Design and Fabrication of Micro-Tip Arrays for Nano-Scale Atom-Probe Tomography

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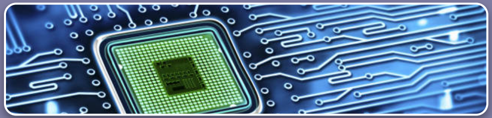
Overview: Atom-Probe Tomography (APT)[1]

What: 3-D characterization analysis with subnanometer resolution

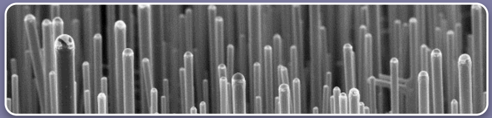
Why: Superior compositional sensitivity relative to prevalent techniques (TEM, SIMS)

Applications:



Microelectronics
Semiconductors

Alloys
Superalloys

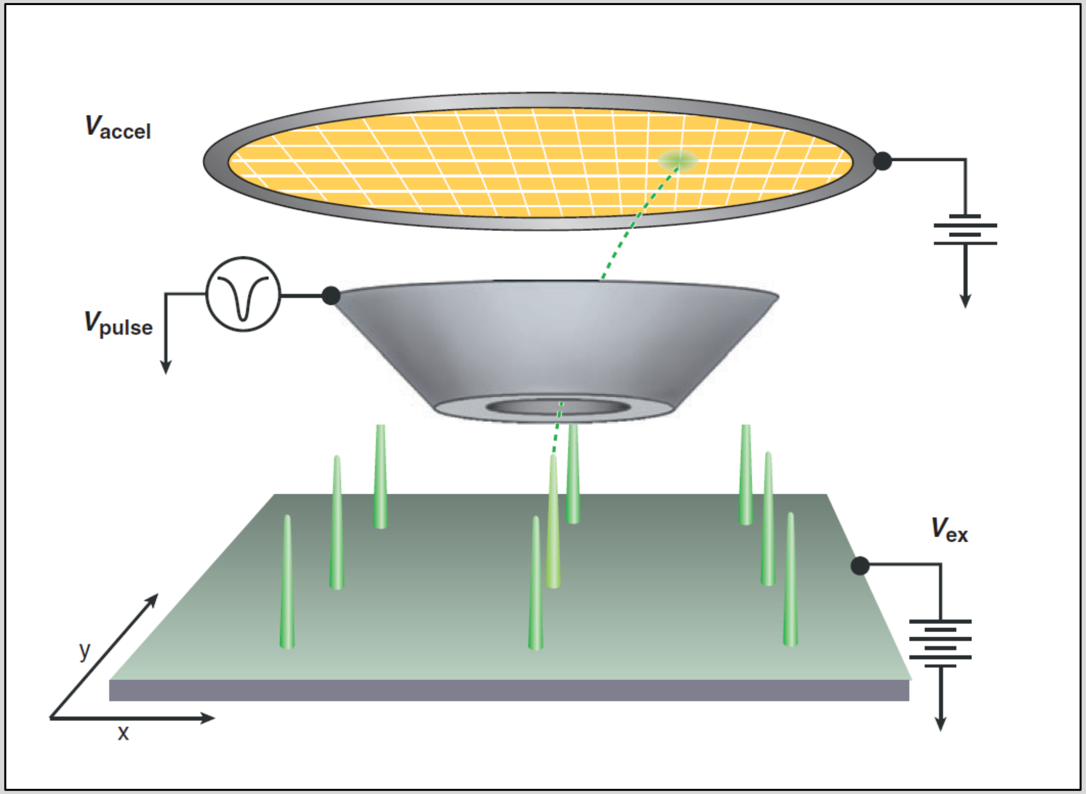


Nanomaterials

Image sources: from left to right, see References [2], [3], [4].

Schematic of APT Mechanism (Seidman[5])

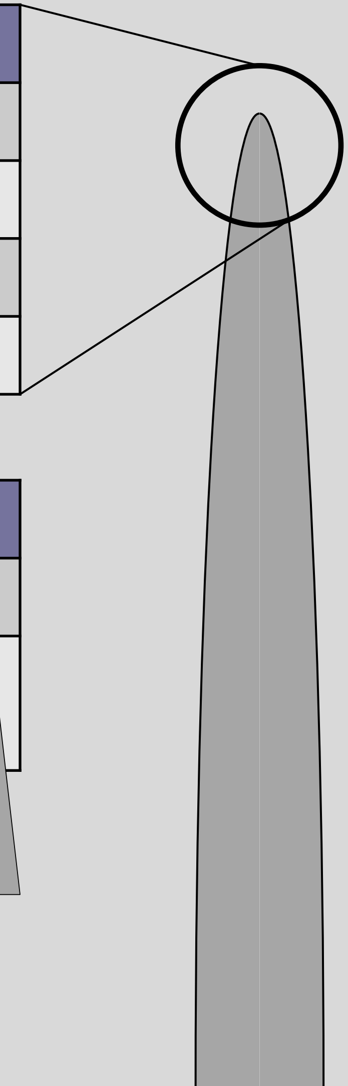
- Laser/voltage pulse generates electric field
- **Field evaporation** of atoms as ions detected, measured, analyzed
- Requires **needle-shaped specimen**



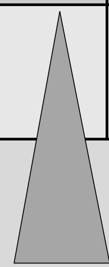
Specimen preparation is the major limitation for wide-spread APT use.

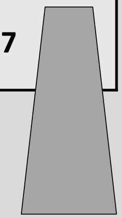
Specimen Preparation Factors

Functional Factors	Parameters[6]
Electric field strength	30-50 V nm^{-1}
Needle tip radius	20-150 nm
Semi-angle taper	< 10˚
Material resistivity	< 0.05 Ω cm

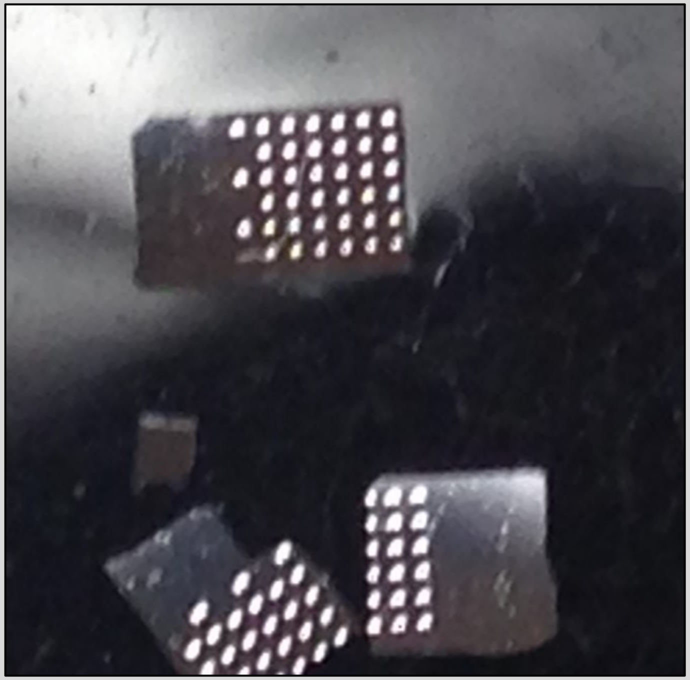


Other Factors	Options	
Throughput	Single needles	**Micro-arrays**
Shape	Pre-sharpened micro-tips (PSMs)	**Flat-top micro-posts (FTM)[7]**





Reconsidering Commercial Arrays

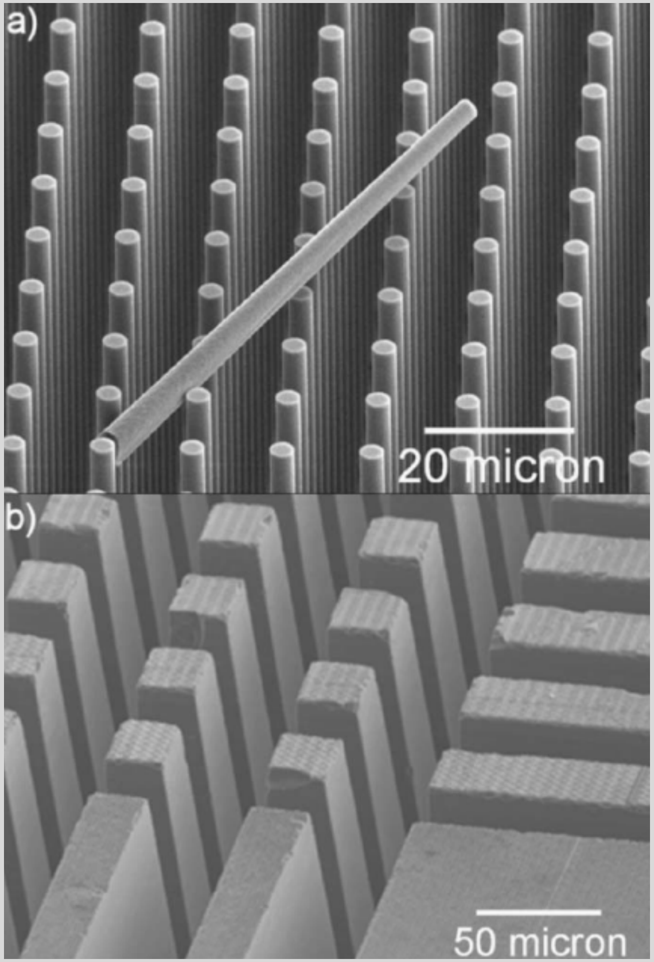


Broken micro-tip arrays.

Information source: communication with Dieter Isheim, Northwestern University (July 2017).

1. **Cost:** $250 / 22-tip array
2. **Design:** marker posts for tip identification interfere with specimen visibility
3. **Fragility:** tips are unprotected in the event of manual mishandling of an array
4. **Flexibility:** customization beyond commercial manufacture may allow joint analysis between APT, TEM, and other characterization equipment

PROJECT OBJECTIVE: Fabricate a microarray satisfying APT parameters.



SEM images of posts cut with (a) reactive-ion etching and (b) a dicing saw, from Kelly *et al.*[3]

Design: Two Tiers

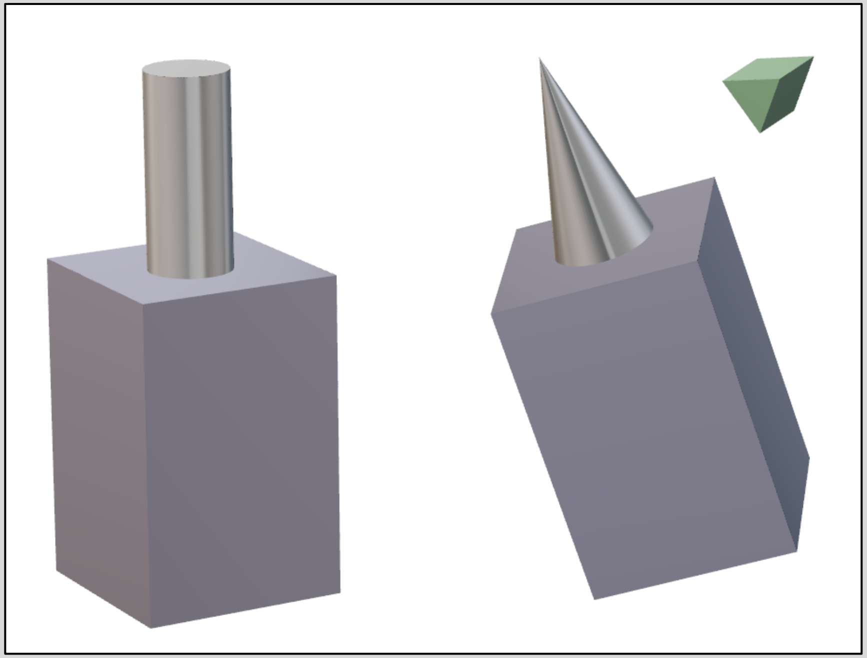
Total Height: 150-180 µm

Top Layer: FTM/needles

- 40 µm height
- ~2 µm tip platform

Base Layer: Angular structure

- 100-200 µm sides
- 110-140 µm height



Simple 3-D mock-up illustrating early design concepts.

Process Overview:

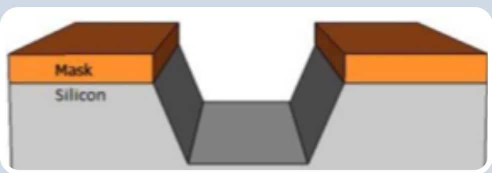
1. Fabricate needles
2. Fabricate base
3. Ensure needles are protected during (1.) and (2.)

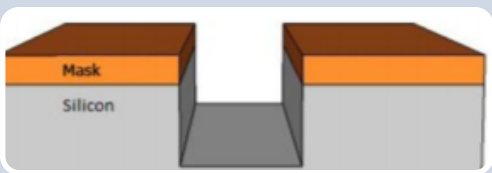
Micro-Fabrication Methods

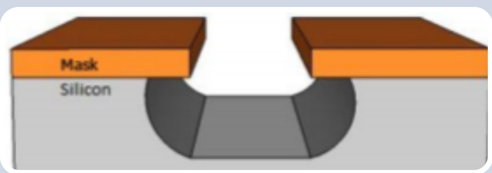
Masking

- Photolithography
- Film deposition (PECVD)

Etching



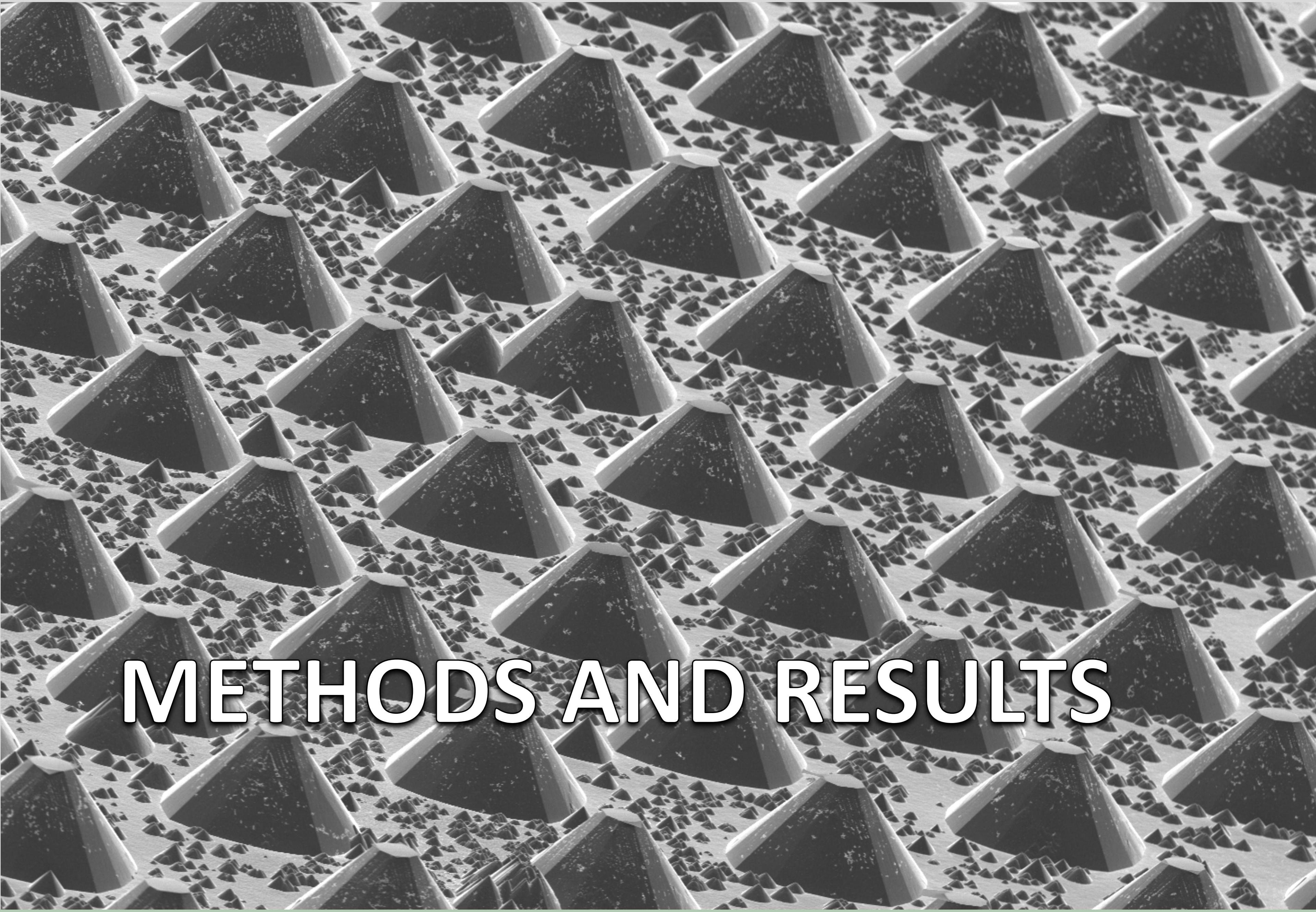




Wet Chemical KOH Etch	Deep-Trench DRIE (SF_6 / CF_4)	Isotropic DRIE (O_2 / SF_6)

Image source: see reference [7].

METHODS AND RESULTS

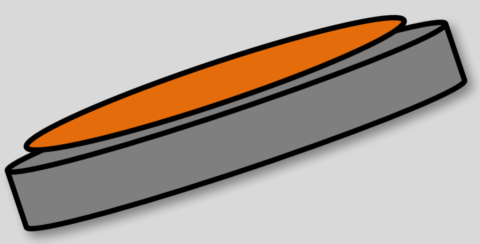


Fabricating: Needles

Multiple etching processes generate the desired needle geometry.

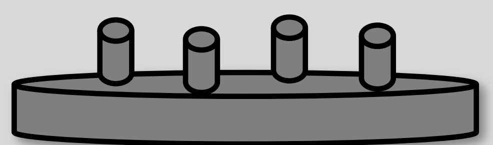
Wafer preparation

- Spin-coat photoresist
- Expose with laser writer



Use DRIE to produce cylindrical columns

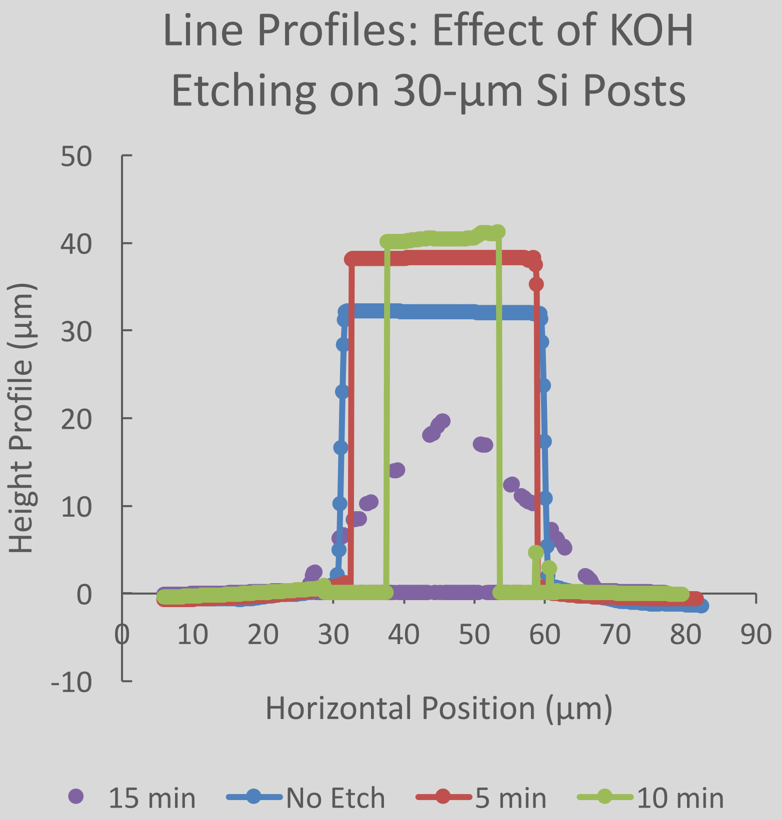
- Short periods of deep-trench recipe
- Results in ~40-µm-tall posts

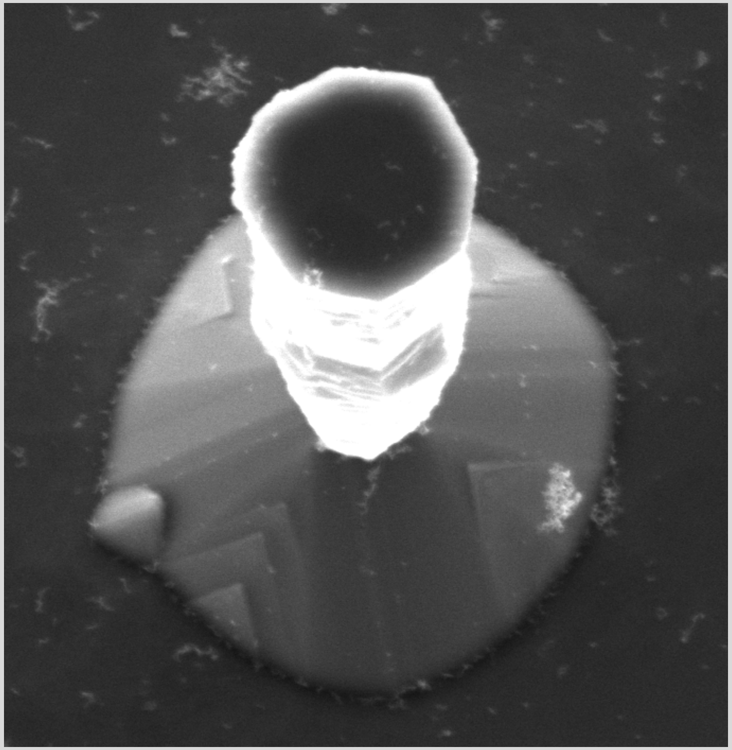


Etch to needle points

Method 1: Wet KOH Etching

Goal: sharpen cylinders into FTMs or needles.

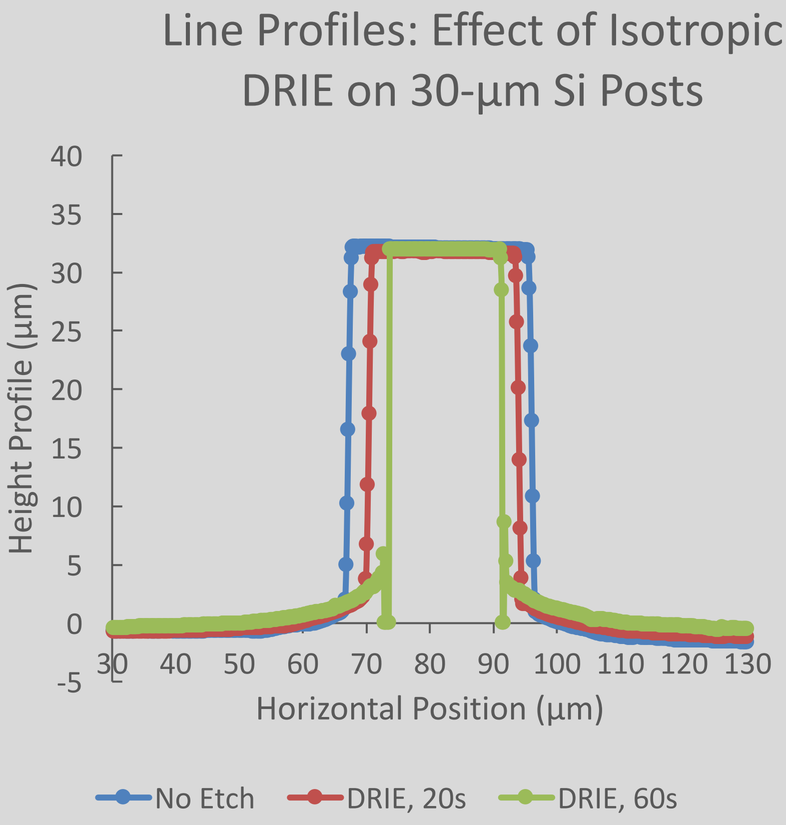


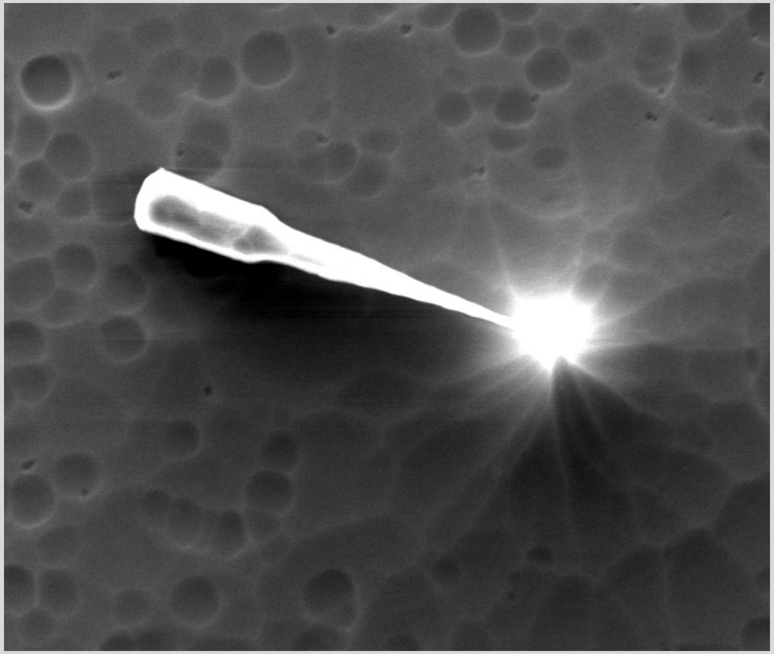


SEM image of KOH-etched Si post, 4448X.

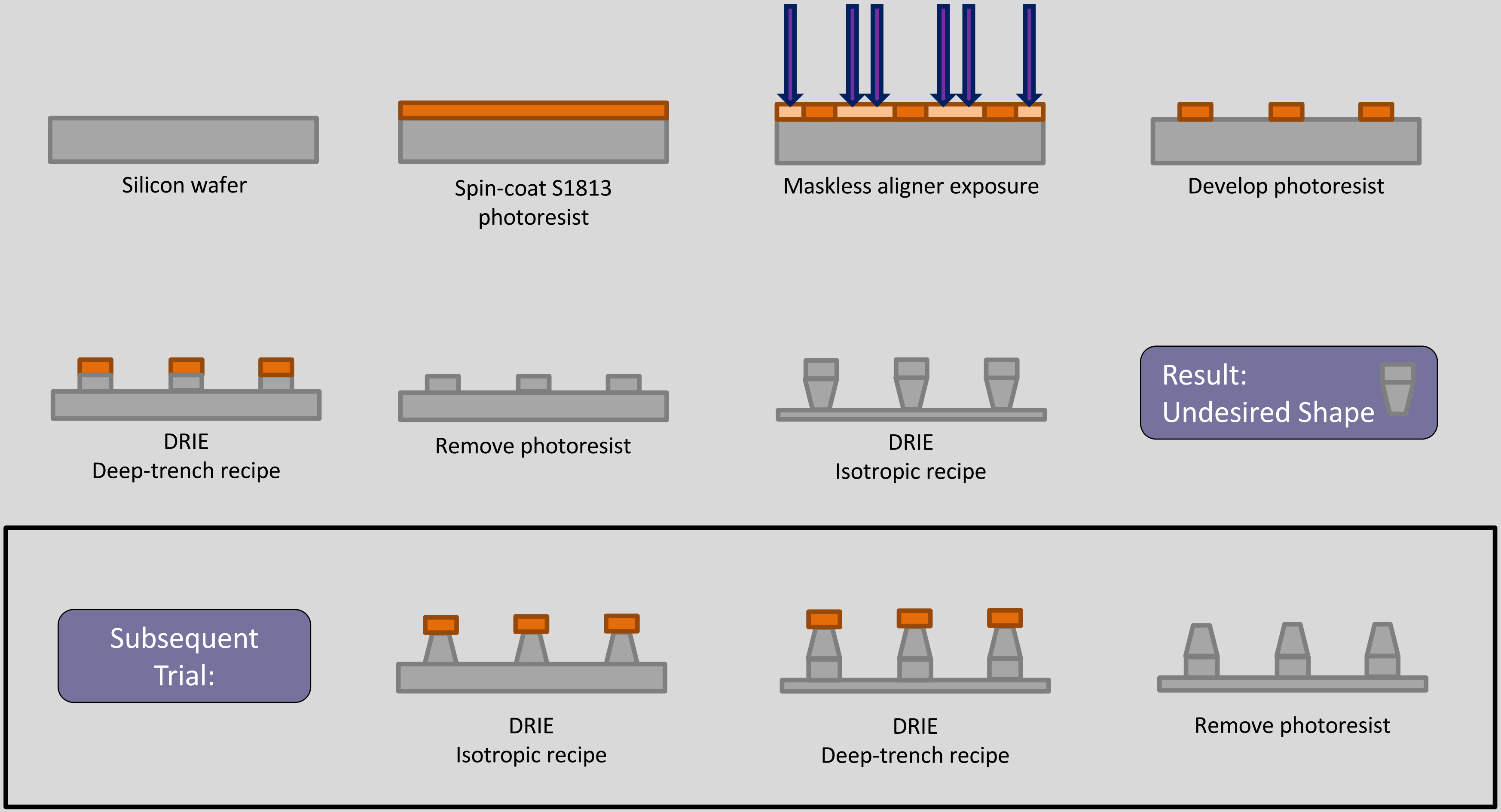
Method 2: Isotropic DRIE

Goal: sharpen cylinders into FTMs or needles.

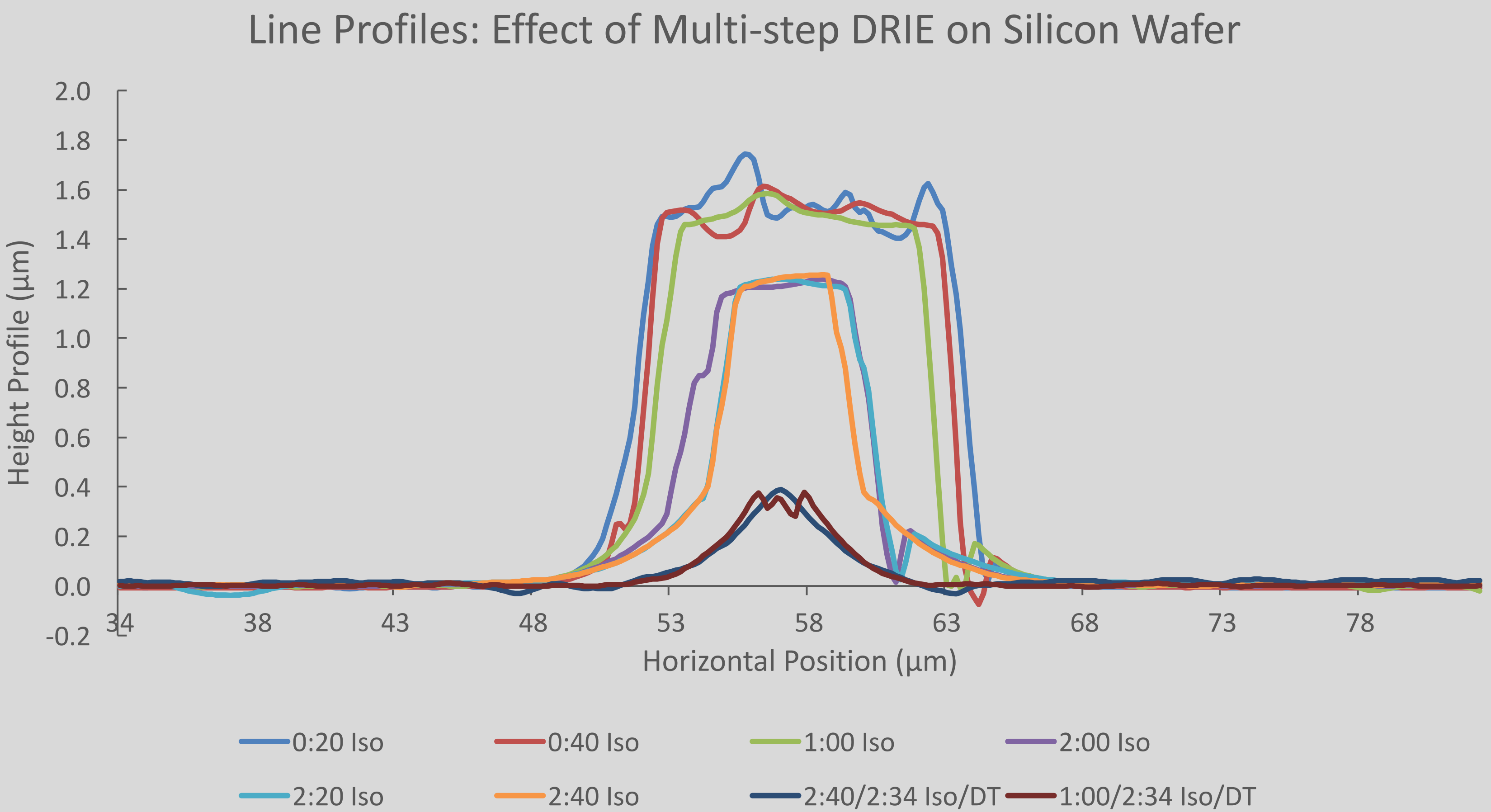




SEM image of DRIE on 15-µm Si post, 4695X.


Silicon wafer
Spin-coat S1813 photoresist
Maskless aligner exposure
Develop photoresist
DRIE Deep-trench recipe
Remove photoresist
DRIE Isotropic recipe
Result: Undesired Shape
Subsequent Trial:
DRIE Isotropic recipe
DRIE Deep-trench recipe
Remove photoresist

Method 3: Reverse-Order DRIE



Needle Structures: Conclusions

- **Wet chemical KOH etching is not viable** for producing needle-like structures at the desired scale
- **Isotropic DRIE is viable** for producing a needle-shaped structure, but will not result in the desired height without further processing

Looking ahead:

- Continue to explore deep-trench and isotropic DRIE parameters

Fabricating: Base Structures

Simulation: KOH Etch of Base Structures

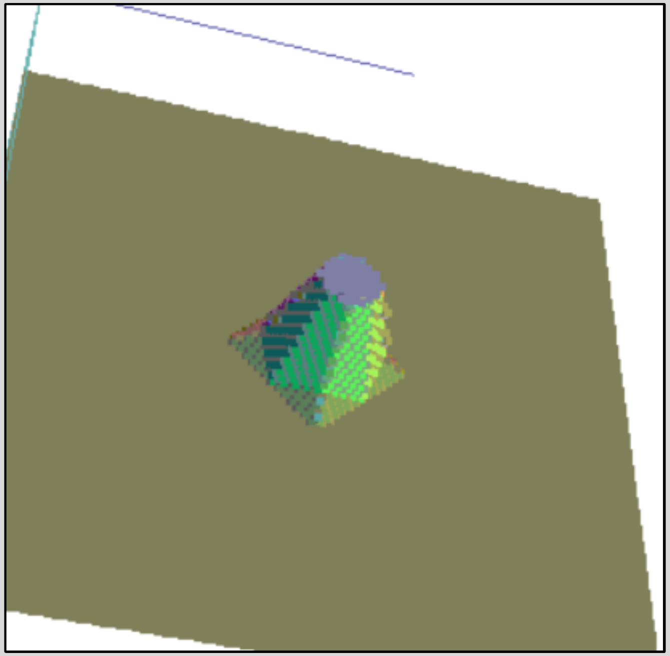


Fig. 9: Simulation of 30% KOH wet etch on Si using Anisotropic Crystalline Etching Simulation (ACES) software.[9]

Parameters:
<100> face and <110> edge wafer
5-minute increments at 1.4 μm/min

(Note: tested etching rate for the procedure of this project is approximately half the simulation rate.)

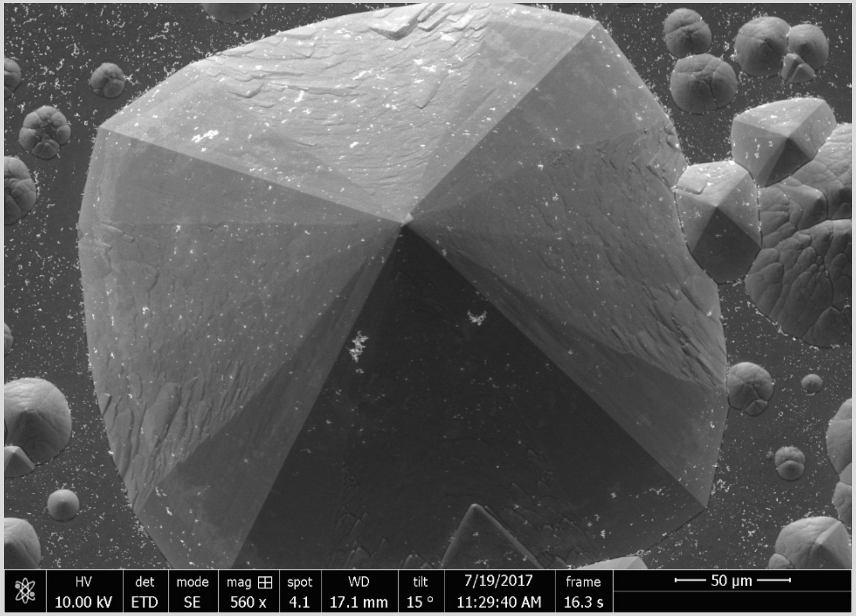
Fabricating: Base Structures

- Etching rate of 30% KOH soln. at 85°C: **~40 µm/hr**

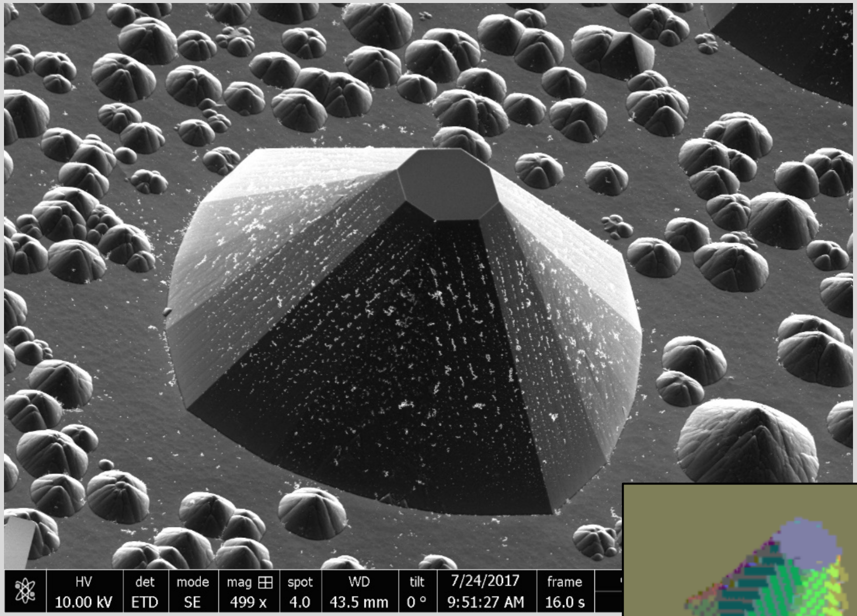
- **3 hrs of KOH etching** will produce a 120-µm structure

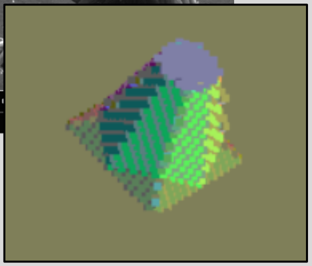
Fabricating: Base Structures

3 Hr KOH Etch (100 μm square)
Over-etched



2 Hr KOH Etch (100 μm square)
Correct geometry





Base Structures: Conclusions

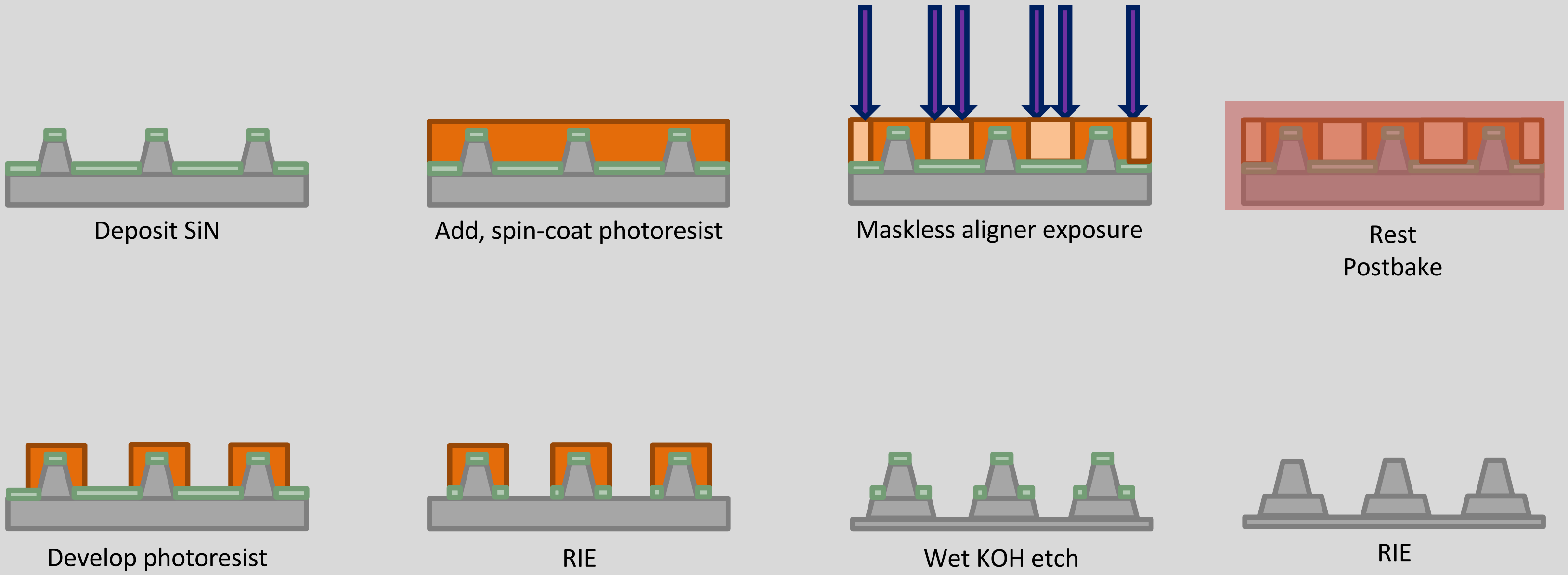
- **Wet chemical KOH etching is viable** for producing large structures to support needles
- **3 hours of etching** (under stated conditions) will produce the desired height

Looking ahead:

- Use larger mask size to retain platform

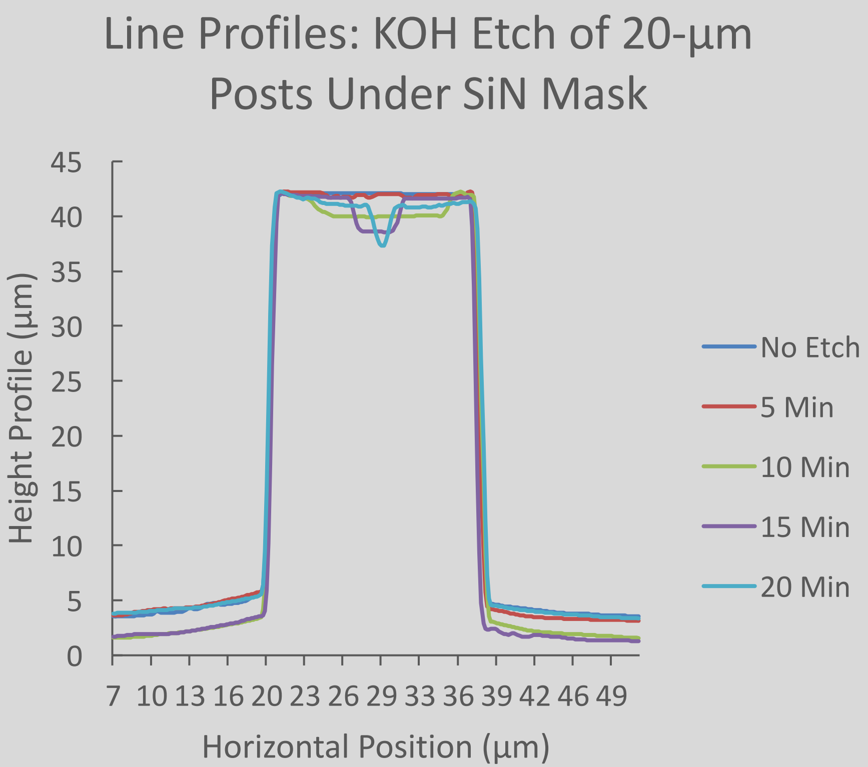
Test: Needle Protection

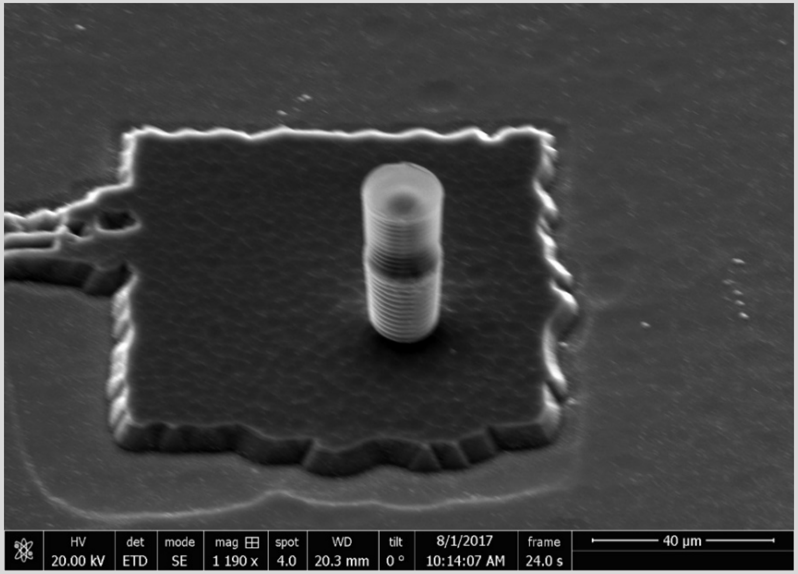
Q: Is SiN masking effective at protecting upper needle structures during KOH etching of the base?



Test: Needle Protection

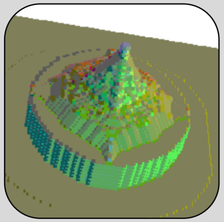
Result: adequate protection, inadequate photoresist





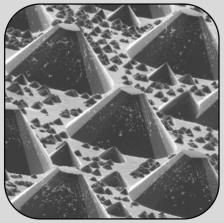
Conclusions and Further Research

Microfabrication techniques can produce components of a **multi-tiered APT micro-array**



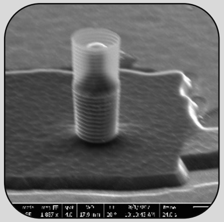
DRIE (isotropic and deep-trench) for needle structures

Optimize height



KOH etching for base structures

Use larger mask



SiN masking to protect upper structures and combine the two processes

Use thicker photoresist

Acknowledgements

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SHyNE Soft and Hybrid Nanotechnology Experimental Resource

NUFAB NORTHWESTERN UNIVERSITY MICRO/NANO FABRICATION FACILITY

NUANCE Atomic and Nanoscale Characterization Experimental Center

NUCAPT Northwestern University Center for Atom-Probe Tomography

References and Image Sources

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