Preparation of ultra-smooth platinum films

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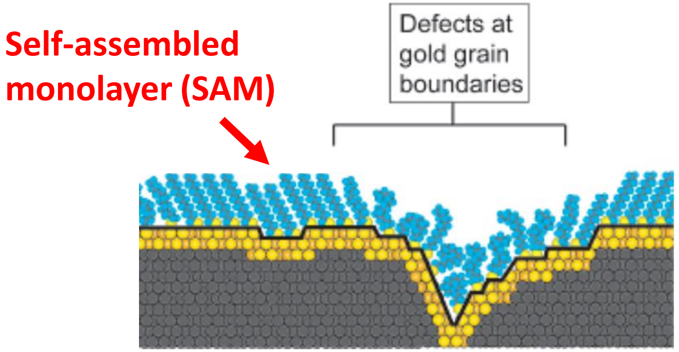
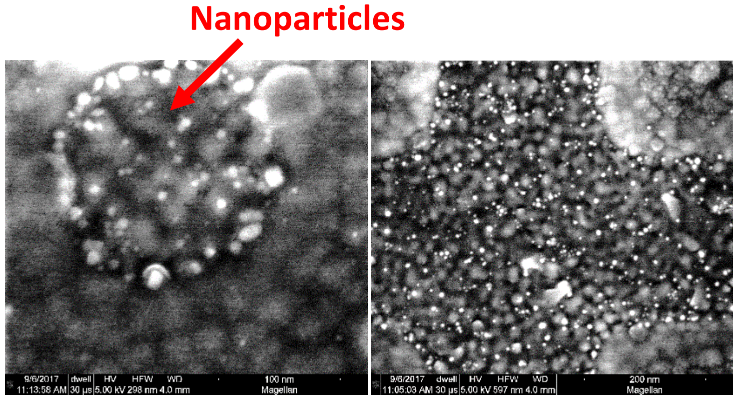
Motivation

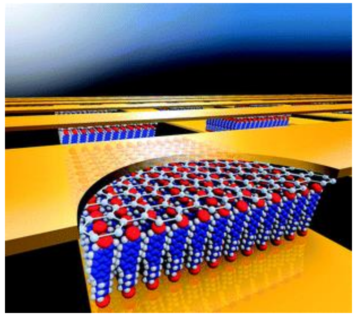
Why ultra-smooth films?

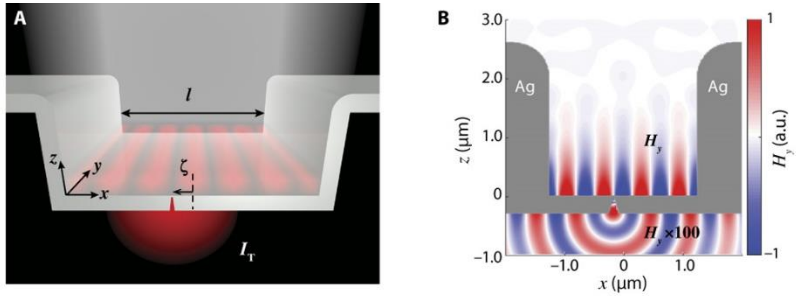
- Grain boundaries ➔ defects and surface energy variation ➔ non-uniformity





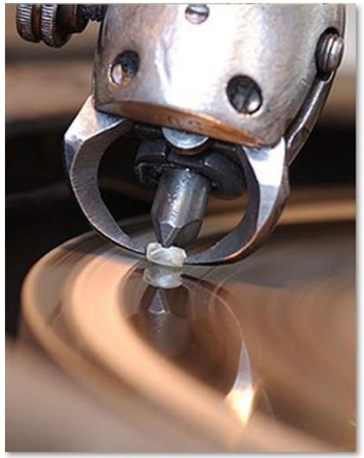
- Improved device **performance**, **yield** and **uniformity**
- Applications in:
  - Molecular electronics
  - Plasmonics
  - MEMS



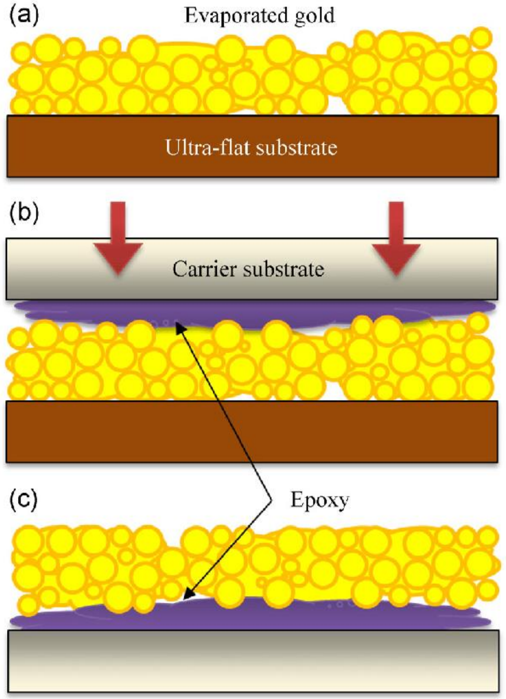


Approaches to obtaining ultra-smooth metal

Polishing

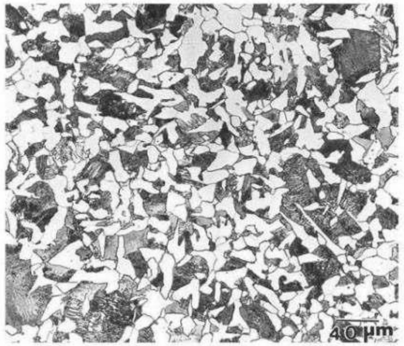


Utilizing a smooth interface: *Template stripping*



Controlling morphology:

- Very small grains
- Very large grains



- Metallic glass (amorphous)

Outline

3 different methods:

1) **Chemical Mechanical Polishing**
2) **Patterned Template Stripping**
3) **Sputter deposition + Annealing**

- How it works
- Process flow
- Results
- Observations and issues

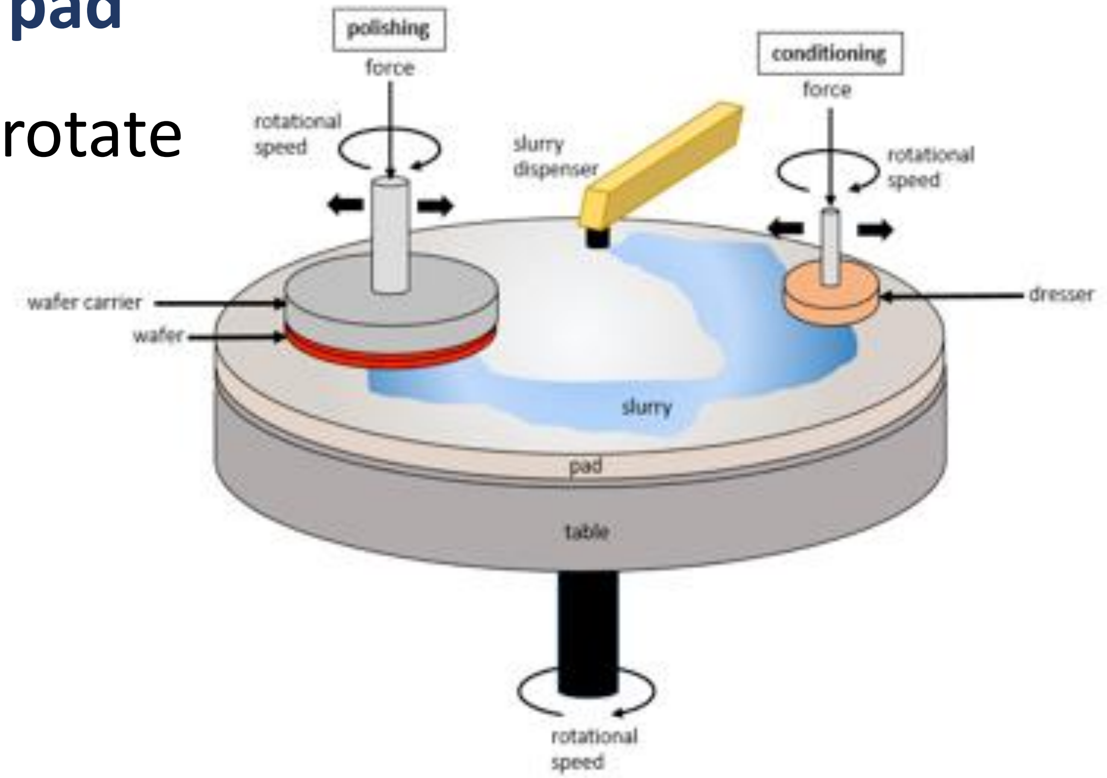
- Characterization of films
- Summary of processes developed

1) Chemical mechanical polishing

1) Chemical mechanical polishing (CMP)

<u>How it works</u>:

- Chemical etching + abrasive polishing
- Wafer is pressed facedown onto **polishing pad**
- Slurry is added as **polishing head** and pad rotate
- **Slurry** typically comprises:
  - Colloidal abrasive
  - Corrosive chemical



1) Chemical mechanical polishing (CMP)

At the SNF

- The Tool: POLI-400L
- Silica slurries available:
  - Ultra-sol S10 → for Si
  - Ultra-sol 2EX → for SiO_2

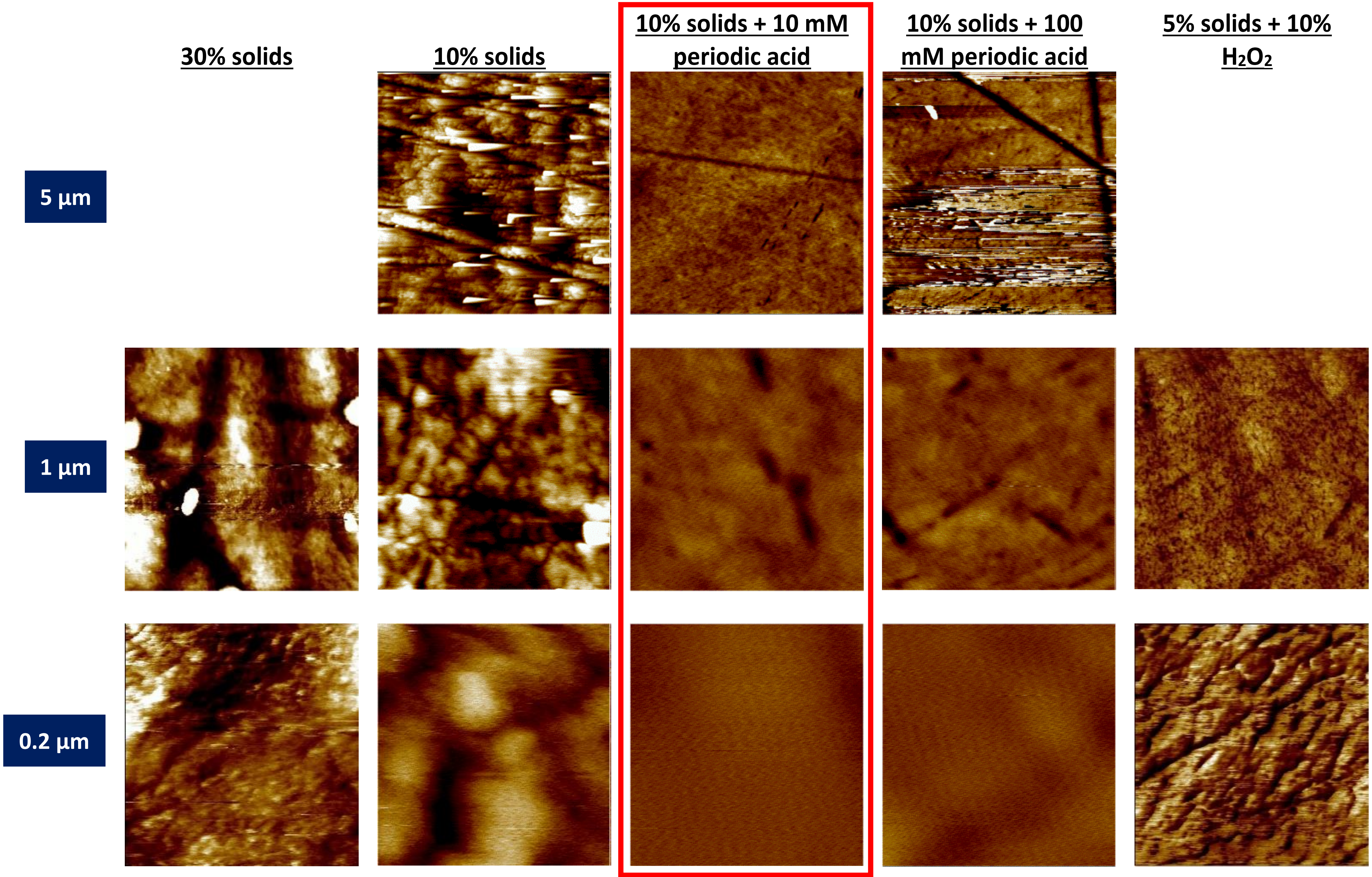
Compare several different slurry formulations:

- Undiluted slurry (30% solids)
- Diluted slurry (10% solids)
- Diluted slurry with ***peroxide*** added
- Diluted slurry with ***periodic acid*** added

Challenges in polishing platinum:

- Pt is very unreactive → difficult to remove
- No commercially available slurry

Results for Ultra-etch S10



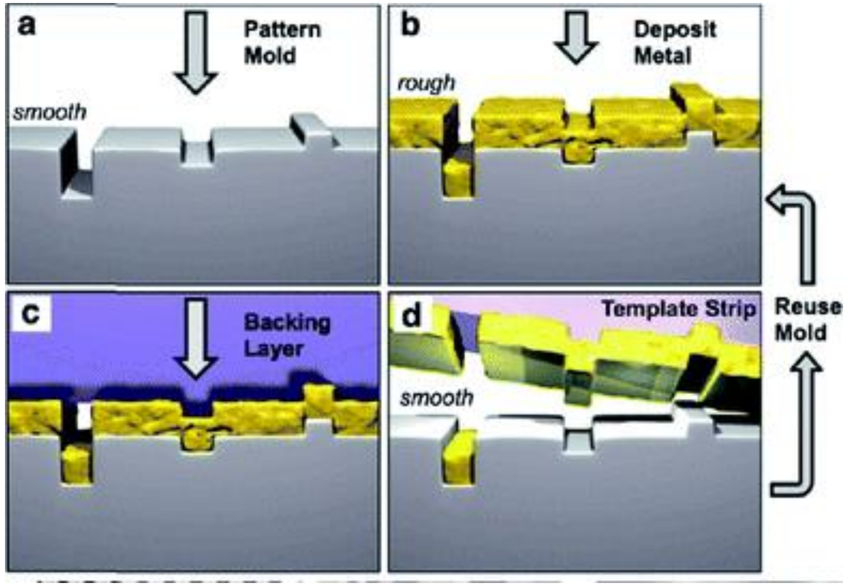
Polish time: 2 min

Pressure: 150 g/cm^2

- RMS roughness down to 0.3 nm
- Some scratches
- Periodic acid seems to help

2) Patterned template stripping

2) Patterned template stripping



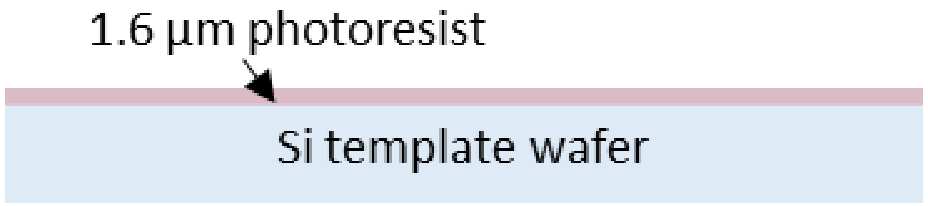
- **Specifications**:
  - Thermal stability (up to 300 °C)
  - Solvent resistance
  - Ease of patterning sparse devices
  - Scalability

This rules out typical epoxies, which have lower T_g

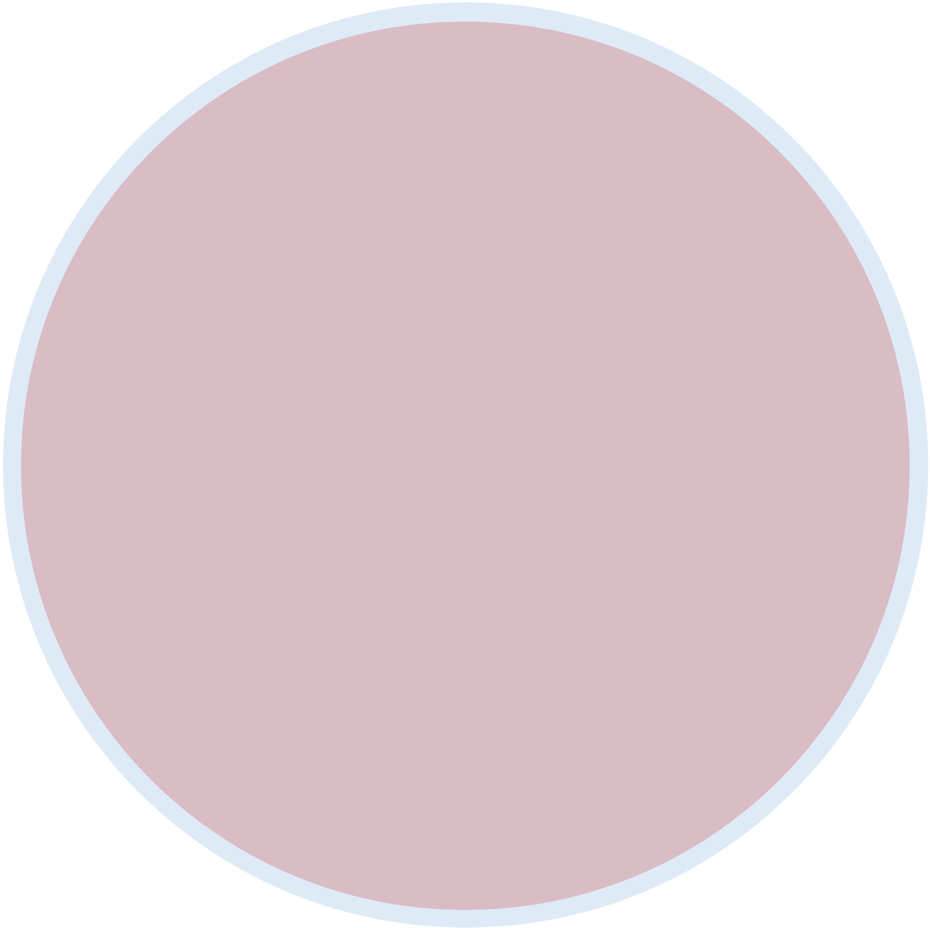
- Tried different silica-based adhesives
  - Liquid glass ➔ too thick, too messy
  - Tacky, UV-curable adhesive ➔ difficult to get continuous film
  - Spin-on glass (SOG) ➔ Proof-of-concept!
- **Tools: Heidelberg, svgcoat/dev, PT-DSE, AJA evap, Headway**

2) Patterned template stripping: Method

1. Spin 1.6 µm of photoresist on Si wafer



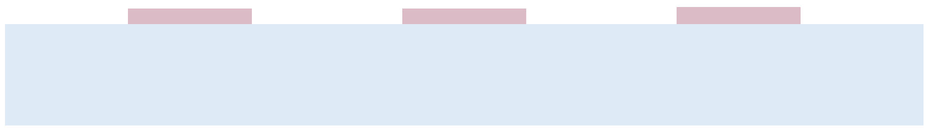
CROSS-SECTIONAL VIEW



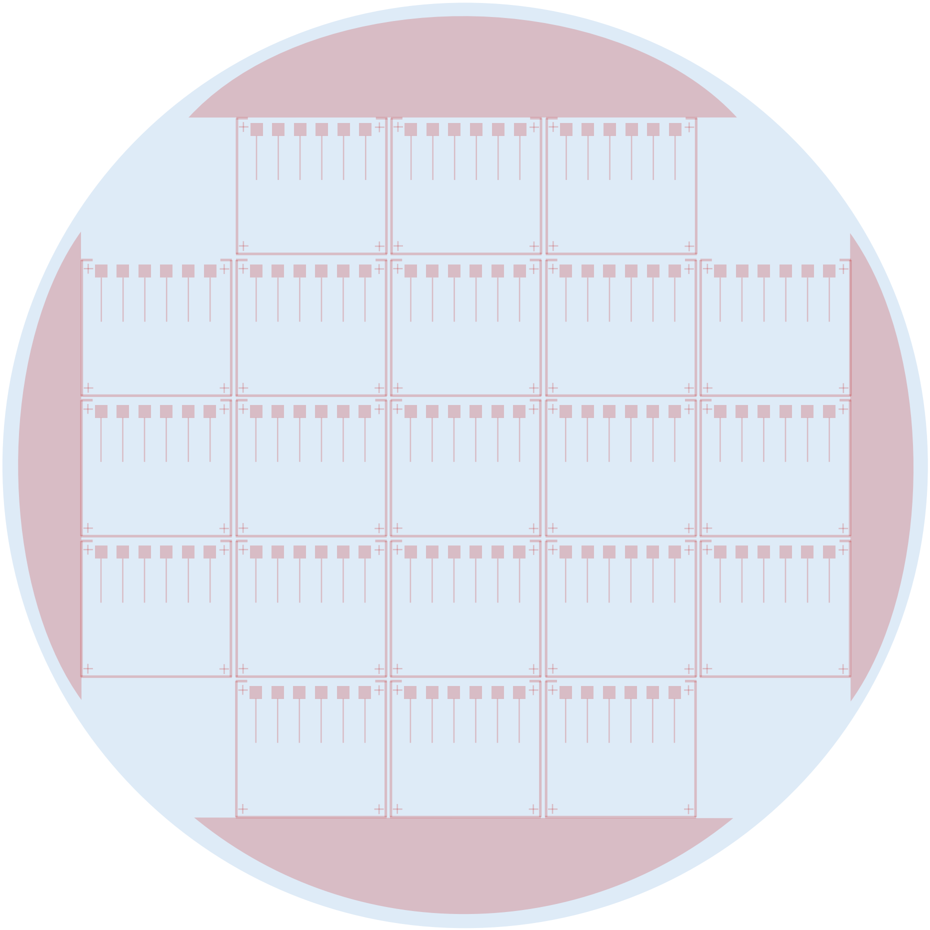
TOP VIEW

2) Patterned template stripping: Method

2. Expose and develop resist



CROSS-SECTIONAL VIEW

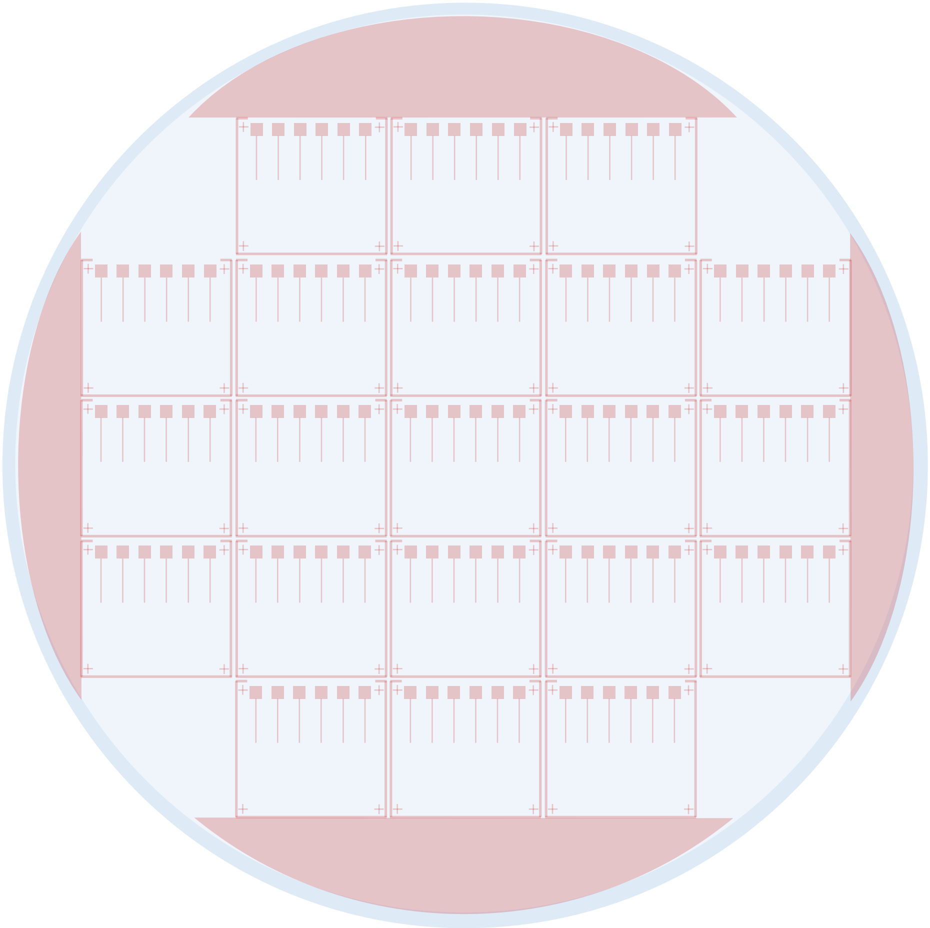


TOP VIEW

2) Patterned template stripping: Method

3. DRIE etch of silicon in exposed areas

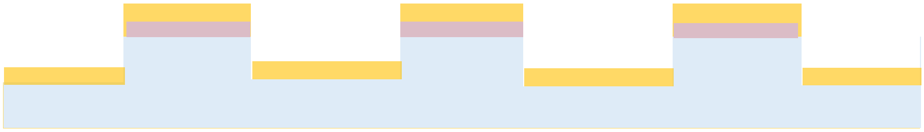
CROSS-SECTIONAL VIEW



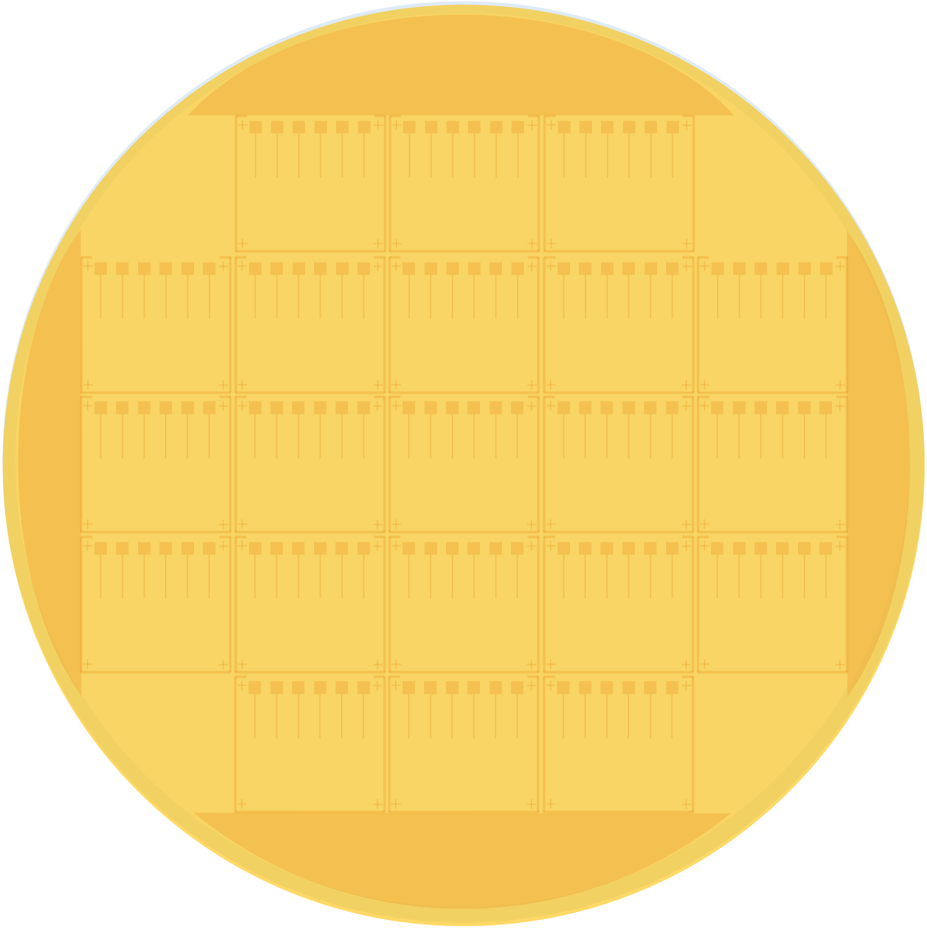
TOP VIEW

2) Patterned template stripping: Method

4. Deposit thin adhesion layer of Cr (10 nm) / Au (10 nm)



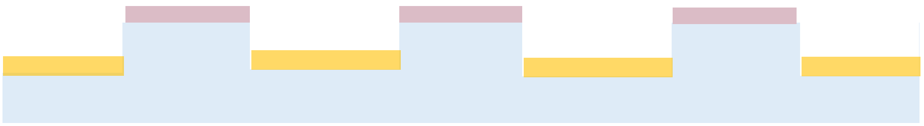
CROSS-SECTIONAL VIEW



TOP VIEW

2) Patterned template stripping: Method

5. Lift-off to remove metal from raised pattern → piranha clean



CROSS-SECTIONAL VIEW

TOP VIEW

2) Patterned template stripping: Method

6. Deposit Pt (70 nm) / Ti (8 nm)

CROSS-SECTIONAL VIEW

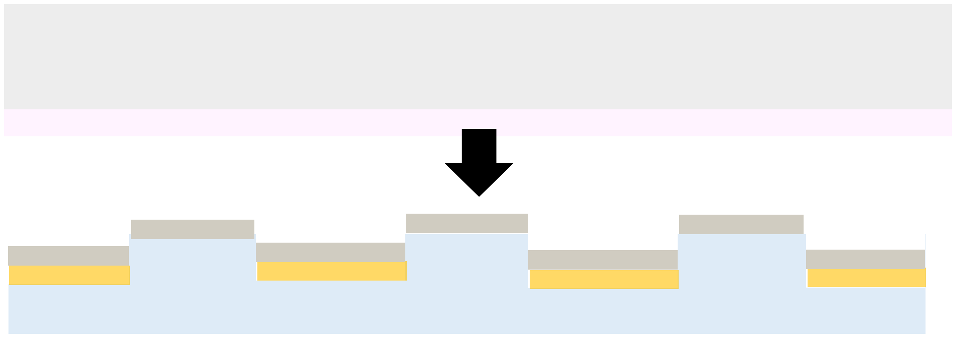
TOP VIEW

2) Patterned template stripping: Method

7. Spin SOG onto clean glass substrate (2000 rpm, 30 s)

CROSS-SECTIONAL VIEW

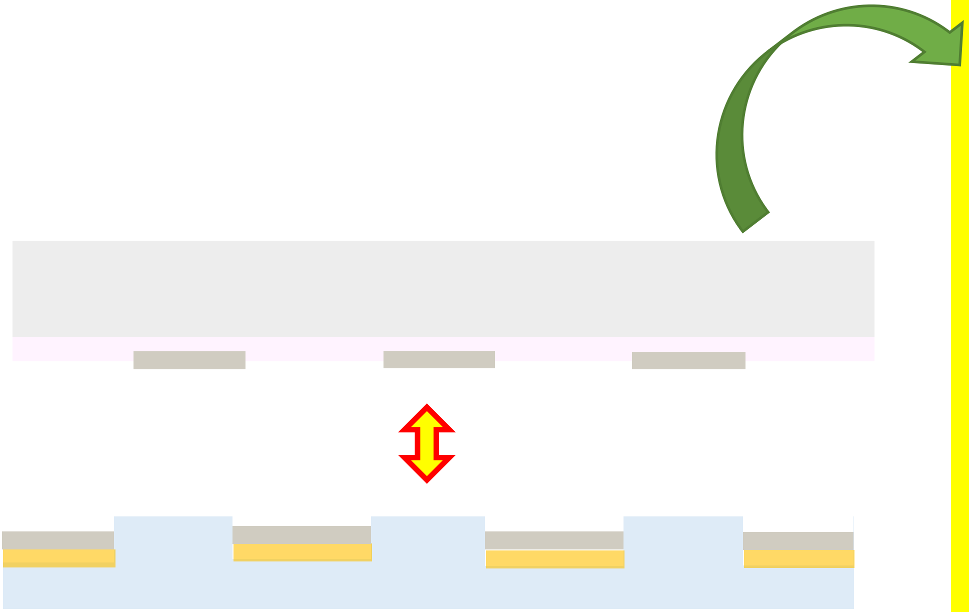
8. Immediately place glass substrate facedown onto template. Apply pressure and cure (120 °C → 200 °C)



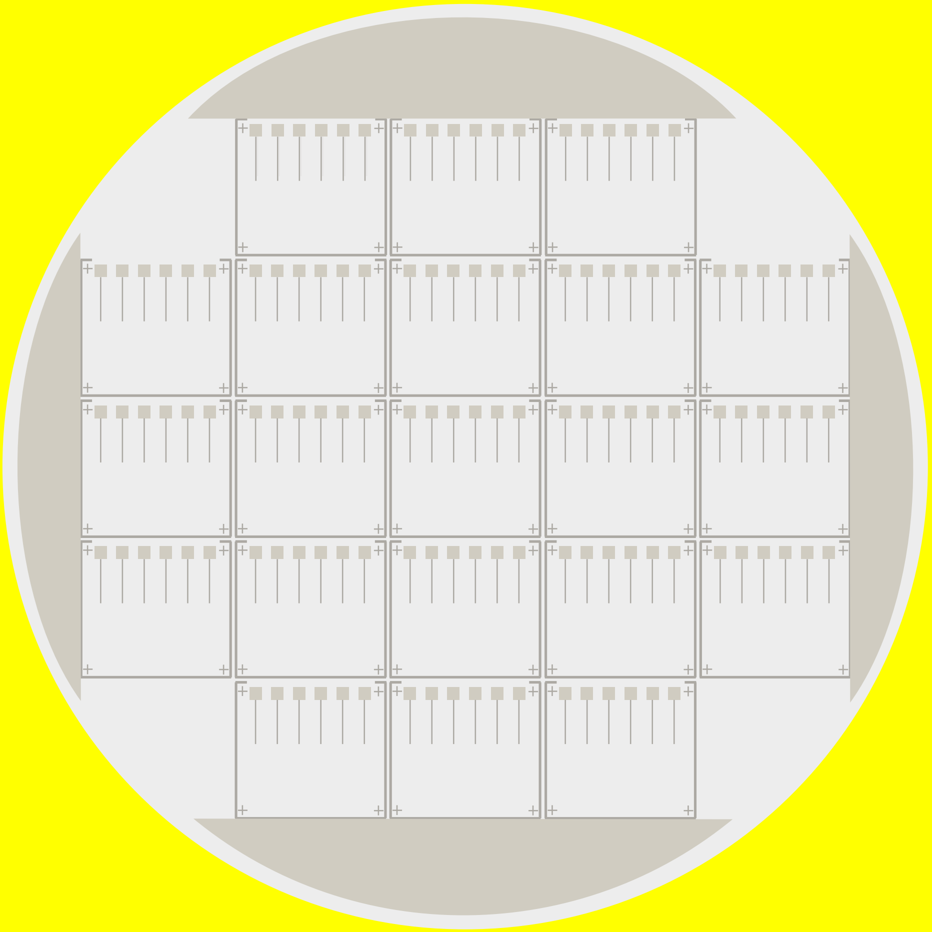
CROSS-SECTIONAL VIEW

2) Patterned template stripping: Method

9. Separate template and substrate using scalpel



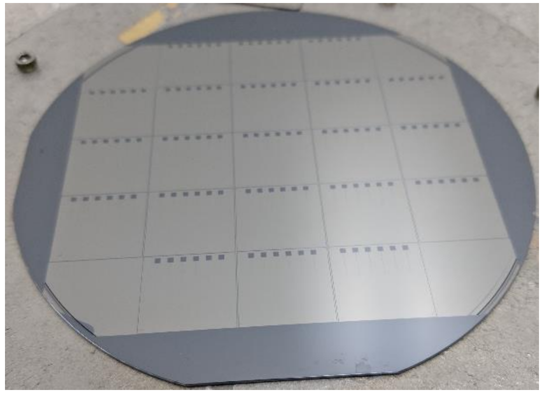
CROSS-SECTIONAL VIEW



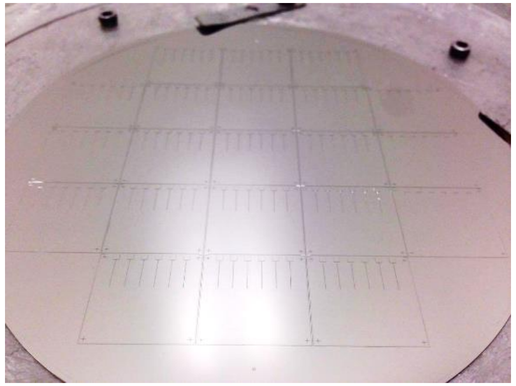
Substrate with patterned template stripped film!

2) Patterned template stripping

Template preparation

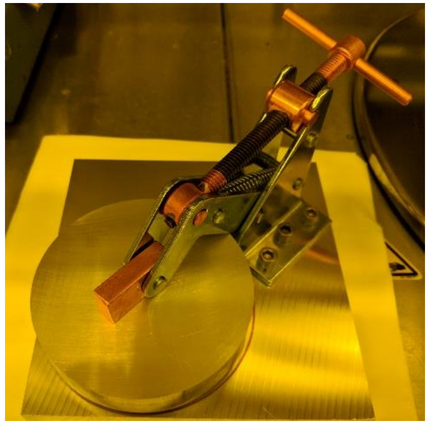


After selective lift-off of adhesion layer



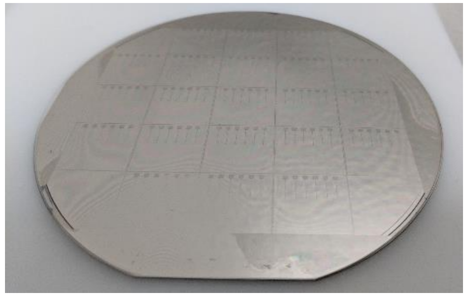
Pt / Ti metallization

Clamp & Cure

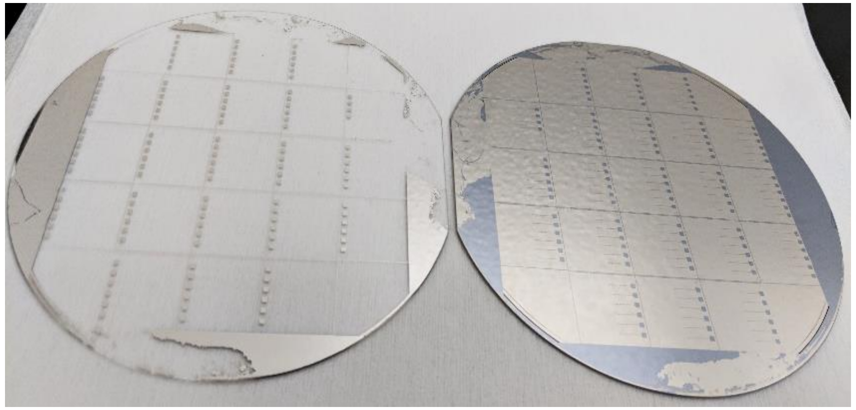


Custom clamp setup

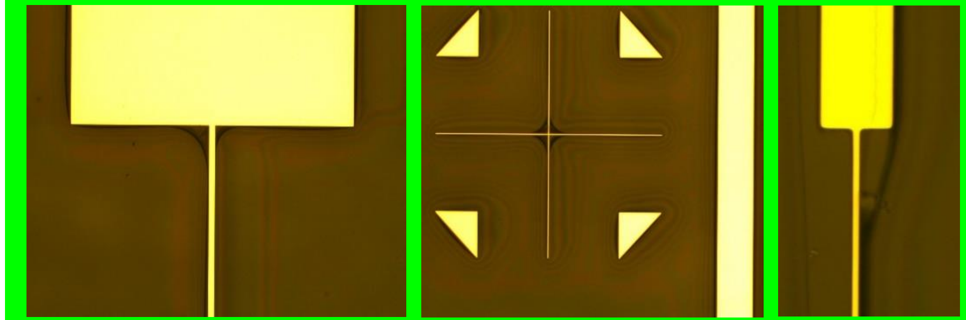
Oven



Wafer stack



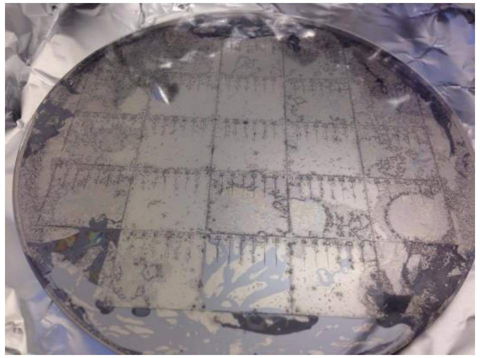
Good yield down to 20 µm, some 2 µm features transferred!



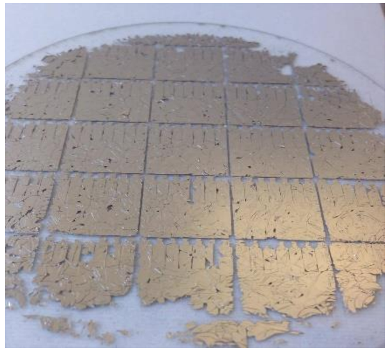
2) Patterned template stripping

Issues encountered

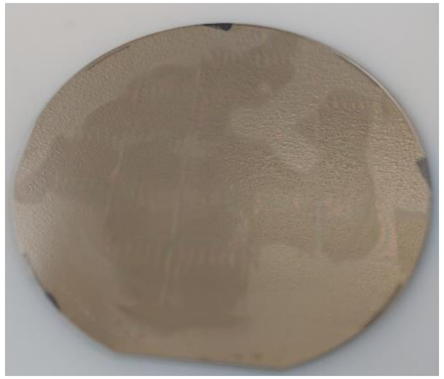
Unsuitable adhesive



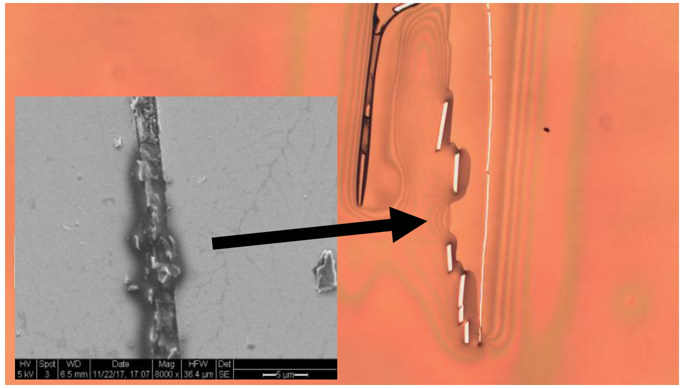
Inverse pattern transfer → solved with adhesion layer



Substrate stuck to template

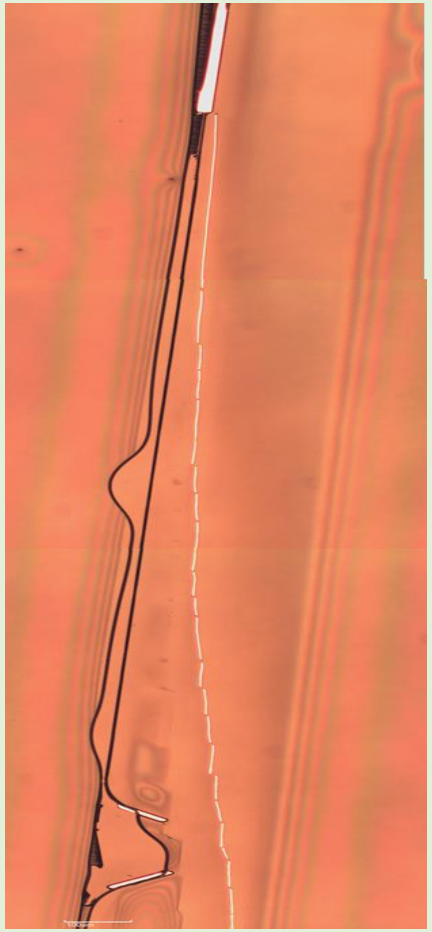


Collapse of fine template structures



Tradeoff between <u>etch depth</u> and <u>mechanical</u> <u>stability</u>

Ongoing issues



Shearing of long thin wires due to **non-uniform pressure** applied by clamp

- Try different clamp setup with better alignment
- Use thicker wires

3) Sputter deposition + Annealing

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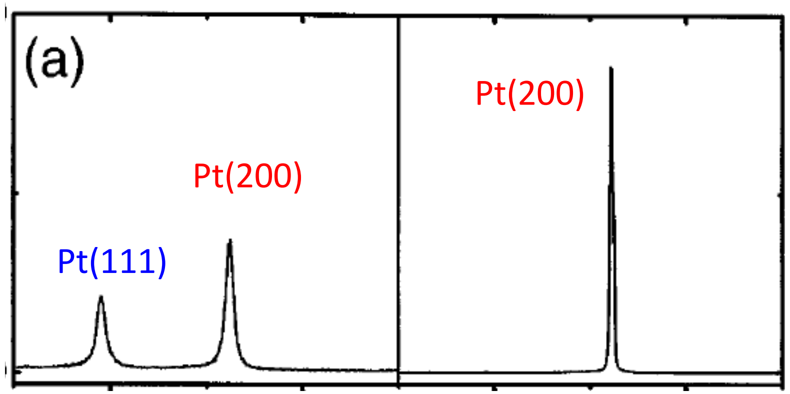
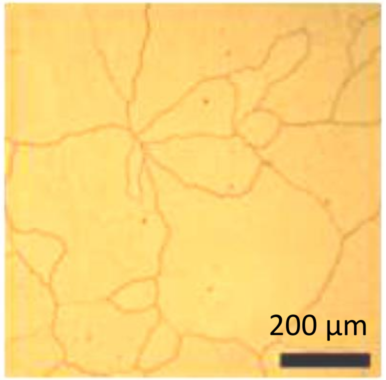
Principle

- Pt films are usually (111)-oriented
- Adding oxygen incorporation during deposition increases strain, causing (200) & (220) planes to become energetically favorable
- Annealing above 750 °C drives **abnormal grain growth (AGG)** to form <u>millimeter-sized</u> grains

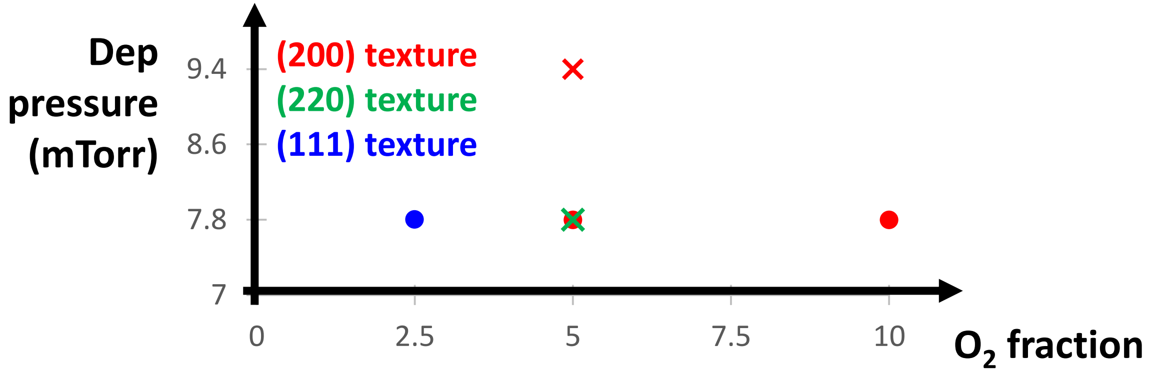
From literature:

Before anneal *After anneal*





Preferred orientation of films for various sputter conditions, after 1 h anneal at 1000 °C

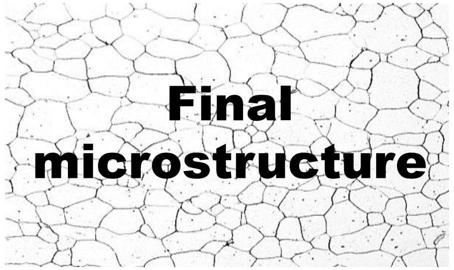


3) Sputter deposition + Annealing

- **Tools used: Lesker, Tylan9 (furnace)**
- Parameters kept constant
  - Substrate (1 x 1 cm quartz sample)
  - DC power
  - Deposition time
  - Anneal temperature + time ➔ no strong effect above certain values
- Parameters varied
  - Sputter pressure
  - O_2 fraction
  - Temperature

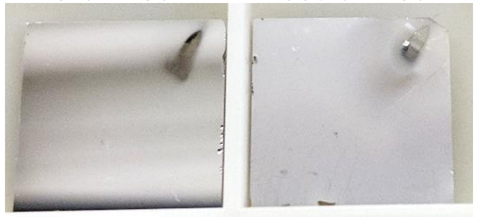
These determine:
- **O_2 incorporation**
- **Film stress**
- **As-deposited morphology**

Anneal



- Parameters reported in literature produced rough films with a matte appearance
- Succeeded in growing large grains (~hundreds of µm) using modified set of parameters

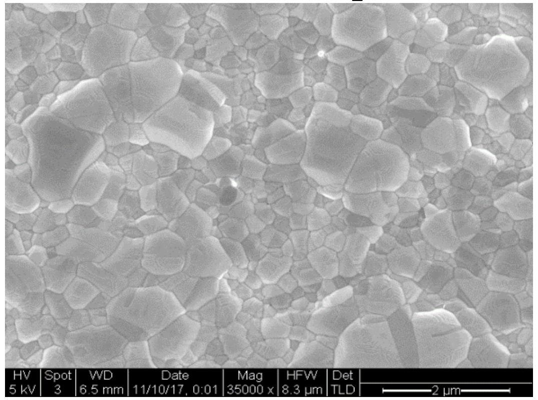
Pre-anneal *Post-anneal*

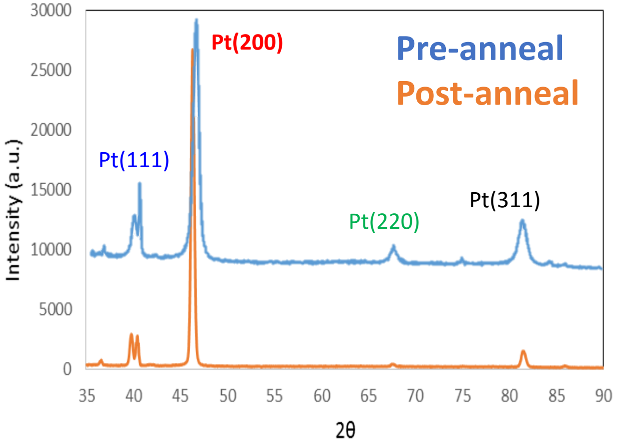


3) Sputter deposition + Annealing

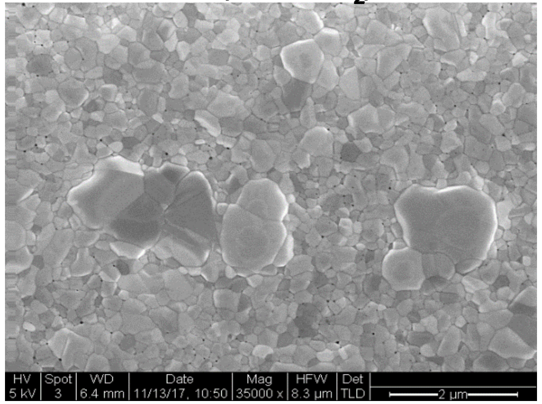
Wide range of microstructures obtained using various parameters:

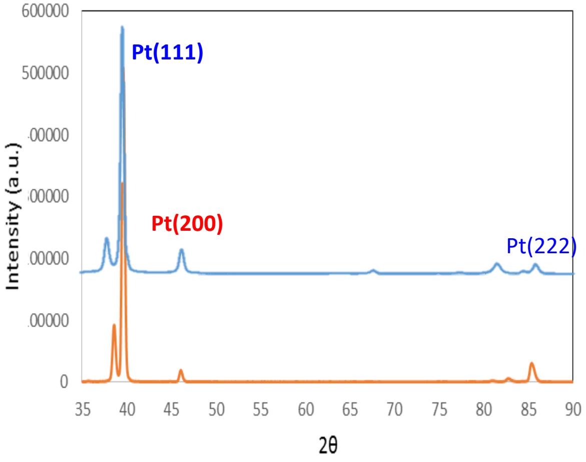
W6: 9.4 mTorr, 9.1% O_2, 50 °C



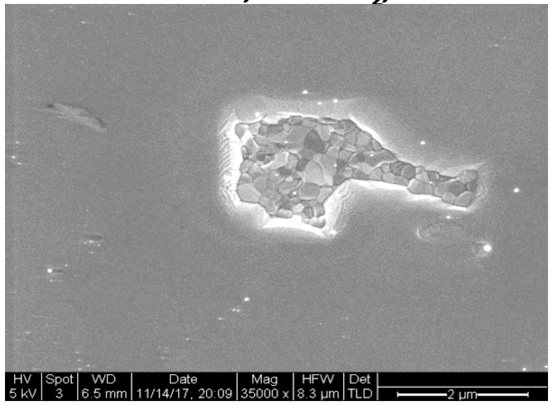


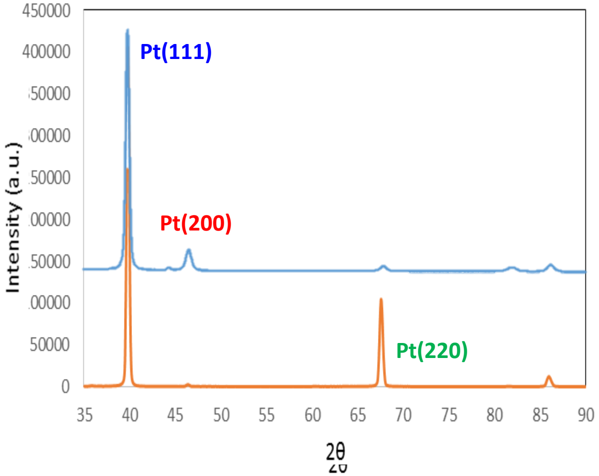
W8: 7.8 mTorr, 9.1% O_2, 30 °C



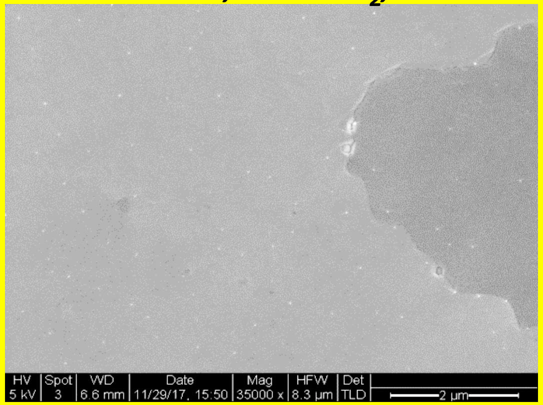


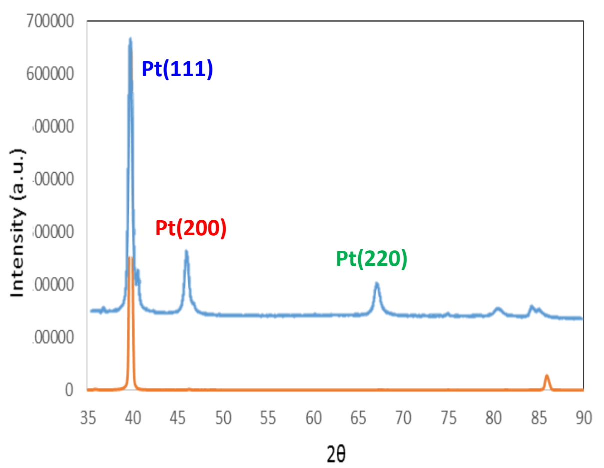
W9: 7.8 mTorr, 2.5% O_2, 30 °C





W15: 6 mTorr, 10.3% O_2, 30 °C





Degree of (200) texture seems to increase with O_2 fraction & pressure

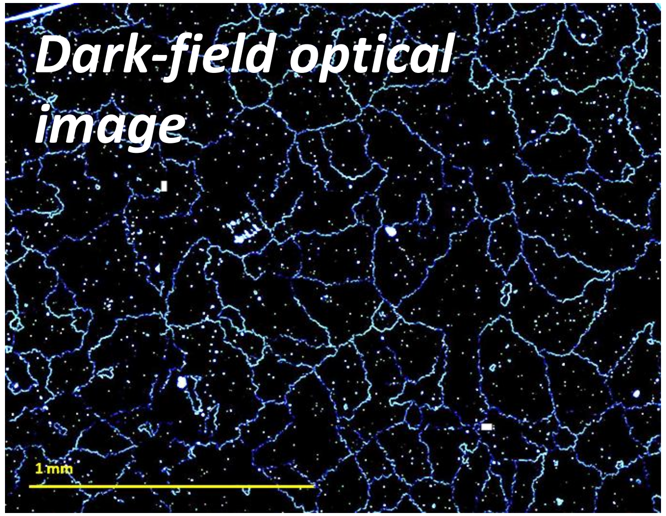
Increasing (200) texture ←

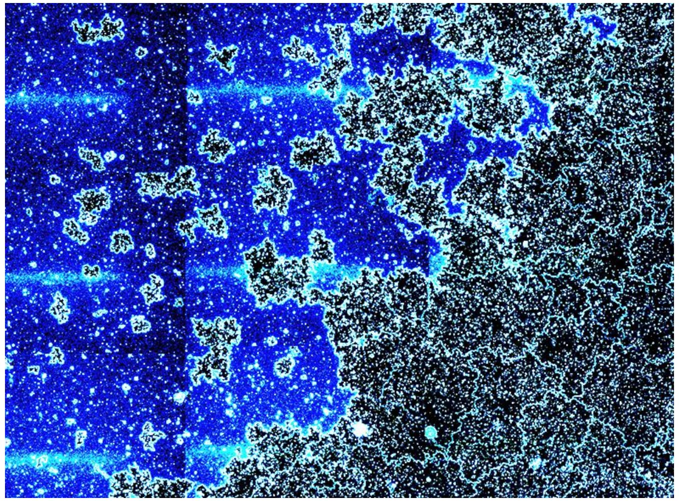
→ **Increasing (111) texture**

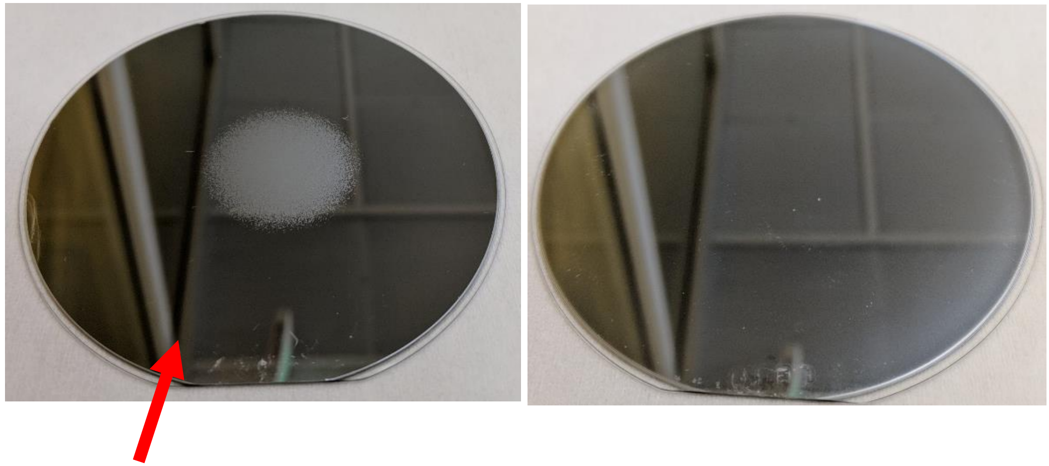
3) Sputter deposition + Annealing

- Continuous large grain growth was observed with (220)- and (111)-oriented grains, *not (200)* as reported
- This suggests that AGG can be induced in all 3 textures, depending on the interplay of various film properties
- AGG seems to be very sensitive to film stress

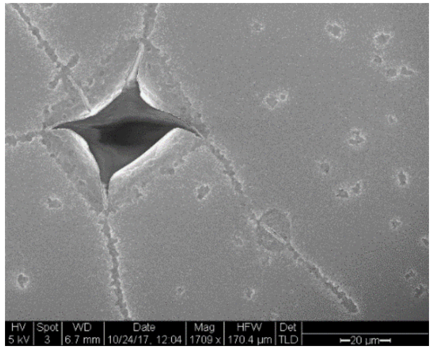
➔ *can lead to non-uniform morphology across a sample*







Parameters which gave large grains on 1x1 cm samples

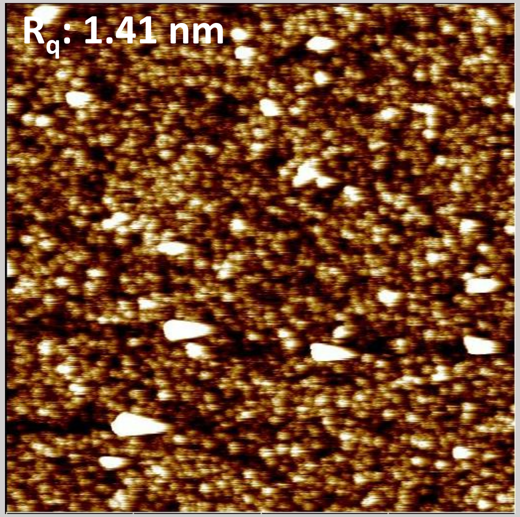


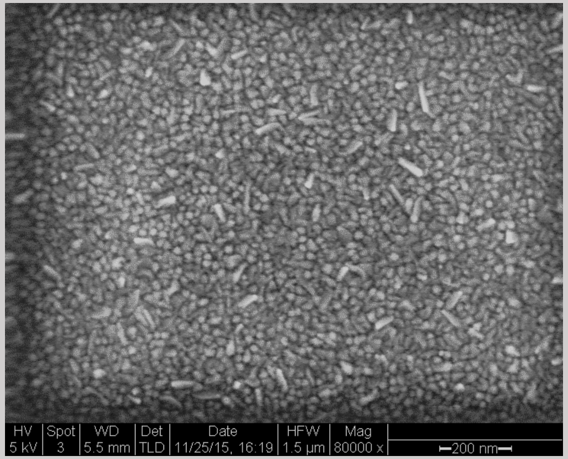
Larger grains at buckled reigions

Summary

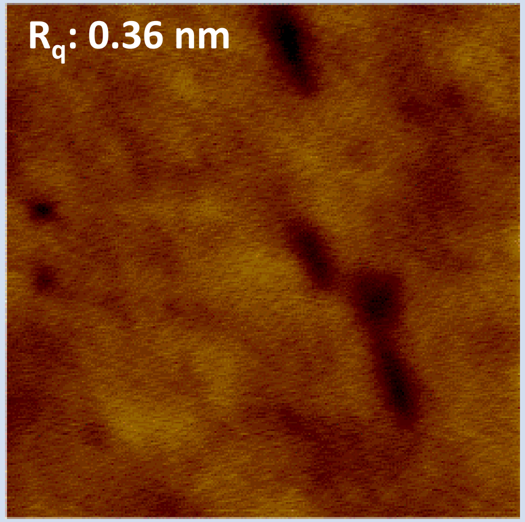
Comparison of methods

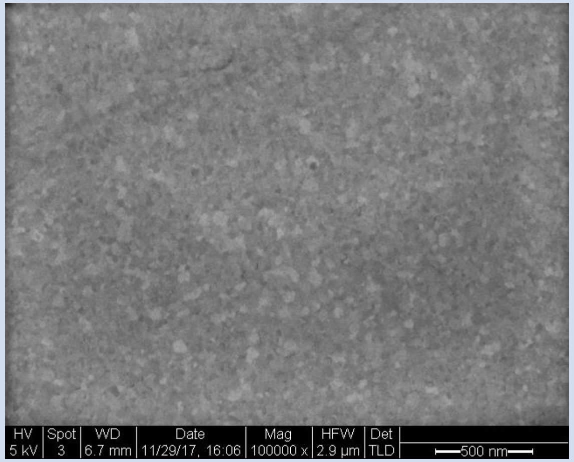
Standard evaporated platinum film (150 nm)



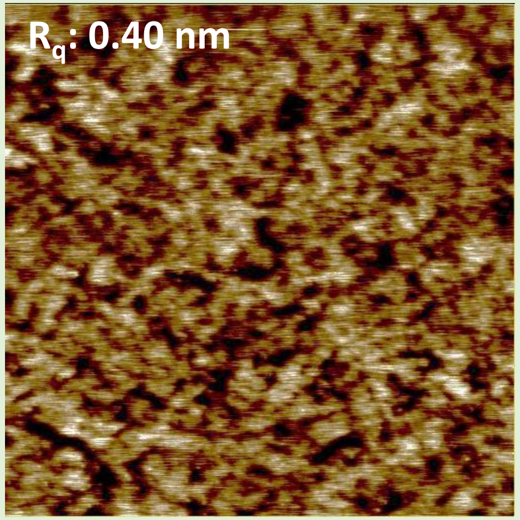


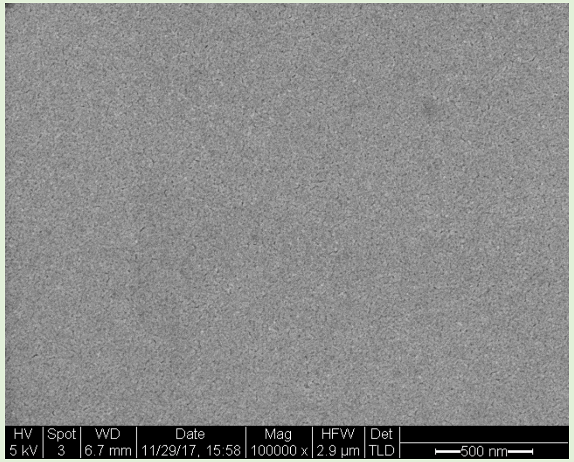
Chemical Mechanical Polishing



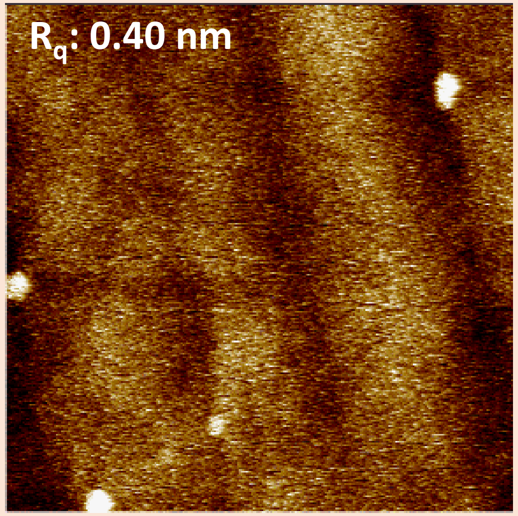


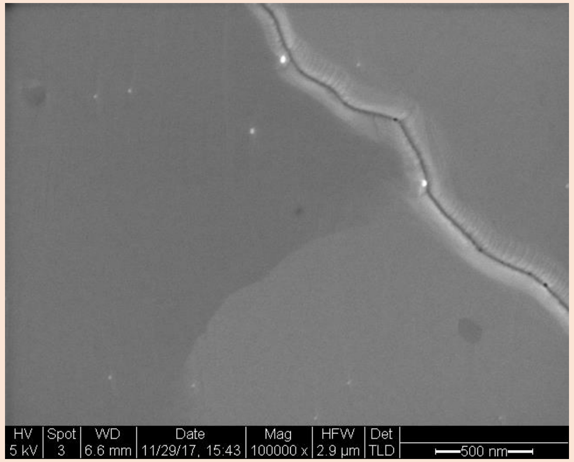
Patterned Template stripping





Sputter deposition + annealing





Summary of optimal processes (so far) for ultra-smooth platinum

Chemical Mechanical Polishing	Patterned Template stripping	Sputter deposition + annealing
• **<u>Polishing parameters</u>:** ❖ **Polishing head pressure: 150 g/cm^2** ❖ **Polishing pad pressure: 250 g/cm^2** ❖ **Pad rotation: 50 rpm** ❖ **Head rotation: 30 rpm** ❖ **Polishing time: < 2 min** • **<u>Slurry</u>:** **1. Dissolve periodic acid in DI water to a concentration of 10 mM** **2. Add 2 parts DI water (with periodic acid added) to 1 part slurry, as obtained from SNF. Either Ultra-Sol S10 or 2EX is fine.**	• Pattern template wafer (Si prime), with desired pattern to be transferred *not* exposed • Resolution down to 20 µm comfortable; for smaller features depends on aspect ratio of feature (sensitivity to shearing) • DRIE etch (~ 8 µm deep) • Deposit 10 nm Cr / 8 nm Au • Overnight liftoff in acetone, *gentle* sonication if needed, IPA clean • Piranha clean (5 min, 3:1 piranha) • Deposit 70 – 100 nm Pt / 8 nm Ti. If thick film, maybe allow break for film to cool to avoid delamination • Plasma clean of glass wafer • Spin SOG 400F @ 2000 rpm, 30 s • Place glass wafer facedown onto template, clamp (not too hard!!) • Cure for 1h @ 120 °C, 1h @ 200 °C • Allow wafer stack to cool; separate with scalpel	• **Quartz wafer** • **Plasma clean in Plasm-Etcher (next to Lesker) for ~ 3 min** • **<u>Deposition conditions</u>:** ❖ **KJ Lesker** ❖ **Adhesion layer: Ti @ 200 W, 3 mTorr, 50 sccm Ar / 5 sccm O_2, 5 min** ❖ **Film: Pt @ 170 W, 6 mTorr, 50 sccm Ar / 6.2 O_2 (11%), room temperature, 30 min** • **<u>Anneal conditions</u>:** ❖ **Tylan9, recipe M1000** ❖ **1000 °C for 1 h**

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