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MICRO-331 Microfabrication technologies

J. Brugger
& teams

Micro for nano Part I

Nanoscale MEMS or NEMS

- What is different when going from micro to nanoscale?
- Nanoparticles (0D), nanowires (1D), nanoflakes (2D)
- New functionality of devices that are unique to the nanoscale size
- Nanoscale imaging by scanning probe microscopy (=nano-MEMS)
- Microfabrication of nanotips
- Nanoscale fabrication (top down)
- Nanoscale fabrication (bottom up)

What is different at the nanoscale

- Wolfgang Pauli said once: **“God made the bulk; the surface was invented by the devil.”**
- High surface-to-volume ratio, less bulk effects, more surface effects
  - Electronic properties (surface scattering, coulomb repulsion, thermal management)
  - Mechanical properties (elasticity, plasticity,…)
  - Magnetic properties (domains, temperature stability, …)
  - Photonic properties (wave characteristics, resonances, …)
  - Chemical properties (reactivity, catalytic,…)
- More on nanoscience, nanotechnology and nanoengineering in the Master course “Nanotechnology” by G. Boero, J. Brugger; spring semester

Nanoparticles (0D)
nanowires (1D)
nanoflakes (2D)

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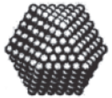
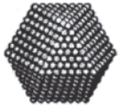
Properties of nanoscale «objects»

Micrometer scale «objects» have properties typically identical to those of larger «objects» (i.e., they have "bulk" properties).

Which properties might be significantly different in nanoscale «objects» ?

- Optical (color, transparency,….)
- Electrical (conductivity,….)
- Mechanical (hardness,…)
- Thermal (melting point,…)
- Chemical (reactivity, reaction rates, …)
- Magnetic (superparamagnetism,…)

Full-shell Clusters		Total Number of Atoms	Surface Atoms (%)
1 Shell		13	92
2 Shells		55	76
3 Shells		147	63
4 Shells		309	52
5 Shells		561	45
7 Shells		1415	35

Why properties of nanoscale «objects» are different from those of macroscopic «objects » ?

- Surface-to-volume ratios are larger
- Gravitational force is negligible
- Quantum effects might be important
- Random thermal molecular motion is more important
- …

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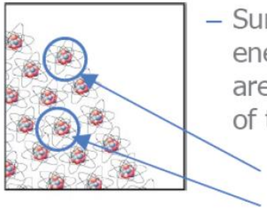
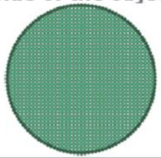
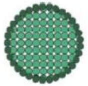
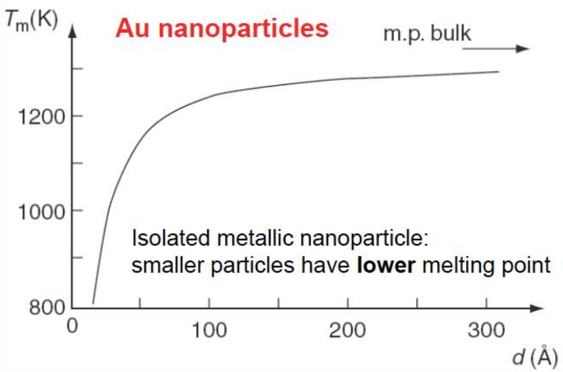
Example: Nanoparticles melting point

- **Melting Point (Microscopic Definition)**
  - Temperature at which the atoms, ions, or molecules in a substance have enough energy to overcome the intermolecular forces that hold the them in a "fixed" position in a solid

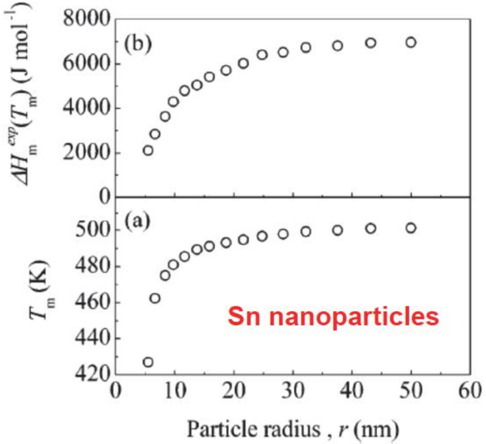


  - Surface atoms require *less* energy to move because they are in contact with *fewer* atoms of the substance

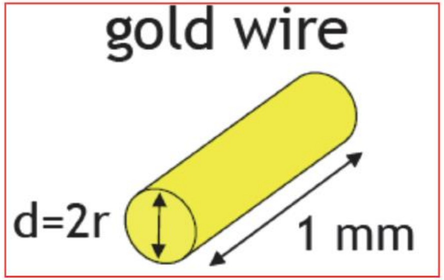
	At the macroscale	At the nanoscale
The majority of the atoms are…	…almost all on the inside of the object 	…split between the inside and the surface of the object 
Changing an object's size…	…has a very small effect on the percentage of atoms on the surface	…has a big effect on the percentage of atoms on the surface
The melting point…	…doesn't depend on size	… is lower for smaller particles



(Note: Metallic nanocrystals in a continuous matrix: smaller particles have **higher** melting point)



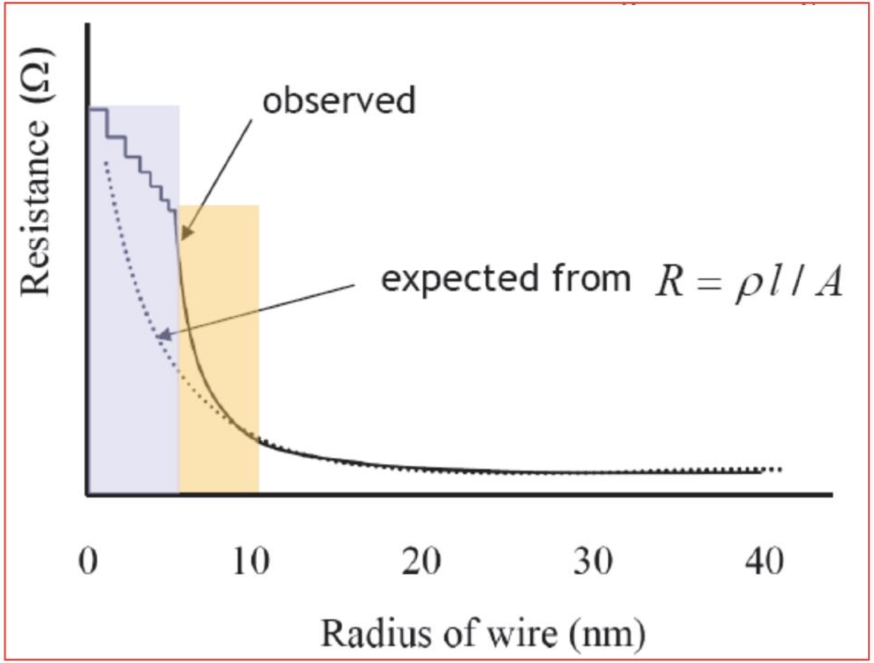
Conductance quantization: experimental evidence



Conductance quantization: first observed in 1988.
1. B. J. van Wees et al."Quantized Conductance of Point Contacts in a Two-Dimensional Electron Gas" *Physical Review Letters* 60, 848-850, 1988.
D. A. Wharam et. Al. "One-dimensional transport and the quantisation of the ballistic resistance," *Journal of Physics C: Solid State Physics* 21, L209-L214, 1988.

mesoscopic effects
- surface scattering

quantum effects



Content for today and tomorrow

- Nanoscale MEMS, or NEMS
- What is different when going from micro to nanoscale?
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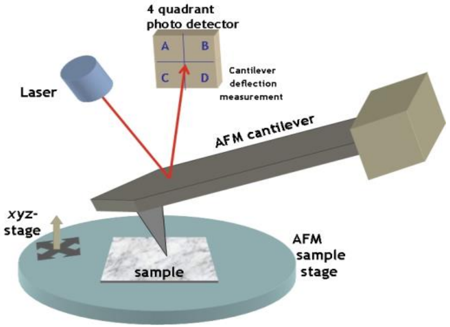
- Wrap up

Microscopy = Probing a surface / detecting the reaction

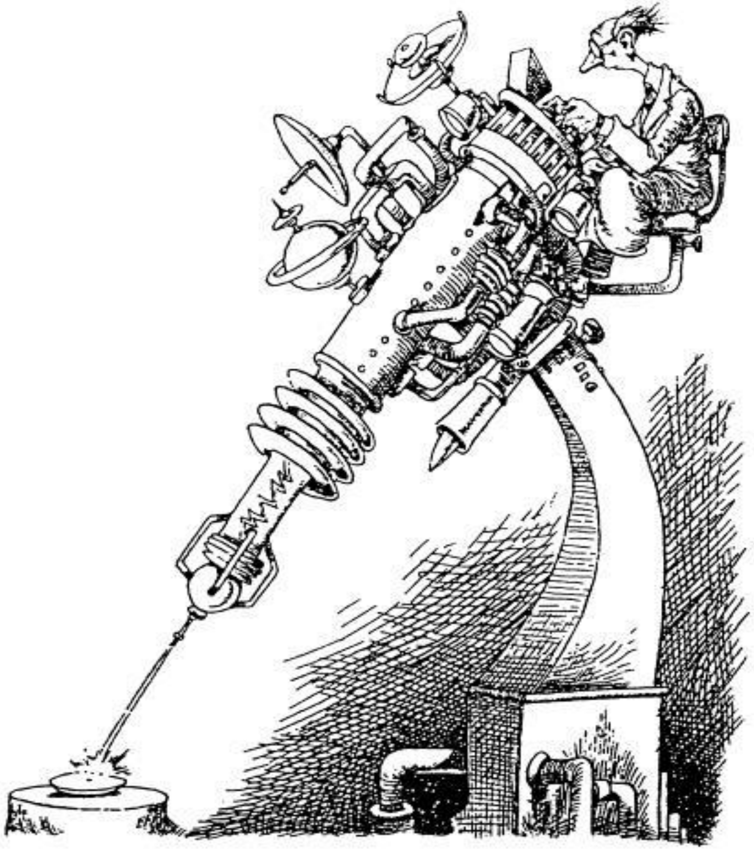


Optical ("light") microscope
'Probe': Light that is reflected from or transmitted through sample.

Electron microscope
'Probe': Electrons (shorter wavelength) that are re-emitted from or transmitted through sample.

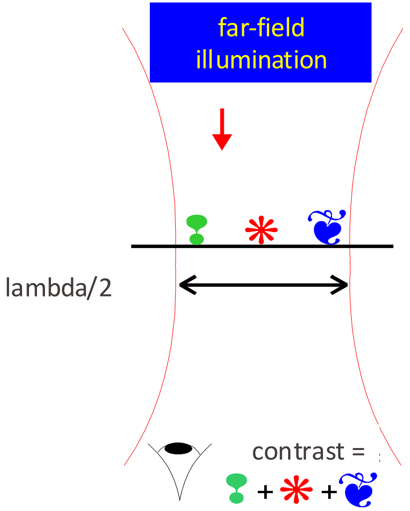
Scanning Probe microscope
'Probe': tip that interacts locally with surface (force, electrons, photons)

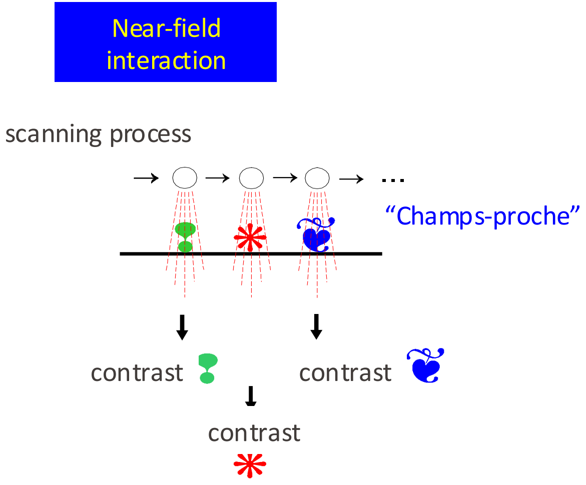
Q1: why do we need big machines to image or fabricate small objects?



Scanning Probe Microscopy

- conventional microscopy
  - wavelength
  - resolution prop. lambda
- probe microscopy
  - spatial dimension
  - resolution = f (a,d,...)





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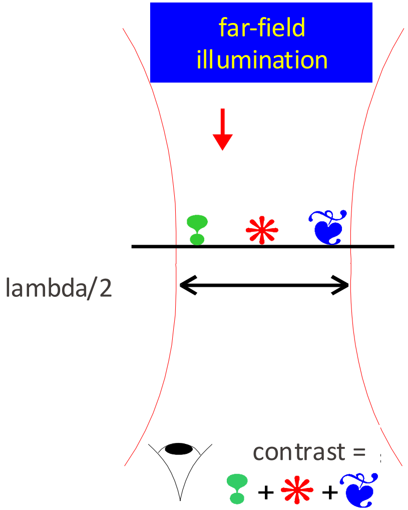
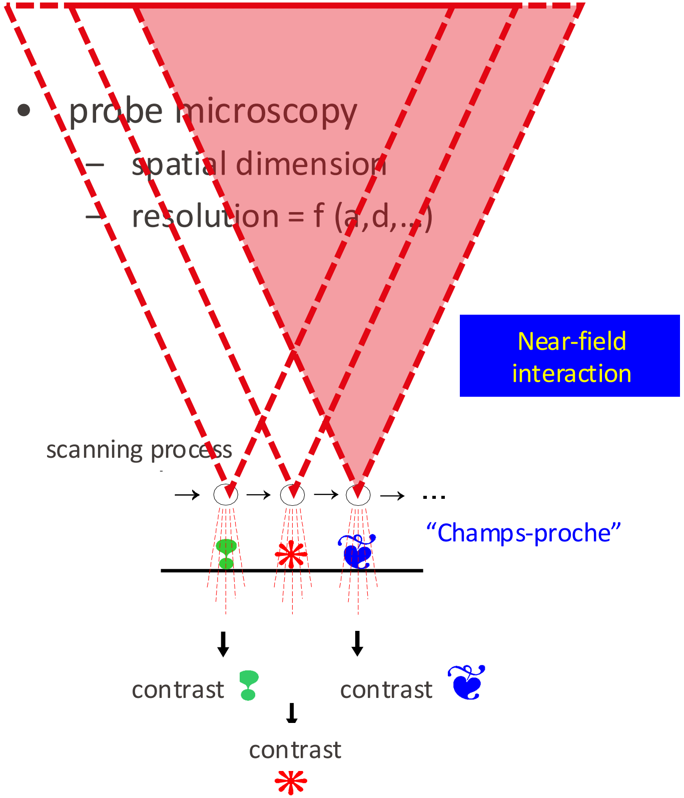




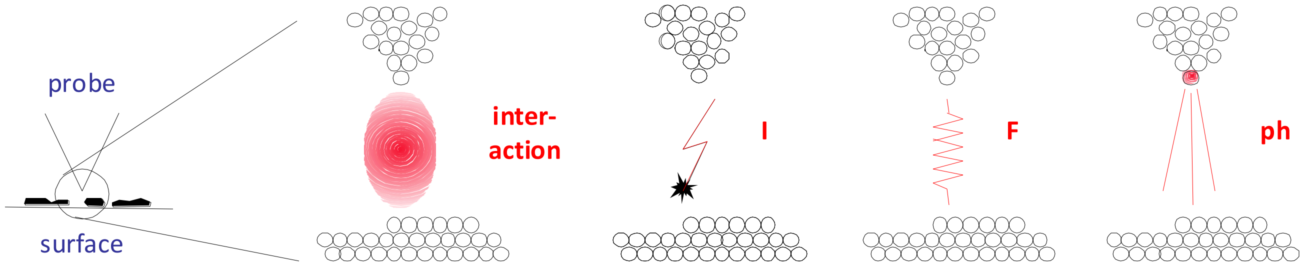
Scanning Probe Microscopy

SCANNING: x,y

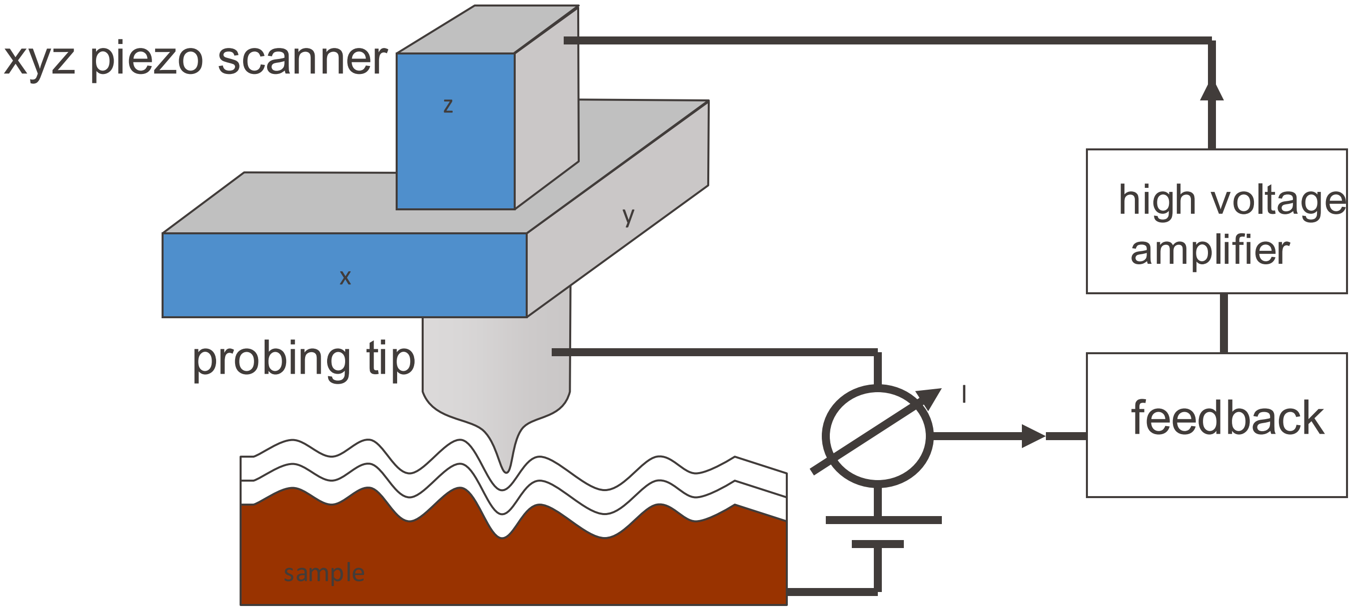
PROBE: spatial confinement

Different types of interaction between probe and surface:

- Current I (Scanning Tunneling Microscope) (Nobel Prize 1986)
- Forces F (Atomic Force Microscope)
- Photons ph (Near-field Scanning Optical Microscope)



Scanning Tunneling Microscope



I = C V exp (-B d) ; C,B = cte ; V = voltage ; d = gap

1 Angstrom gap change ~ 10x current change !!

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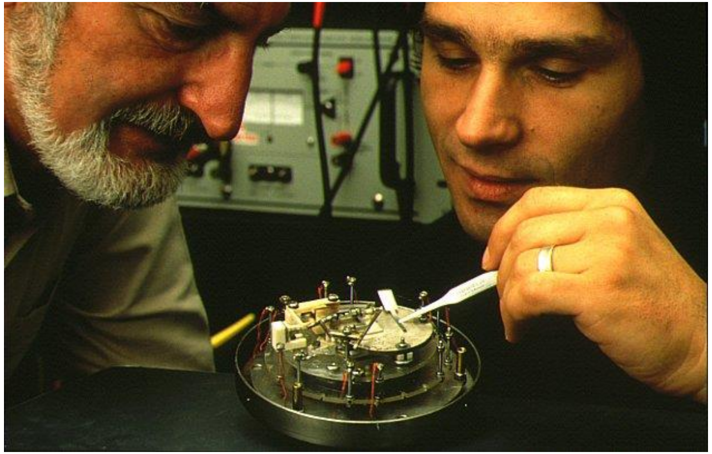
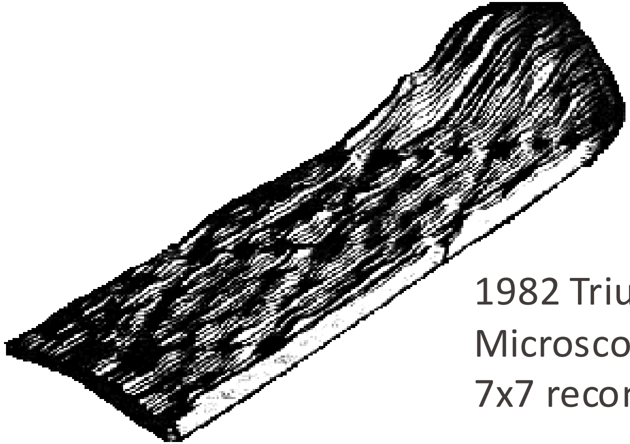
First STM Instrument

Great example of a micro-technique product



Exact copy of first Scanning Tunneling Microscope of Binnig and Rohrer (original has not preserved).

Nobel Prize in Physics 1986

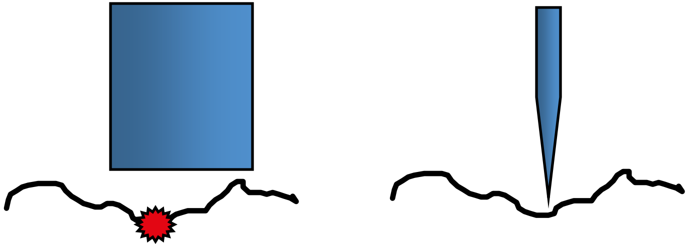


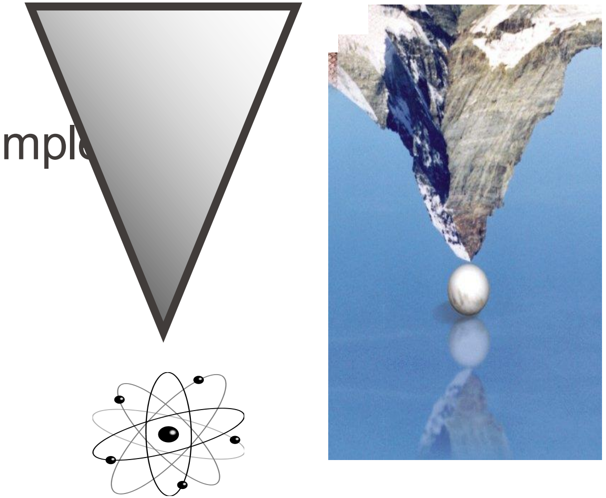
1982 Triumph of Scanning Probe Microscopy - image of silicon surface 7x7 reconstruction.

the Tip is the key

Q2: how sharp is the tip?
Q3: how can you fabricate tips?

- The resolution is determined by:
  - the dimension of the probe
  - the distance of the probe to the sample

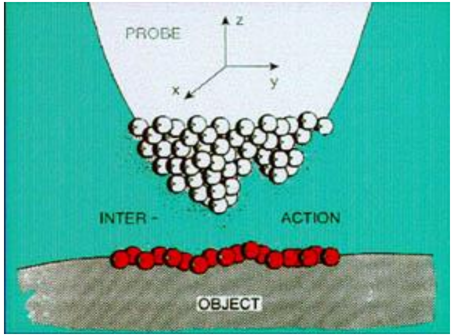


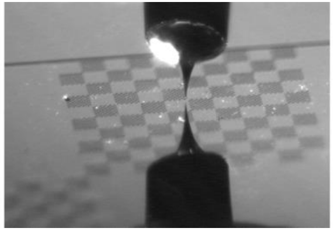


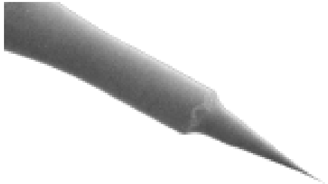
SPMs are surface microscopies

STM Probes

- How to make sharp STM tips?
  - Wire of W, Pt-Ir
  - 200 um diameter
  - cut or etch
  - → 400 Å diameter tip
  - hand-made
  - no microfabrication process



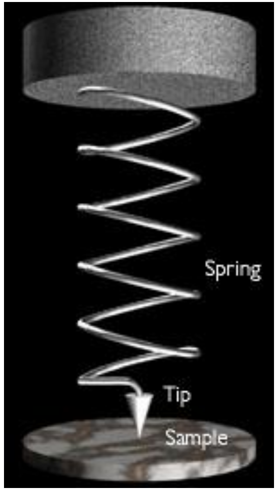




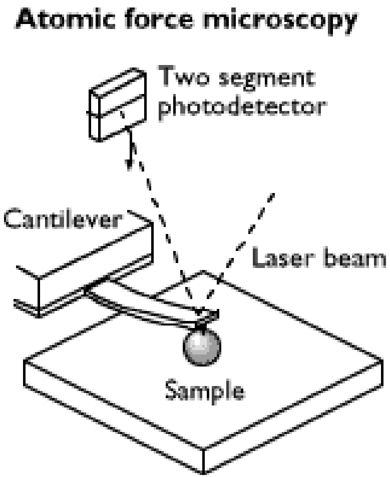
For STM, most of the time, the tips are hand-made just before the measurement. The important part is the end of the tip, which must be very thin. Another method based on an etching process is also sometimes used.

The Atomic Force Microscope

Q4: What forces are we talking about?

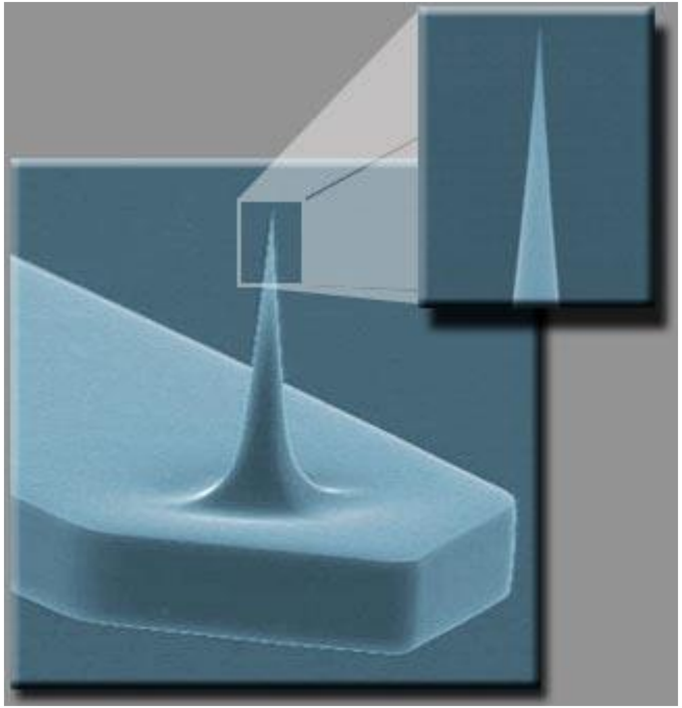


Model



Implementation

Microfabrication of sharp tips for AFM

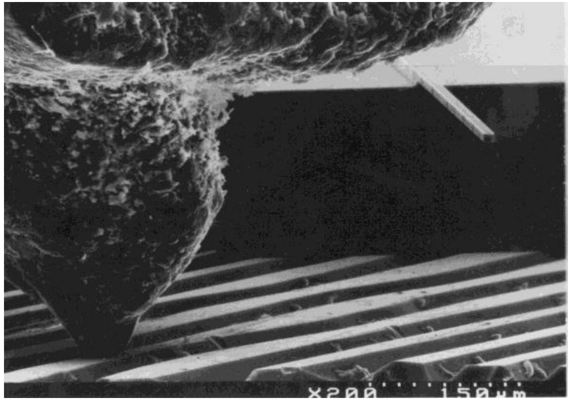


Tip height: 7 micron

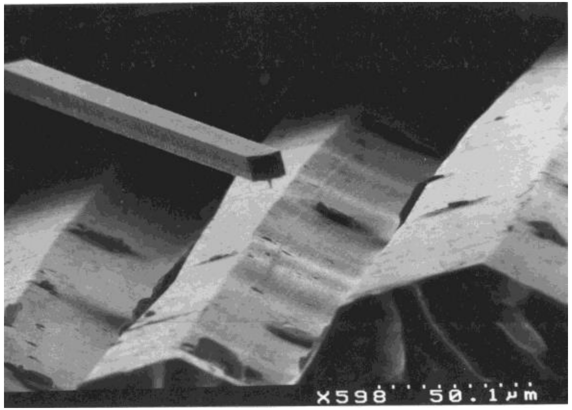
Radius: < 10 nm

Tip integrated in a cantilever

The Atomic Force Microscope



- AFM probe tip
- 1000 times smaller than a record player needle
- 33 rpm vinyl disc record
- Precision machined diamond needle
- Micromachined Silicon AFM cantilever



Microfabrication of nano-tips

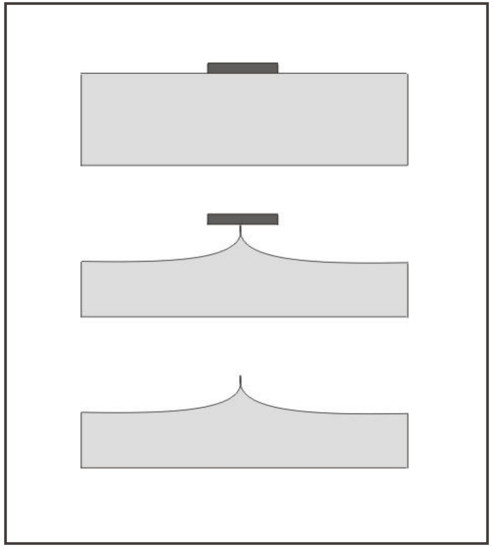
Inspired from Nature
Les Pyramides d'Euseigne
Valais, Switzerland

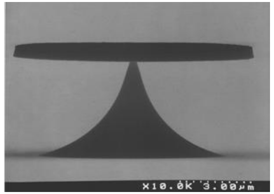
Microfabrication of nano-tips

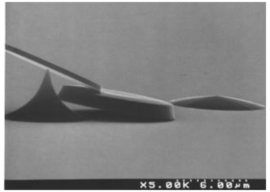
Erosion in the lab

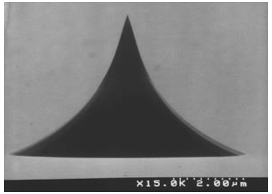
Direct tips

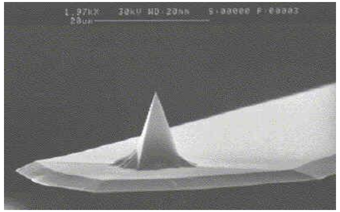
- *E.g. Silicon*
- *Define etch mask*
  - *nitride, dioxide, resist*
- *Wet etching (examples)*
  - *potassium hydroxide (KOH), anisotropic*
  - *HNO3:HF, isotropic*
- *Dry etching*
  - *plasma*
  - *fluorine, chlorine based*











Microfabrication of cantilevers

- Cantilever = force transducer
- Important parameters
- Spring constant k
- Resonance frequency f
- Practical values
- E: Young's modulus (material constant) E_{si} = 1.7 E11 N/m^2
- I: cross sectional moment of inertia (square cross section I = w t^3/12)

Forces: Micro/nano/pico Newton

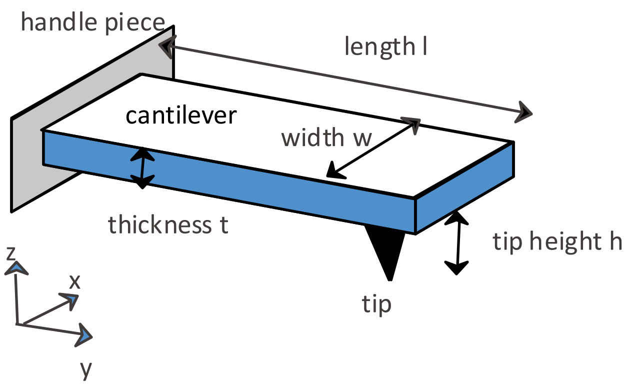
general

k = 3 E I / L^3

$$f = \frac{1}{2\pi}\sqrt{\frac{k}{m_{eff}}}$$

Rectangular shape

$$k = \frac{Ewt^3}{4l^3}$$



k: 0.01-10 N/m
f: > 10kHz

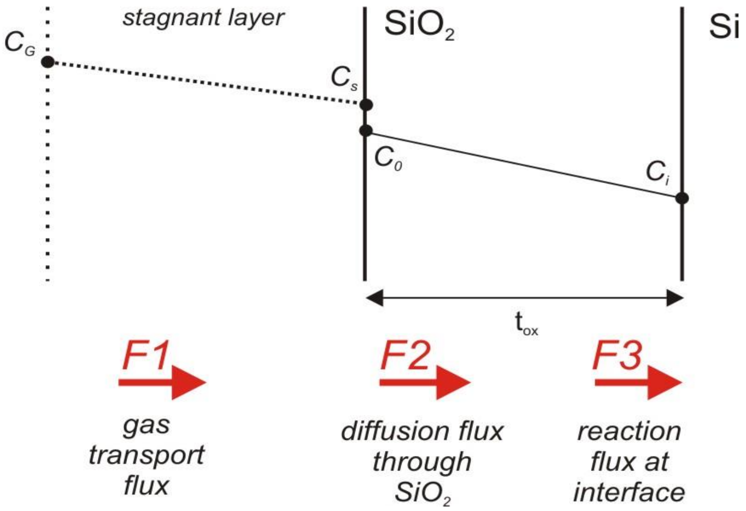
How to make VERY VERY sharp tips

- Typical tips after microfabrication have 20-30 nm radius
- Sharpening by oxidation

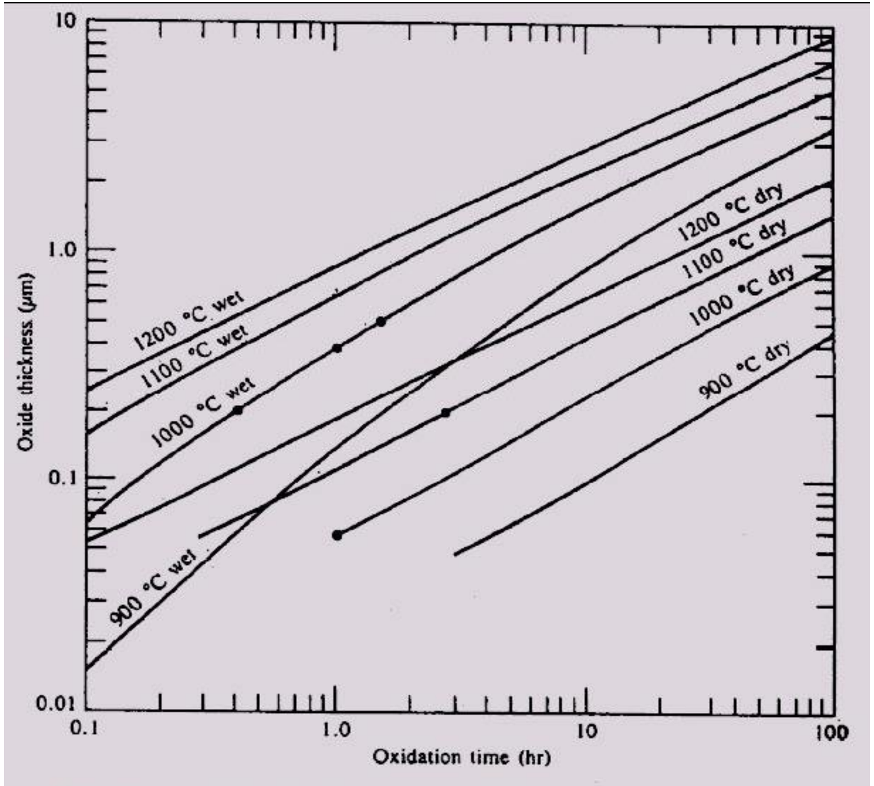
Q5: What happens during thermal oxidation of silicon?

Basics of Thermal Oxidation

- Oxide growth kinetics based on *Deal-Groove Model*
- For short oxidation times: $t_{OX} \sim t$
- For long oxidation times: $t_{OX} \sim \sqrt{t}$
- Oxide growth slows down with increasing thickness



Basics of Thermal Oxidation



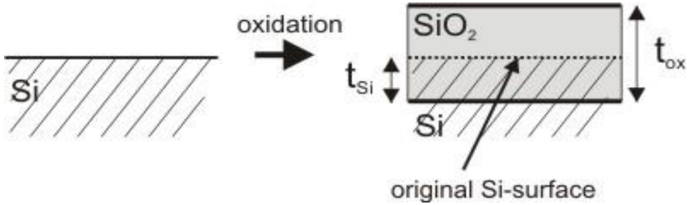
Wet and dry silicon dioxide growth for ⟨100⟩ silicon

In practice:

Use oxidation charts to determine thickness

Basics of Thermal Oxidation

- Thermal oxidation of Si
- Si is consumed during growth of SiO_2

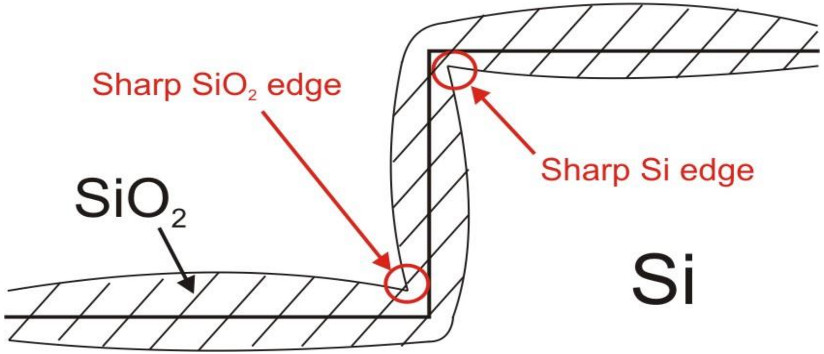


$$t_{Si} = t_{ox} \cdot \frac{N_{ox}}{N_{Si}}$$

N_{ox} : molecular density of SiO_2
N_{Si} : molecular density of Si

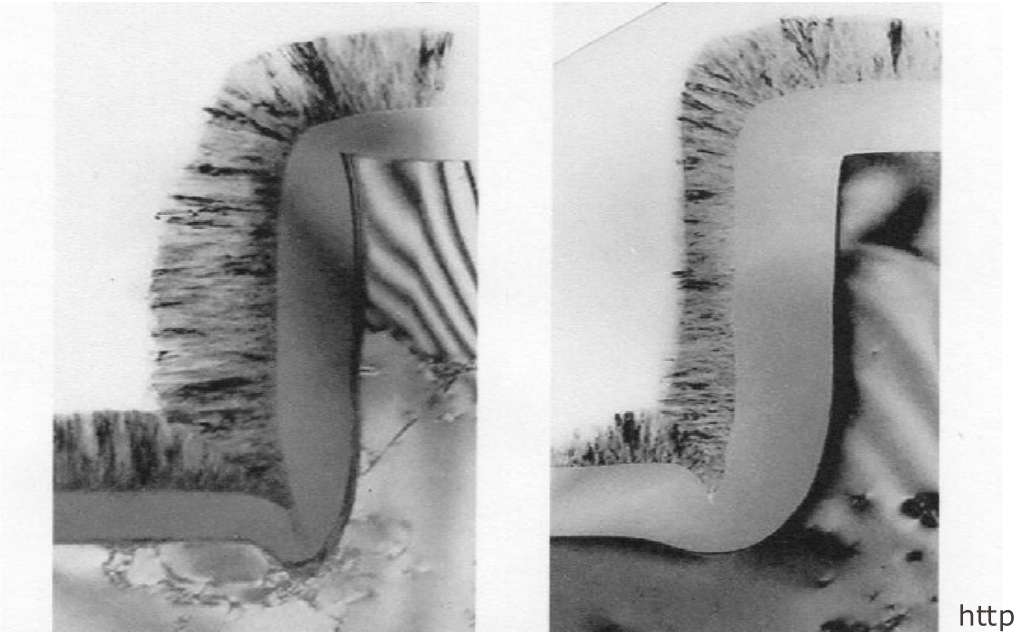
$$= t_{ox} \cdot \frac{2.3 \cdot 10^{23} \text{ molecules/cm}^3}{5 \cdot 10^{22} \text{ atoms/cm}^3} = 0.46 \cdot t_{ox}$$

- Oxide growth at an edge profile :



Oxidation at sharp corners

- TEM cross section of oxidized silicon



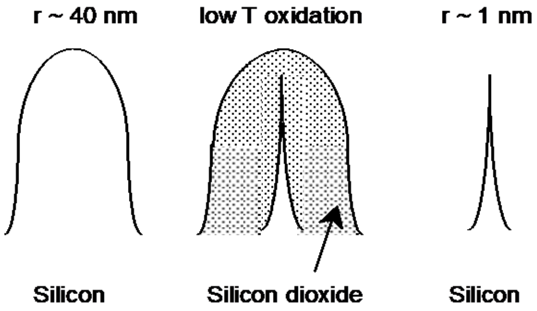
http://web.mit.edu

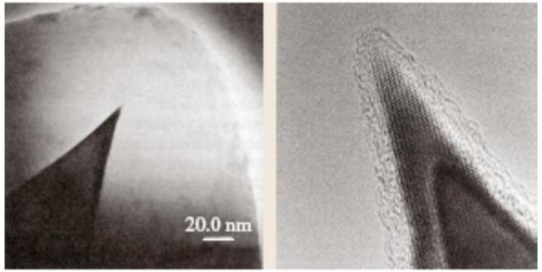
950°C oxidation

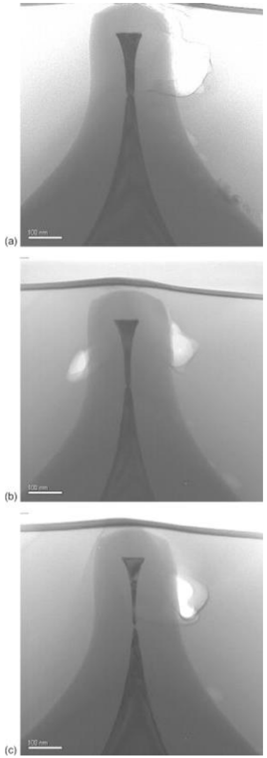
1100°C oxidation

Tip sharpening

- Self-terminating step:
- Example: Silicon tip
- ‘low’ temperature oxidation sharpening
- T<950 deg C
- anomalous oxide growth of SiO2 at regions with high curvature radii







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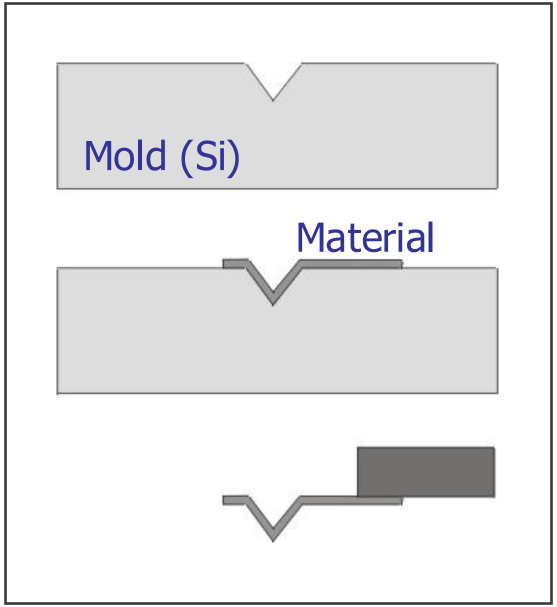
Molded tips (indirect etched)

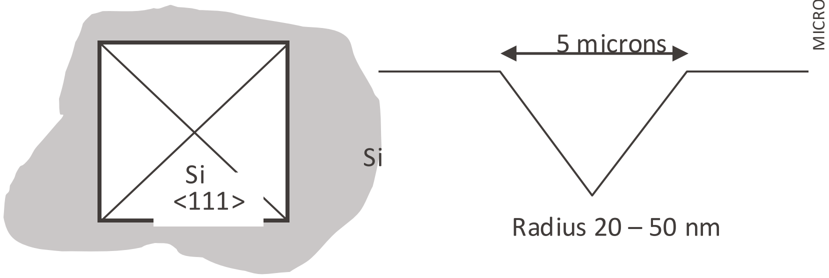
- Define mold by etching
  - inverted pyramid (anisotropic etching KOH)
- Automatic etch stop at <111> etch planes Si
- Good large area uniformity
- Filling mold with appropriate material
  - Silicon nitride
  - Silicon oxide
  - Metal
  - Polymers
- Release process of thin film
- keep mold, replication

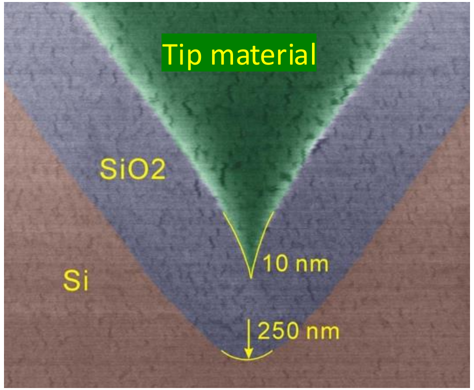


Q6: Oxidation sharpening of molds?

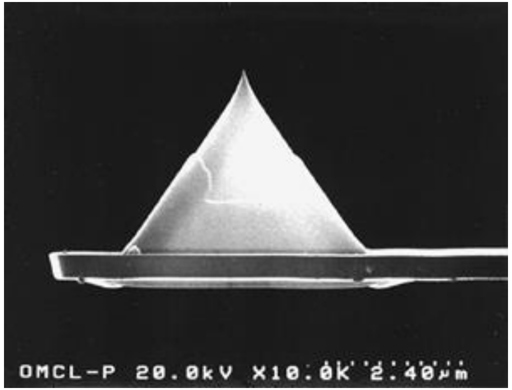
Moulded Tips for AFM

- Moulds by anisotropic Si etching (e.g. KOH, TMAH)
- Oxidation of the mold
- Deposition of the tip material (e.g. metals, polymers) into the SiO_2 covered mold





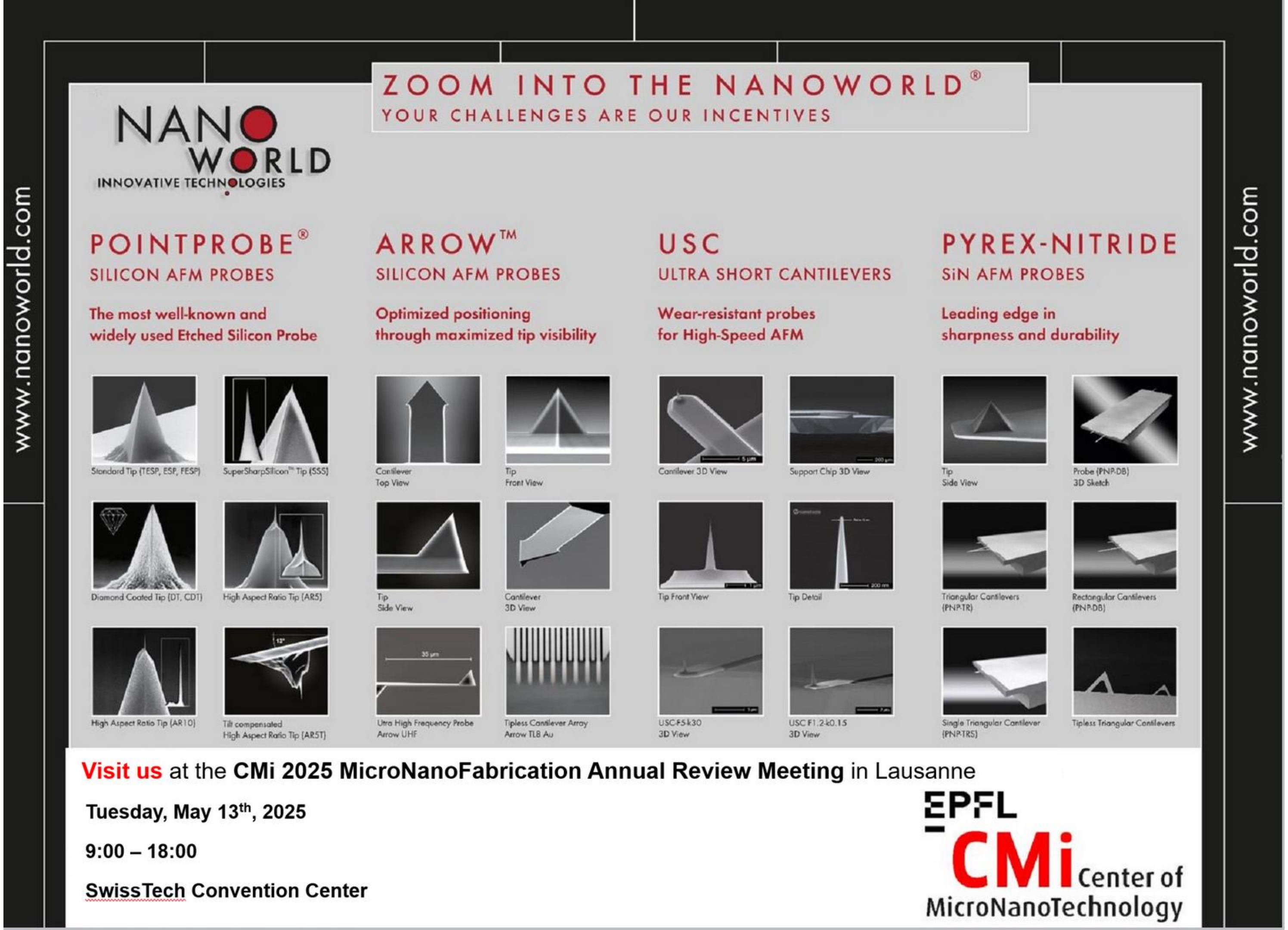
release



Take away message

- Use standard Microfabrication (i.e. microscale lithography) to fabricate nanometer precise objects, nano-tips in this case
- Exploit processes such as diffusion control, etch anisotropy, self-limiting/self-sharpening effects
- Allows for cost efficient nanoscale features with wafer scale processes
- No need for expensive equipment such as electron beam lithography

https://www.nanoworld.com/contact-us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PYREX-NITRIDE
SiN AFM PROBES
Leading edge in sharpness and durability
Standard Tip (TESP, ESP, FESP)
SuperSharpSilicon™ Tip (SSS)
Cantilever Top View
Tip Front View
Cantilever 3D View
Support Chip 3D View
Tip Side View
Probe (PNP-DB) 3D Sketch
Diamond Coated Tip (DT, CDT)
High Aspect Ratio Tip (AR5)
Tip Side View
Cantilever 3D View
Tip Front View
Tip Detail
Triangular Cantilevers (PNP-TR)
Rectangular Cantilevers (PNP-DB)
High Aspect Ratio Tip (AR10)
Tilt compensated High Aspect Ratio Tip (AR5T)
Ultra High Frequency Probe Arrow UHF
Tipless Cantilever Array Arrow TL8 Au
USC-F5-k30 3D View
USC-F1.2-k0.15 3D View
Single Triangular Cantilever (PNP-TRS)
Tipless Triangular Cantilevers
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Tuesday, May 13th, 2025
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