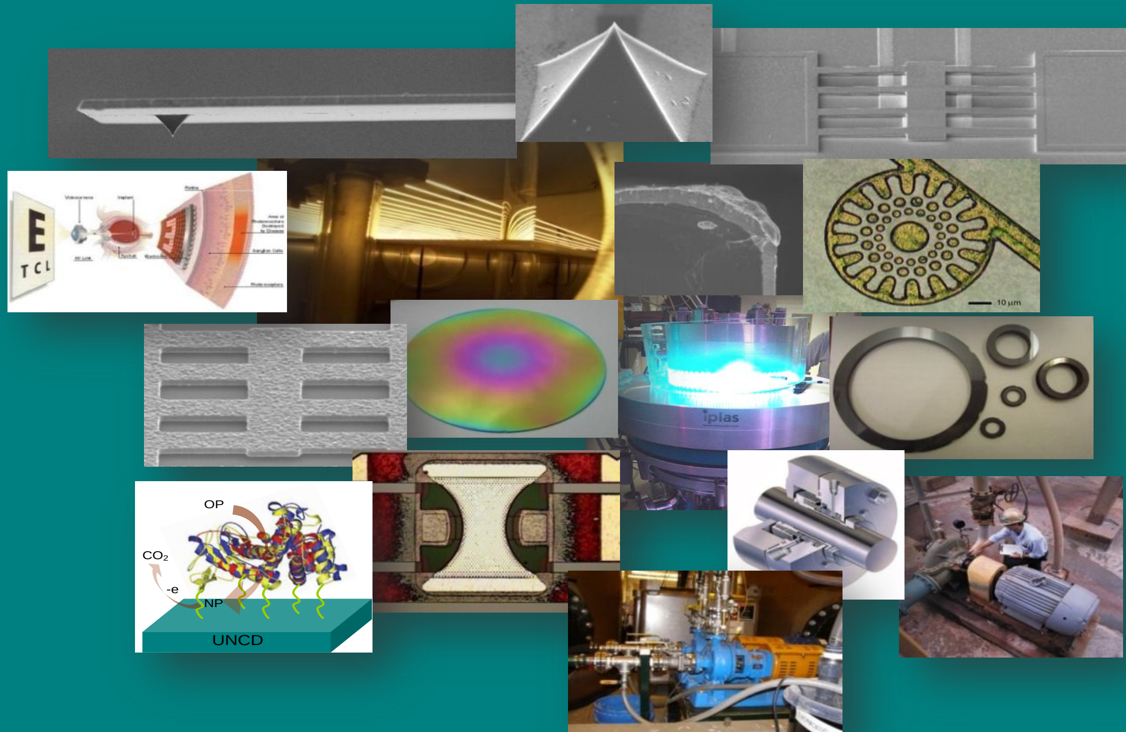

ICP-RIE of Ultrananocrystalline Diamond

ADVANCED DIAMOND TECHNOLOGIES, INC.

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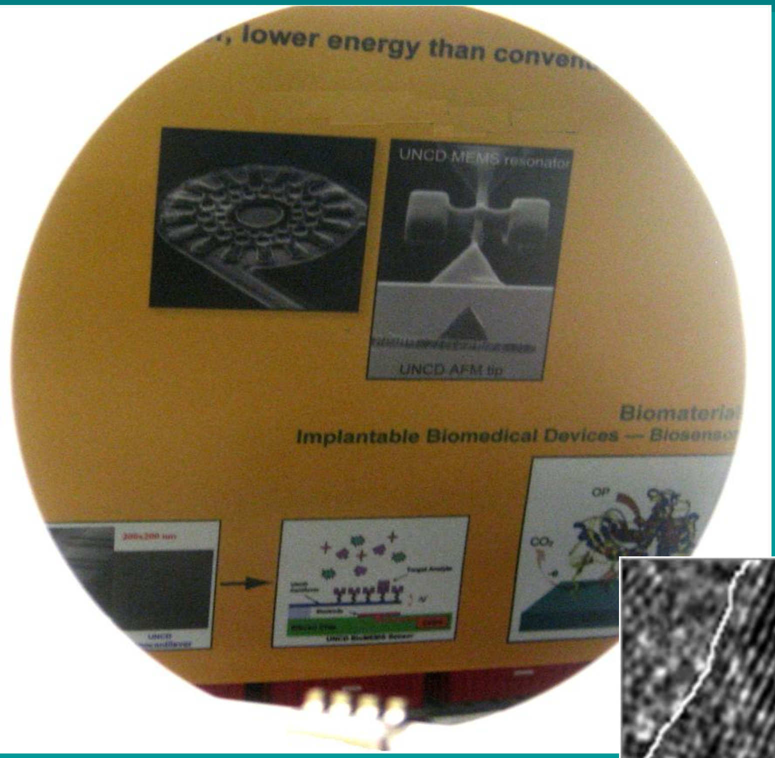
*We haven't even scratched the surface*SM

NCCAVS Plasma Applications Group Meeting, San Jose, Febr 10, 2010

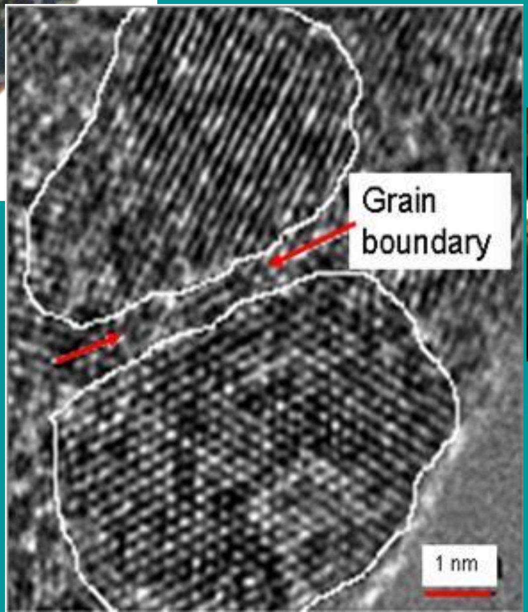
Outline

- Motivation
- Preliminary thoughts on direct etching
- Optimization of diamond etching with ICP-RIE
- Discussion of tip etching results
- Conclusions

Ultrananocrystalline diamond (UNCD)

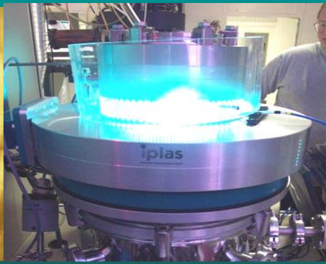


- Smooth: 3-20 nm rms as grown, <1 nm "Horizon"
- Thickness: 40 nm to 20 μm; <5% uniformity
- Tunable electrical and thermal conductivities
- High Young's modulus (~900 GPa)
- Biocompatibility
- Up to 300 mm wafers
- Most of bulk diamond mechanical properties



- Deposited at 400-900 ºC

HFCVD



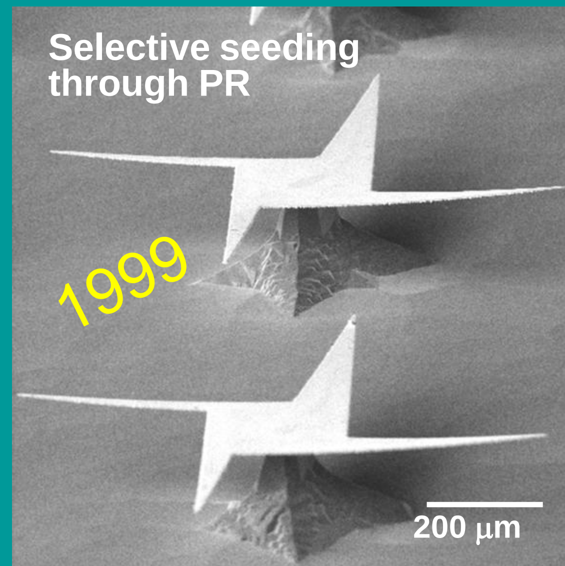
MPCVD

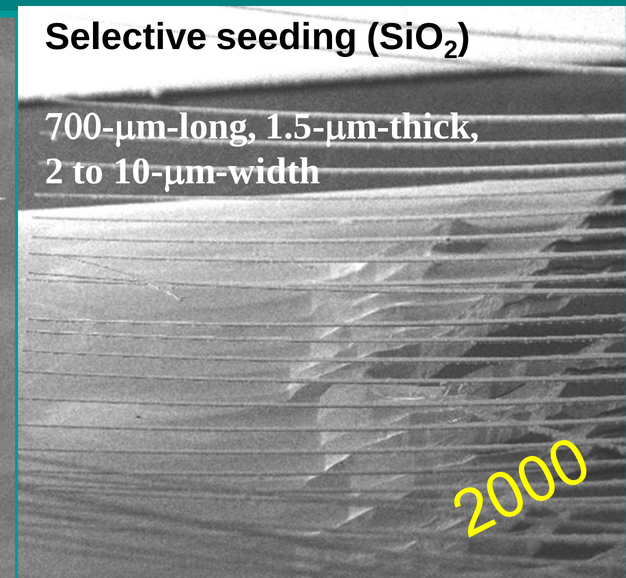
UNCD micro-manufacturability

- Selective seeding through photoresist or SiO_2 mask

  Rounded edges

  ~1-2 μm resolution

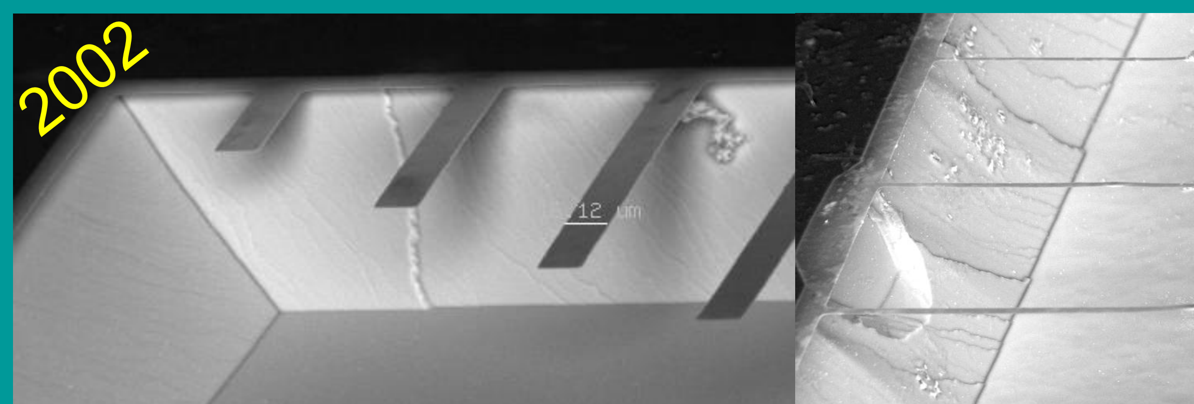




- O_2-RIE through Al mask

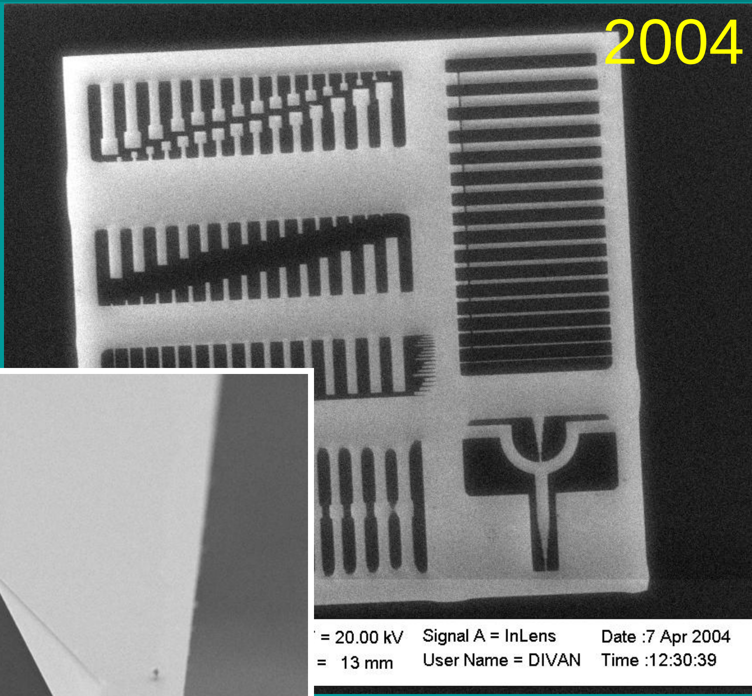
  Vertical edges, rough at the nanoscale

  ~ 1-2 μm UNCD

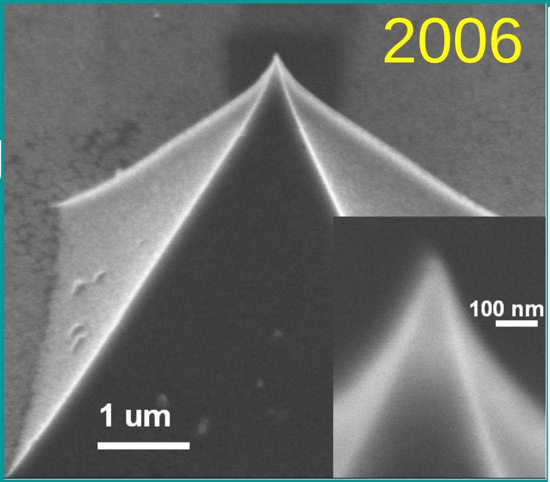


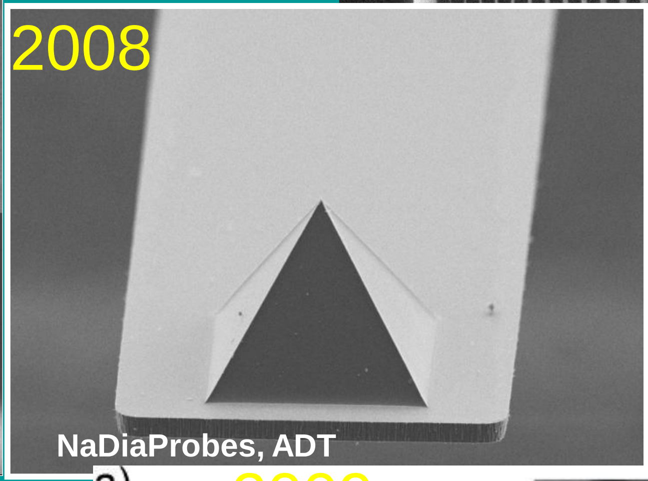
UNCD nano-manufacturability

- E-beam lithography, lift off (Al, Ni), O_2-RIE of UNCD, release by KOH (XeF_2) etch

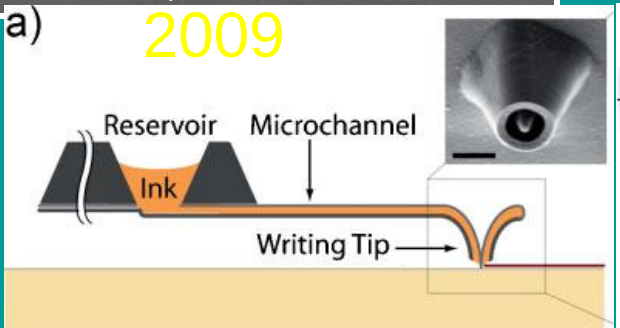


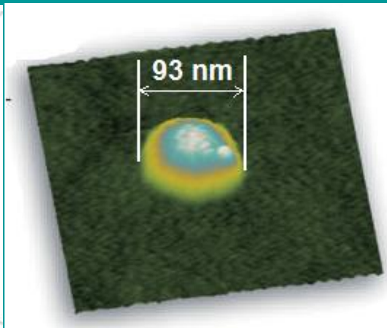
- Molding



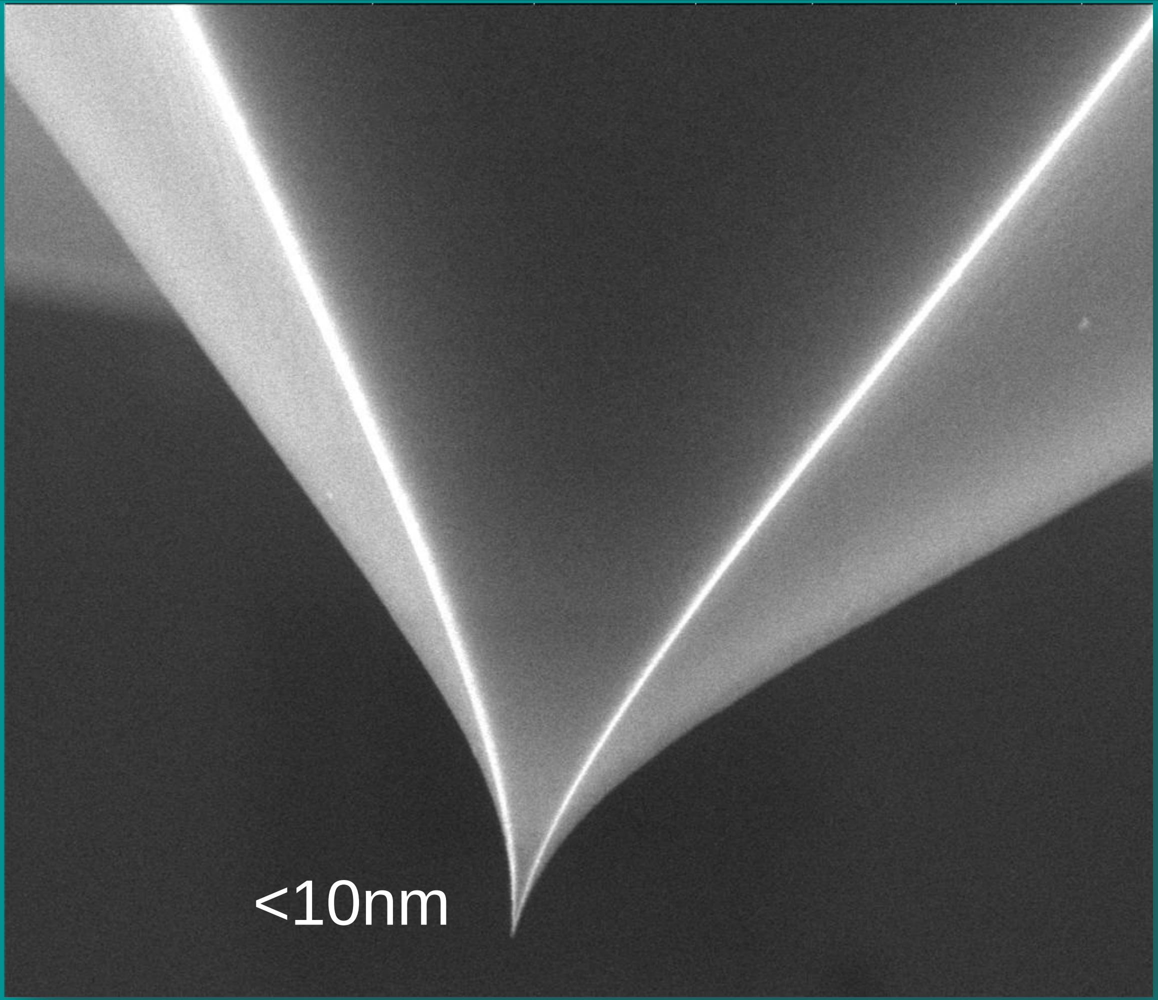


- Tip-based nanopatterning of seeds and growth



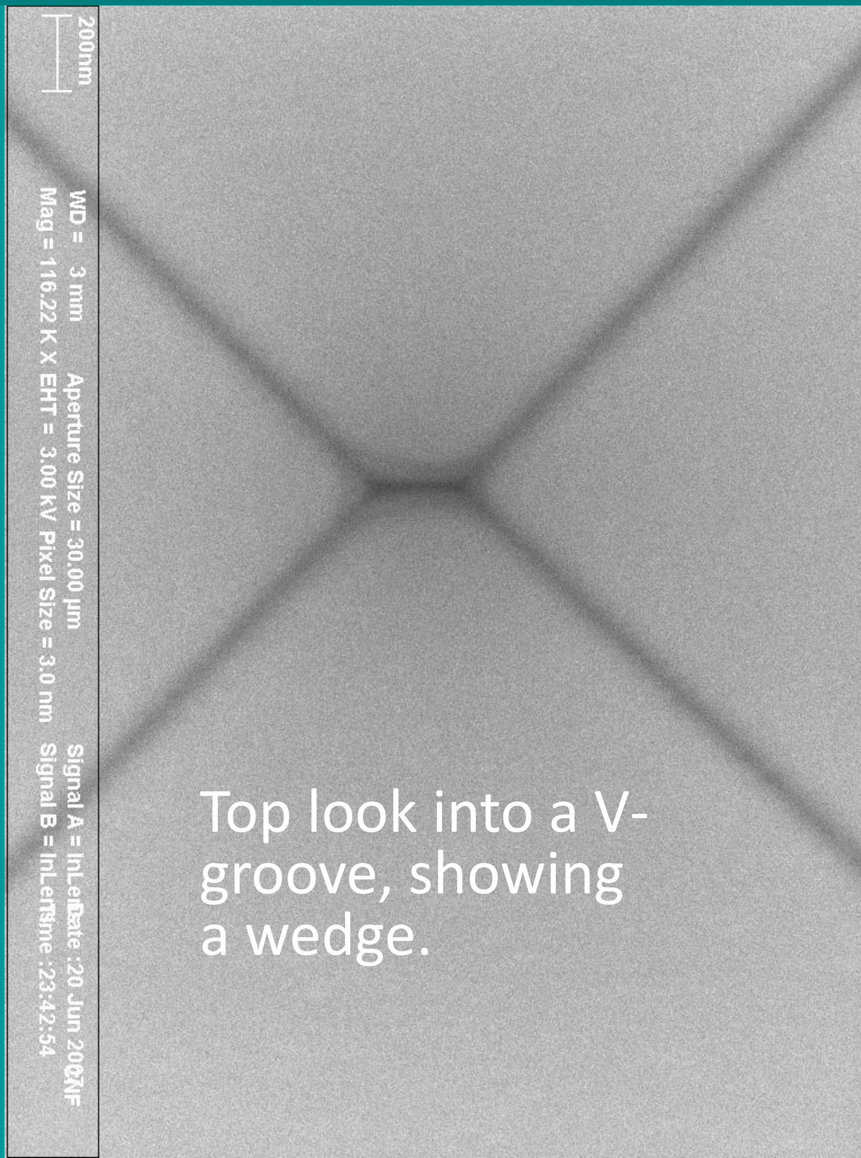


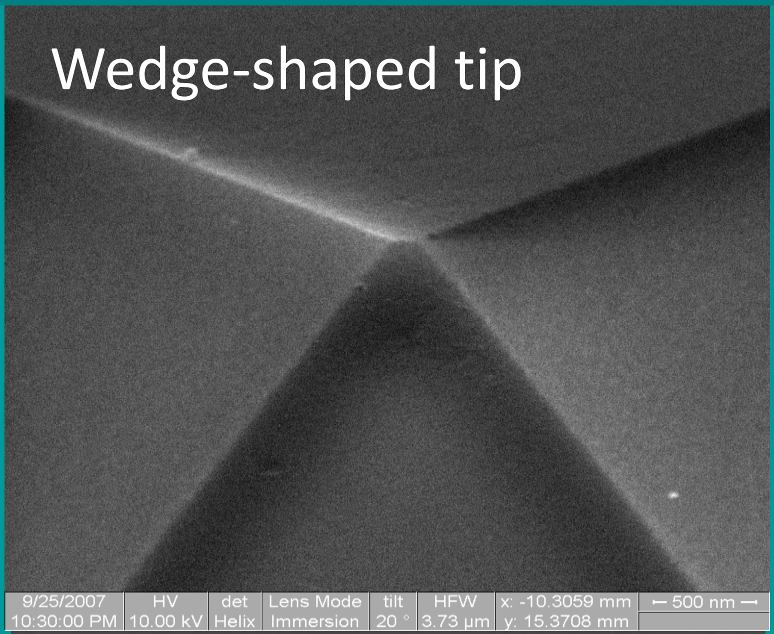
Why is molding not good enough?

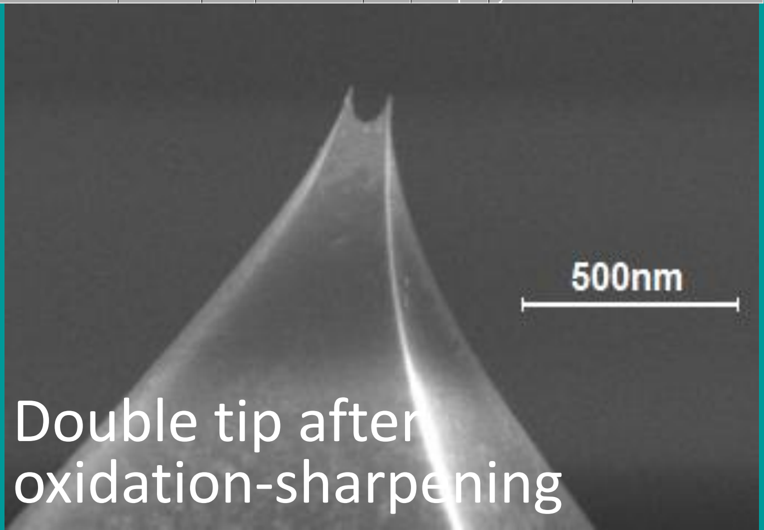


It can be spectacular, but…

Why is molding not good enough?

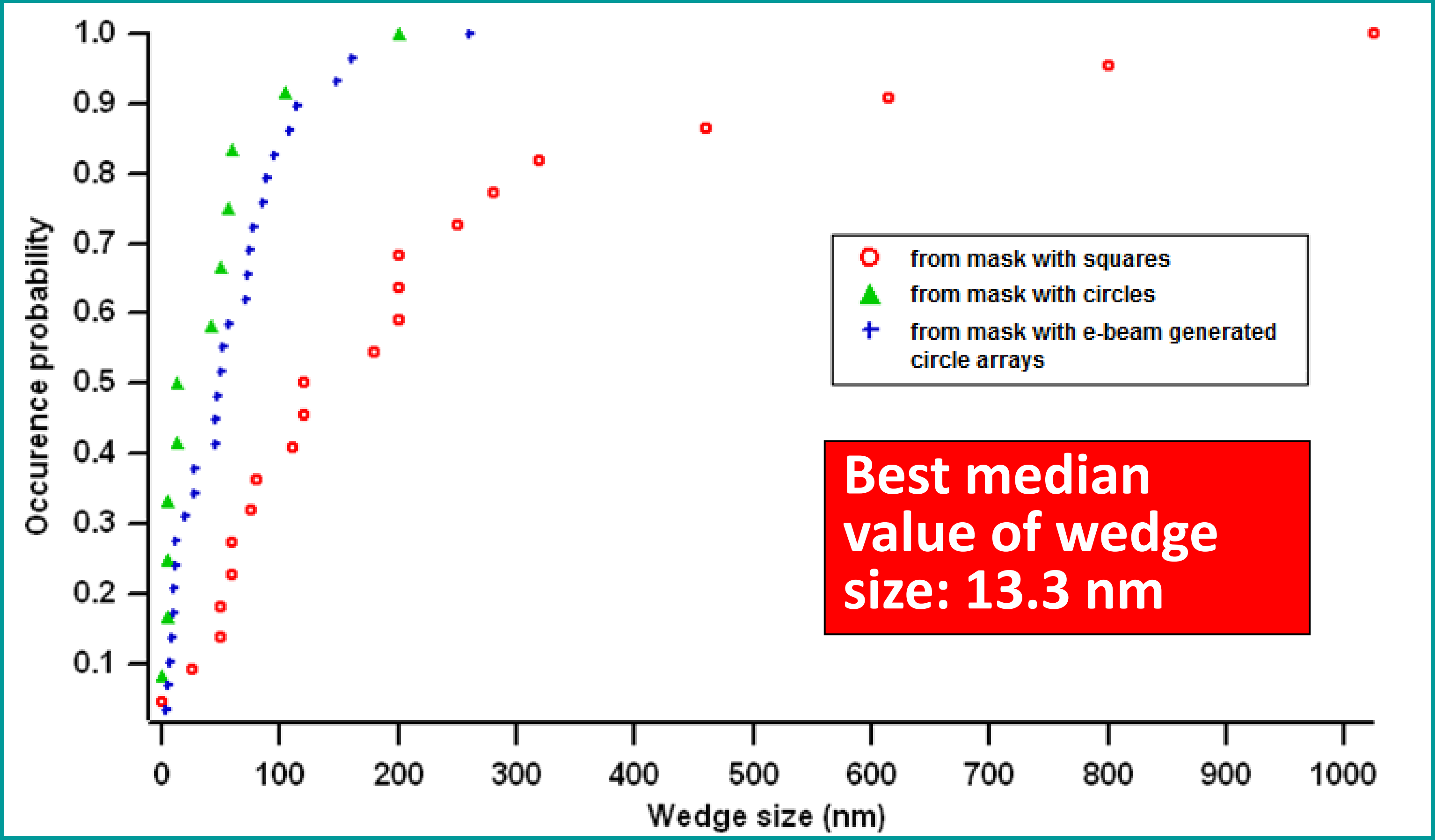






Why is molding not good enough?

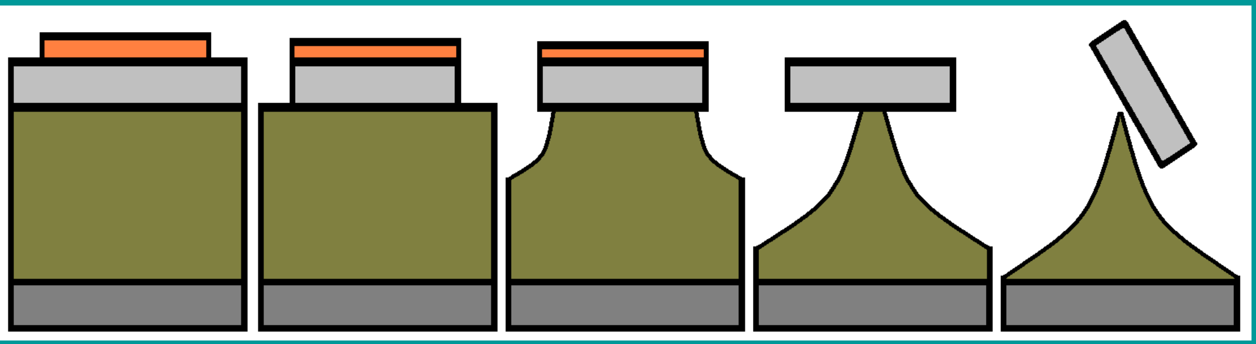
Statistics

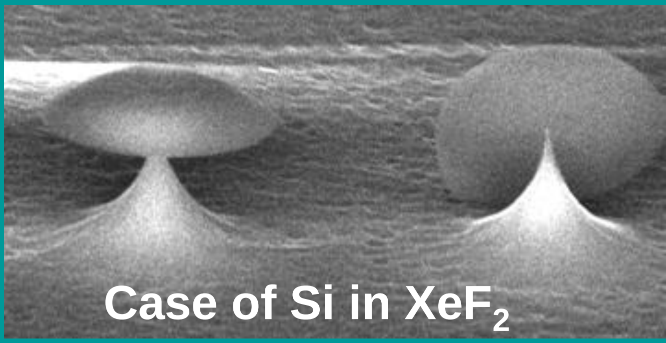


Preliminary thoughts on direct etching

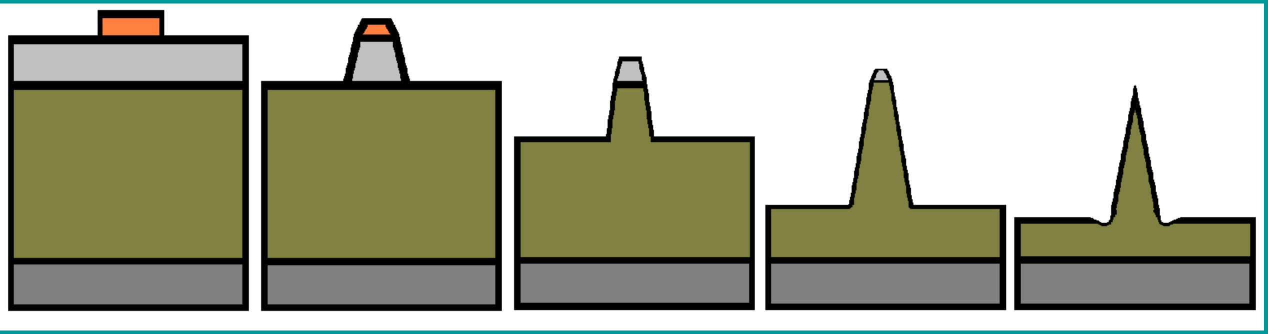
Two ways:

- Isotropic underetching





- Slope amplification by differential etching & sharpening



3-7 μm diamond

UNCD ICP-RIE – needs optimization

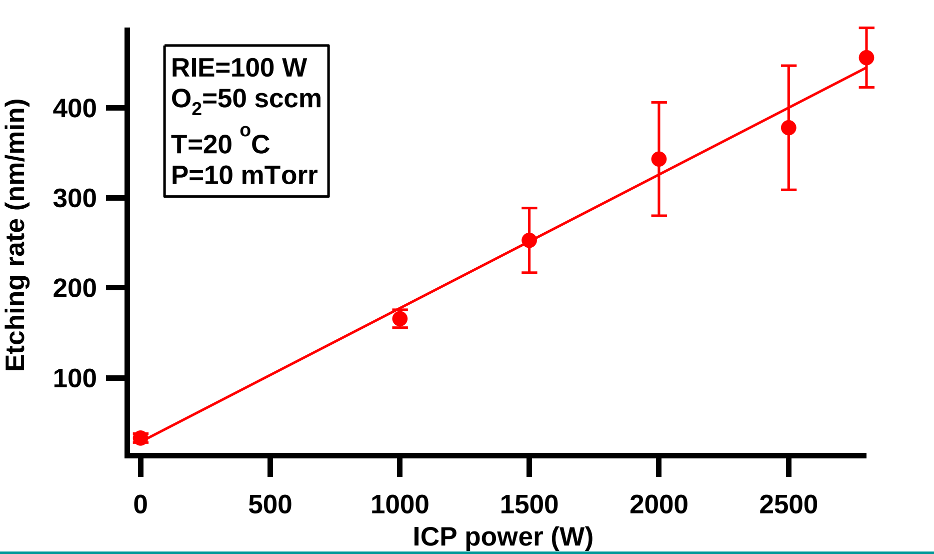
Our experience:

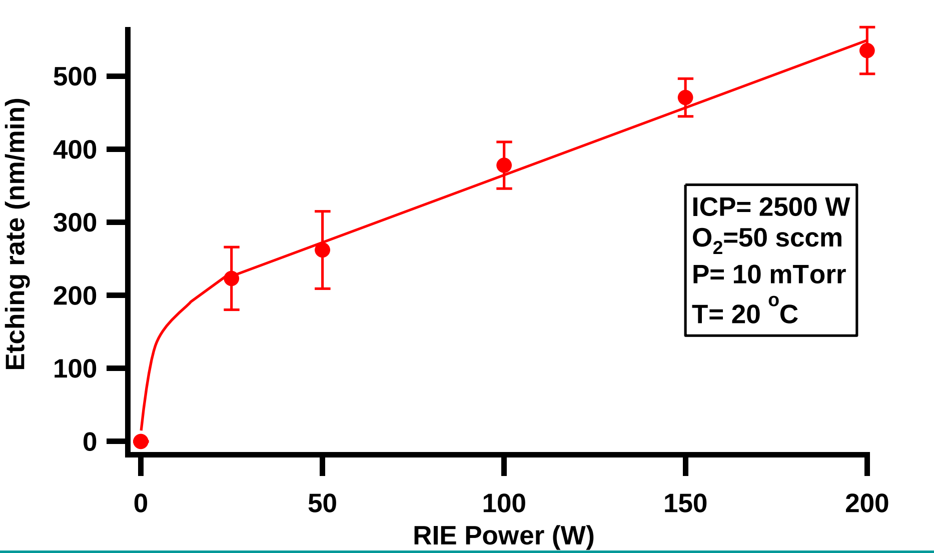
- RIE : 1μm in 30 min (33 nm/min)
  O_2:CF_4 (50:2 sccm), 300 W, 20 mTorr
- ICP-RIE: 1 μm in 10 min (100 nm/min)
  O_2:CF_4 (50:2 sccm), ICP= 2800 W, RIE=100 W, 20mTorr

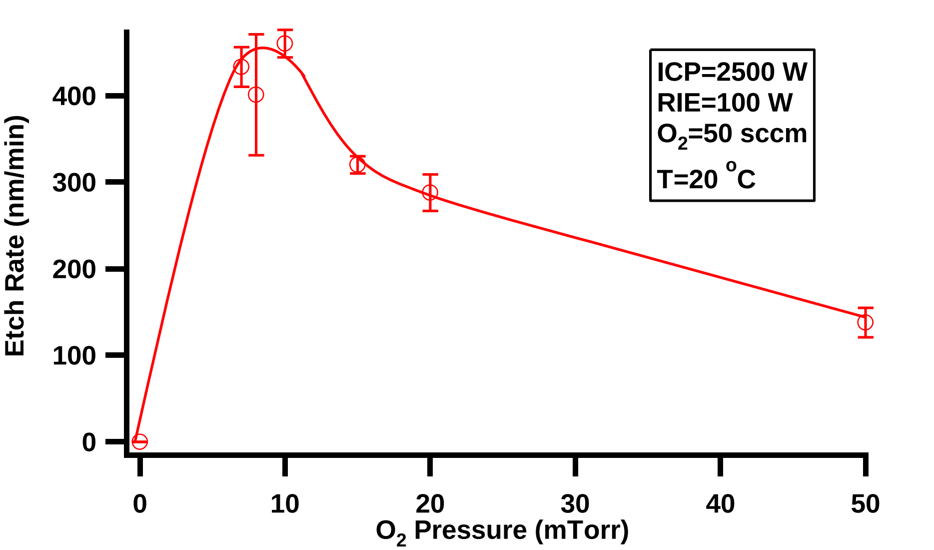
Literature:

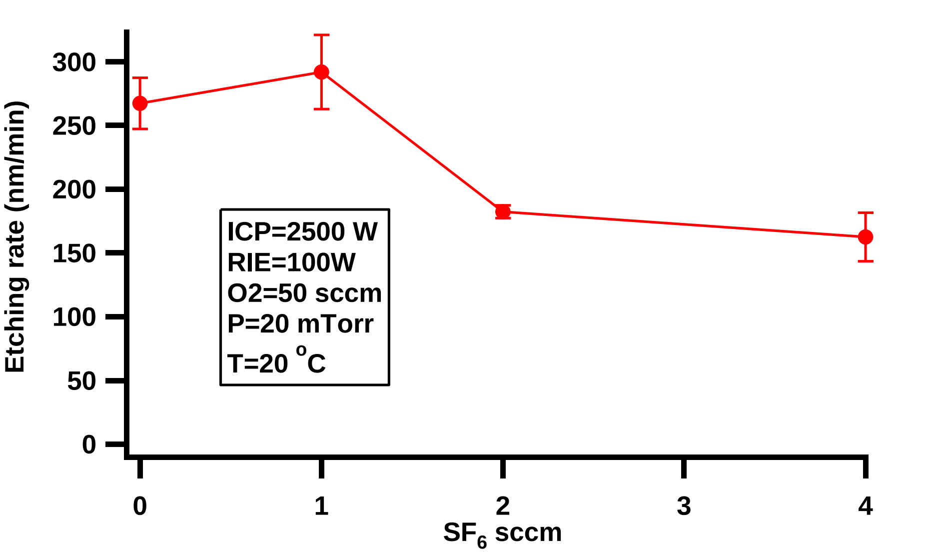
- O_2-RIE: 30-40 nm/min
- ECR O_2 plasma, 400 nm/min
  (Pearton et.al. Electron. Lett. 28, 822 1992.)

Optimization of ICP-RIE, Oxford 100



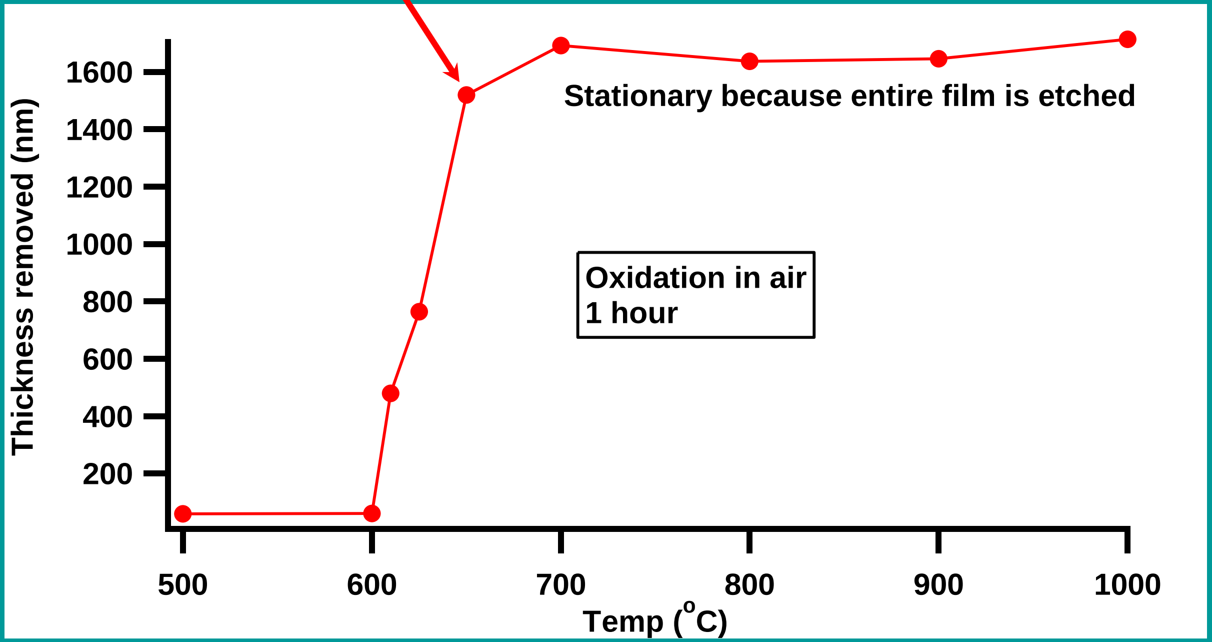




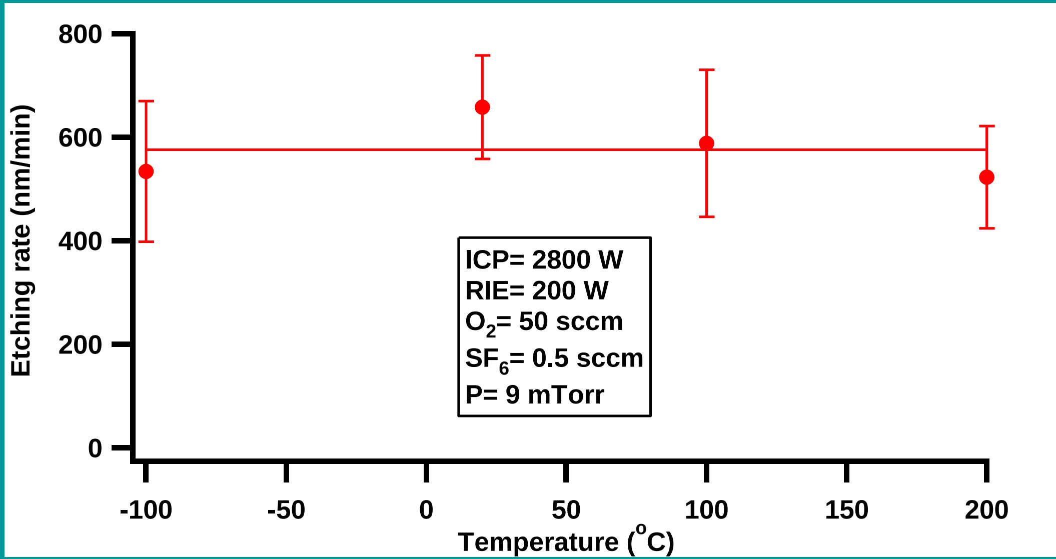


Thermal oxidation of UNCD in air

- **Diamond etches at > 600 ºC in air**
  (but slowly: 25 nm/min)

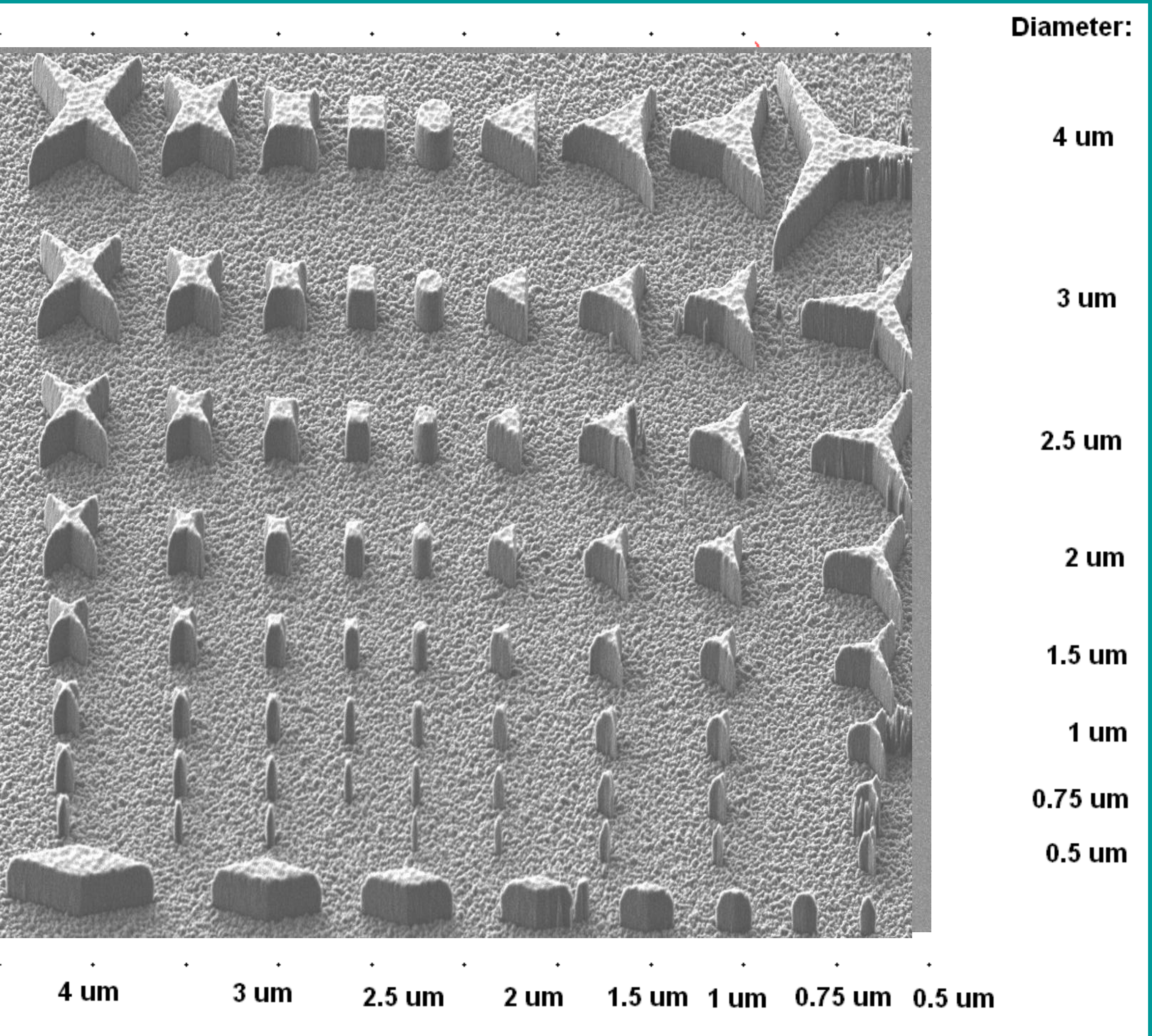


ICP-RIE rate with temperature



- Almost no temperature dependence
- Isotropy unlikely in this regime
- Machine tripped above 300 ºC (cooling problems)
- Do the isotropic etching by thermal oxidation sequentially

Investigating isotropy: test mask



- 100 μm x 100 μm field
- 3.5 μm UNCD/350 nm PECVD SiO_2
- Exposed with Raith 150, (30 keV, 60 um aperture, 1554 pA, dose 400 μC/cm^2)
- Resist: ma-N 1405 (MicroChem) @ 2000 rpm, baked at 100ºC for 90 sec, thickness 350 nm, developed 30 sec in mD533S
- SiO_2 etched by ICP-RIE (CHF_3-Ar plasma)
- UNCD etched half way for rate measurement, then continued to full removal

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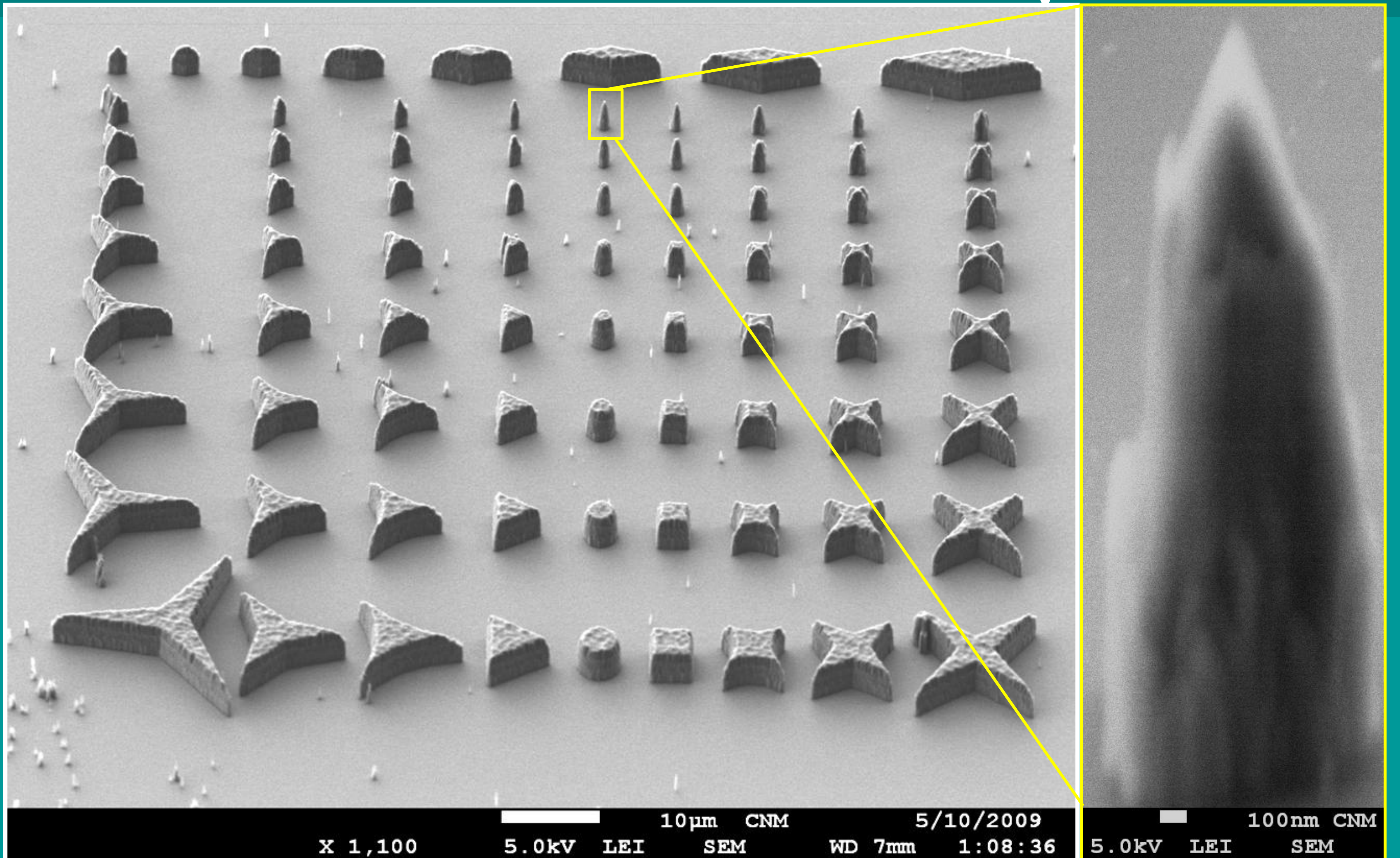
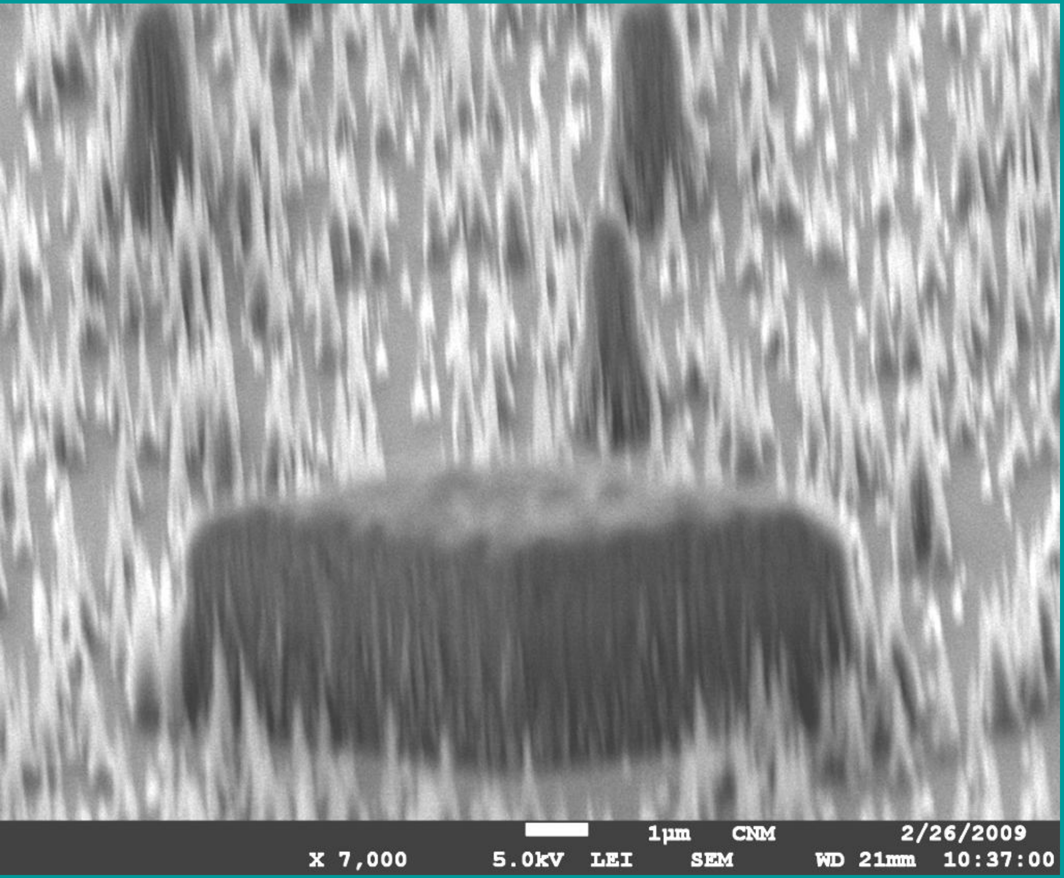
Etched structures on 3.5 μm films



Etched structures: best conditions for isotropy



- **No sign of underetching**
- **No sign of isotropy**
- **Ultra-sharp "grass"**
  originating from SiO_2 in crevasses (~50 nm) on the surface, generating spikes by differential etching mechanism
- **Mostly vertical walls,**
  except the top of structures, where edges are curved by differential etching when the oxide vanishes

Sample etched incompletely at 300ºC
ICP=2500W, RIE=50 W, P=50 mTorr , O_2=50 sccm

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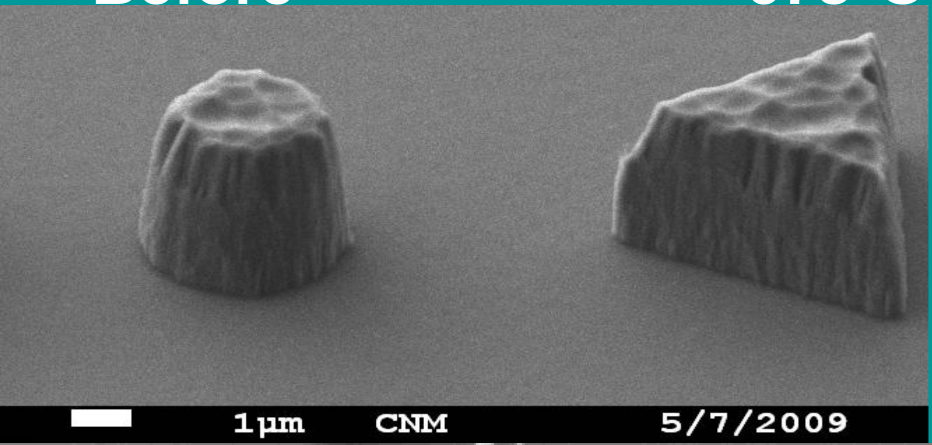
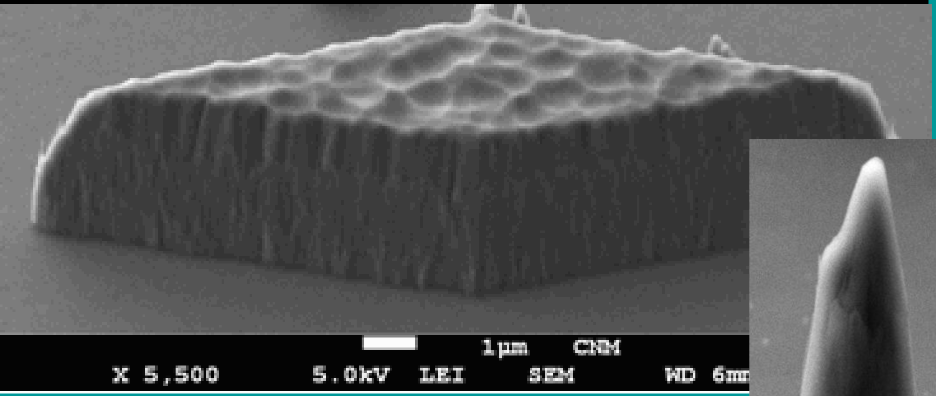
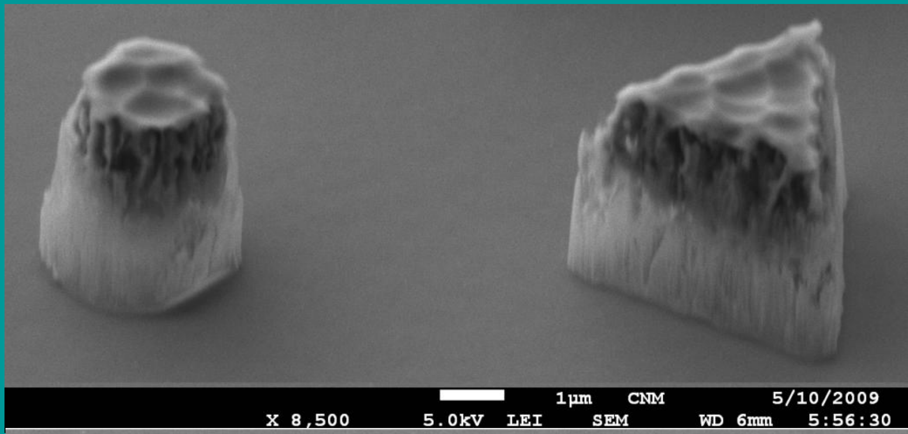
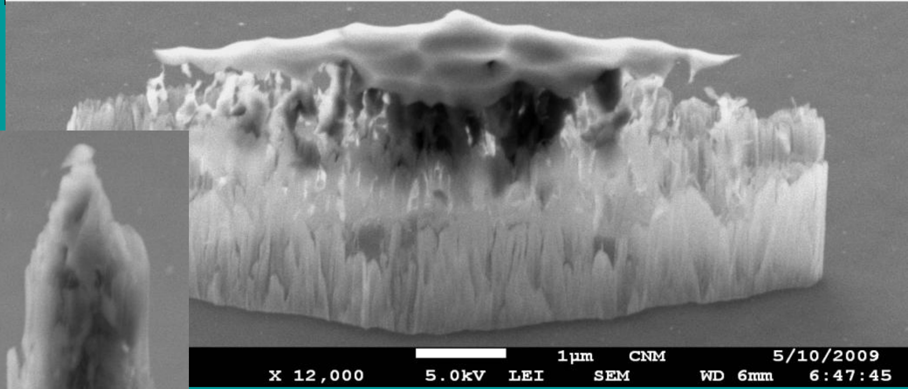
Thermal oxidation after ICP-RIE

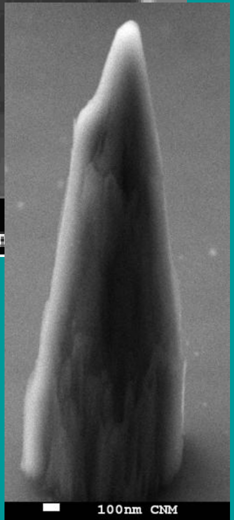
Before **675ºC 30 min, air** **After**

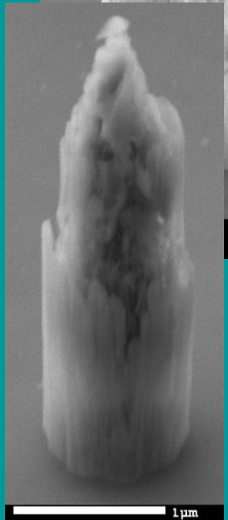








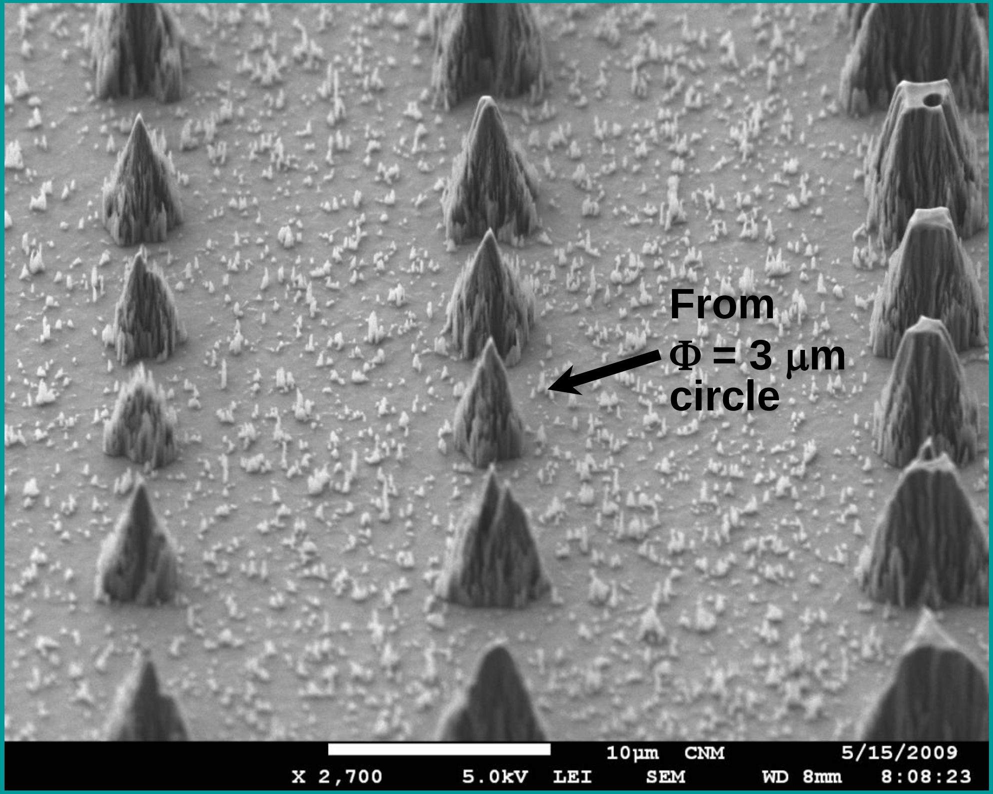


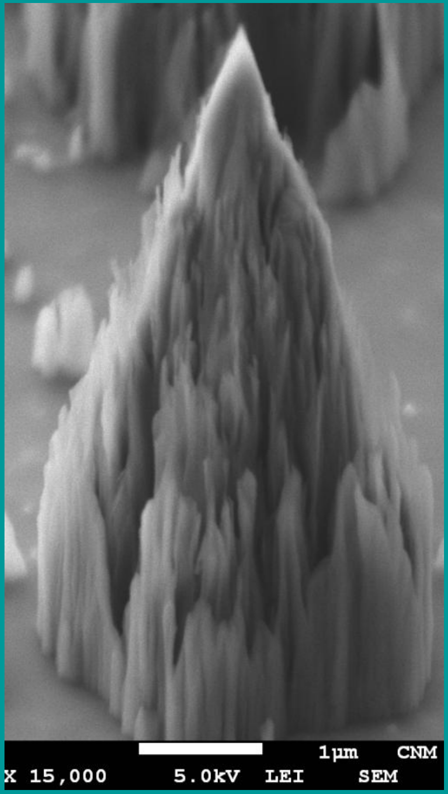


Oxidation proceeds by roughing the surface and generation of cavities

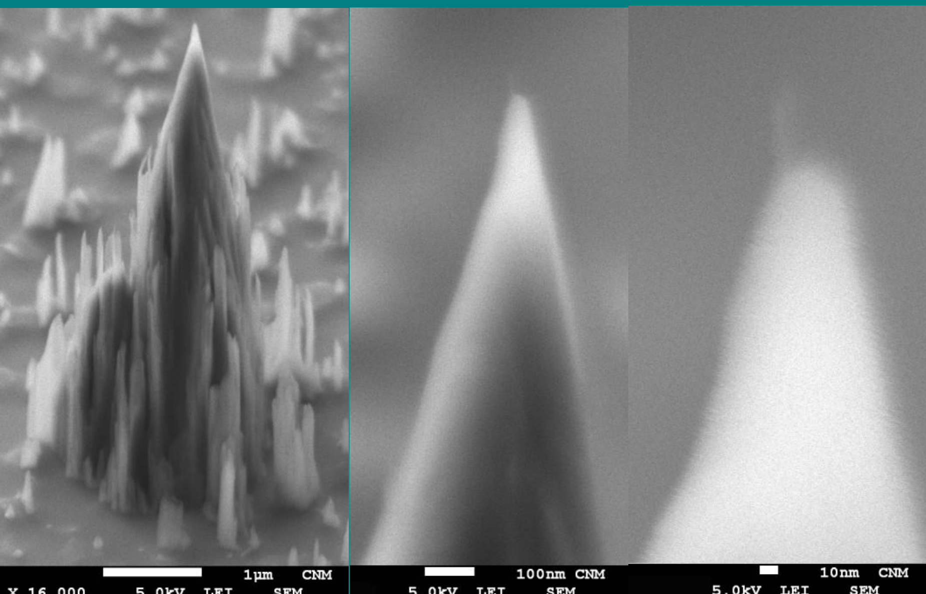
Boosting differential etching

- **6.5 µm of UNCD**
- **350 nm of SiO_2**
- **ICP= 2800W, RIE= 200 W, P= 9 mTorr**
  SF_6= 0.5 sccm, O_2= 50 sccm, T= 20 ºC, 12 min



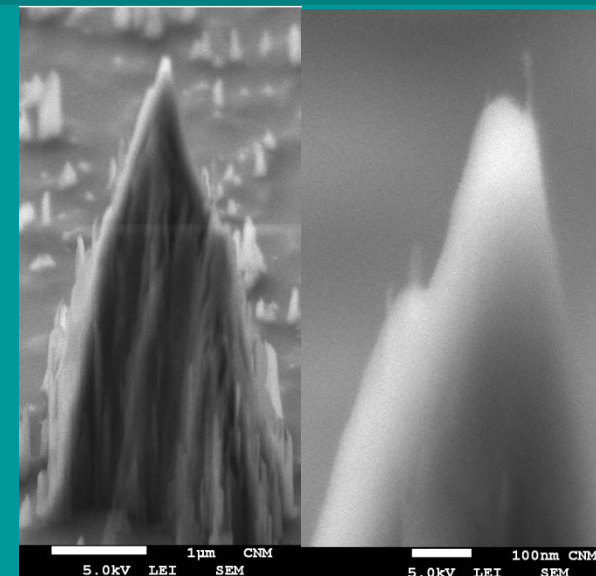


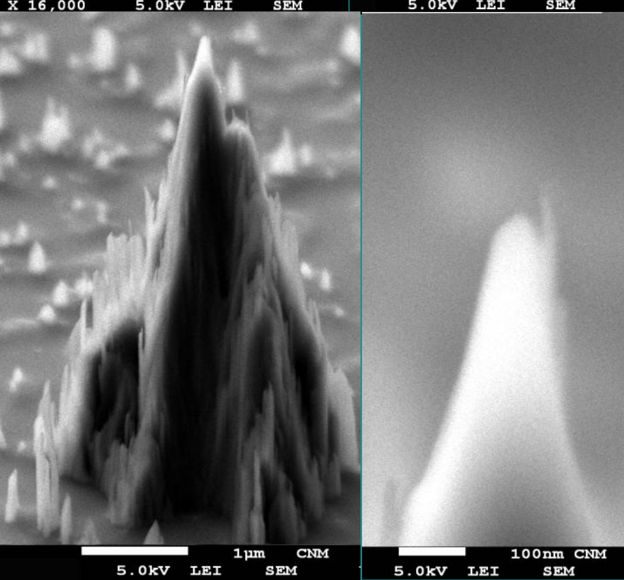
Pleasant surprises: Nanowire ending tips

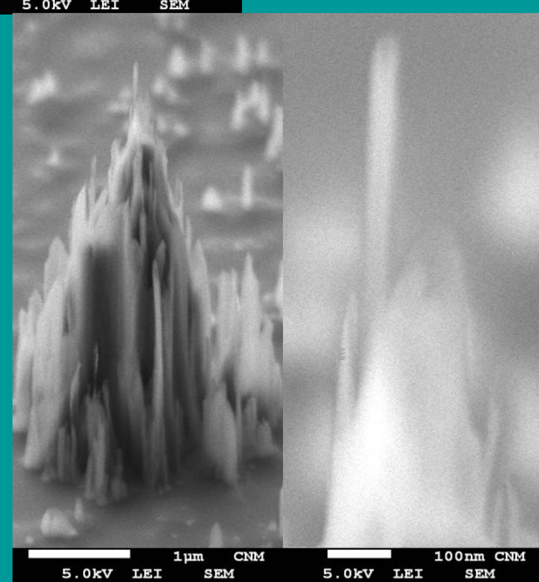


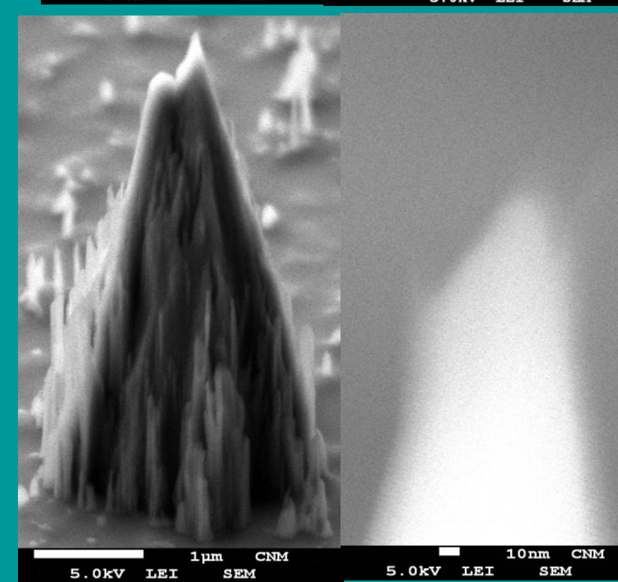
Are not that rare!

14%?

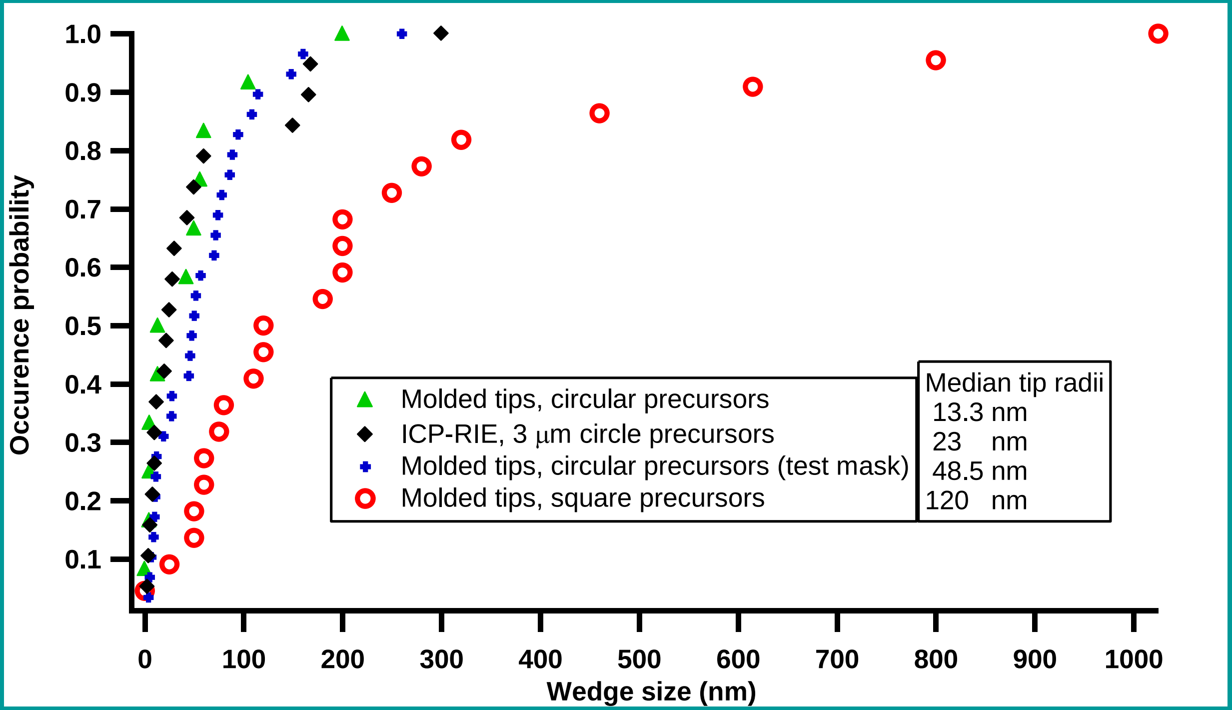








Statistics rules!



- Tips considered: from the 6.5 μm batch, 3 μm circles
- ICP-RIE tips are somewhat worse than the best molded tips
- ICP-RIE tips statistics improved by the NW-ending tips

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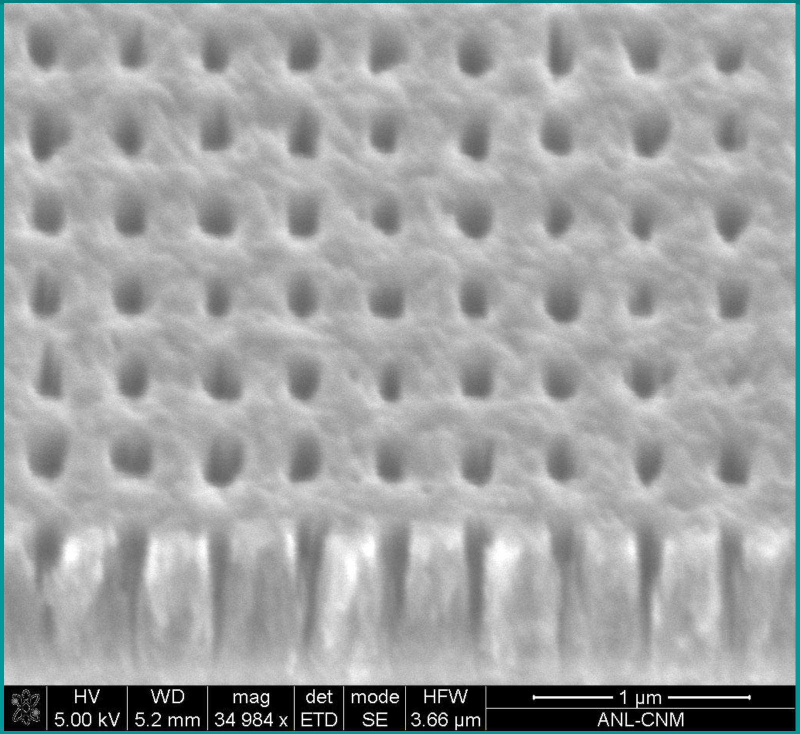
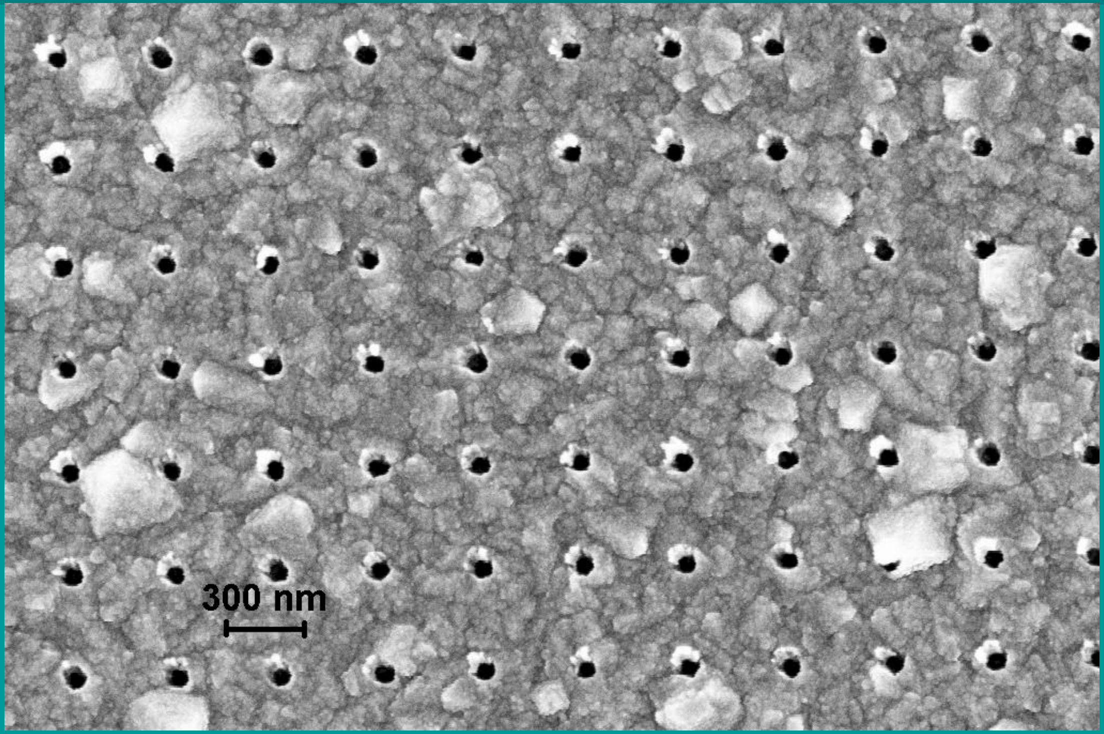
ICP-RIE: etching of pores*





100-nm-diameter pores

80 nm pores

1 µm low stress UNCD

Same ICP-RIE recipe

*to be presented at EIPBN 2010, O.Makarova et.al.

Conclusions

- **Sharp tips can be obtained by ICP-RIE of UNCD, R<10 nm**
- **Isotropic underetching does not work**
- **Differential etching and sharpening work**
- **Post-RIE Oxidation sharpening does not work – roughness**
- **ICP-RIE was optimized: DRIE of UNCD?**
- **Maximum rate (674 nm/min) with ICP=2800W, RIE=200W, P = 9 mTorr, O_2 = 50 sccm, SF_6 = 0.5 sccm, T = 20 ºC**
- **Technique is good for arrays of tips (field emission?), but still inferior to molding for AFM probes**
- **Some AFM probes may benefit from feathery shape – DPN?**

ACKNOWLEDGMENTS

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Q&A