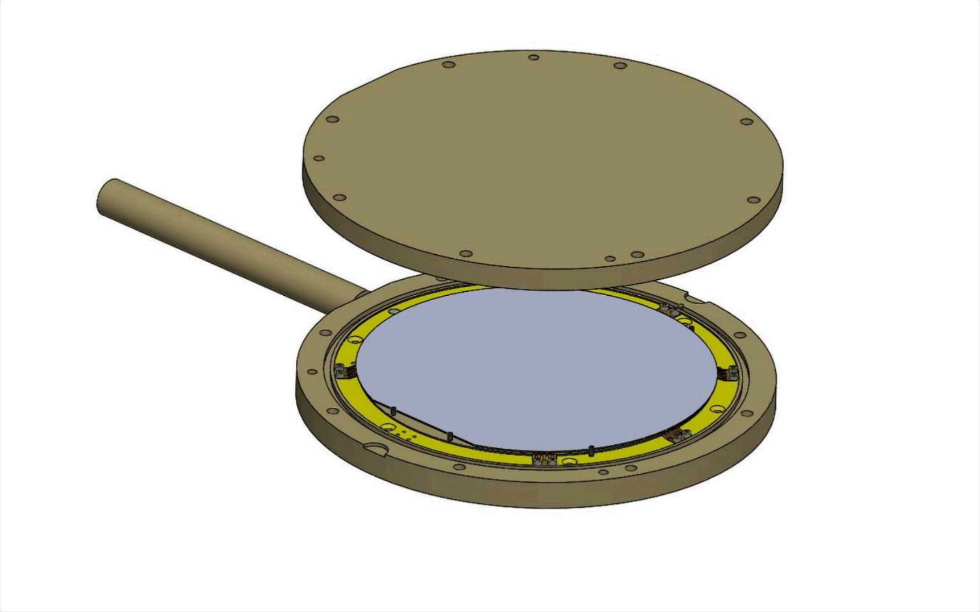






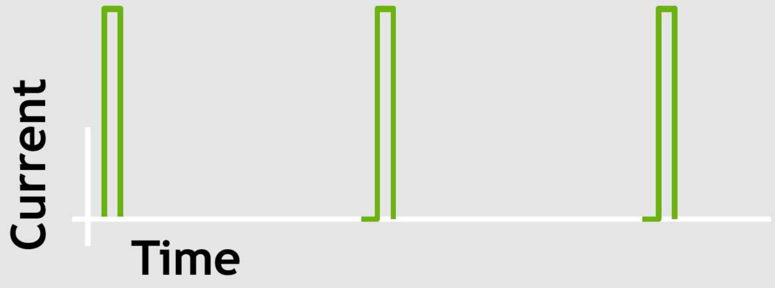
Chemistry Flow and Fixture Modifications to Realize 16in.^{2} Uniformity



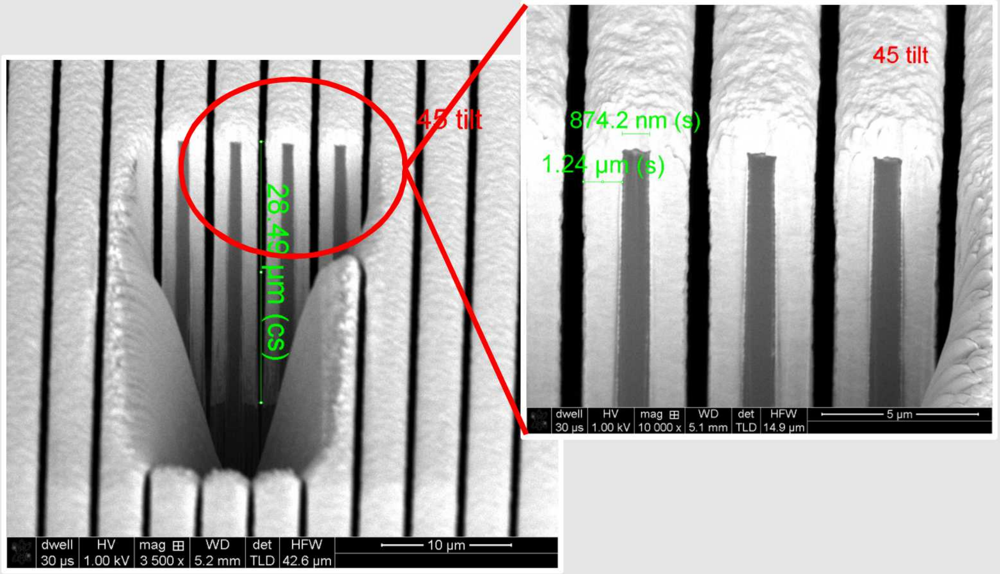


Optimized Pulse Plating Conditions

100 Hz, 3% duty

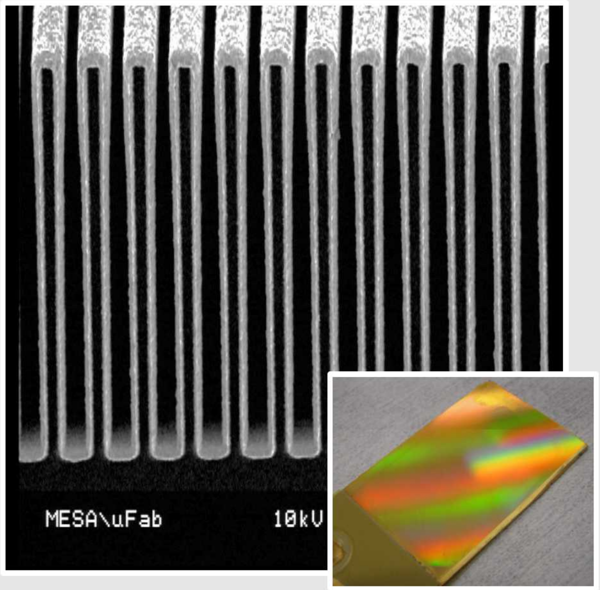


Drastically reduced kinetic and plating rate



28 µm deep Si template

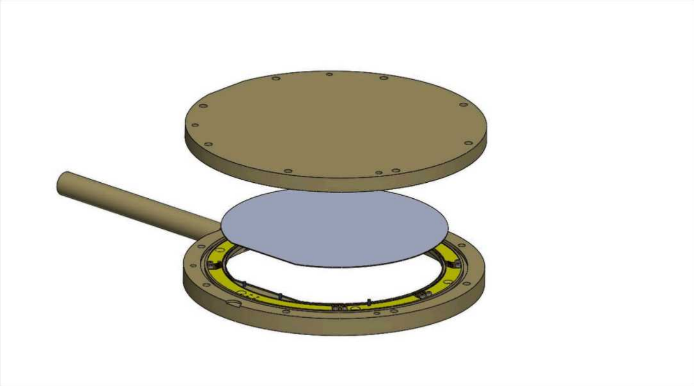
50:1 aspect ratio gratings



- Uniform plating in 28 µm deep Si templates
- Near conformal deposition in 50 µm tall Si gratings have been plated
- Uniformity can be tailored to desired aspect ratio

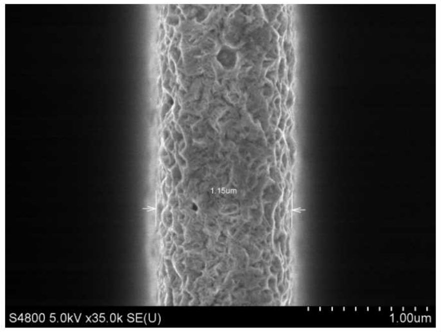
G2 - Au Plating Development

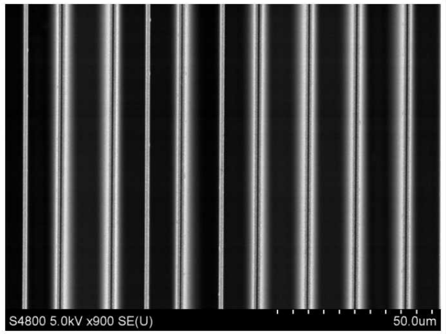
Custom Plating Fixture



- Enables plating of one side of the wafer at a time
- Control of deposition uniformity
- Protect backside grating from damage due to surface tension forces post plating

Initial Plating Attempts

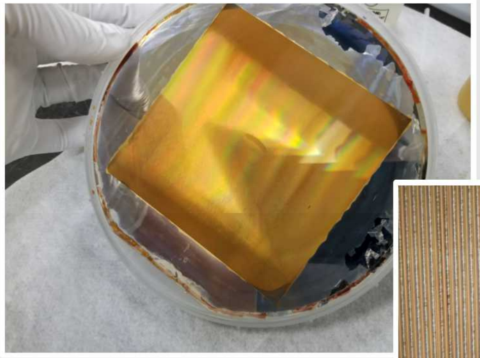


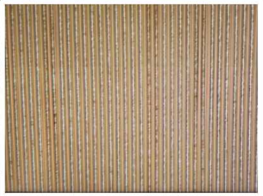


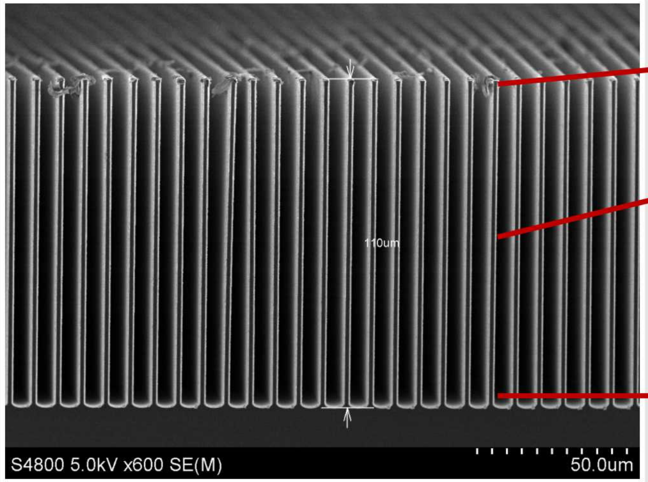
- 0.3 μm deposited in 13 hrs
- Insufficient current applied
- Non-ideal drying process led to grating damage due to surface tension forces

G2 Au Plating Improvements

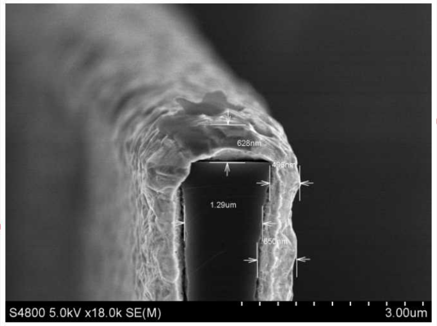
- Increased current density
- Improved rinsing and resist removal process to prevent surface tension induced grating delamination
- 0.6 um plating in 18.5 hrs
- Uniform Au deposition



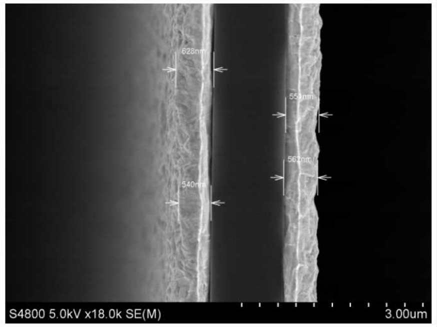




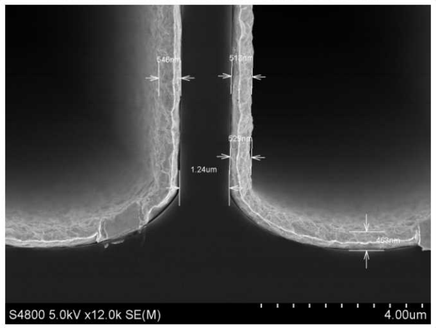
~0.63 um thick Au



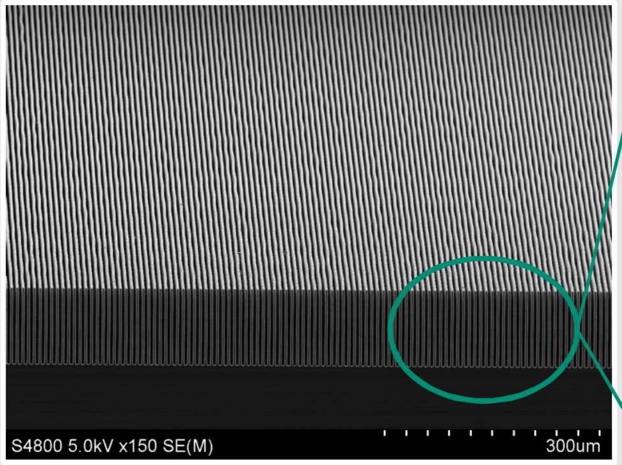
0.56-0.63 um thick Au

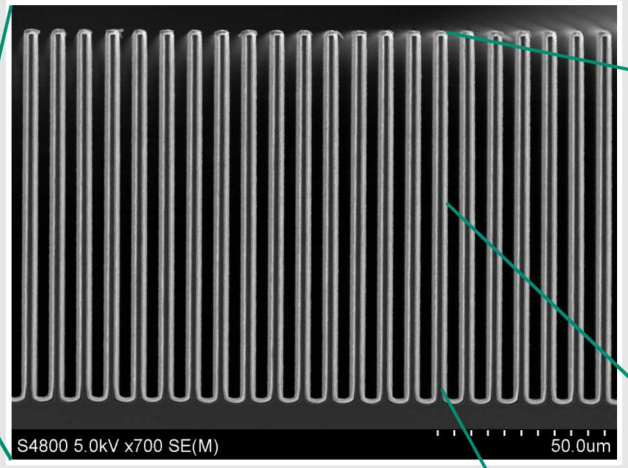


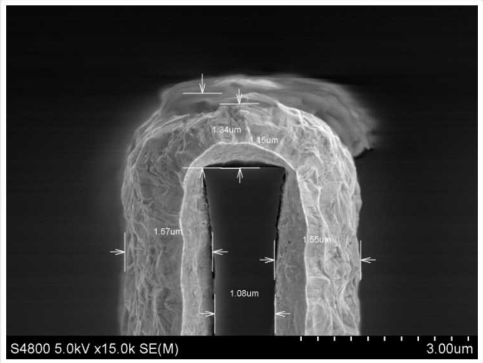
0.53-0.55 um thick Au

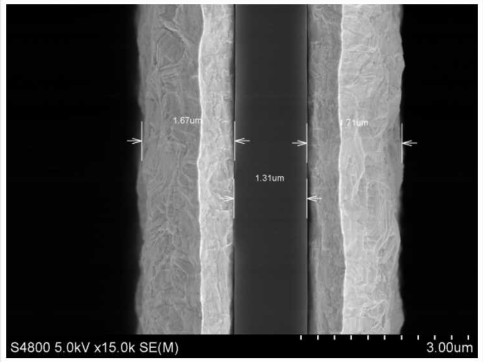


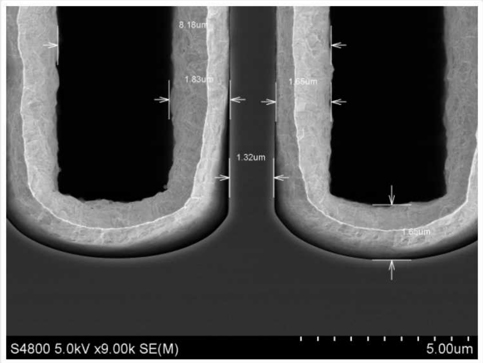
G2 Au Plating Improvements





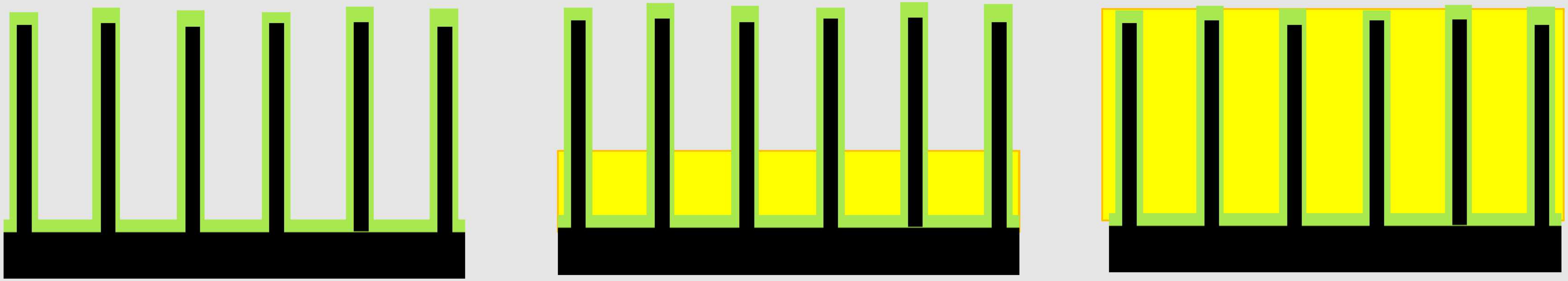


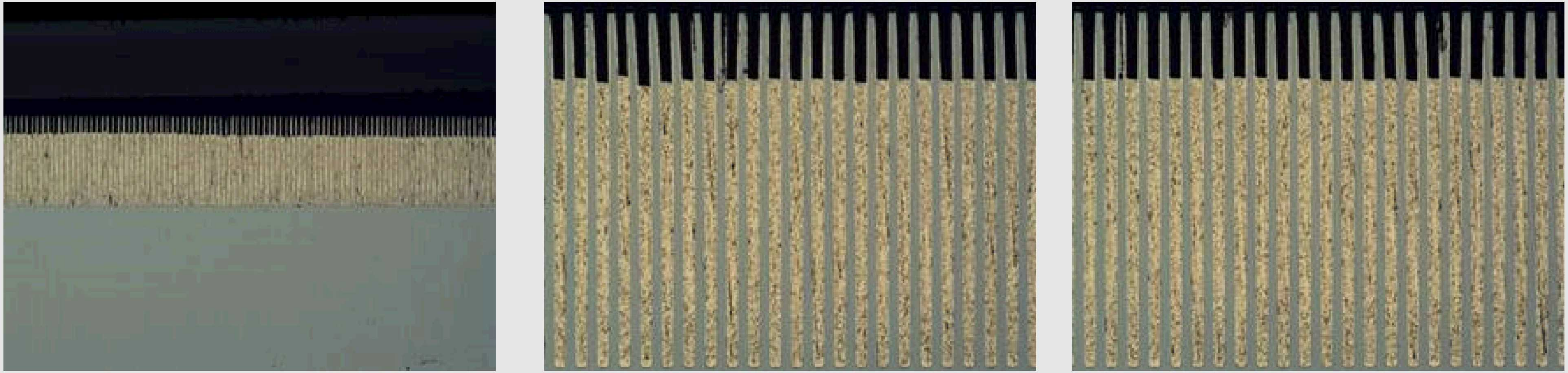




- **67 hr deposition**
- **Uniform Au deposition throughout depth of Si features and across 4 in. x 4 in. area**
- **Backside grating successfully protected during frontside deposition**
- **5 wafers used for double sided plating process development**
    - **strict protocol required for protecting the backside during the resist application, plating, rinsing, and resist removal is needed to prevent the backside grating from breaking**

Gold Super-filling rigid Si templates

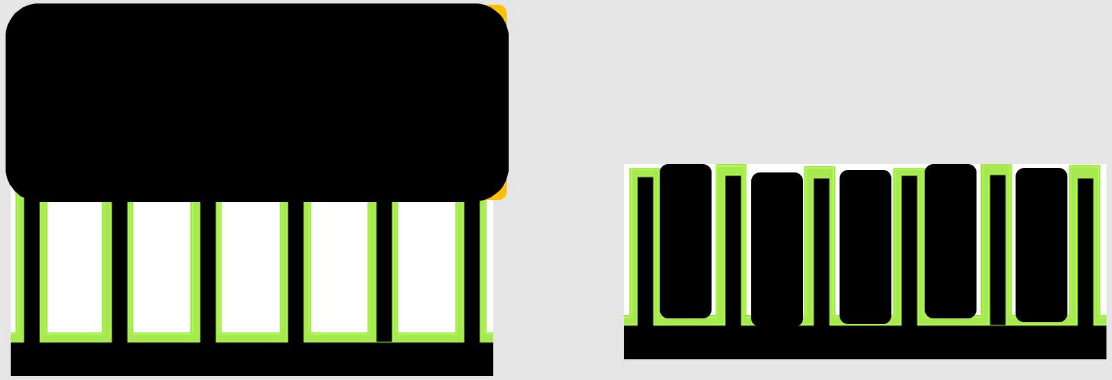


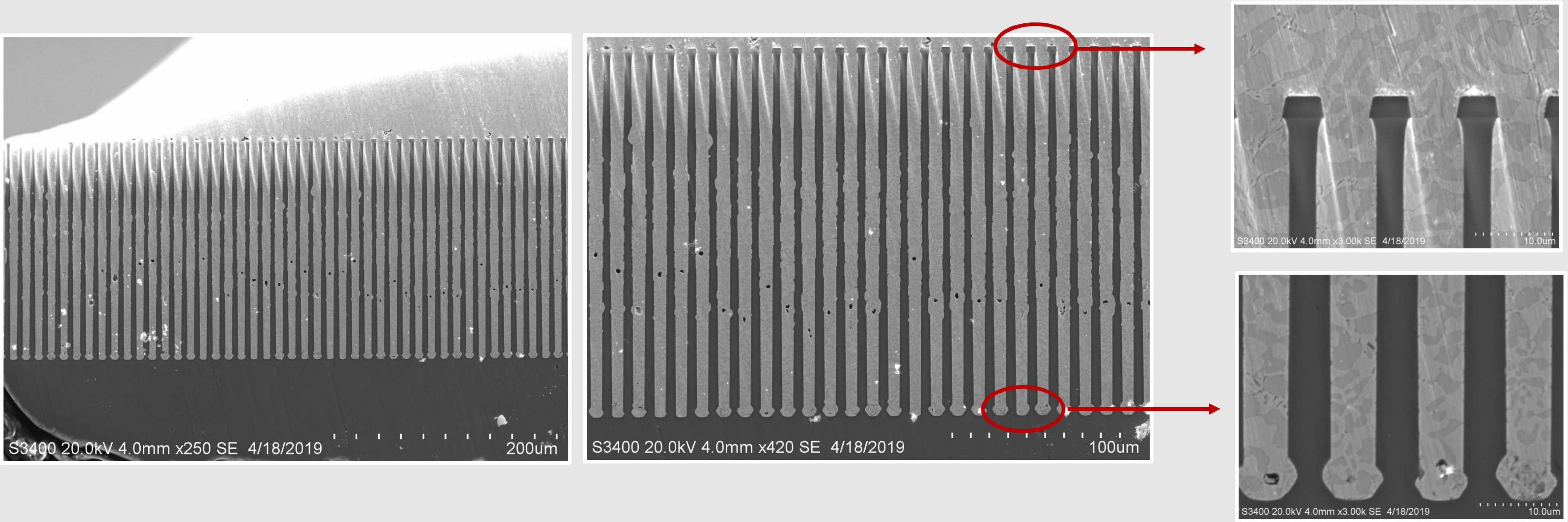


Proprietary electrodeposition techniques promote bottom up growth from a continuous conductive ALD Pt seed metal

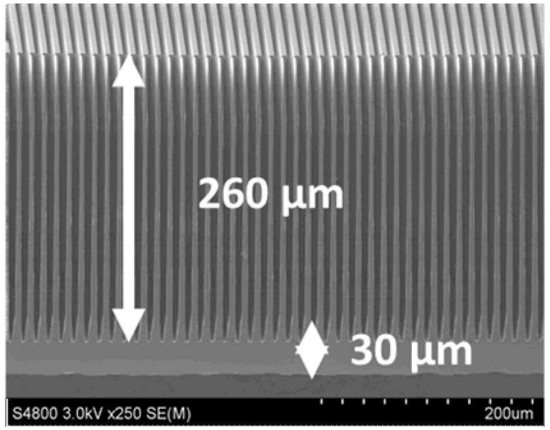
Development gold/tin (Au/Sn) eutectic flood

1. Reflow AuSn to flood trenches of grating
   - Conformally coat Si template with Pt wetting film
   - Solder jet AuSn on top of grating
   - Reflow AuSn at 280°C to fill trenches

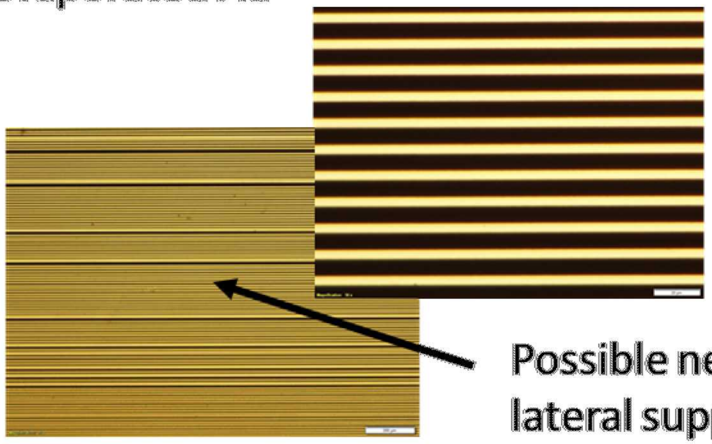




Through etch, plug and plate



Through etched grating 260 µm deep grating with angled ebeam evaporated Au



Possible need for lateral support

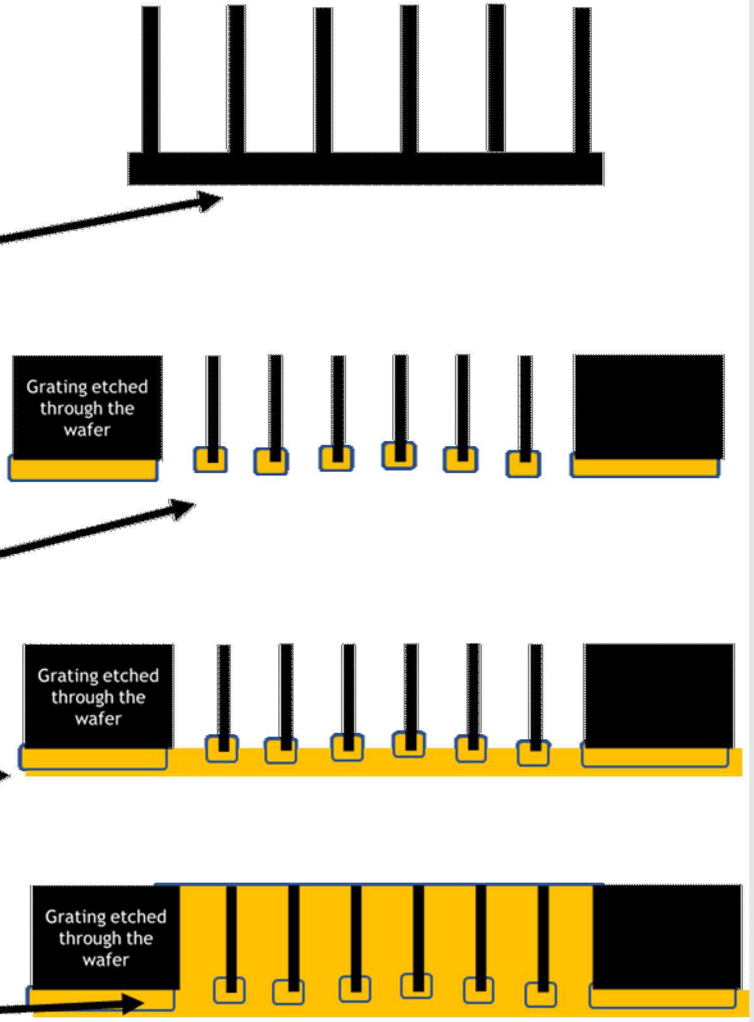
260 µm deep etch in a 290 µm thick Si Wafer

Blanket etch backside to the grating to create a through etched grating

ebeam angled Au evaporation to coat only the bottom of the gratings

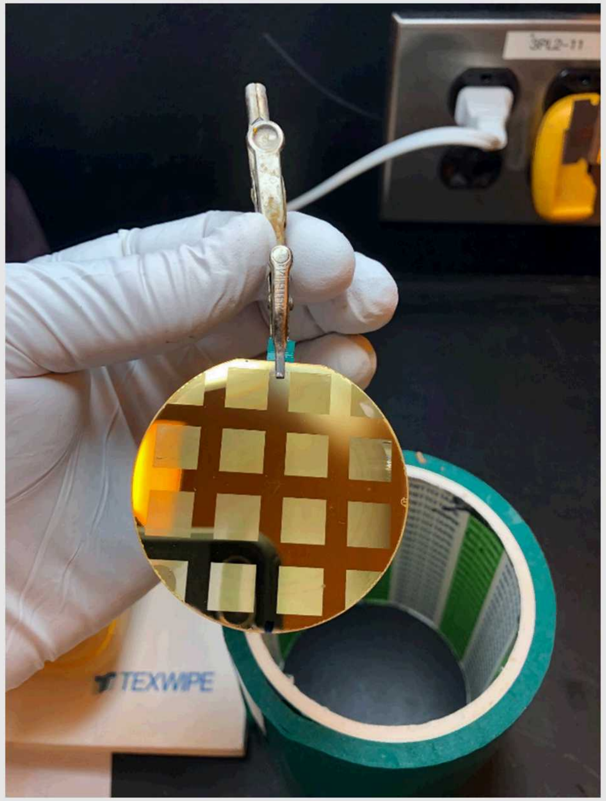
Electroplate Au to plug one side of the grating; need to passivate backside so don't plate downward only up through grating.

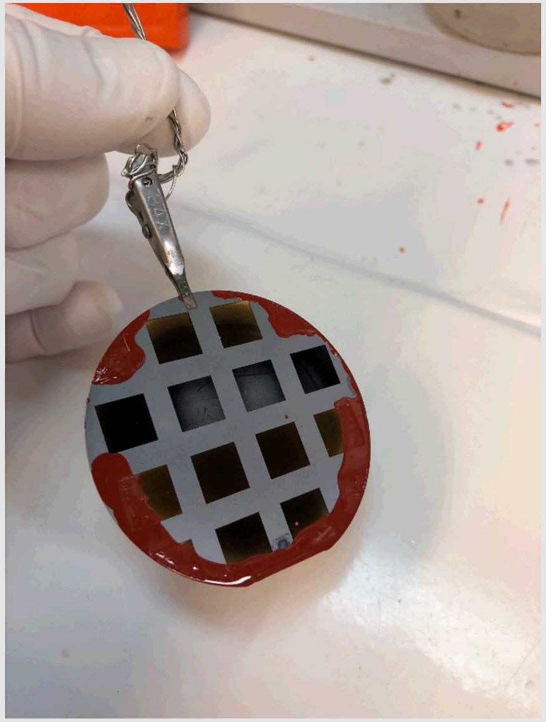
Electroplate Au to fill rigid Si template



DRIE sample 3

- DRIE sample with Al2O3 coating.
- 25 mA pulse plated (700 HZ, 25% duty) backside of grating, estimated area to be 12.5 cm^2
- Connection with alligator clip and applied red varnish on front and back, ready to gold plate from bottom up fill





Summary

- Description of XPCI and our lab based system at SNL
- Shown the difference between regular X-ray images and the phase images of XPCI
- Fabrication techniques of XPCI gratings
  - LIGA
  - Anisotropic KOH etching <110> Silicon
  - Bosch etching
    - Highest aspect ratio
    - Front to back alignment
    - Through etch
- Precision conformal electrodeposition over rigid Si templates
- Super filling using localized bottom up filling
- Plug and plate

Questions?