Scanning Probe Microscopy for III-V semiconductors

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Outline:

- How it works:
  - STM
  - AFM
- Applications for III-V semiconductors:
  - STM
  - AFM

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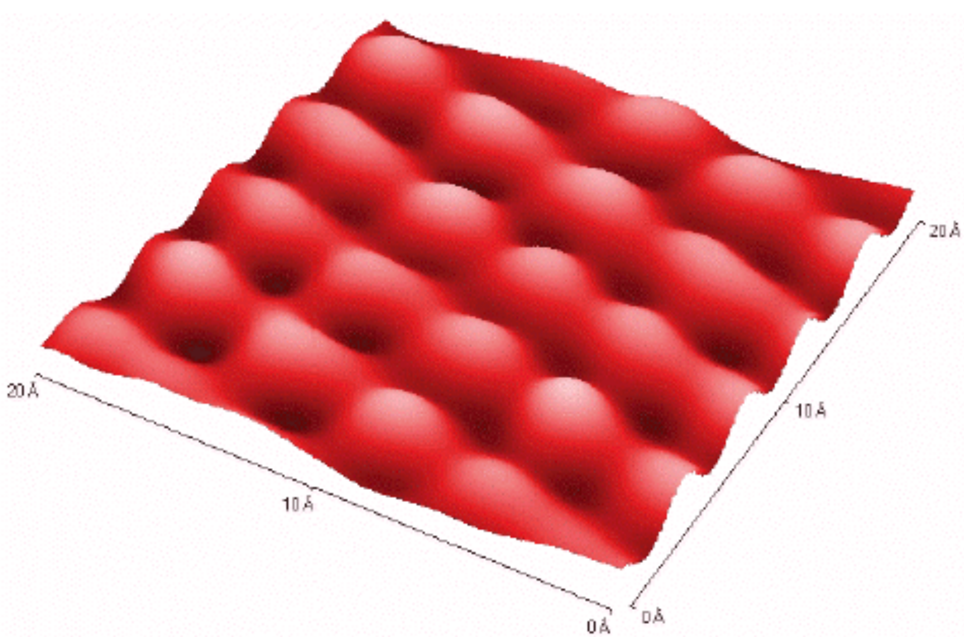
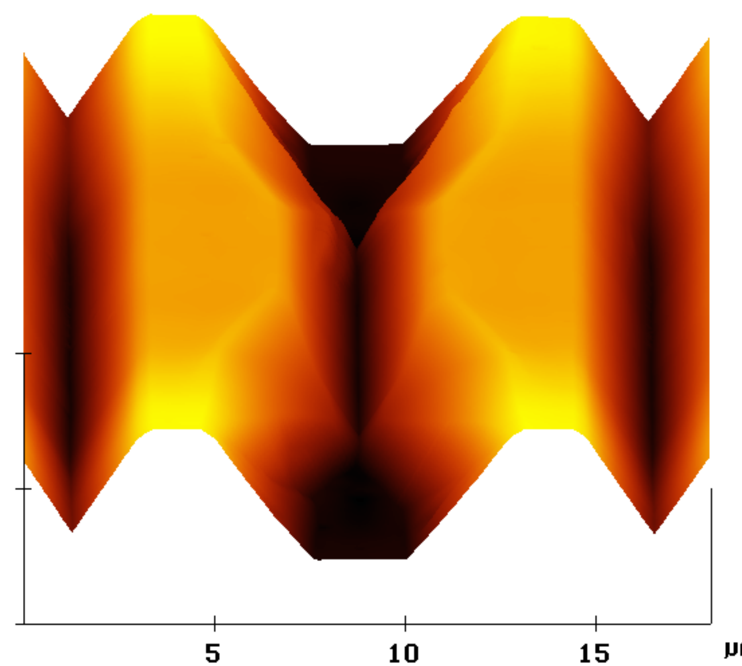
What is SPM ?

- **Scanning Probe Microscopy :**

The characterization of a sample by scanning its surface with a probe, at a small distance

As a result, only surface properties are observable





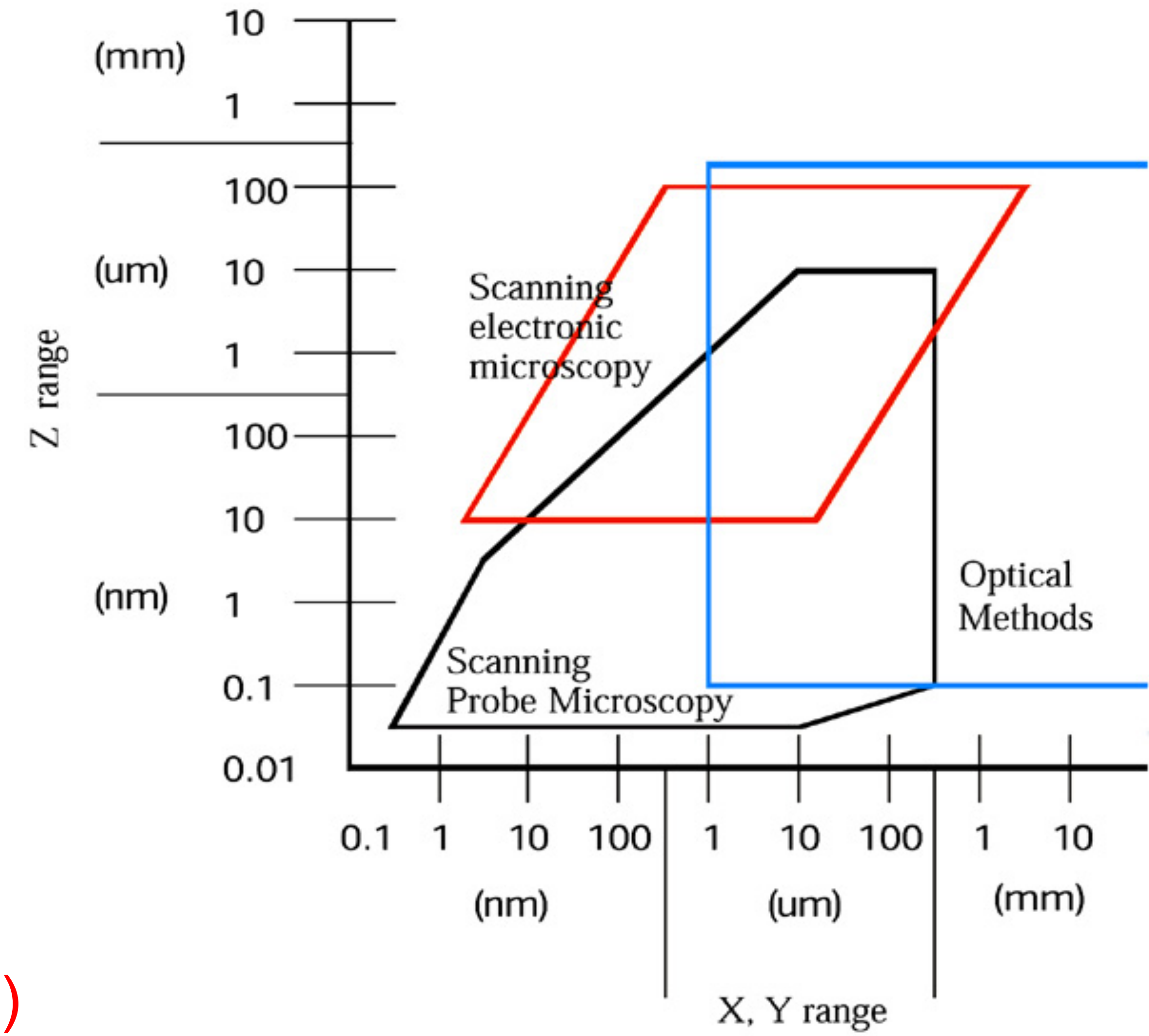
How does SPM compare with other microscopy techniques ?

Microscope	Optical	Confocal	Interference	SEM	TEM	STM	AFM	SNOM
XY resolution	400 nm	150 nm	250 nm	1 nm	0.1 nm	0.1 nm	(0.1) 1-10 nm	<50 nm
Z resolution	-	10 nm	0.1 nm	-	-	0.01 nm	0.01 nm	(0.01nm)
Ambience	air (liquid)	air (liquid)	air (liquid)	vacuum	vacuum	vacuum (air)	air (liquid)	air
Sample preparation	none	none	none	none / coating	polishing, ion milling	none / UHV cleaving	none	none
Damage to sample	none	none	none	Contamination,(heating)	Contamination, heating	none	none (scratch)	none
Price (kFr)	5-30	50-200	50-200	200-700	500-2000	70-300	50-300	70-300

Advantages of SPM

- 3D imaging
- High spatial and vertical resolutions
- No sample preparation
- Simple to operate
- Low-cost

- Main disadvantage : slow (5-20 min/image)



The main challange of SPM: How to get nm resolution?

Potential problems:

1. Tip size
2. High-resolution XY scanning
3. Non-destructive
4. Constant distance from sample
5. Vibrations
6. Thermal stability

Solutions:

1. Short-term interactions
2. Piezo scanner
3. Non-contact
4. Height feedback
5. Rigid structure, isolation
6. Compensation

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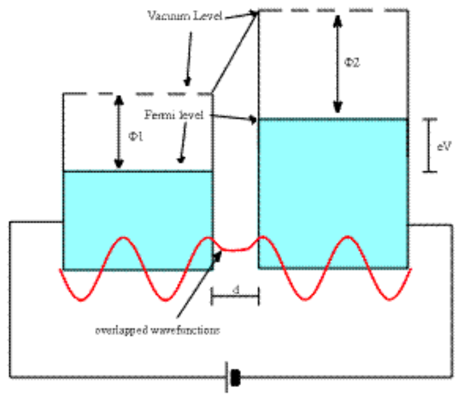
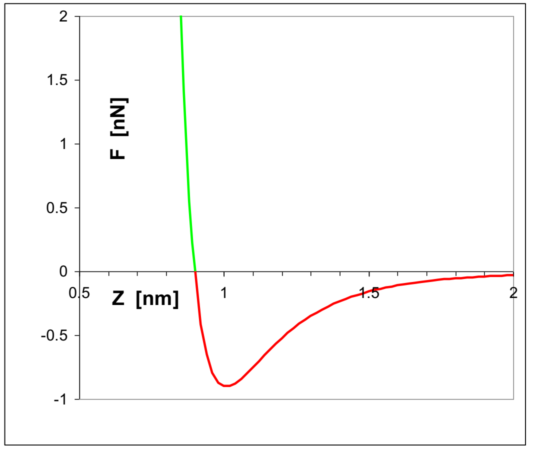
Let's look at the solutions:

1. Short-term interactions: Do you know any?

- Quantum-mechanical electron tunneling: STM



- Van-der-Waals forces: AFM

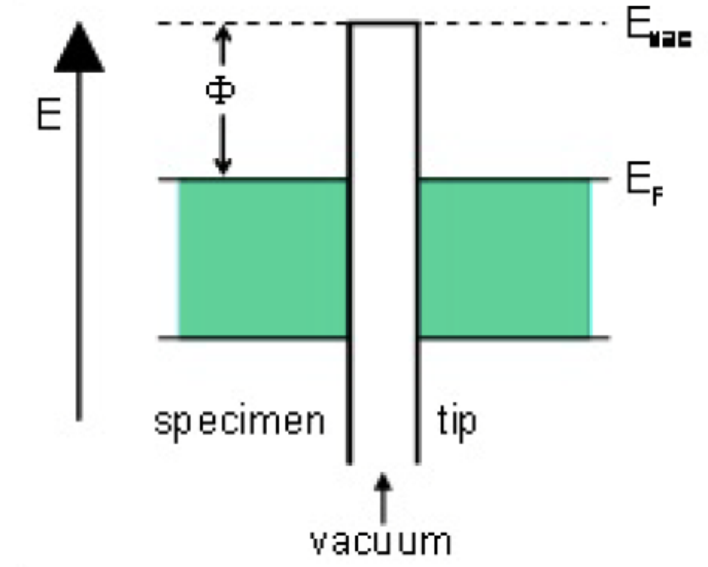


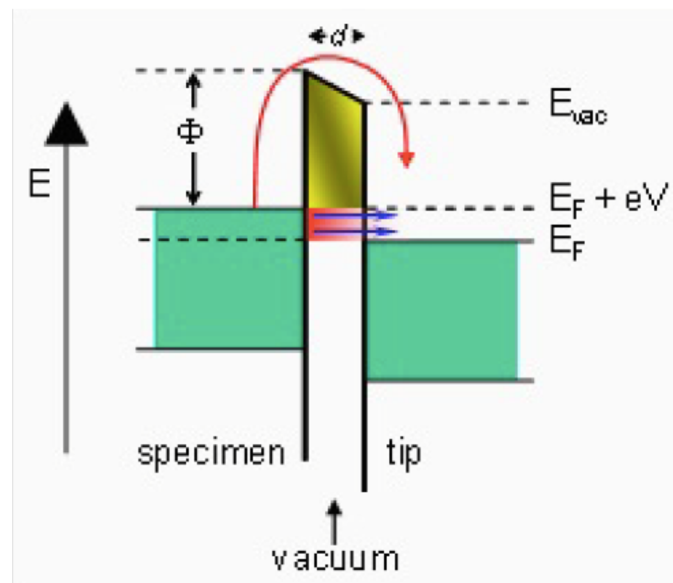
First working STM: Binnig & Rohrer, 1982

G. Binning, H. Rohrer, Ch. Gerber, and E. Weibel, Phys. Rev. Lett. **49**, 57 (1982) "Surface Studies by Scanning Tunneling Microscopy"

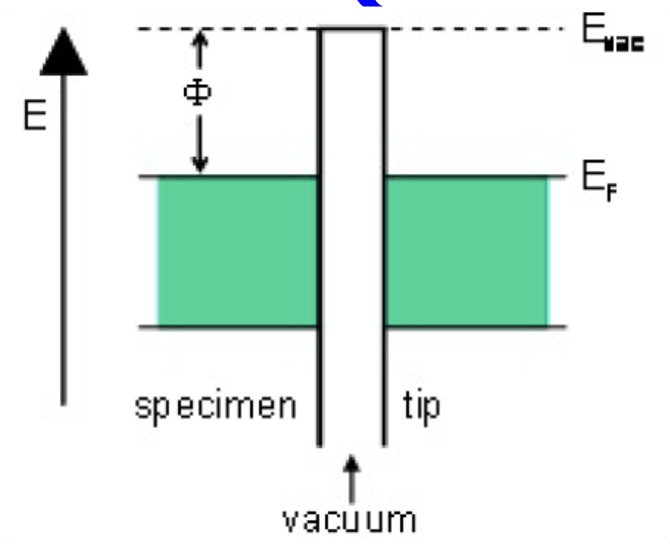
Quantum Tunneling

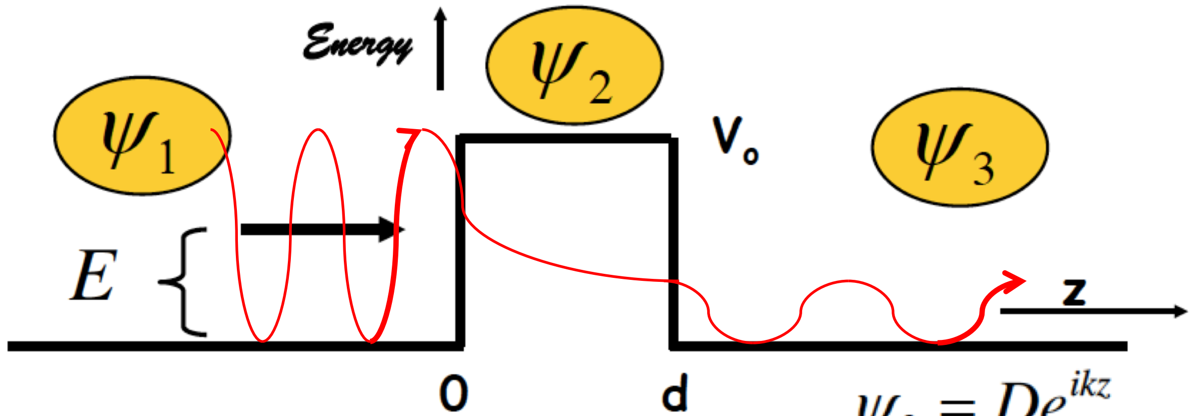
- Electrons in metals fill up energy levels to the Fermi level, which is about 4.5-5.5 eV (the work function) below the Vacuum level.
- External potential between metals shifts the relative Fermi levels, so that electrons can pass from full (left) to empty (right) levels, but… the energy barrier is too high!
- When the metals are close enough, electron wavefunctions can overlap and tunneling current flows





Quantum Tunneling: statics





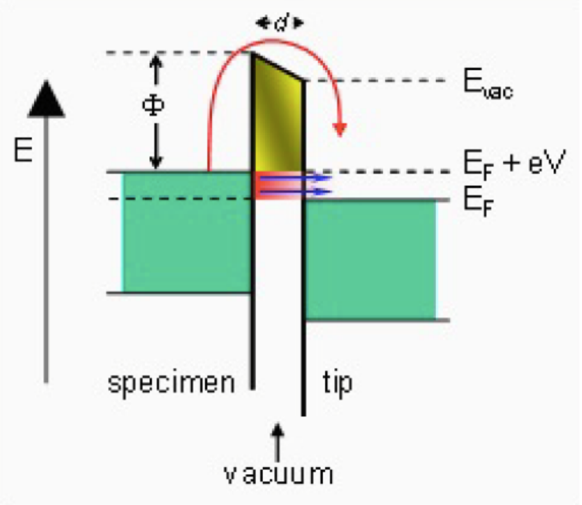
$$\psi_1 = e^{ikz} + Ae^{-ikz}$$
$$U(z) = 0$$
$$k^2 = \frac{2mE}{\hbar^2}$$

$$\psi_2 = Be^{-\alpha z} + Ce^{\alpha z}$$
$$U(z) = V_o$$

$$\psi_3 = De^{ikz}$$
$$U(z) = 0$$
$$k^2 = \frac{2mE}{\hbar^2}$$

- Electron wavefunction is propagating in metal, but exponentially decaying in the vacuum barrier.
- The decay coefficient: $\alpha = \frac{\sqrt{2mV_0}}{\hbar} = \frac{\sqrt{2m\Phi}}{\hbar} \approx 11\ \text{nm}^{-1}$
- The transmission coefficient is: $T = \left(\frac{4k\alpha}{k^2+\alpha^2}\right)^2 e^{-2\alpha d} \propto e^{-2d\sqrt{2m\Phi}/\hbar}$

Quantum Tunneling: dynamics



- When we apply a potential *V* between the metals, current will flow across the barrier.
- The current from the left metal to the right is given by (f = Fermi distribution):

$$J_{l\to r} = \frac{4\pi e m^2}{h^3}\int_0^\infty f(E) D_l(E) D_r(E+eV) T(E)\, dE$$

- The current from the right metal to the left is given by:

$$J_{r\to l} = \frac{4\pi e m^2}{h^3}\int_0^\infty f(E+eV) D_l(E) D_r(E+eV) T(E)\, dE$$

- The net current is the difference:

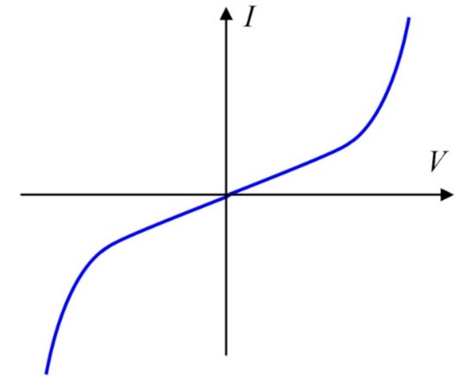
$$J = \frac{4\pi e m^2}{h^3}\int_0^\infty \left[f(E) - f(E+eV)\right] D_l(E) D_r(E+eV) T(E)\, dE$$

Quantum Tunneling: current

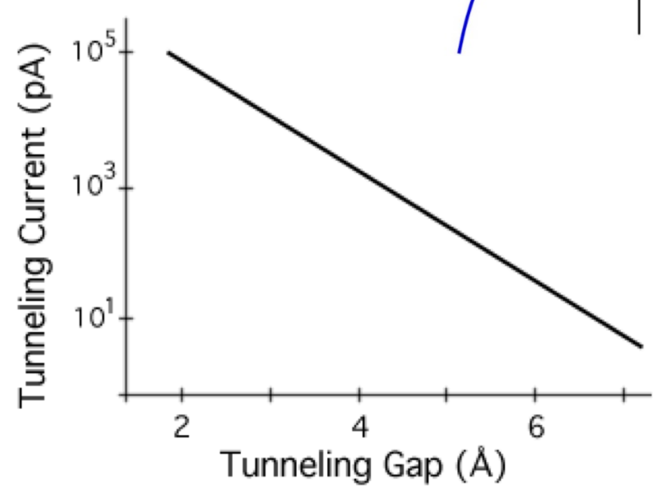
- The current is: $J = \frac{4\pi e m^2}{h^3} \int_0^\infty \left[f(E) - f(E+eV) \right] D_l(E) D_r(E+eV) T(E)\, dE$
- If the voltage is small relative to the barrier, we can use a 1st order expansion for the Fermi distribution function and get:

$$J = \sigma_0 V e^{-2d\sqrt{2m\Phi}/\hbar} \propto V e^{-2\alpha d} \qquad \alpha = \frac{\sqrt{2m\Phi}}{\hbar} \qquad \sigma_0 = \frac{e^2\sqrt{2m\Phi}}{h^2 d} D_l(E_F) D_r(E_F)$$

- The I-V curve is linear, as in an Ohmic contact

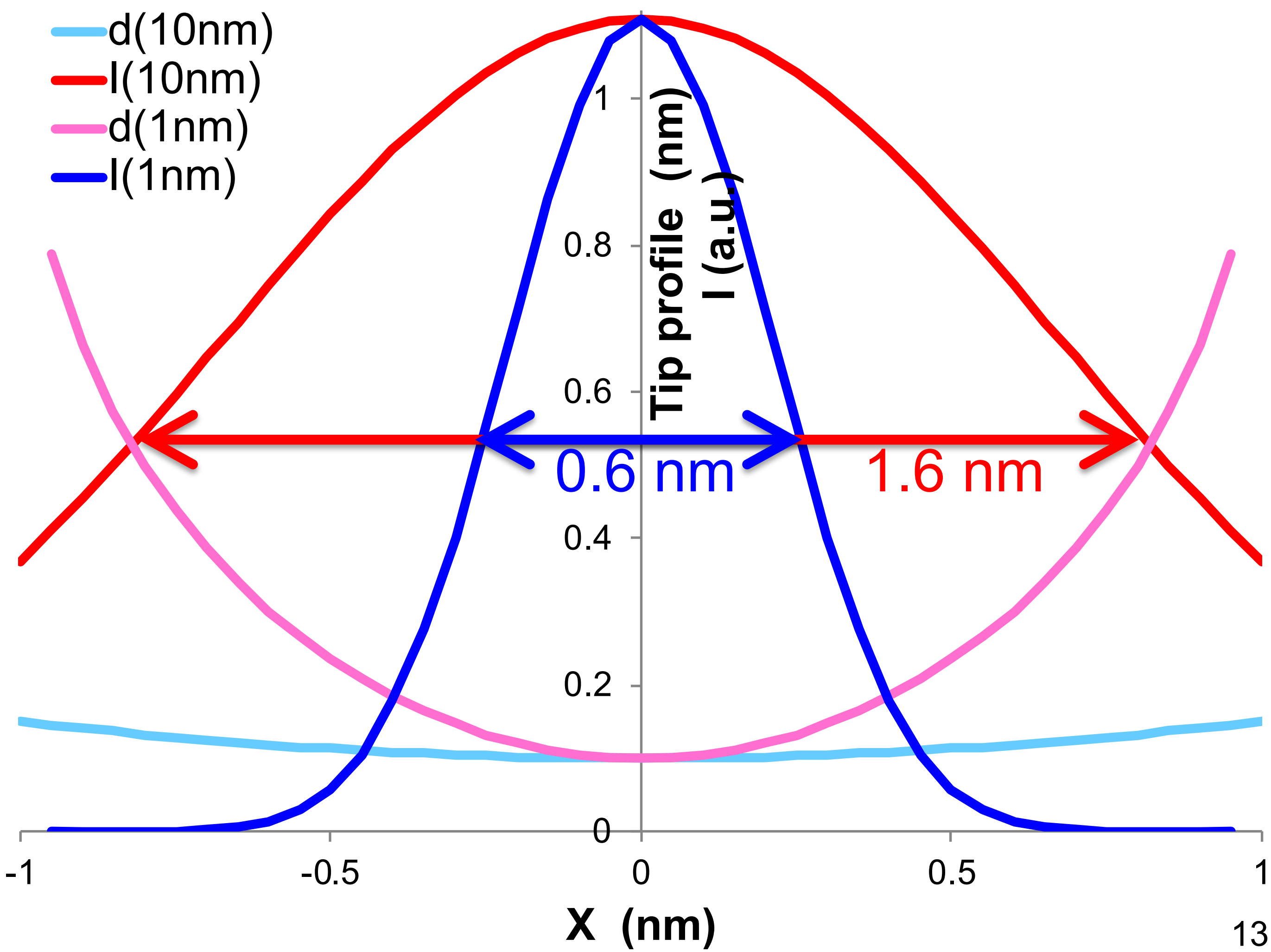


- The short range of tunneling ($\alpha \approx$ 11 nm^{-1}) makes the current very sensitive to the distance:
  - Change of distance of one atomic monolayer = 0.3 nm, gives change of current by x1000!



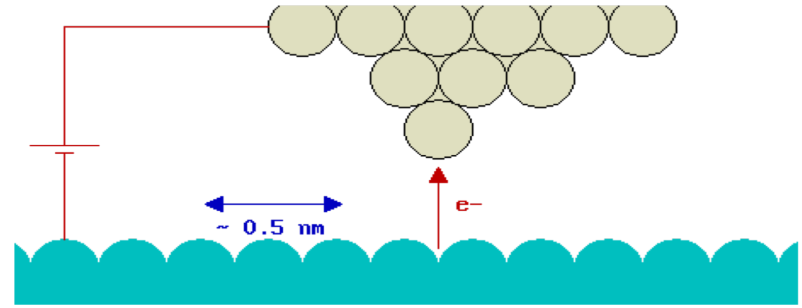
Tunneling between plane and tip

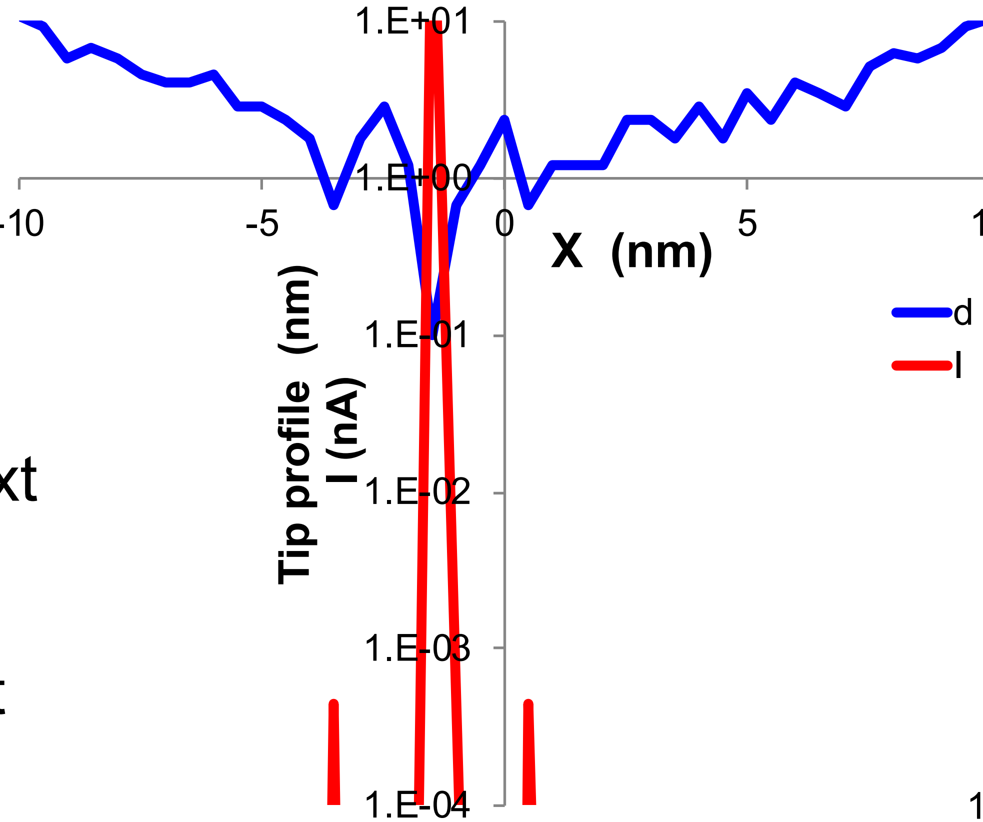
- Tunneling current between plane and half-spherical tip of identical metals, for tip radii of 1 and 10 nm: apparent width is much smaller!



Tunneling between plane and tip

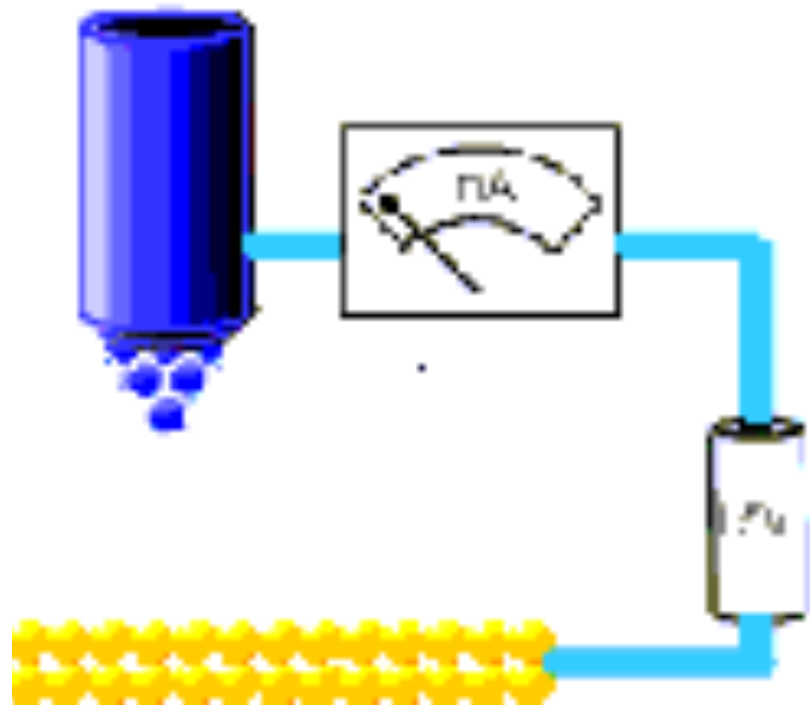
- In a more realistic case, the random atomic nature of the tip will “promote” one atom to produce most tunneling current. Here the tip R=10 nm, h=0.1 nm, lattice constant 0.56 nm
- The current through the lowest atom is bigger by 10^4 than the current from the next one!
- The real “tip” (atom) is not at zero, but it doesn’t matter!

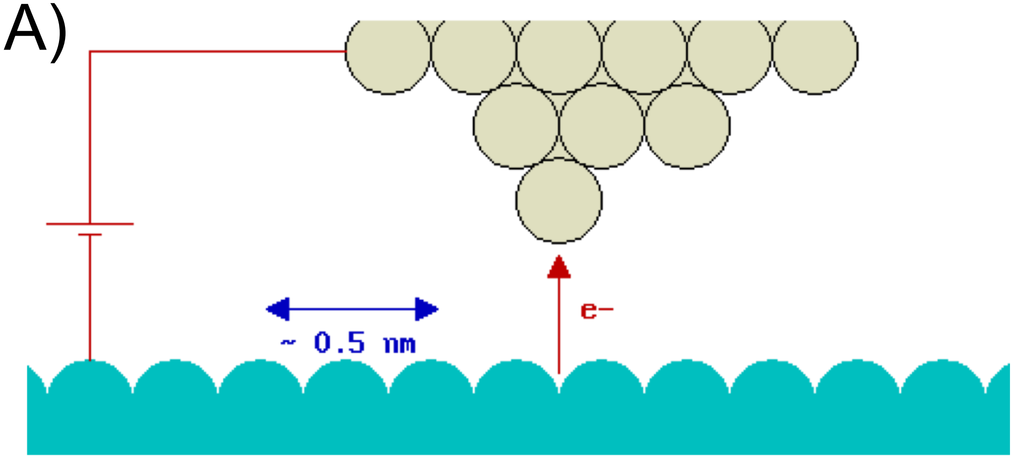




Tunneling as surface probe:

- We approach the sample with a sharp metallic tip, biased to a small potential (1-1000 mV)
- At a very close distance, tunneling current will start to flow between the tip's atoms and the samples' surface atoms
- This current is measurable (nA) at tip-sample distance of 1Å





Tunneling between plane and tip

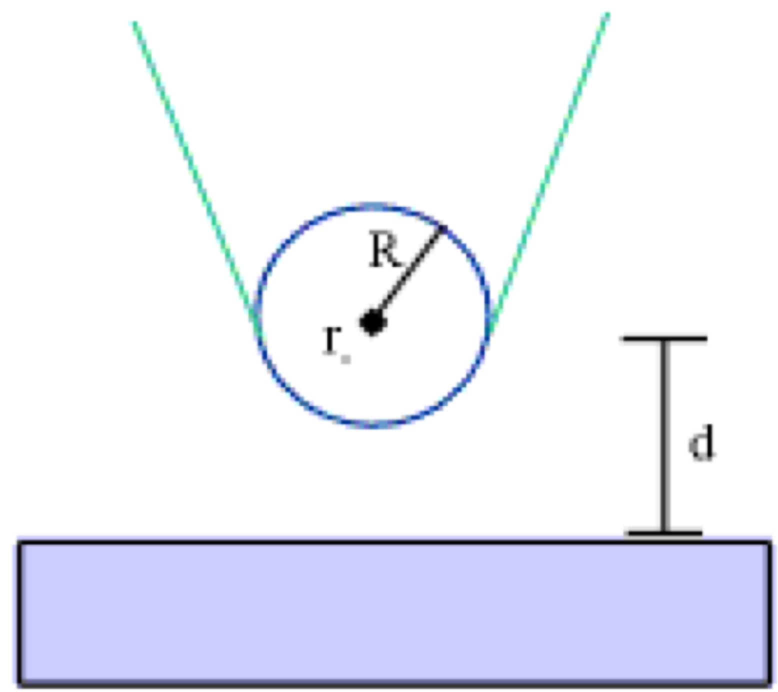
- At low temperatures, between plane and spherical tip of identical metals, we get the tunneling current:

$$I = \frac{32\pi^3 e^2}{\hbar} V\Phi^2 D_T(E_F) R^2 K^{-4} e^{-2Kd} \sum_s |\psi_s(r_0)|^2 \delta(E_s - E_F)$$

- Where D = density of states at tip,

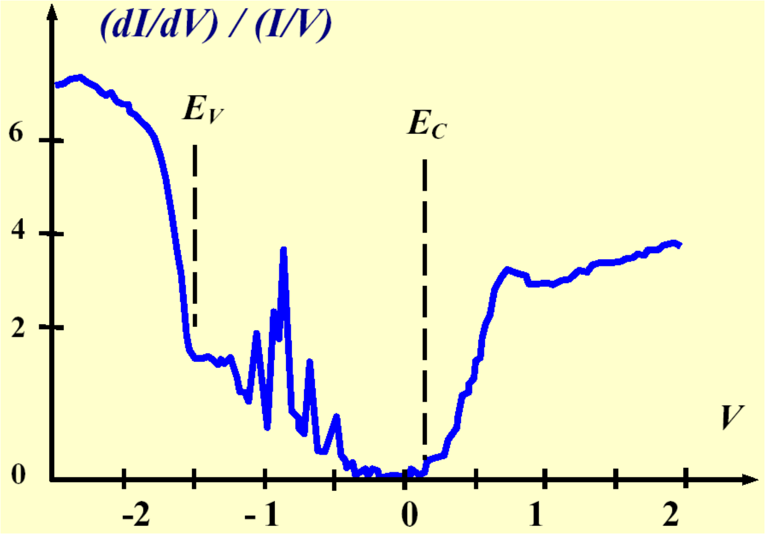
R = tip radius

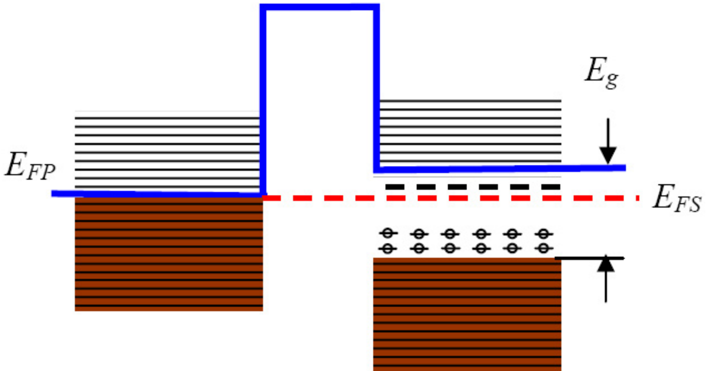
- There is still exponential dependence on distance
- Tip radius plays a secondary role
- DOS can be measured as well:
- STS = Scanning Tunneling Spectroscopy:

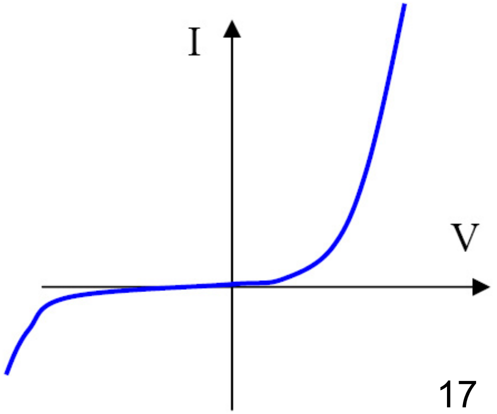


Metal-semiconductor tunneling

- Tunneling between the metallic STM tip and a semiconductor shows the energy gap in the I/V curve (STS)
- In many cases the derivative dI/dV is plotted vs. V to show more clearly the DOS, states in the gap etc.
- Surface states (oxidation) can pin the Fermi level – UHV is needed





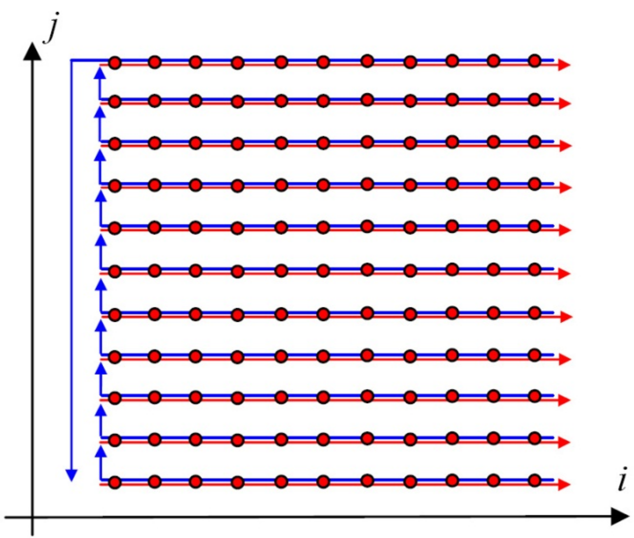


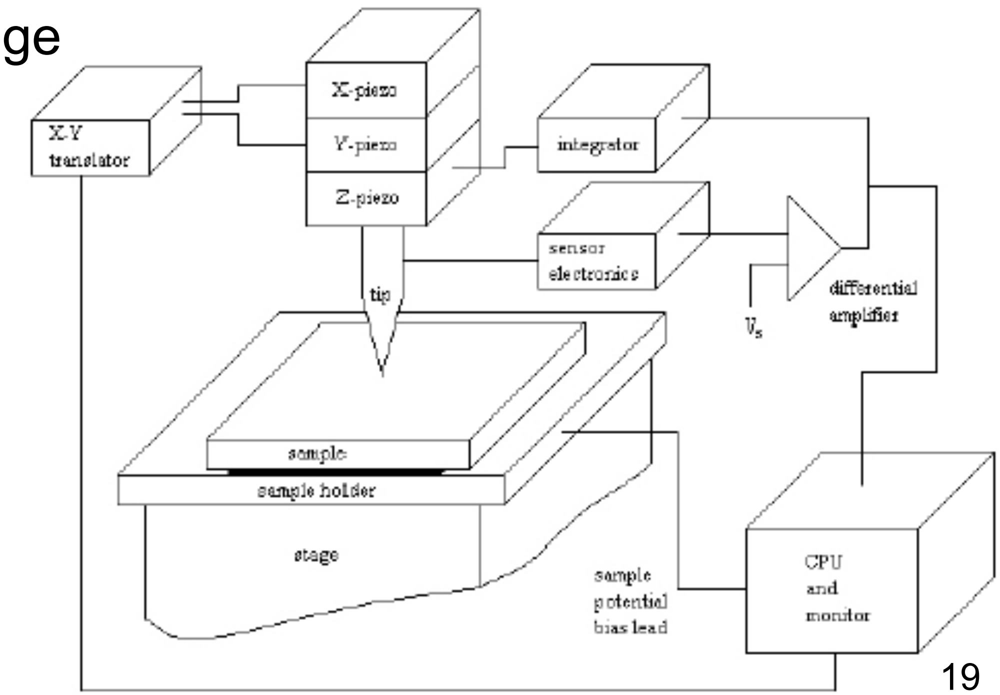
The main challange of SPM: How to get nm resolution?

Potential problems:	Solutions:
1. Tip size	1. Short-term interactions
2. High-resolution XY scanning	2. Piezo scanner
3. Non-destructive	3. Non-contact
4. Constant distance from sample	4. Height feedback
5. Vibrations	5. Rigid structure, isolation
6. Thermal stability	6. Compensation

High resolution: the piezo scanner

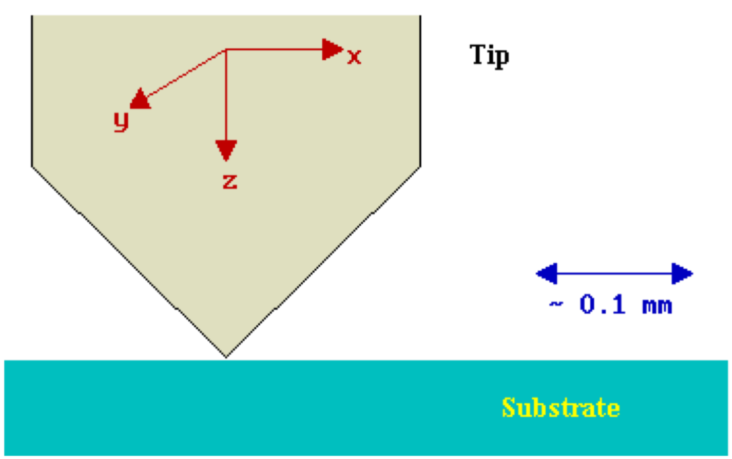
- The X-Y-Z relative movement between tip and sample is controlled by a Piezoelectric Scanner with < 1Å precision
- During the scan, we need to measure and stabilize **tip height**:
  - Measured through tunneling current (very sensitive!)
  - Can be used to control tip height by a **feedback loop**
  - Height is displayed as a 3D image

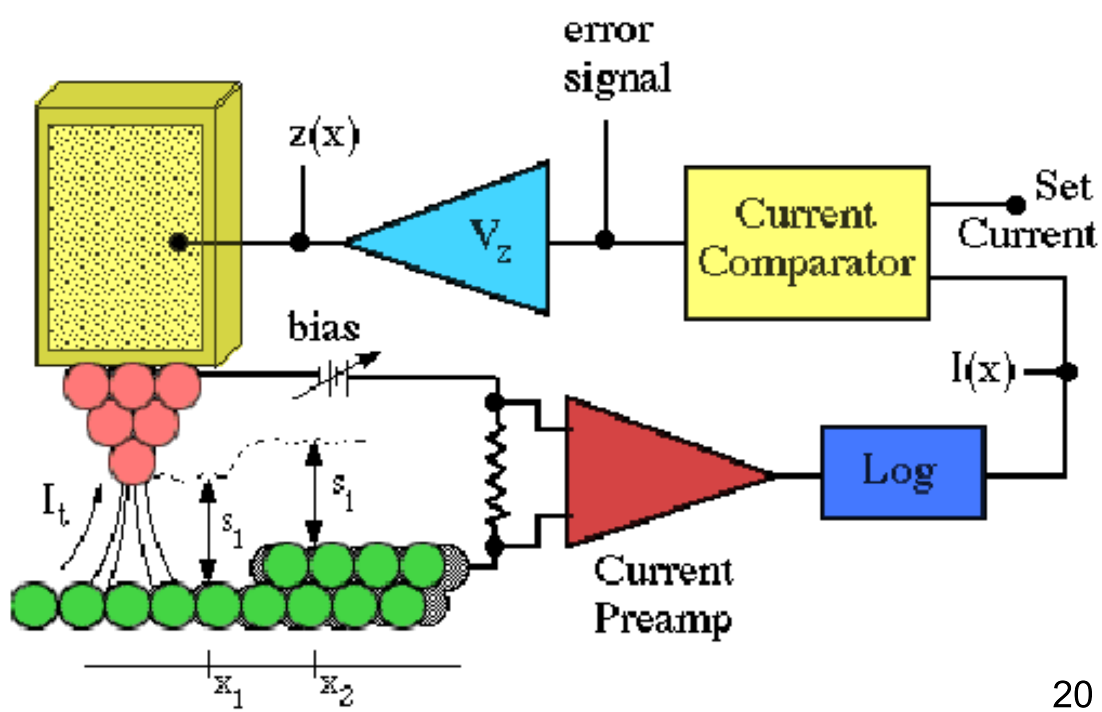




The use of feedback :

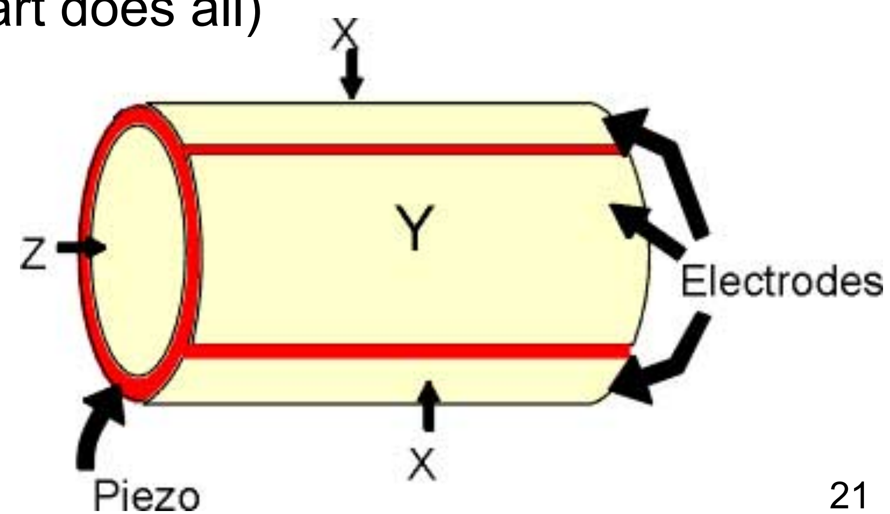
- In STM, current varies exponentially with tip-sample distance
- A log amp gives signal inversely proportional to distance
- The signal is fed back to a Piezoelectric actuator, to keep the same current = same distance
- The piezo tracks the surface of the sample, giving a high-resolution height map





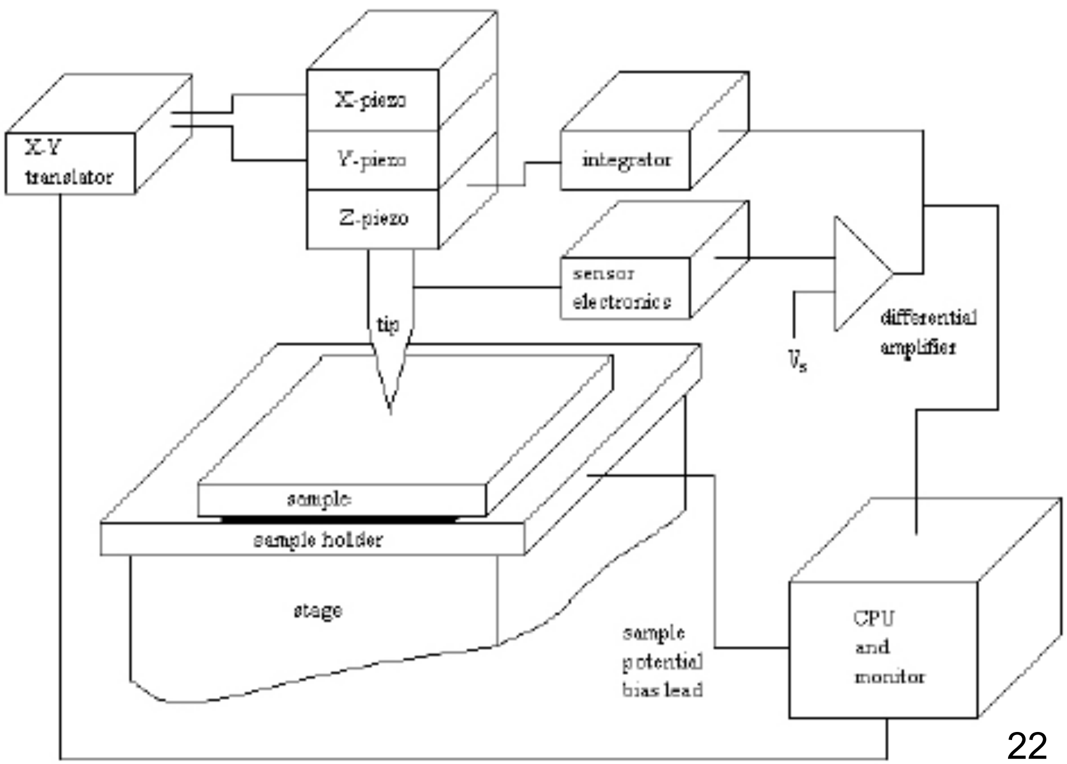
Types of piezo scanners: Tube

- All movements (X-Y-Z) can be achieved by a single tube scanner:
  - Applying opposite voltages to pairs of electrodes bends the tube in one direction (X,Y)
  - Applying voltage to the inner electrode makes the tube contract/expand (Z)
- Advantages of tube scanner:
  - Simple, small, rigid, cheap (single part does all)
- Disadvantages of tube scanner:
  - Non-linear (especially XY)
  - Difficult to add position sensors
  - All scan axes linked



Types of piezo scanners: XYZ

- Cartesian movement is often achieved by using separate Z and XY scanners
- High-quality XY scanners are available, also with high-resolution position sensors (resistive or capacitive) to achieve linear scan
- Z piezo (used for the feedback!) is uncoupled from the XY scan



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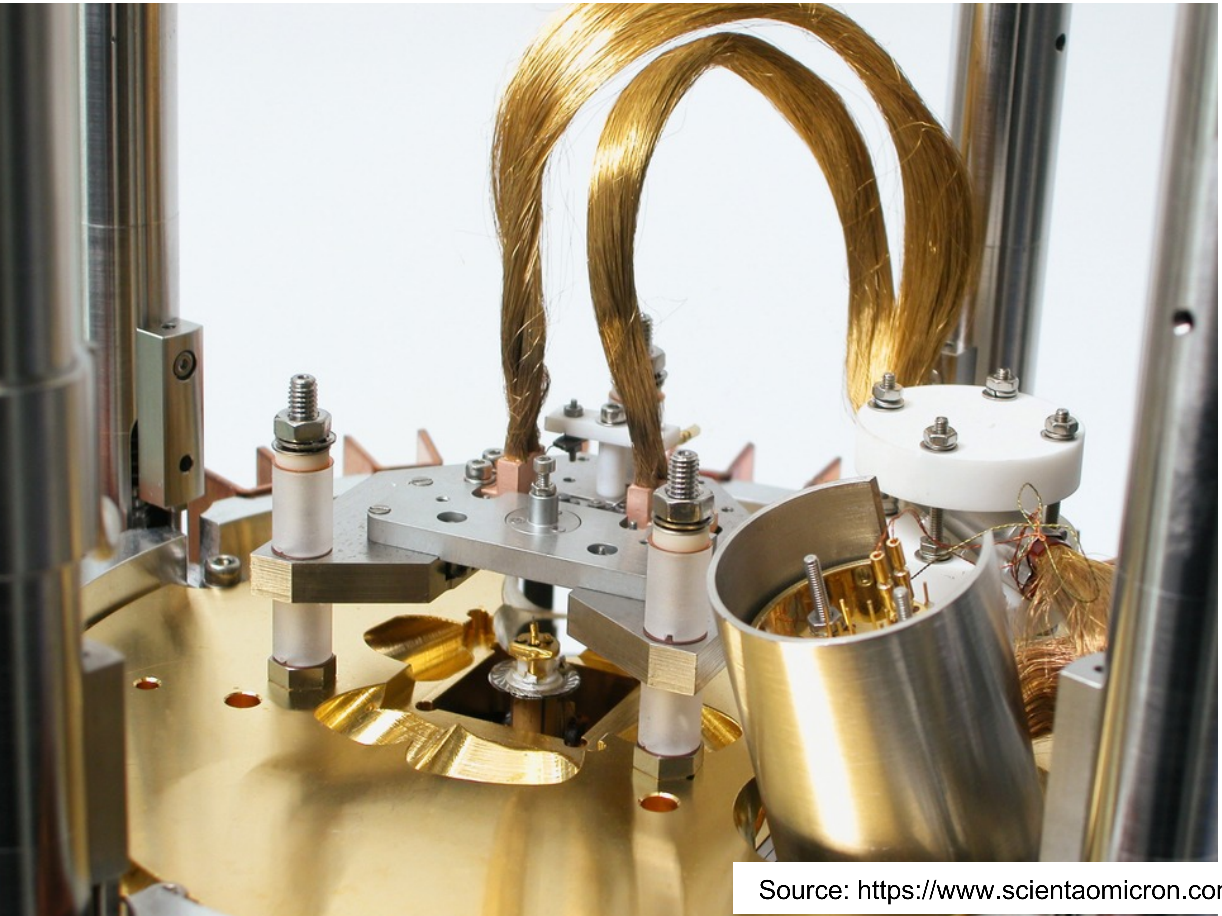
STM construction

- On top of the scanner and tip, we need:
- Mechanical stabilization (vibrations!)
- Environment control (vacuum, low temperature)



UHV LT STM

Source: https://www.scientaomicron.com/en/products/scanning-probe-microscopy

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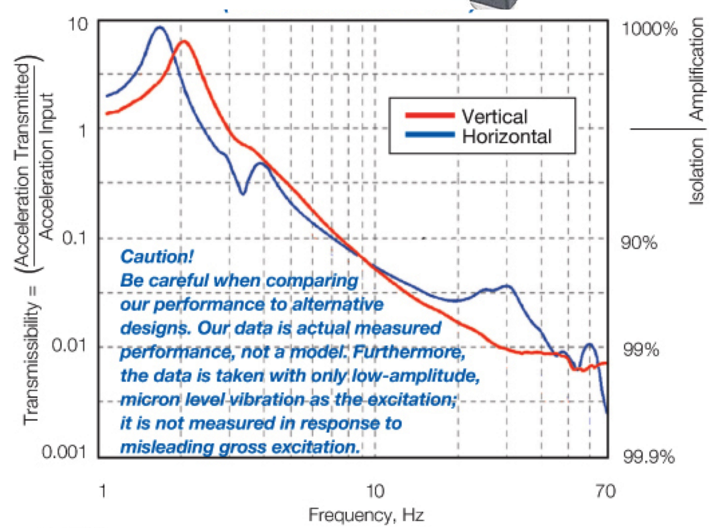
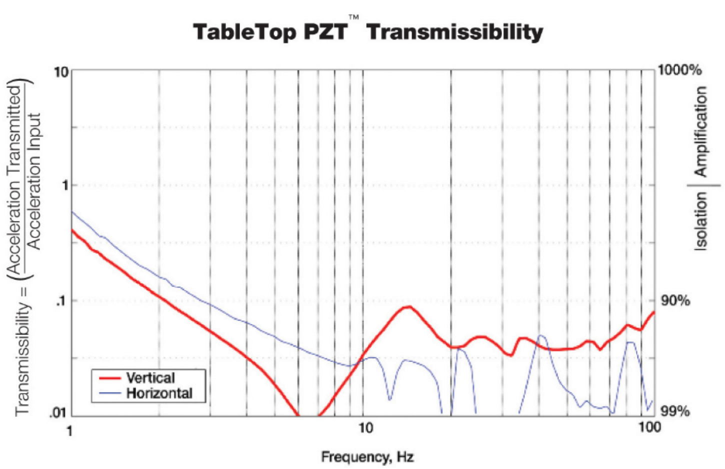
Vibrations

- Passive tables:
  - Passive - needs only compressed air
  - Can support very heavy weights
  - No damping below 2-3 Hz – amplification at resonanc
  - Performance increases with frequency T ~ ω^{-2}
- Active tables:
  - Small size, light weight
  - Good attenuation at low frequencies
  - Limited performance above 10 Hz

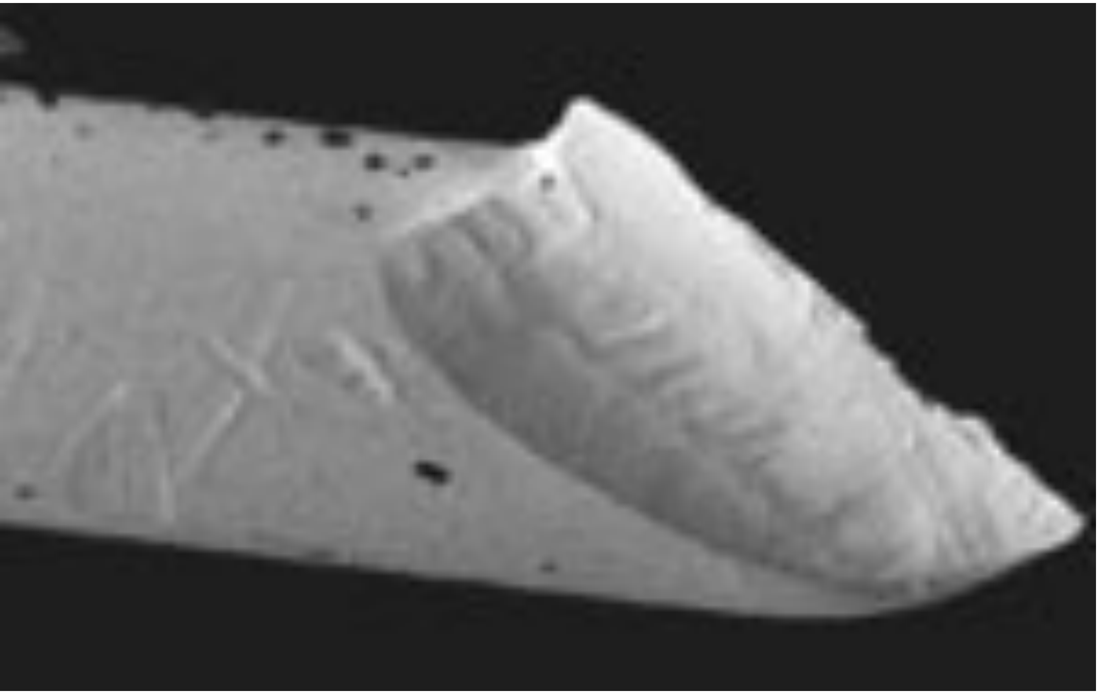




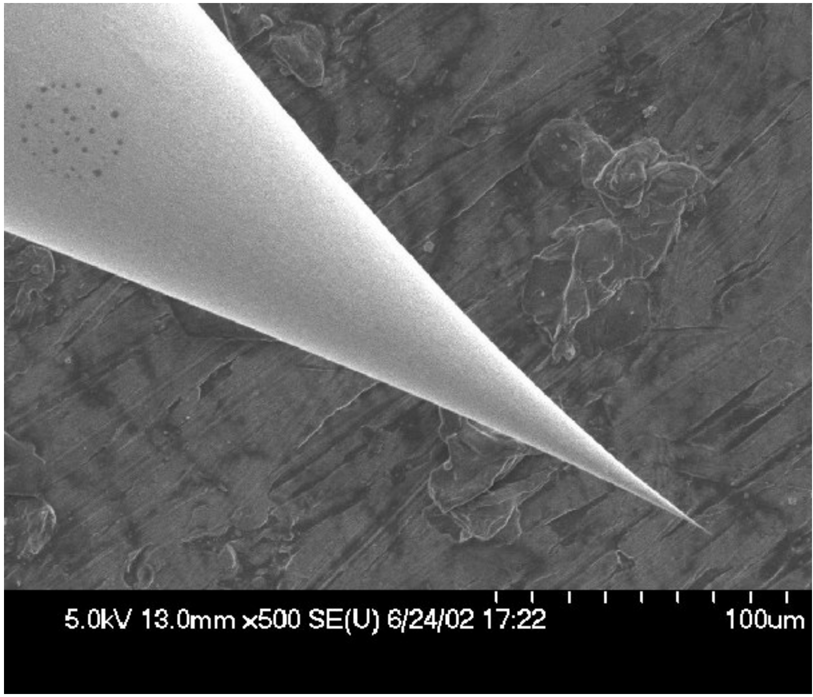
Source: TMC corp., http://www.techmfg.com

STM tips

Which STM tip is better ?



Pt/Ir wire, diameter 0.2 mm, cut with wire cutter

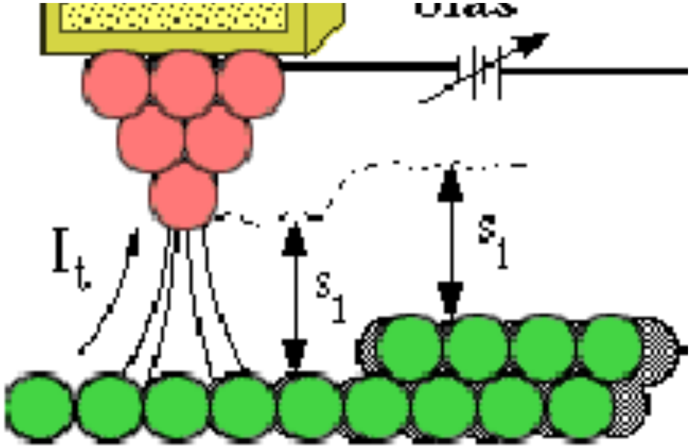


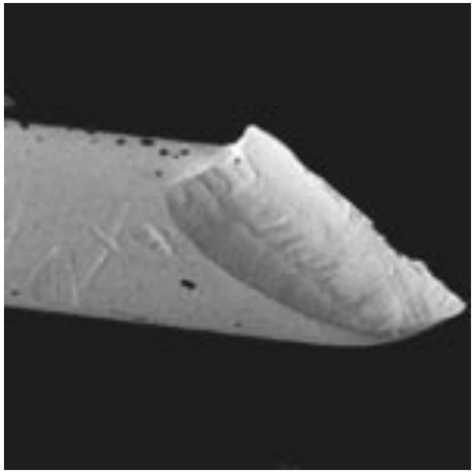
W wire, diameter 0.2 mm, electro-etched in KOH solution

STM tips

Answer: They give the same performance!

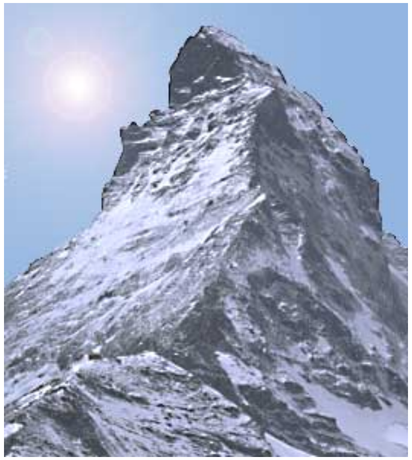
- Tip radius is not very important: rigidity is more important than tip radius!
- Exponential tunneling current dependence “selects” that only the very last atoms of the tip participate in the imaging



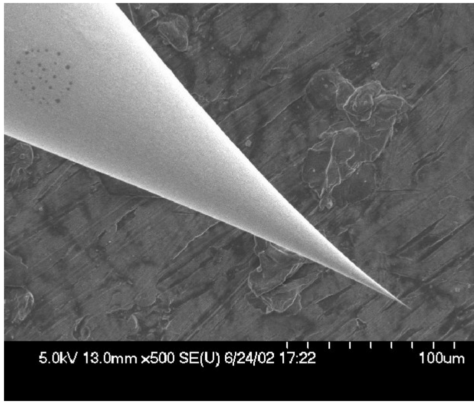


Cut STM tip

"Swiss" STM tip



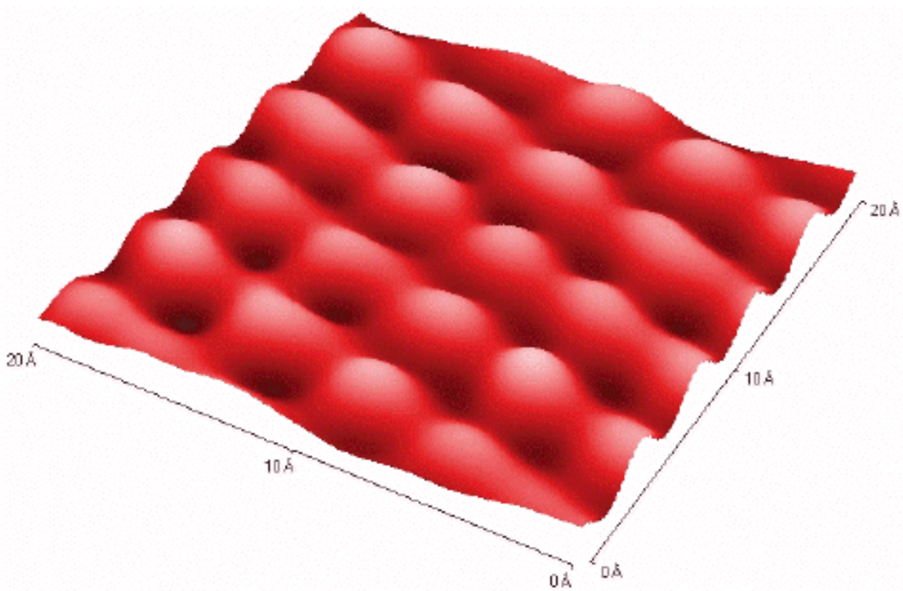
(Same, enlarged x10^8)



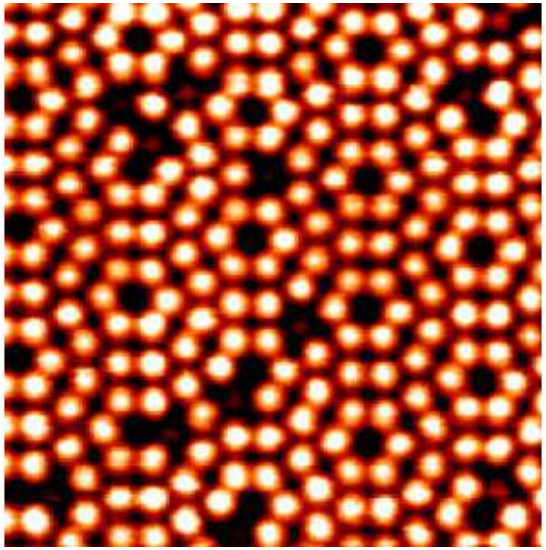
Etched STM tip

STM Images

- STM images can resolve individual atoms, or parts of molecules, on surfaces – preferably under vacuum



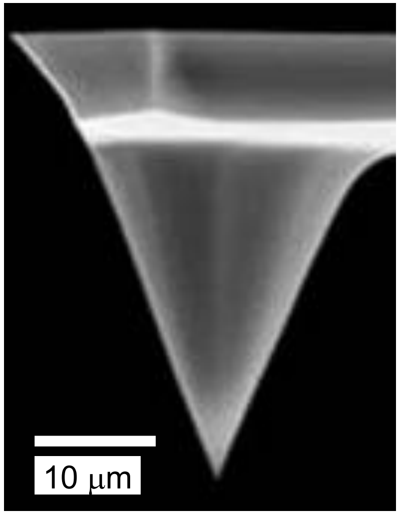
STM Image of graphite atoms



STM Image of Si atoms

More STM Images later …

Atomic Force Microscope: AFM

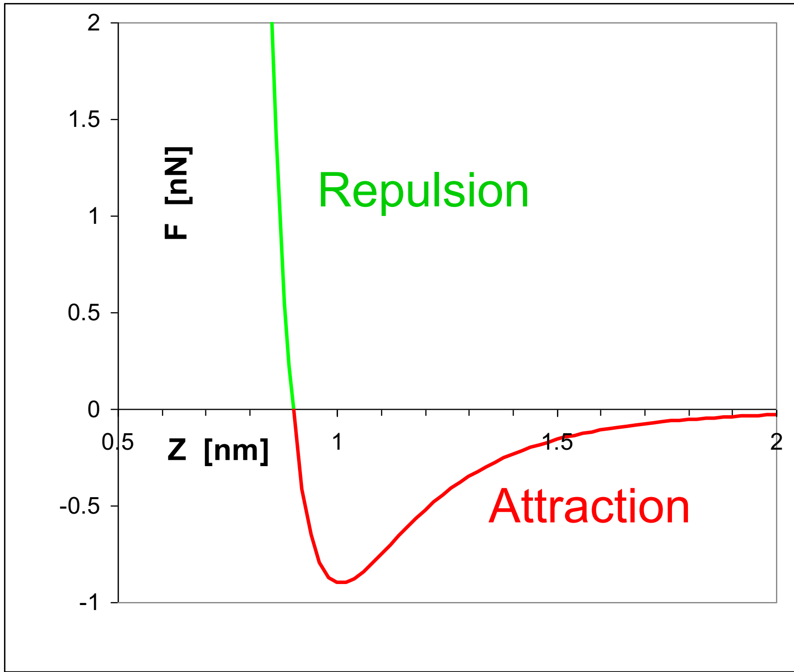


- Developed by Gerd Binnig (1984)
- Uses the Van-der-Waals force between tip and sample as short-term interaction
- Especially the repulsion force is very sensitive to distance:

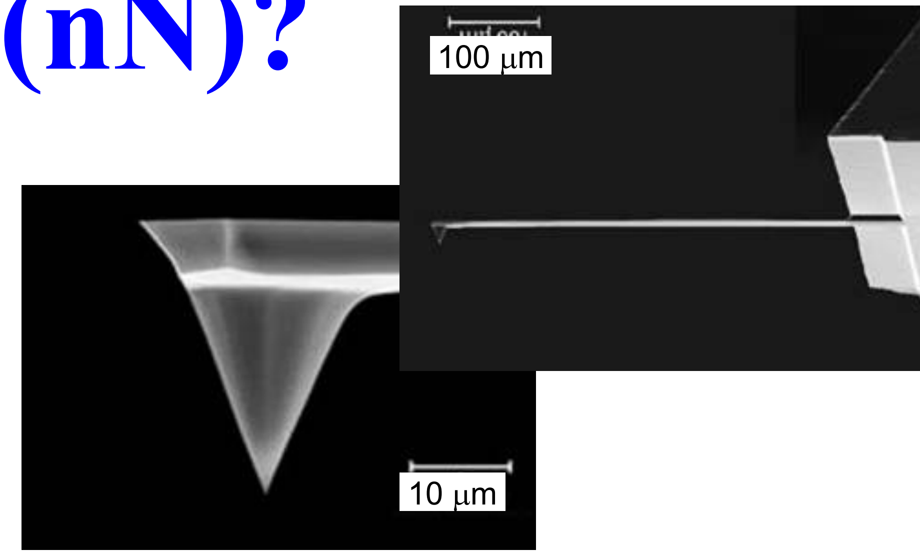
$$F \sim z^{12} \,!$$

(almost as good as exponential) and short-range (<1 nm)

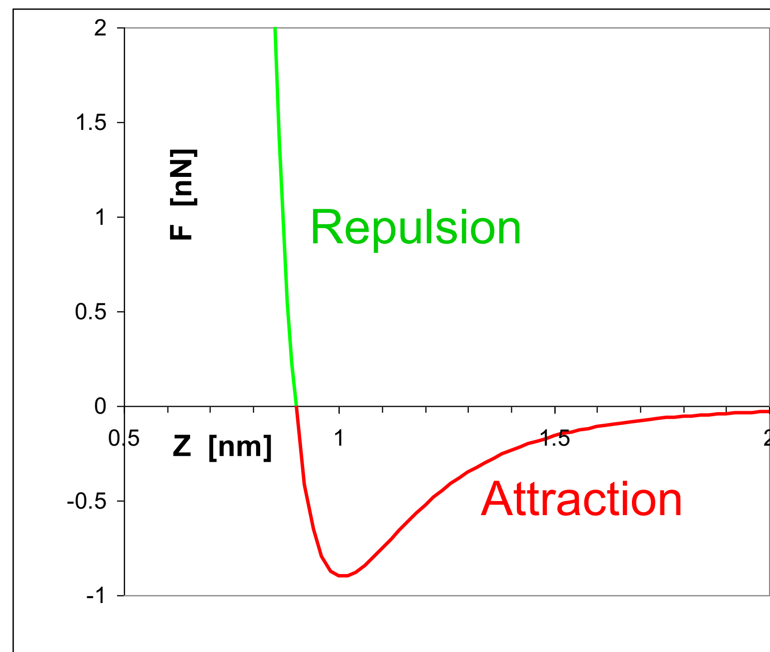
- But: Van-der-Waals force are small: order of nN
- We need a sensitive force sensor



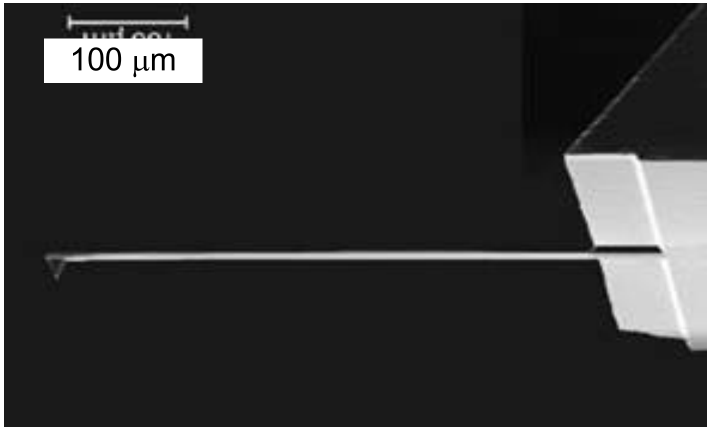
AFM: How do we measure small forces (nN)?



- Solution: use the flexibility of the tip-carrying cantilever
- Material: Si
  - Tough, flexible $E = 2 \cdot 10^5$ N/cm^2
  - Easy to structure by photo-lithography and chemical etching
- The force on the tip moves (flexes) the cantilever.

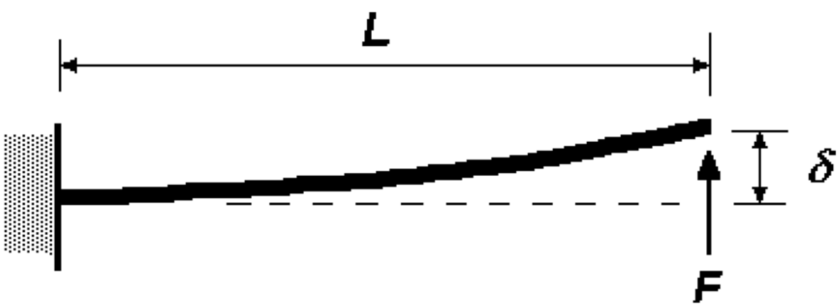


How do we measure small forces (nN)?

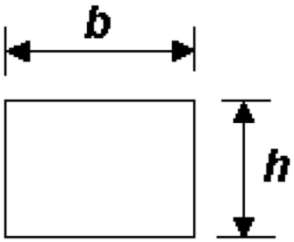


- Deflection of a beam: $\delta = FL^3/3EI$
- For a typical Si cantilever ($L=400\ \mu m$, $b=20\ \mu m$, $h=2\ \mu m$): $\delta = 0.2\ \mu m/nN$
- Next question:

How do we measure a small deflection?



Moment of inertia for rectangular section



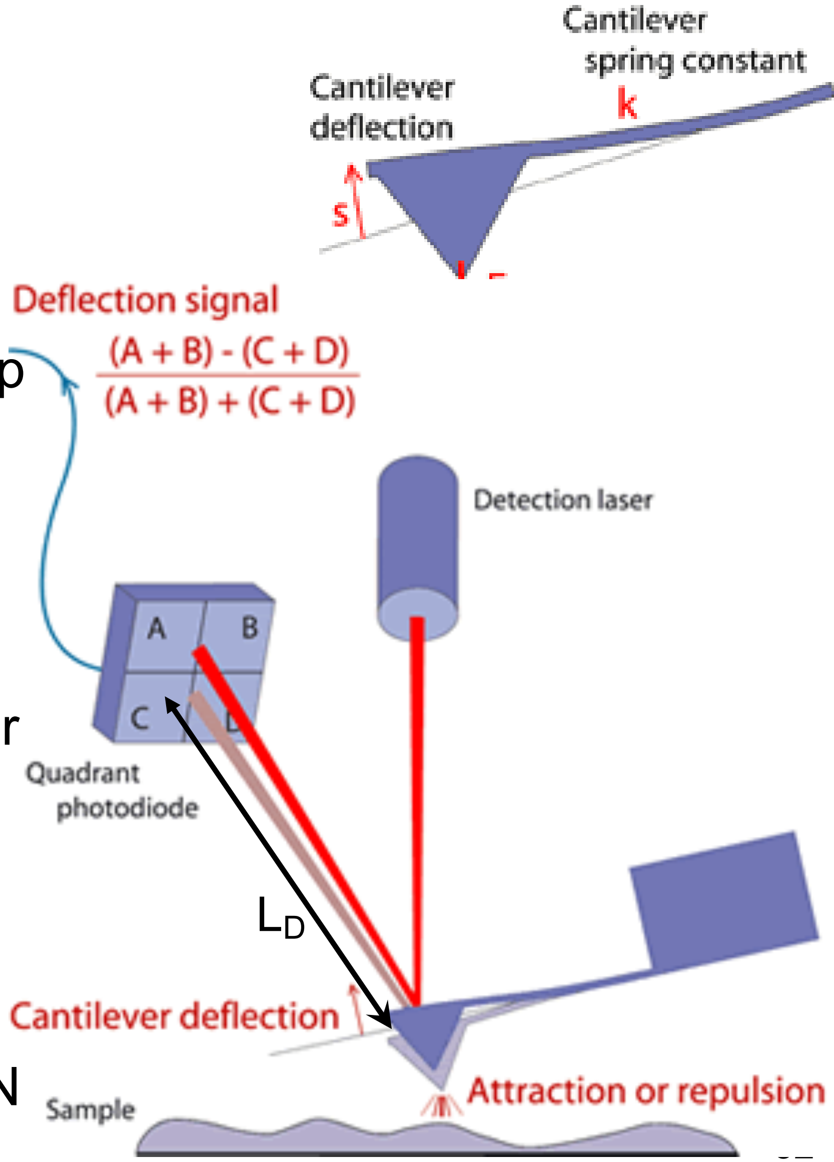
$I = bh^3/12$

where h is the dimension in the plane of bending, i.e. in the axis in which the bending moment is applied.

http://www.clag.org.uk/pics/beams/moment1.gif

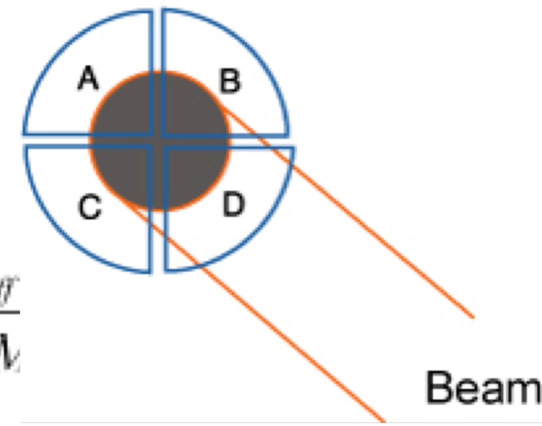
How do we measure small deflections (nm)? With a laser!

- A laser beam is reflected from the cantilever to a position-sensitive photodetector
- The detector signal is proportional to the tip displacement = force
- The tip is scanned over the sample (as in STM) to produce the image
- We saw that the deflection of the cantilever is: $\delta = FL^3/3EI$, typical 0.2 μm/nN
- The reflected laser beam moves by a distance: : $\delta L_D = \delta \cdot L/L_D = FL^2/3EIL_D$
- Typical value (L_D~20 mm): δL_D = 10 μm/nN



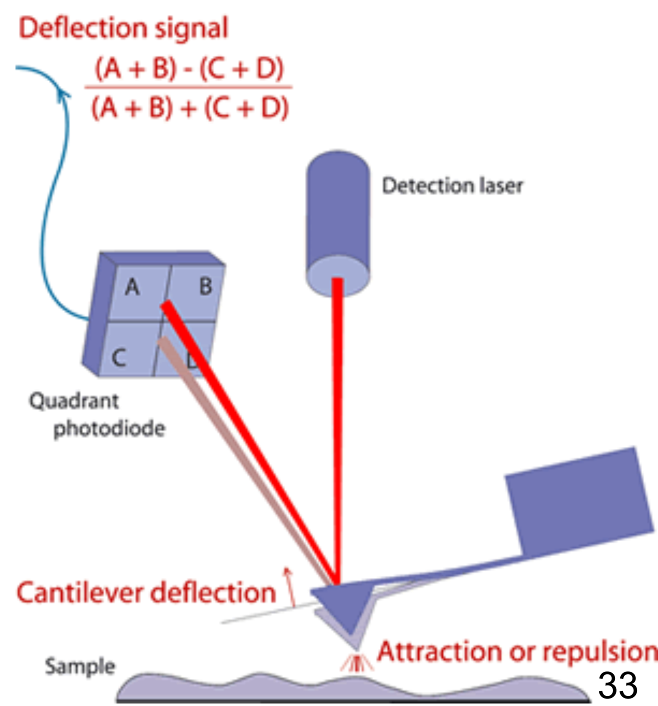
Operation of contact AFM

- The beam movement (10 μm/nN) is easily detectable by a 4-quadrant photodetector:



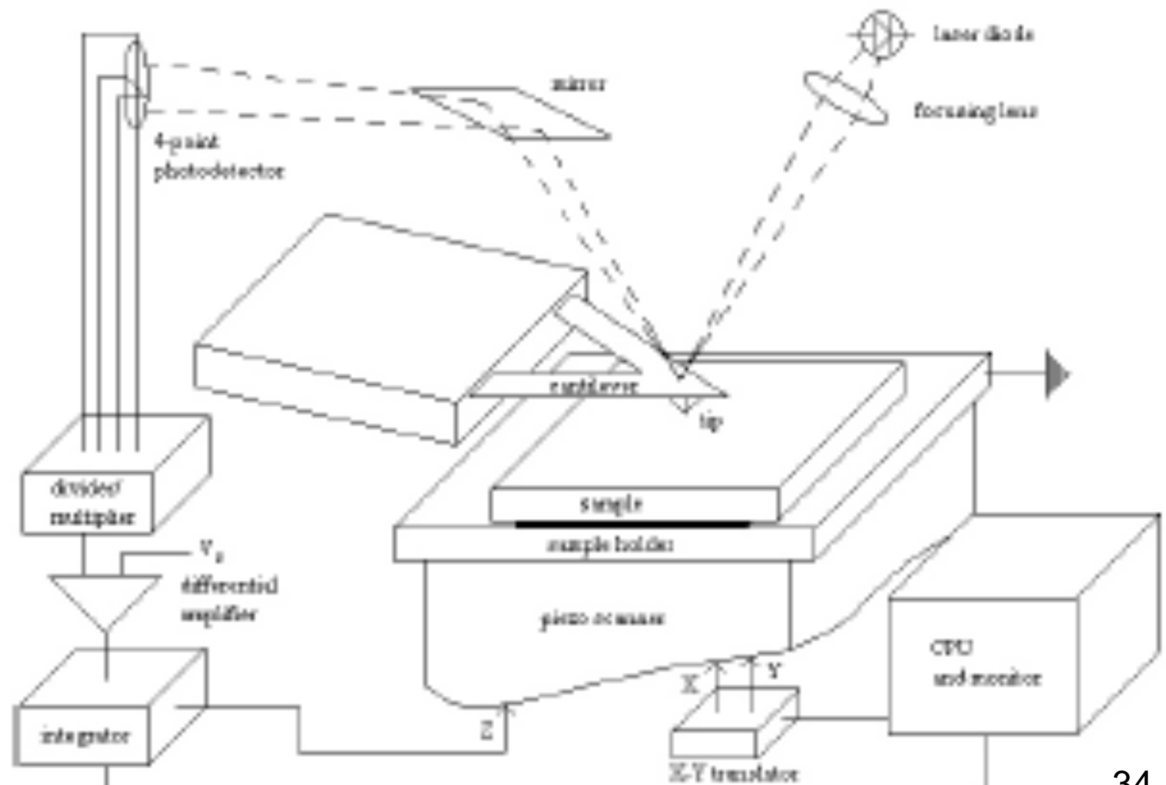
$$X = \frac{(A+C)-(B+D)}{(A+B+C+D)} = \frac{X_{Diff}}{SUM} \qquad Y = \frac{(A+B)-(C+D)}{(A+B+C+D)} = \frac{Y_{Diff}}{SUM}$$

- SUM signal is used for beam alignment
- Y signal is used to detect the cantilever movement (zeroed before contact)
- X signal used to detect lateral forces
- Typical values: Beam diameter 1 mm, P_{refl}=10μW, gives a SUM signal of 10V. Difference of 10 μm/nN (1%) gives a difference signal of 0.1 V



Contact AFM feedback

- As in STM, feedback is used to keep the tip at a constant force from the sample, the height is plotted as image.
- On flat surfaces (to a few nm), constant height can be maintained, and the force is displayed



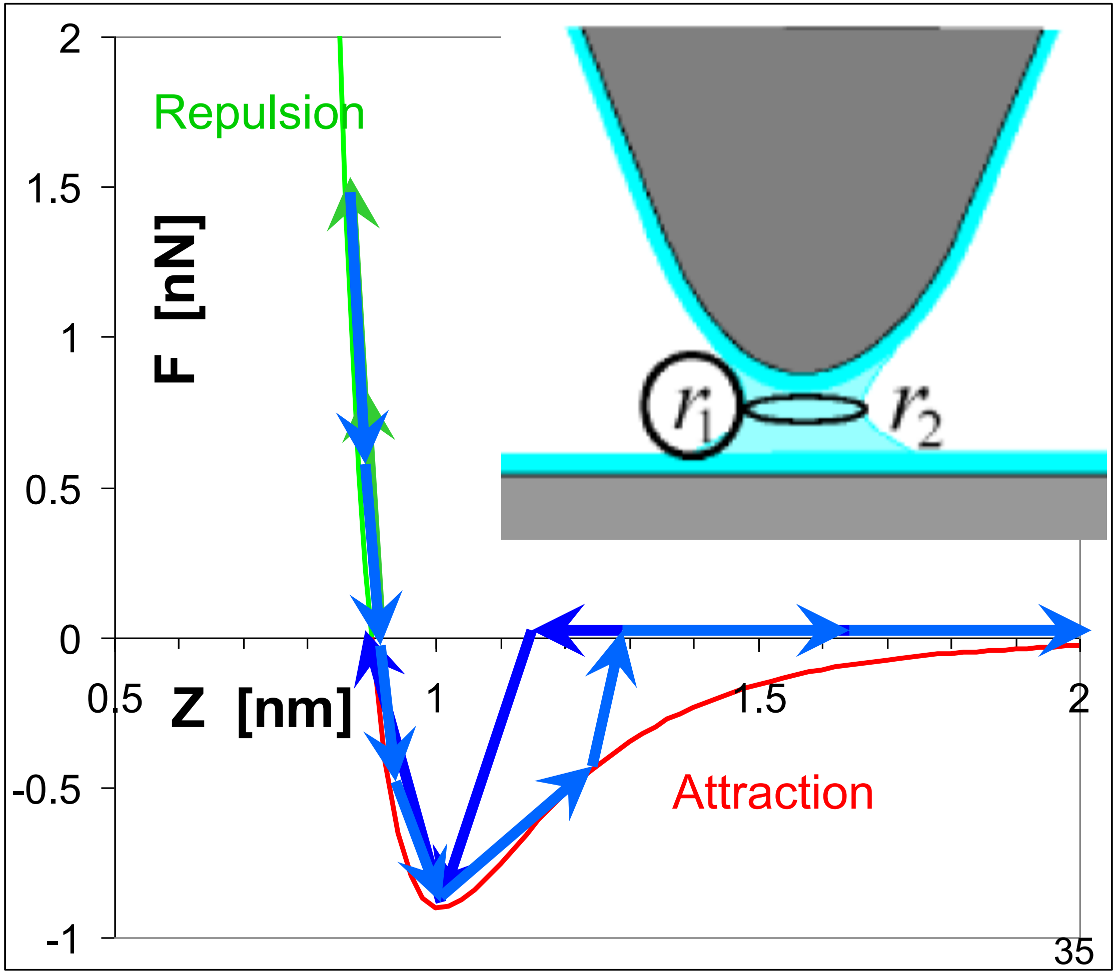
The other side of the force ...

- The Van-der-Waals force between tip and sample: $F \propto \left(\frac{1}{z^6} - \frac{1}{z^{12}}\right)$
- When the tip approaches, the force is attractive, then strongly repulsive

The attractive force:

- Makes the tip “jump” to touch the surface quickly when approaching
- Makes the tip “stick” to the surface when retracting

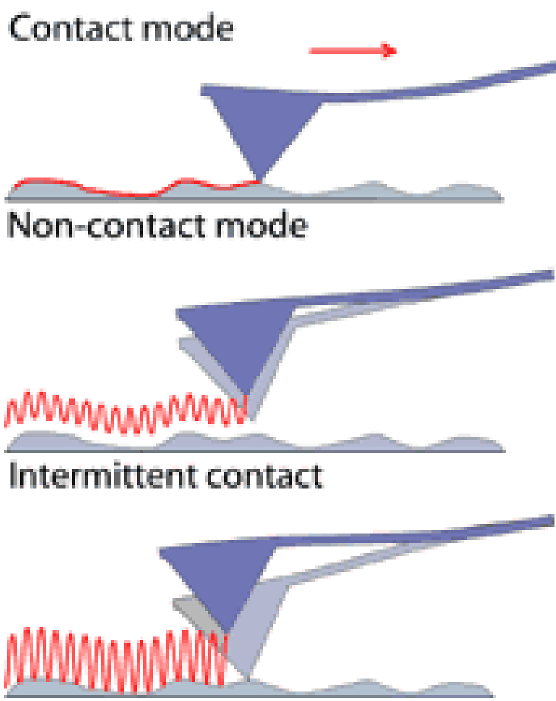
- Result: hysteresis in tip movement !

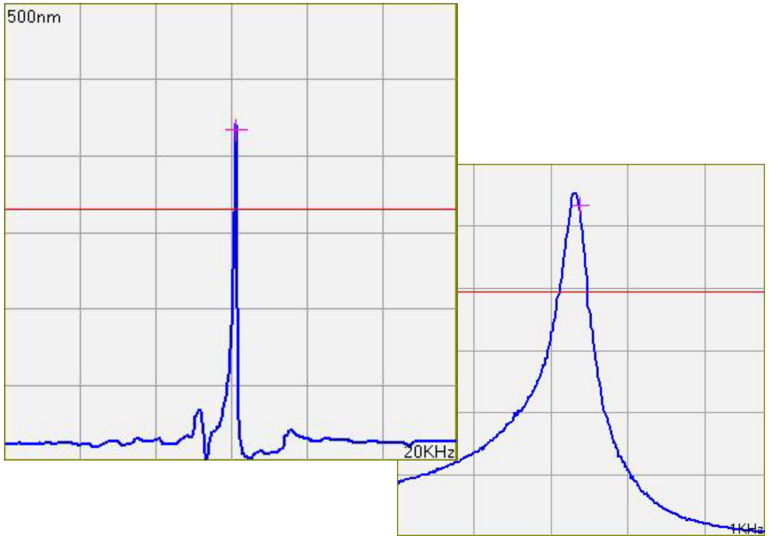


This is most problematic in ambient air, as the sample and tip are covered by a thin (1 nm) water layer.

Non-contact AFM: no sticking!

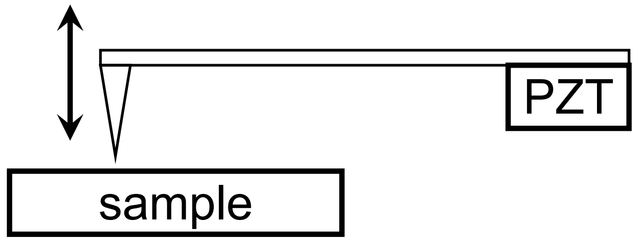
- To avoid the problems of tip sticking, non-contact AFM is used.
- "Pure" non-contact: tip never touches the sample, oscillation amplitude is small
- Intermittent contact, or tapping: large amplitude, at every oscillation cycle the tip touches the sample.
- The cantilever is vibrated by a piezo at its resonance frequency: $f_0 = \sqrt{K/\mu}$ where K = force constant, μ = cantilever mass
- The laser beam reflected onto the position-sensitive detector moves at the resonance frequency
- The PSD signal has an AC component, showing the tip's oscillatory motion
- At resonance, the transducer needs to supply minimum energy to maintain oscillations



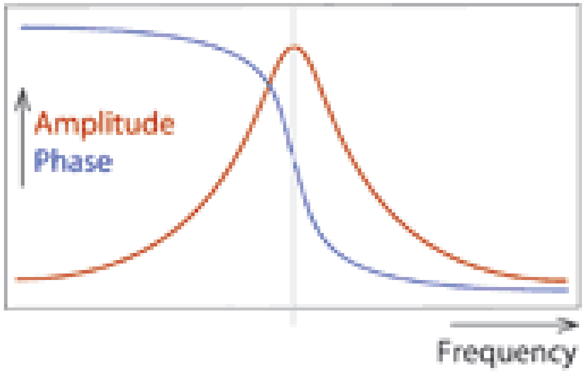


Feedback in non-contact AFM

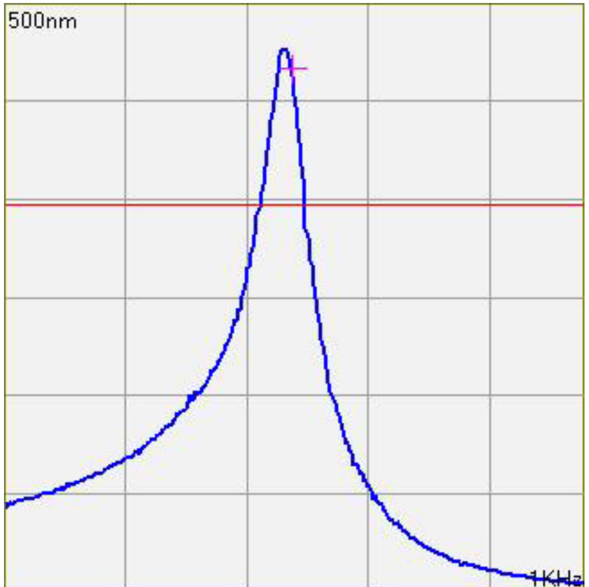
- What changes by tip-sample forces ?



- Changes in the vibrations'
  - frequency
  - phase
  - amplitude

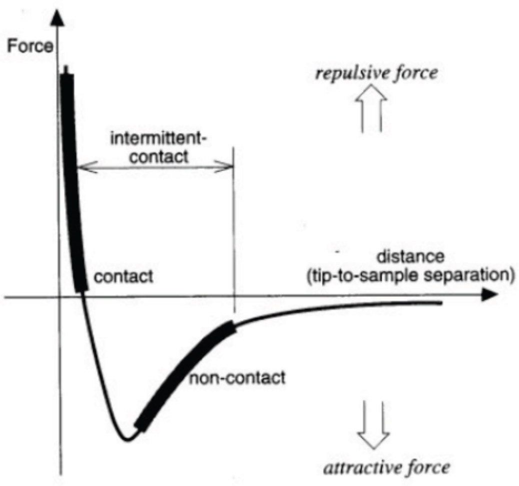


- In NC-AFM, usually frequency is used for feedback. In IC-AFM, usually the amplitude change is used
- Sometimes lock-in (phase) detection is used, e.g. to plot height + phase images

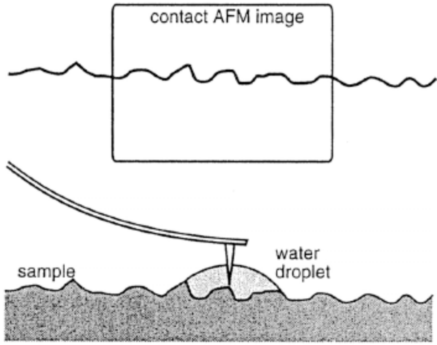


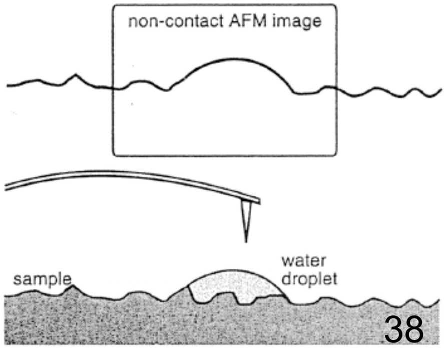
Force, resolution in non-contact AFM

- NC and IC AFM use different force regimes:
  - AFM works with repulsive forces, at close distance (<0.5 nm)
  - NC-AFM works with the attractive force, at a larger tip-sample distance (1-10 nm)
  - IC_AFM works with the repulsive force, at smaller distance, like contact AFM (0.5-2 nm)

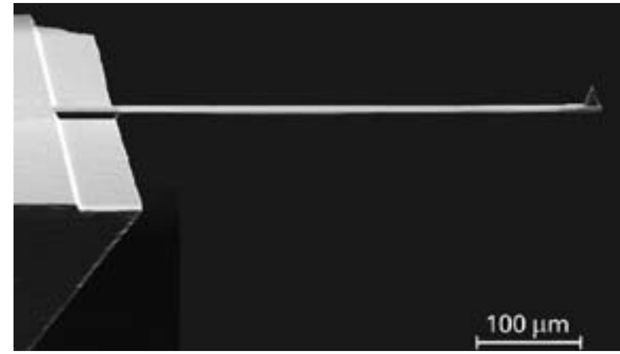


- Results:
  - Contact AFM has the highest resolution (atomic, like STM), but uses high forces (1 μN- 1 nN) which can scratch the surface
  - NC-AFM uses less force (1 pN-1 nN), good for delicate surfaces (polymers), but has lower resolution
  - IC-AFM uses higher force (1 μN- 1 nN), good for hard and rough surfaces, has higher resolution

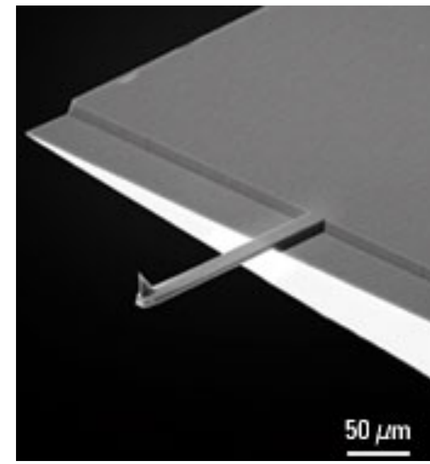




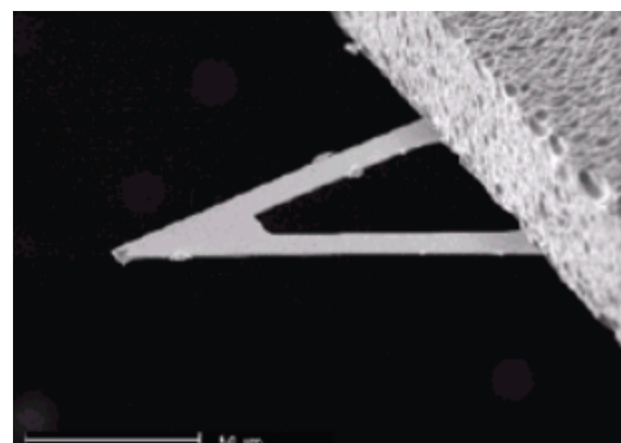
Types of AFM cantilevers



- For contact mode, usually a long cantilever is used to increase sensitivity (low K).
  - Typical values: K = 0.1-1 N/m, L = 250-400 µm
  - Resonance frequency is low (50 kHz)



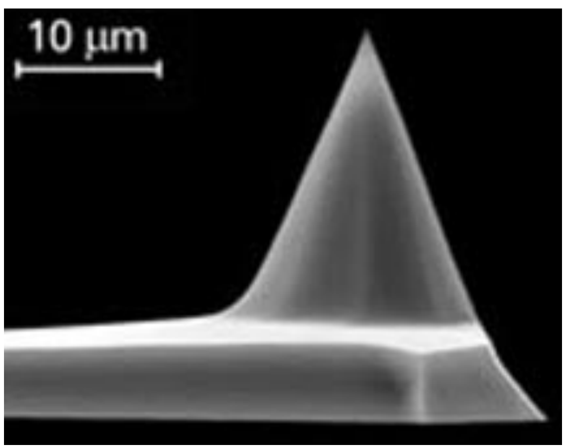
- For non-contact mode, usually a short cantilever is used to increase the resonance frequency.
  - Typical values: K=10 N/m, L = 100 µm
  - f_0 = 200-400 kHz



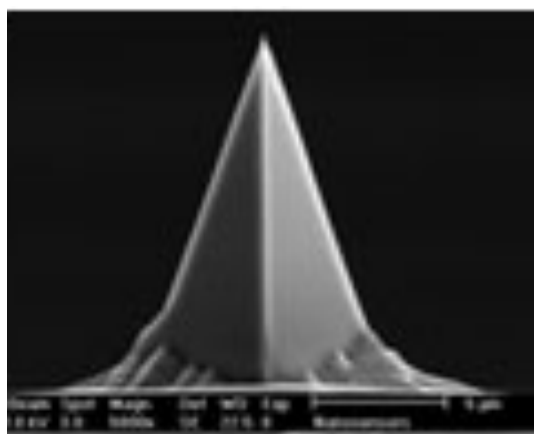
- Sometimes, two-beam cantilevers are used, e.g. to measure lateral forces

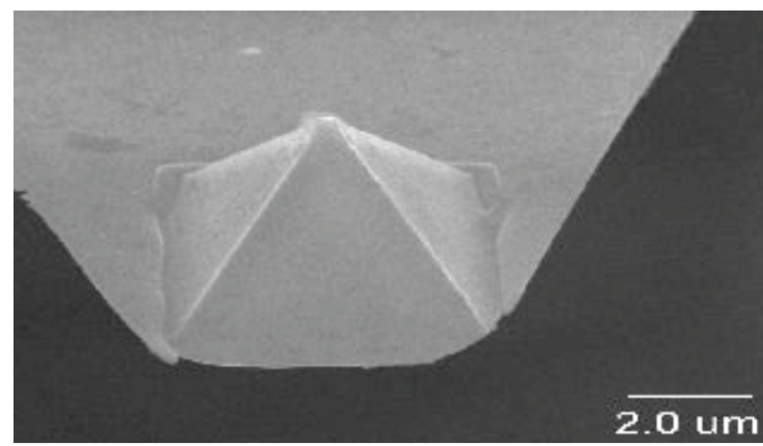
Standard AFM tips

- Most AFM tips are made from Si, by photolithography and directional etching.
- The tip is pyramidal in shape, with height ~20 μm and sidewall angles of ~20° to the normal
- The standard tip radius is on the order of 5-10 nm
- In some cases Si_3N_4 is used



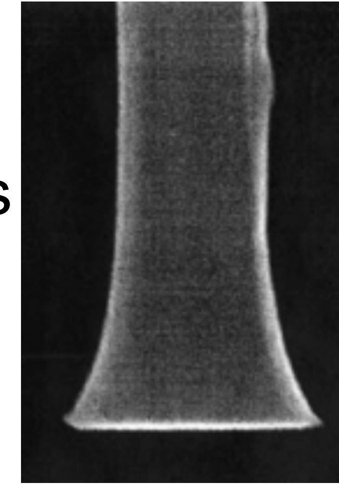
Si tip: Side view


Si tip: Front view

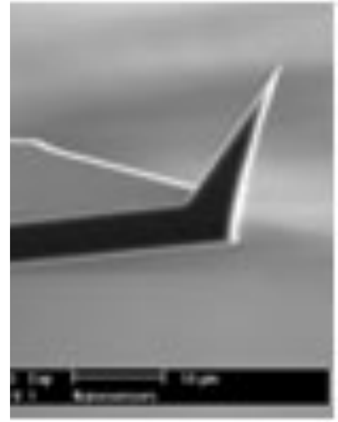


Si_3N_4 tip: Oblique view

Special AFM tips

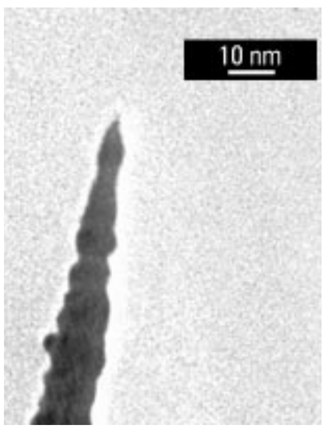


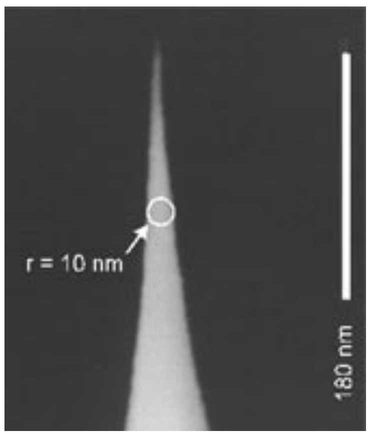
- For metrology : "elephant foot" to measure sidewalls



- Inclined tips to probe edges:
- Ultra-sharp tips,

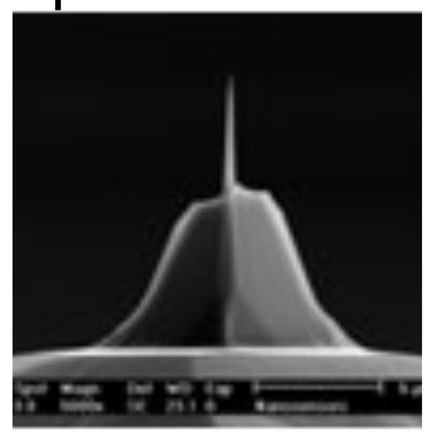
  down to 1 nm

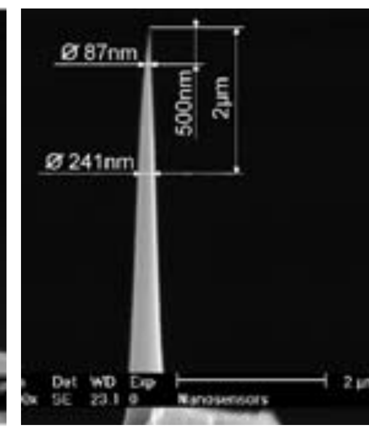




- High aspect-ratio tips to

  probe trenches and holes





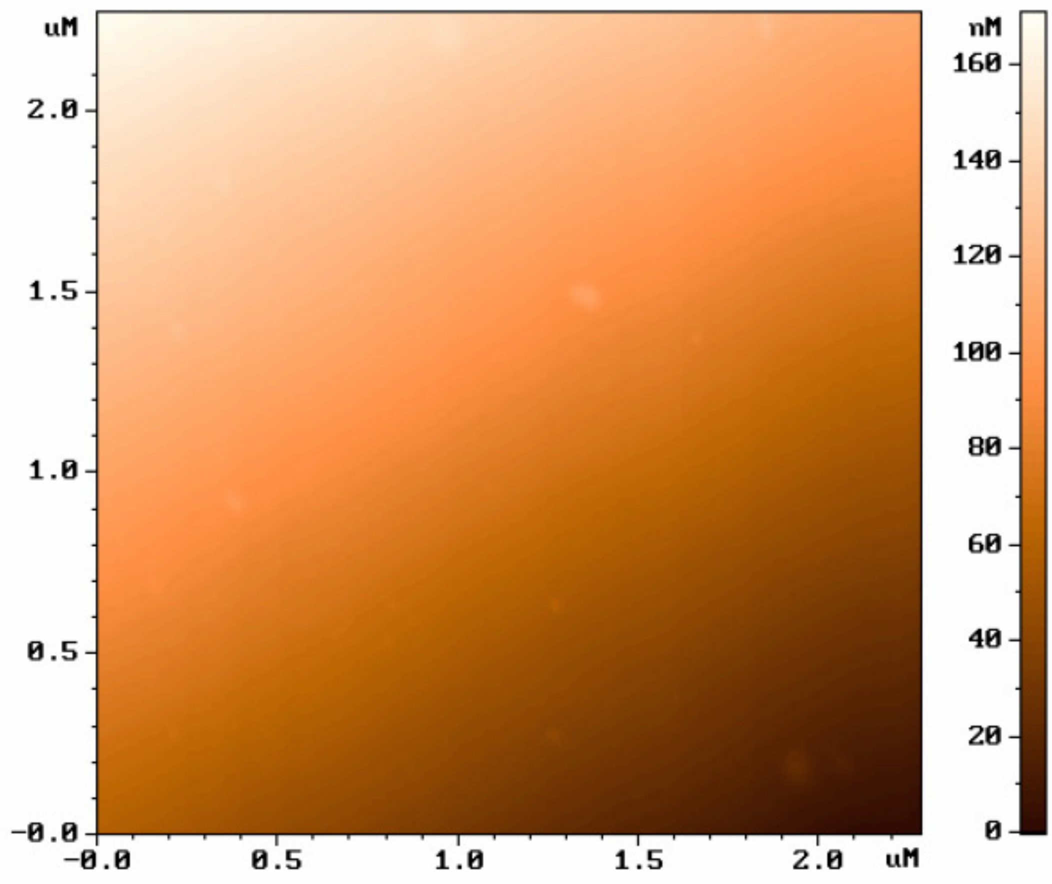
- Diamond tips for hardness testing and long life (low resolution!)
- Different coating on tips : conducting, magnetic etc.

Image processing

- SPM images are rarely perfect “as taken”
- “Basic” image processing:
  - Removing sample tilt, scanner non-linearity, tip jumps, noise
- “Advanced” image processing:
  - Filtering, deconvolution, finding & characterizing objects
- Measurements:
  - Size, distance of features
  - Line profiles & their characterization
  - Roughness
- Calibration
  - Scan (axes) calibration
  - Tip characterization

Basic Image Treatment

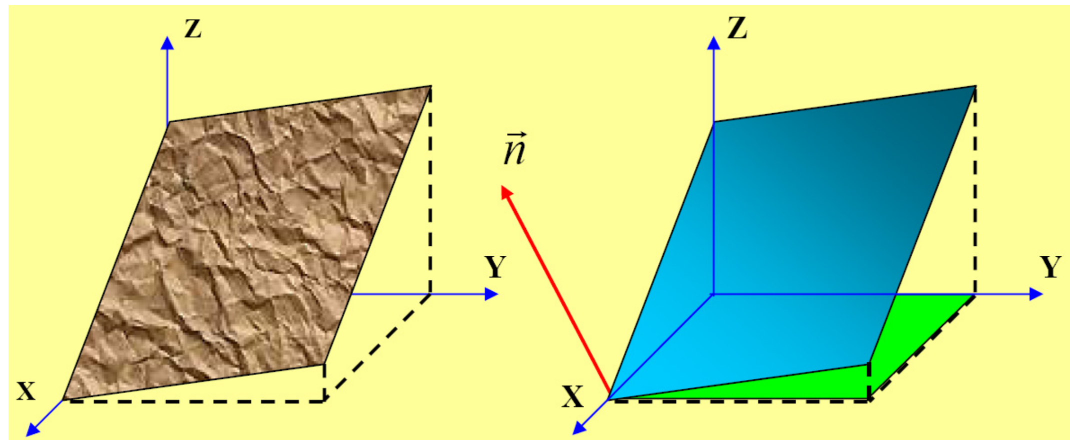
A newly-taken AFM image of a flat sample looks like this:



WHY ?

Basic Image Treatment: plane-fit

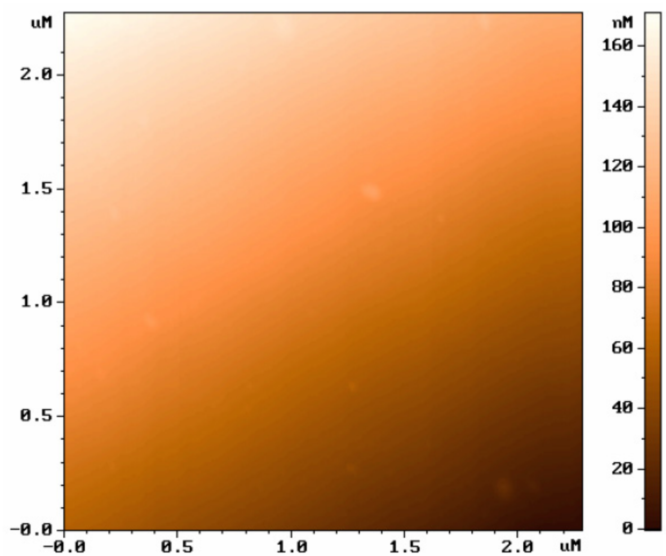
The sample is never horizontal! A 10mm sample mounted with one side higher than the other by 0.1mm, will give an image slope of 100nm over a 10 μm scan!



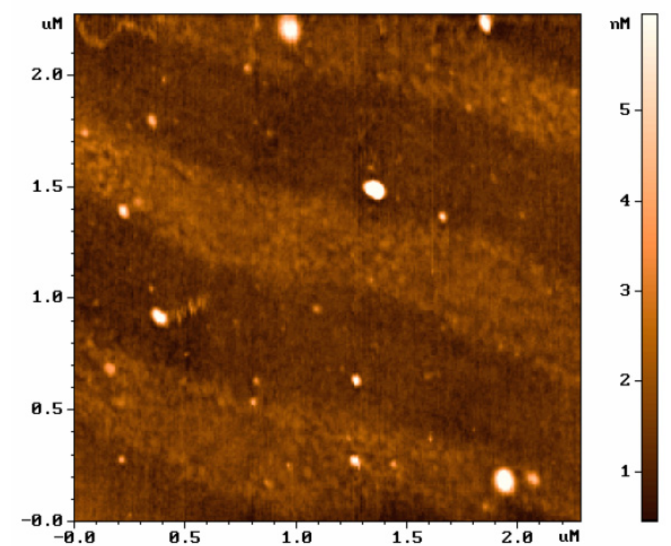
The plane-fit correction: An average plane is calculated by LSQ fit of the image to:

ax+by+cZ=0

This plane is subtracted from the image, to "planarize" it



"Before"

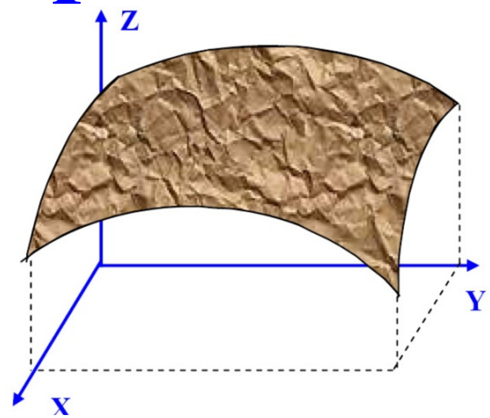


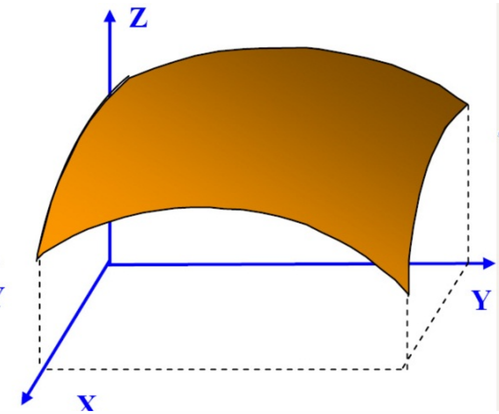
"After"

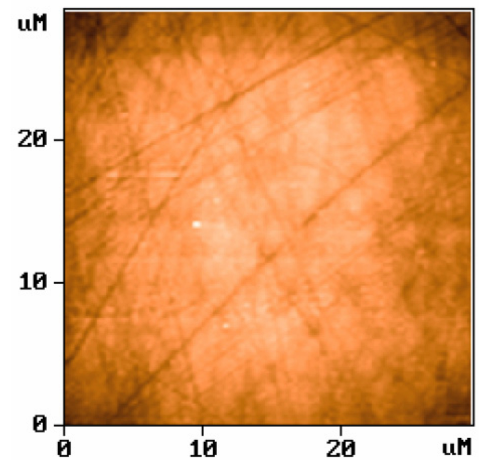
2^{nd} order plane-fit

- The piezo scanner can have non-linearities (especially tube scanners), leading to changes in Z values across the scanned plane
- In this case, a simple plane fit is not enough to correct the image, which is curved.

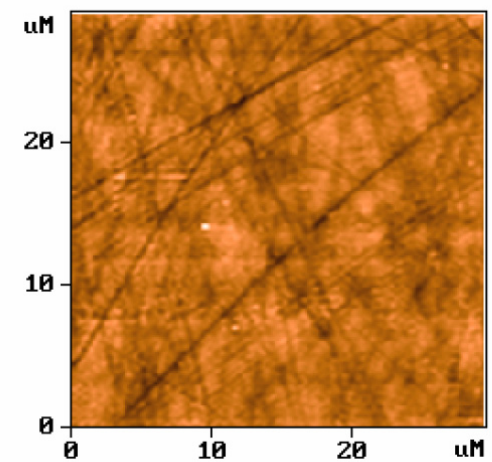
The 2^{nd}-order (and sometimes even higher order) plane-fit correction subtracts from the image a LSQ-fitted curved plane





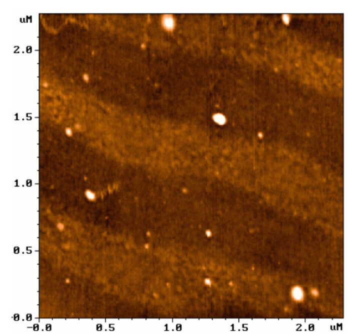


After 1^{st} order planefit



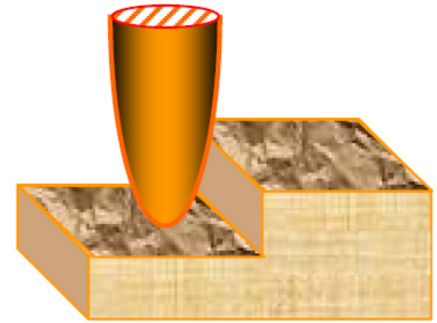
After 2^{nd} order planefit

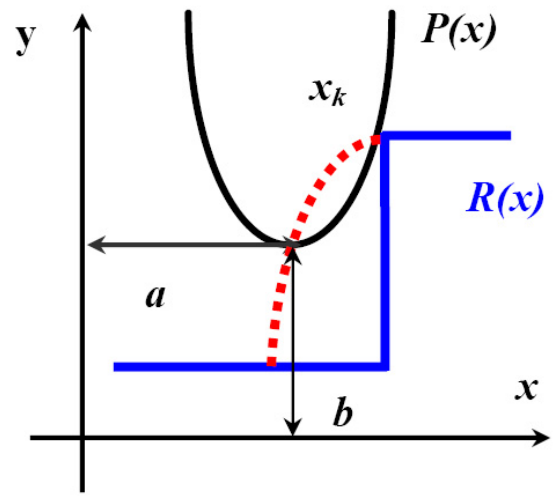
Caution: in some cases, high-order plane-fit correction can remove real image features (e.g. sample undulations)

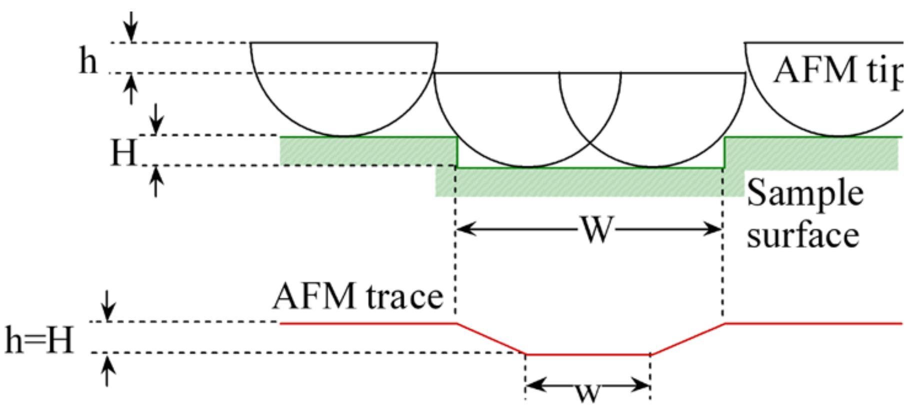


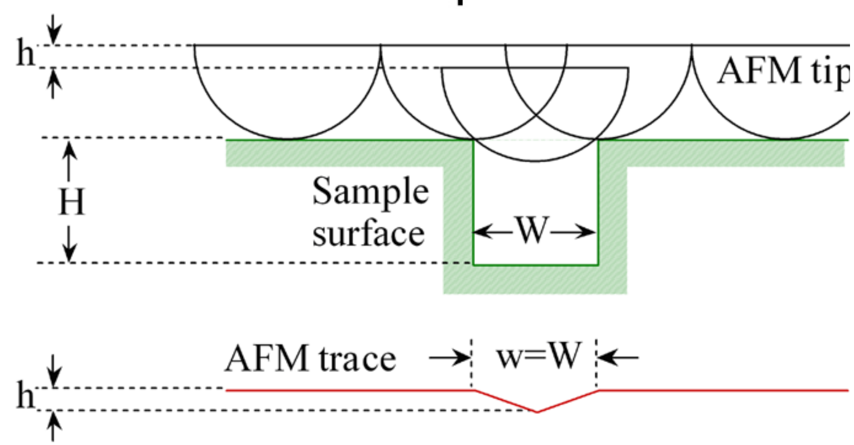
Effect of the tip size on the Image

- The AFM tip is never atomically sharp (standard R~10 nm), especially if it's broken !
- The image is the result of a geometrical convolution of the tip and sample
- The tip size will increase the apparent step width, but not its height
- A blunt tip will not penetrate a deep trench – a triangular image will result, shallower than the real depth.



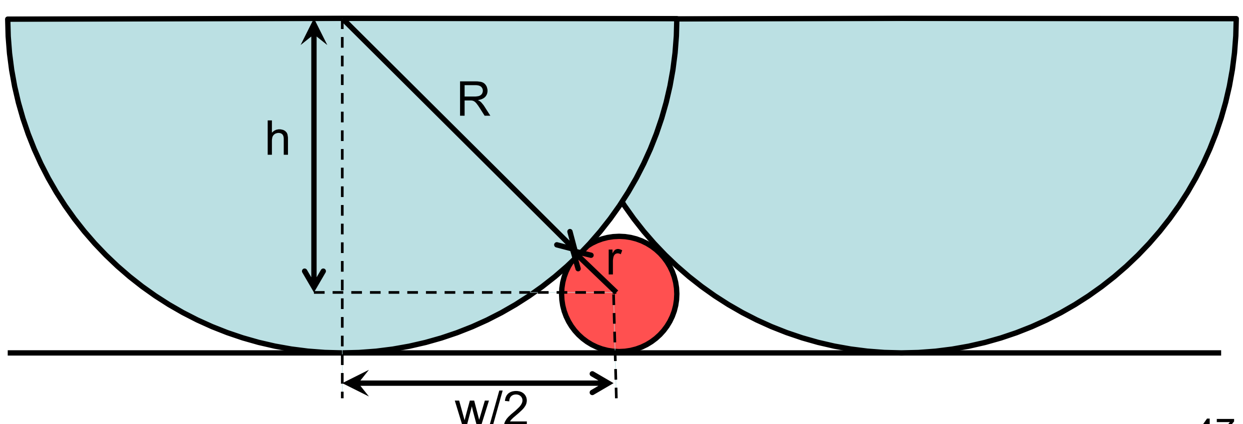






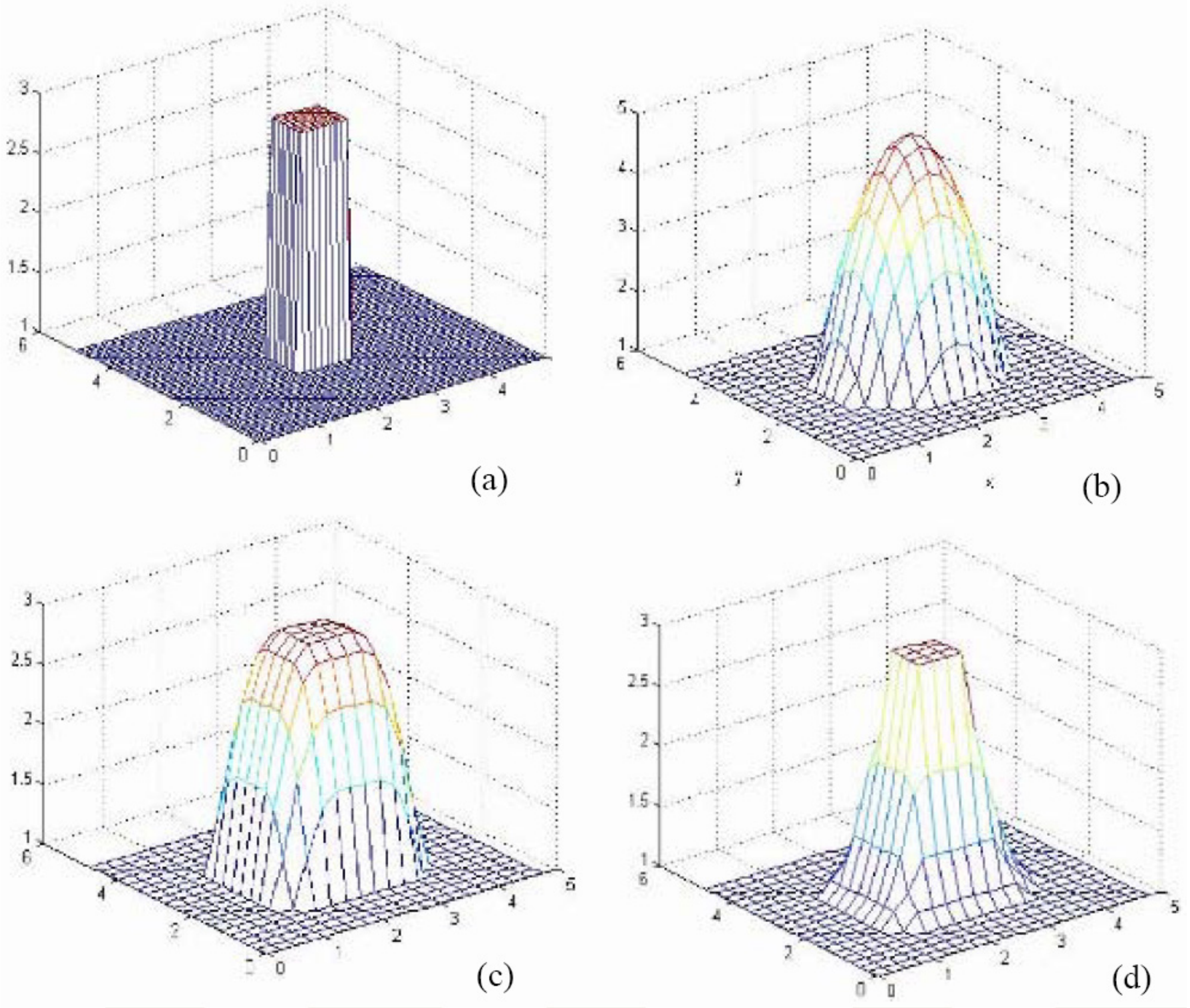
Effect of the tip size on the Image

- Example: Semi-circular tip, radius R, measures a molecule (or particle), radius r < R
- The apparent width is measures between the points where the tip touches the molecule from each side
- Geometrical calculation shows: $w = 2\sqrt{(R+r)^2 - (R-r)^2} = 4\sqrt{Rr}$
- Typical values: r = 1 nm, R=10 nm, giving w = 12 nm!
- The tip size will increase the apparent step width, but the height is correct!



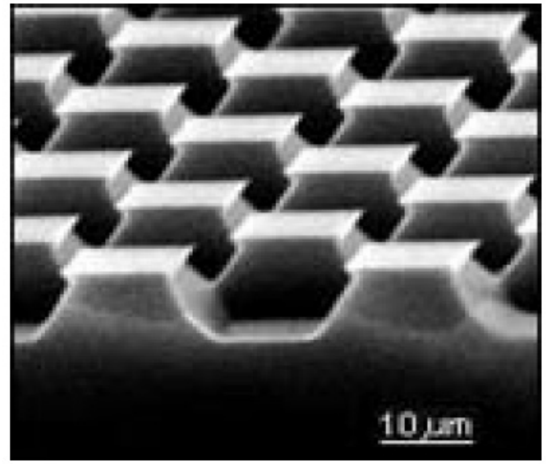
Example of tip deonvolution

- A square object (a) is scanned with a similar-size rounded tip (b).
- The resulting image (c) combines features of both
- The object can be partially restored by deconvolution (d)

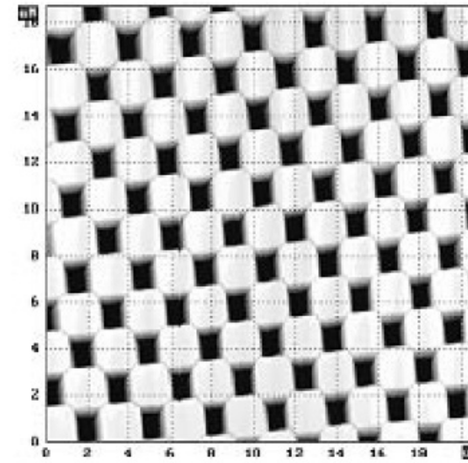


AFM calibration

- Special "calibration standards"
  - Checkerboard pattern to calibrate XY scale, linearity

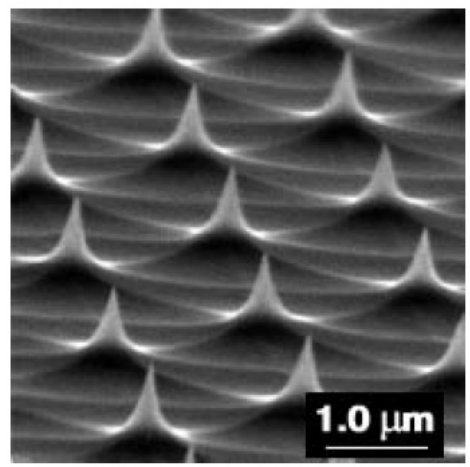


SEM picture of sample

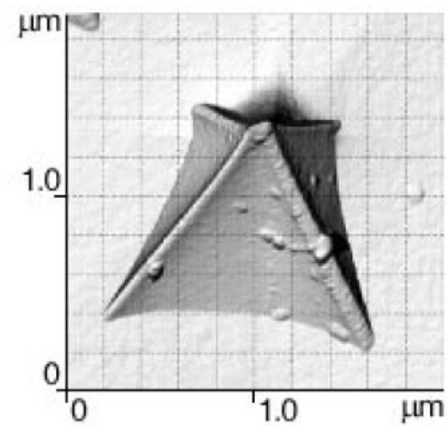


AFM image

  - Sharp “tips” to calibrate tip shape



SEM picture of sample



reconstructed tip shape

- It's time-consuming to calibrate every tip – useful only for critical applications (metrology)

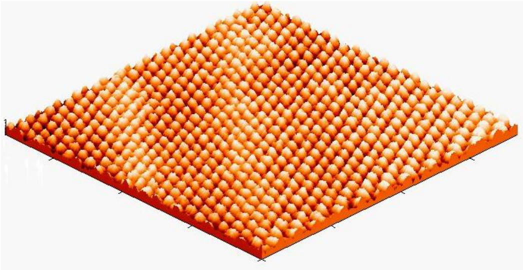
Advanced modes - SPM

- The AFM can measure small forces (< 1 nN), so any phenomenon that can be translated to force can thus be measured by the AFM
- The tip is very close to the sample, so it can be used to interact with the sample while scanning
- The general name : Scanning probe microscopy - SPM

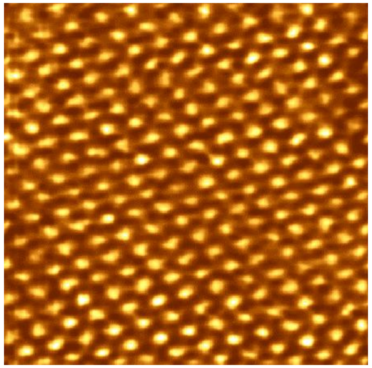
- Friction : LF-AFM
- Electric field : EFM
- Voltage : KF-AFM
- Current : I-AFM, CAFM
- (Spreading) Resistance : SSRM
- Capacitance : SCM
- Magnetic field : MFM
- Temperature
- Magnetic field (Hall)
- Chemical interaction
- Optical excitation and detection : SNOM
- Lithography

LABORATORY OF nanoSTRUCTURES

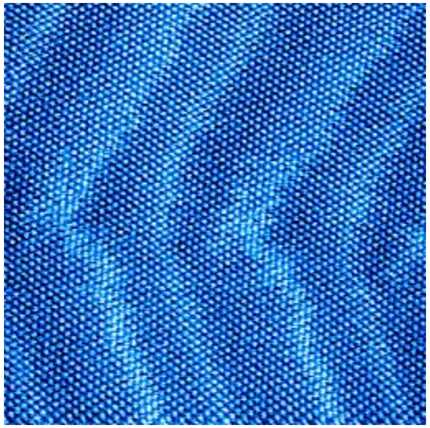
STM Applications



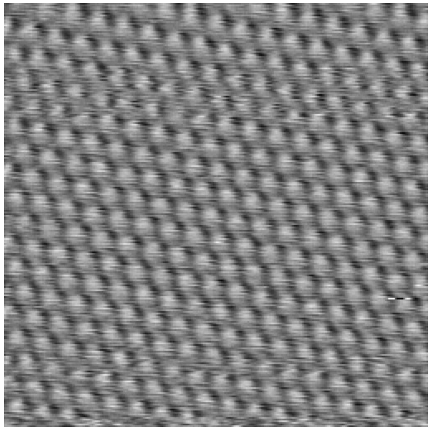
- STM is most useful in LT/UHV:
  - Naturally forming oxide layer distorts surface states
  - Water layer interferes with tunneling current
  - Thermally induced atomic vibrations distort image
- Many MBE growth systems have a UHV STM (sometimes LT STM) coupled to the growth chamber
- STM can show growth morphology, electrical properties, doping, on the atomic scale



Au (111) atoms (3x3 nm)

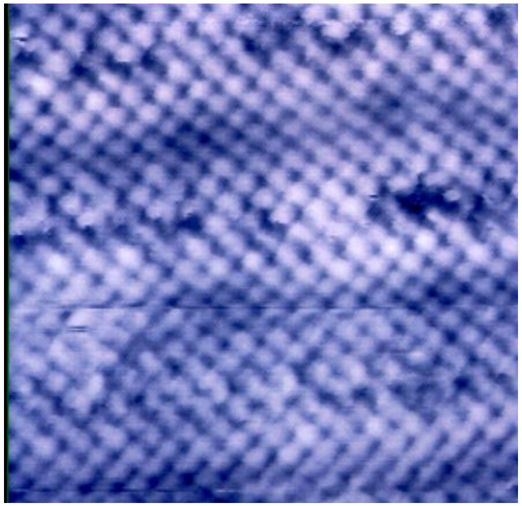


Au (111) atoms (17x17 nm)

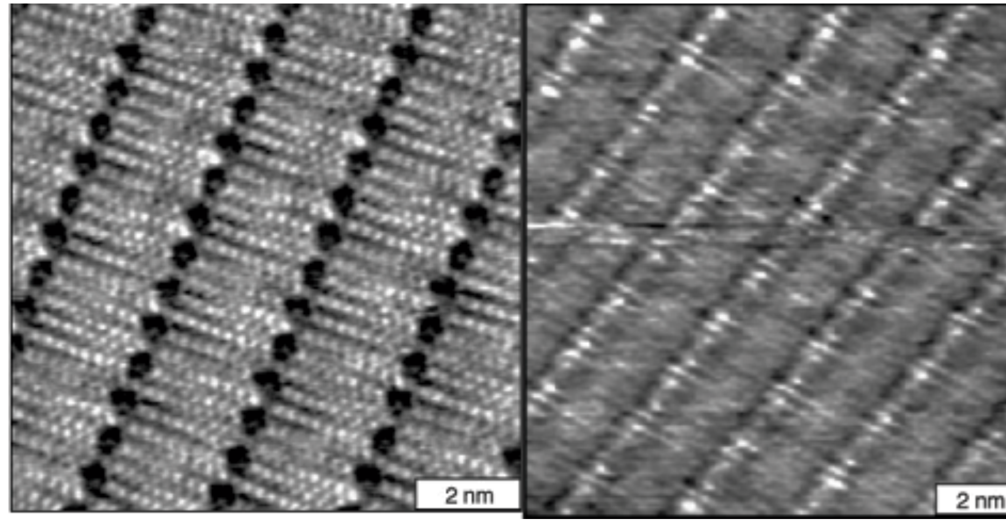


Ag (111) atoms

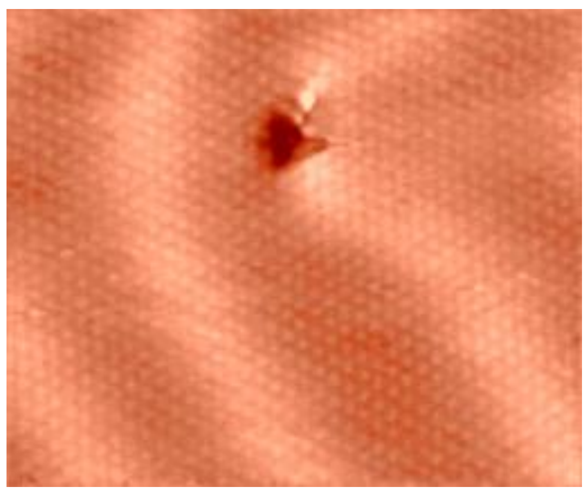
Some STM Images (1)



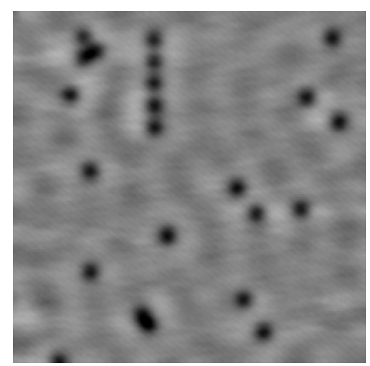
BSCO HTSC superconductor at 4K



STM Images of molecules. Right – tip is modified to interact with specific part of molecule

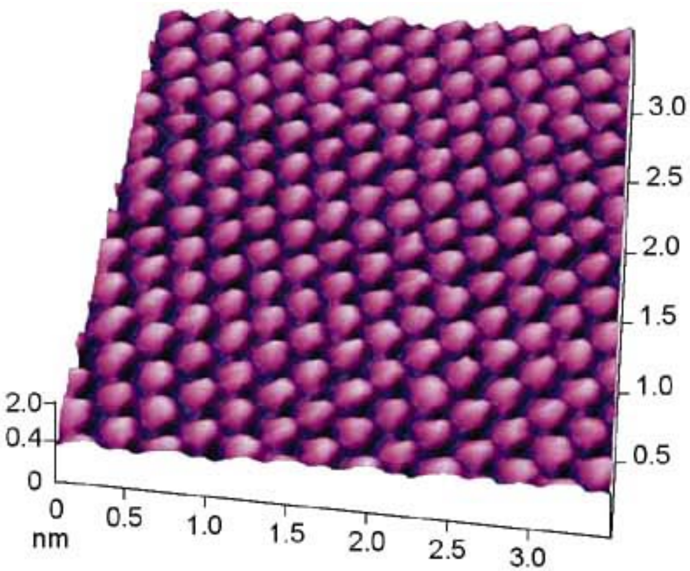


Co atoms (dark) on Au (111) surface

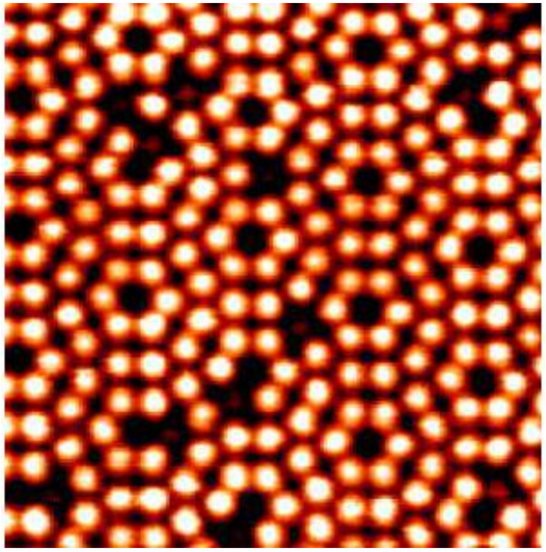


Co atoms (dark) on Ag surface

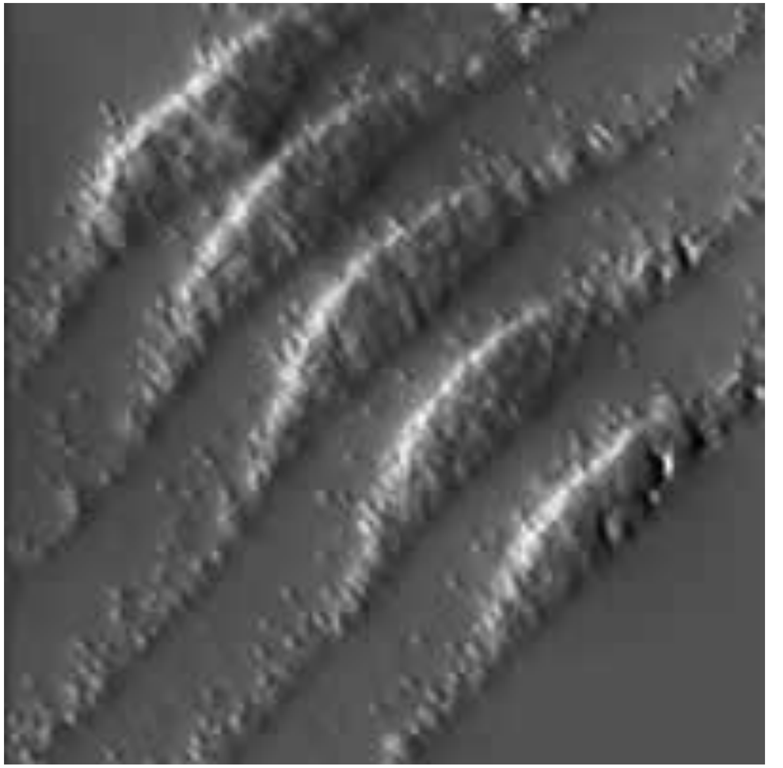
Some STM Images (2)



Graphite atoms

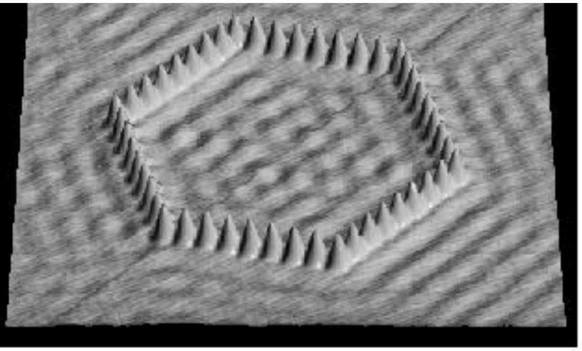


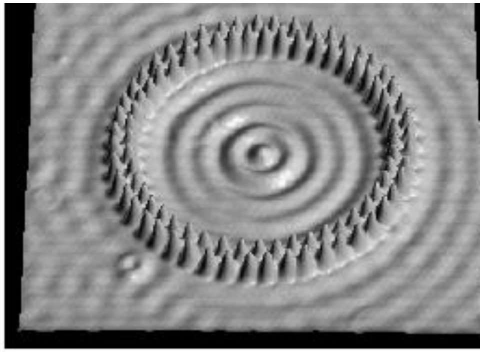
Si atoms

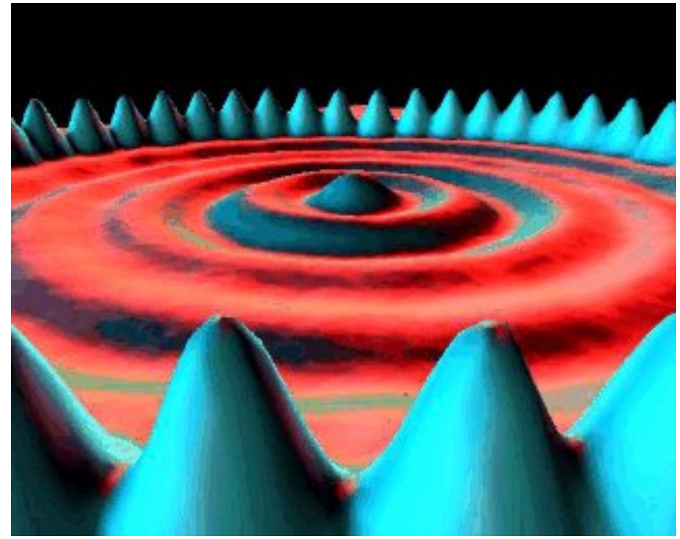


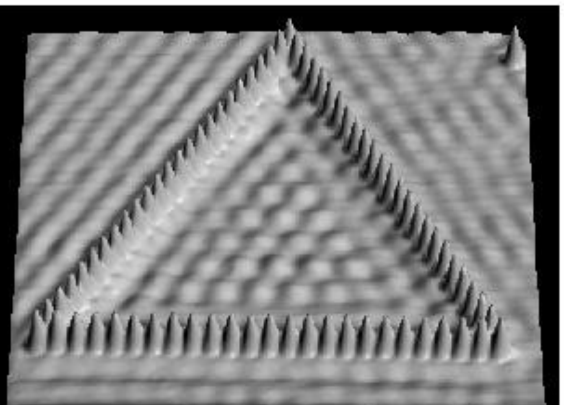
Stacked InGaAs quantum dots

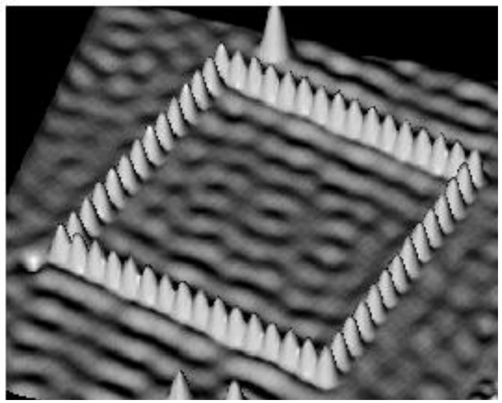
Some STM Images (3)





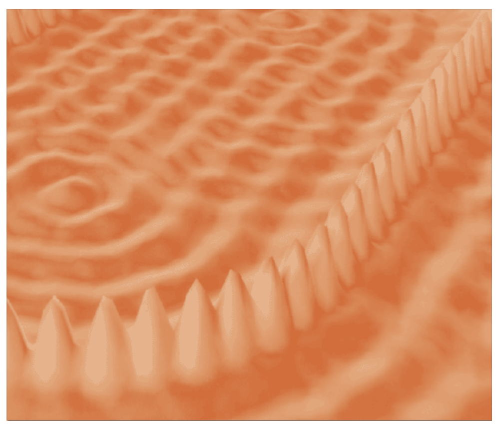


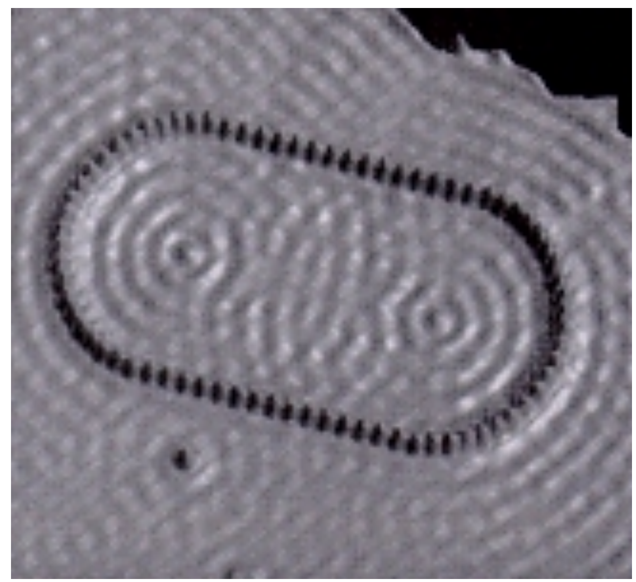




Fe atoms manipulated by STM

Note the quantum interference patterns !





The rat race …

Detailed example of STM imaging

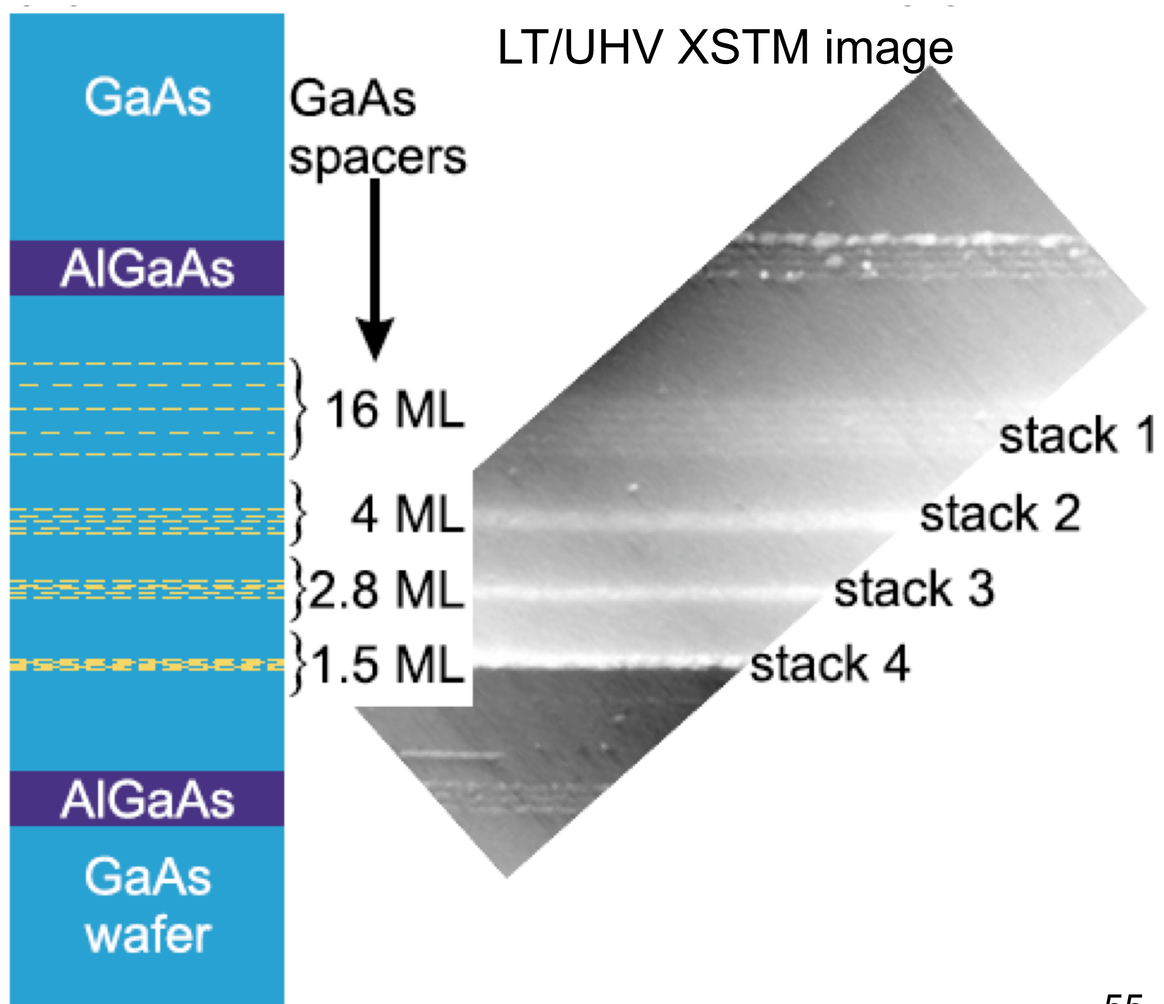
Atomic structure and optical properties of InAs submonolayer depositions in GaAs,

A. Lenz, H. Eisele, J. Becker, J.-H. Schulze, T. D. Germann, F. Luckert, K. Pötschke, E. Lenz, L. Ivanova, A. Strittmatter, D. Bimberg, U. W. Pohl, M. Dähne,

B. JVST. B 29, 04D104 (2011)

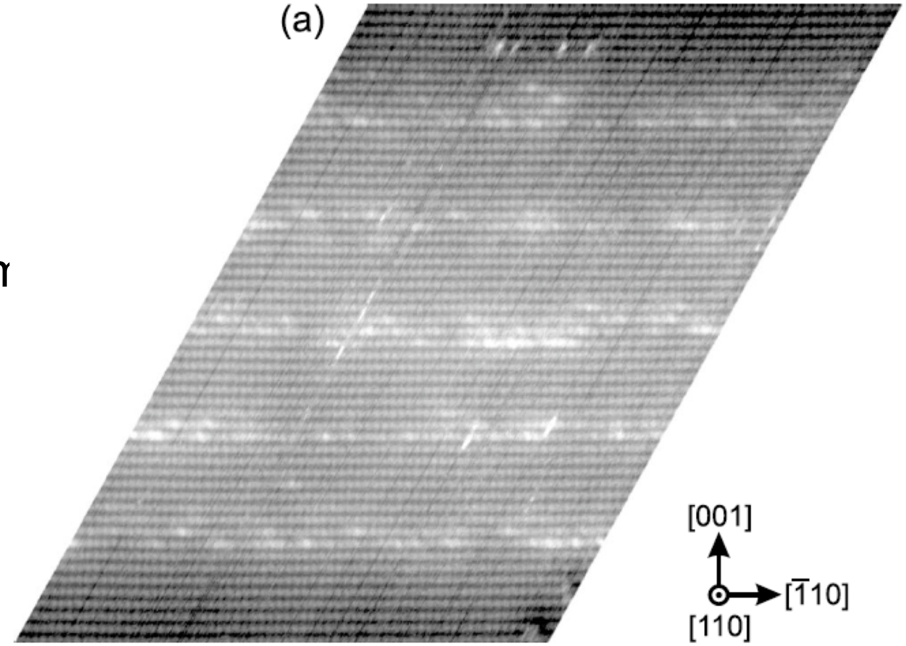
Sample structure:

- GaAs growth by MBE
- AlGaAs barriers at extremities ("garbage collection")
- Four "stacks", each of five 0.5 ML InAs layers in GaAs
- Different spacing between InAs layers in stack: 1.5-16 ML.
- Sample cleaved in UHV, STM of cross-section (XSTM)
- InAs brighter than GaAs

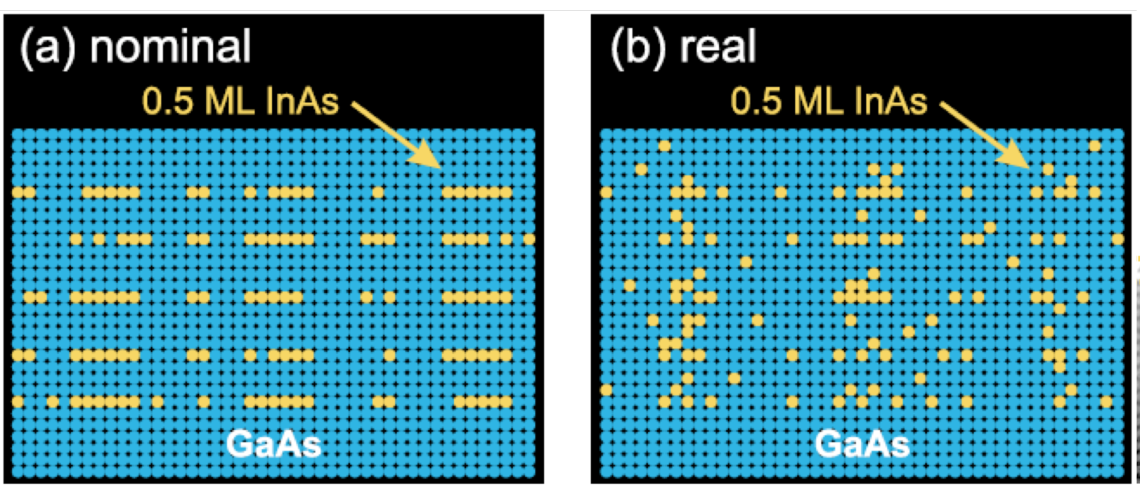


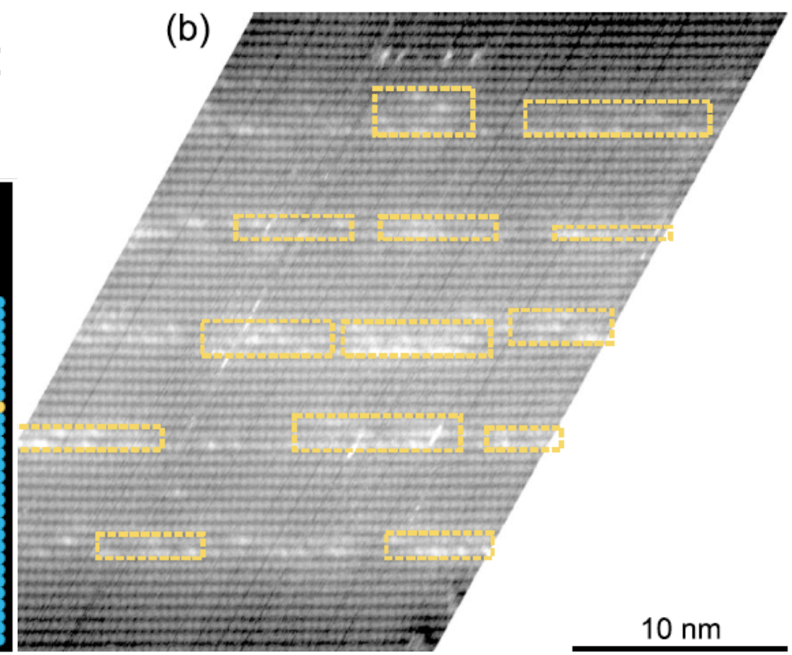
XSTM images of stack 1 (16ML)

- We can distinguish single atoms!
- InAs atoms are brighter than GaAs:
  - Strain release after cleave pushes atom above surface
  - Higher electron state density -> higher tunneling current



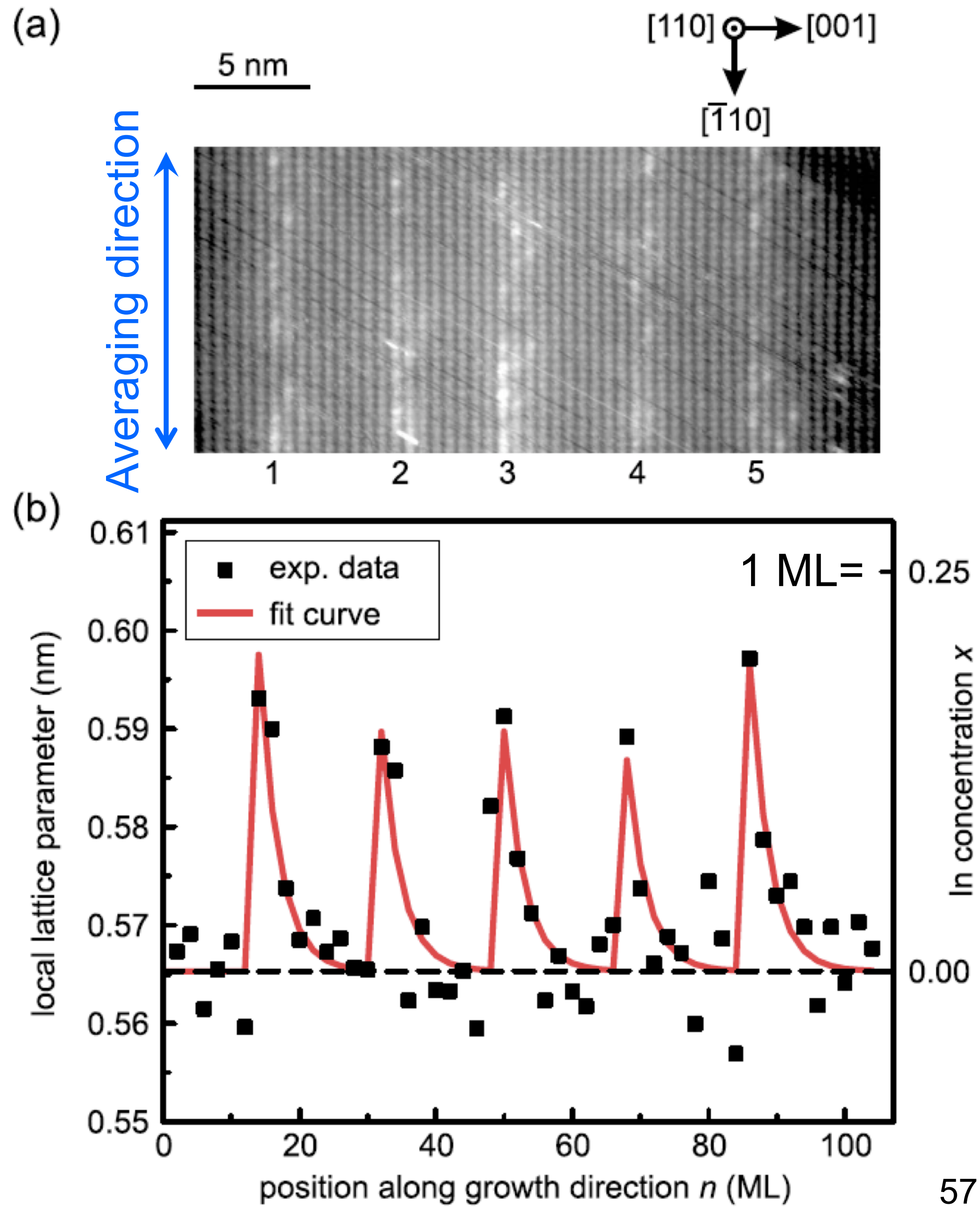
- InAs atoms tend to "cluster" (fig.b) in height: evidence for some diffusion in the bulk





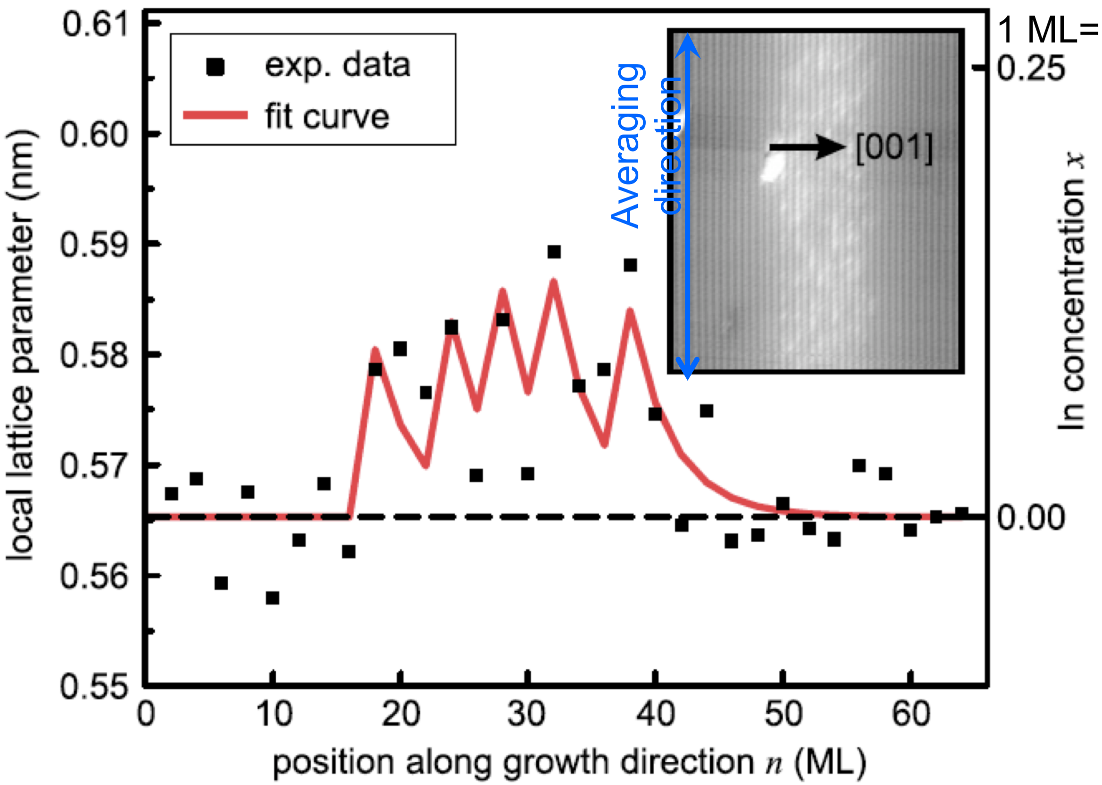
XSTM images of stack 1 (16ML)

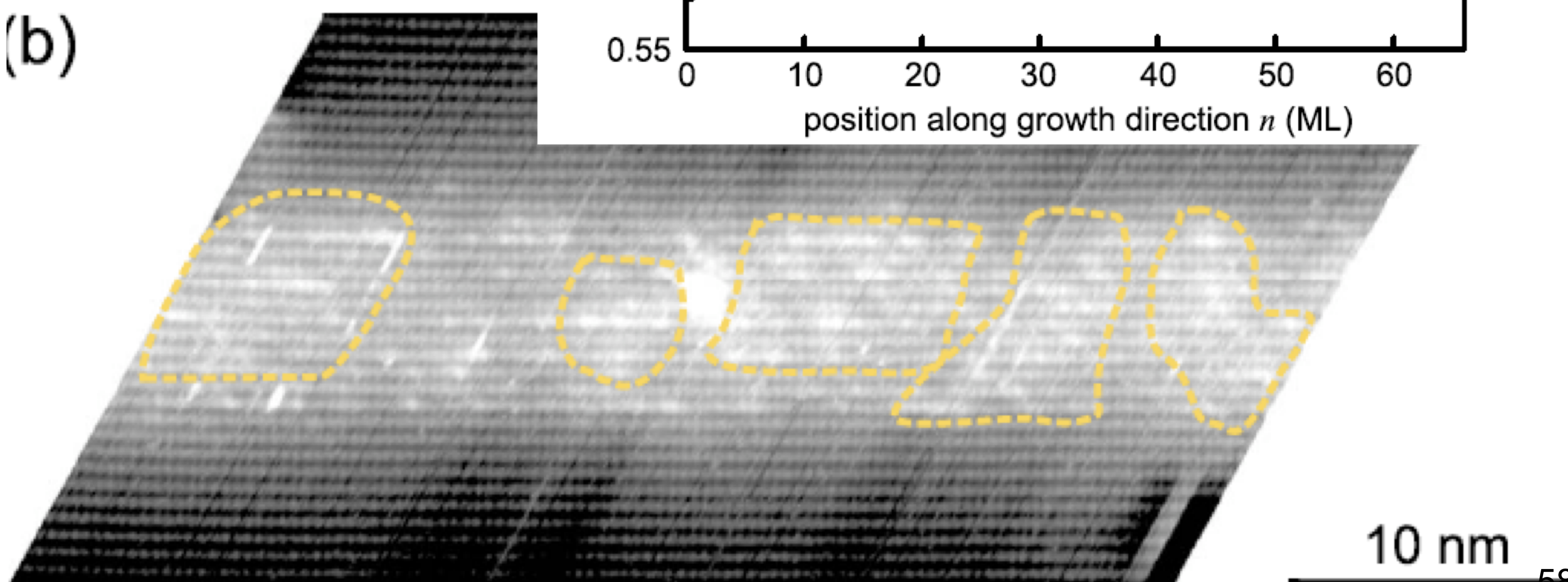
- We can quantify the In contents by measuring local lattice parameter between rows (averaged along the rows)
- Accuracy: ~1 pm!



XSTM images of stack 2 (4ML)

- The same can be done on a stack with smaller distance between inAs layers, leading to denser clustering



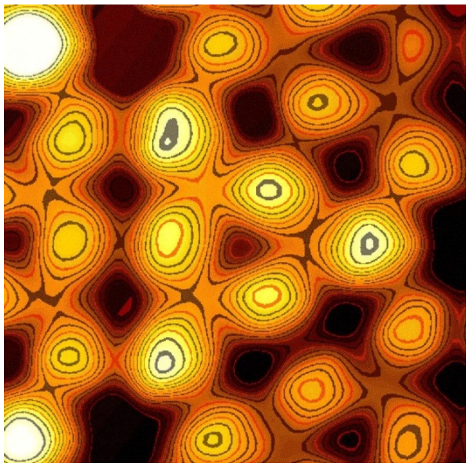


AFM Applications

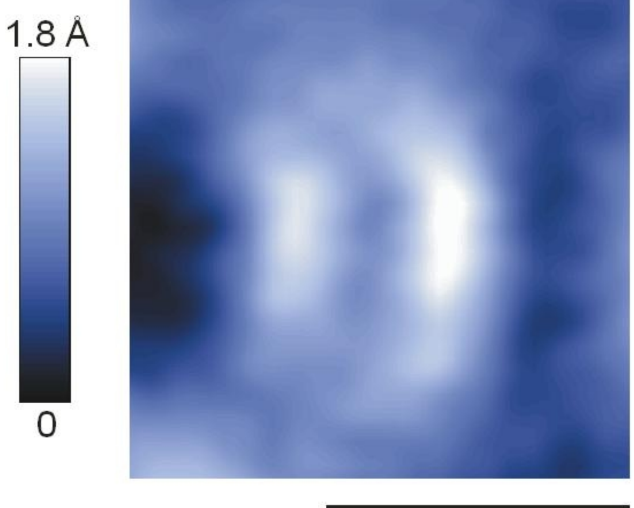
- AFM is used more frequently than STM:
  - Easier to use and to obtain an image
  - Can work at ambient air/temperature
- Uses of AFM in characterization:
  - Substrate characterization before growth (especially non-planar substrates)
  - Growth surface characterization (morphology)
  - Cross-sectional imaging (layer structure)
  - Advanced electrical modes (doping)

AFM atomic resolution Images

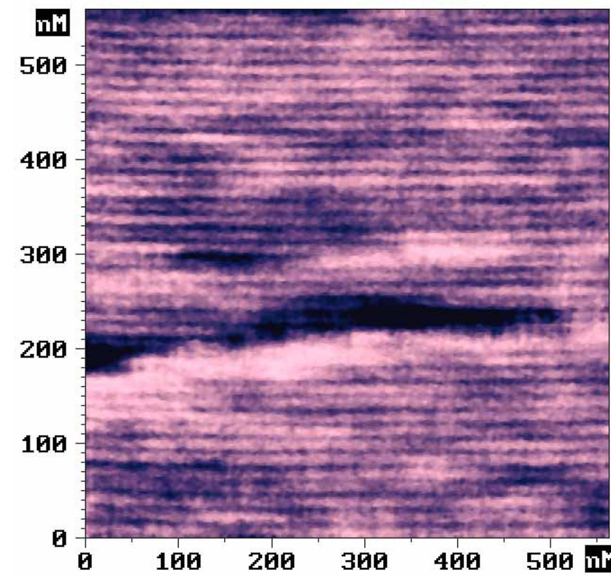
- AFM is also capable to produce atomic resolution images :



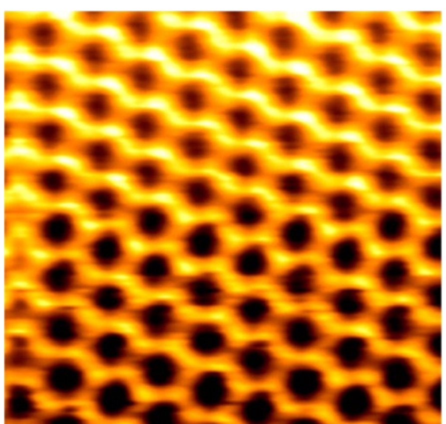
Silicon



Sub-atomic resolution by AFM



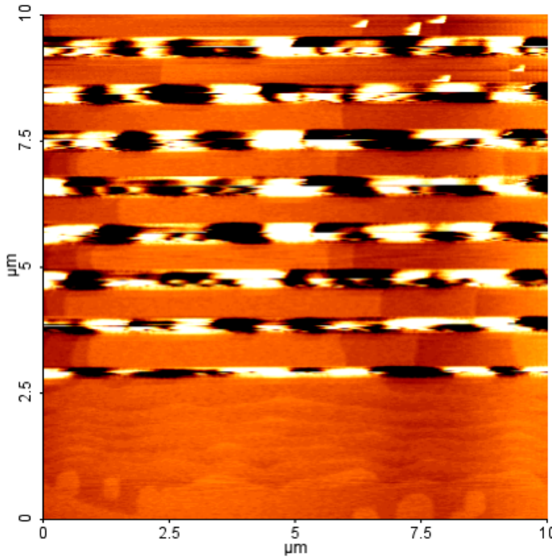
GaAs cross-section

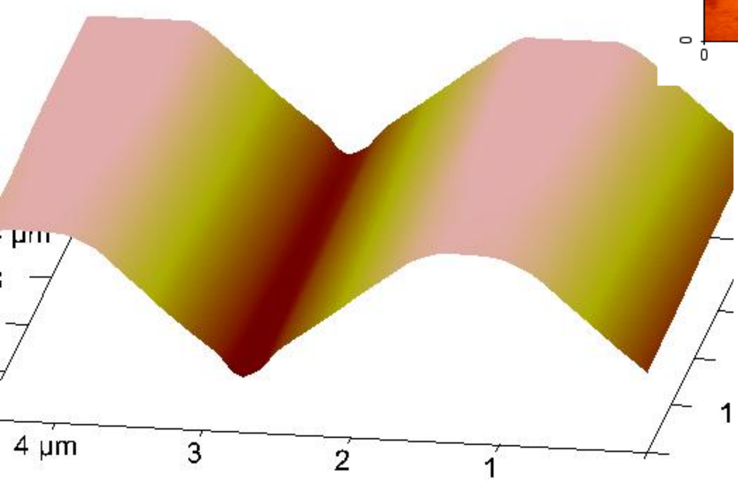


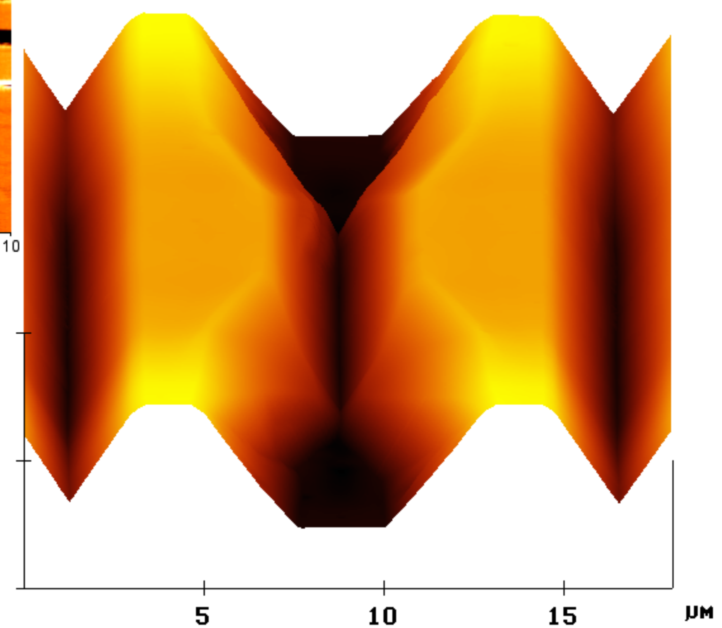
Graphite

Substrate characterization

- Example: etched V-groove substrate
- The quality of surfaces is easily determined by AFM

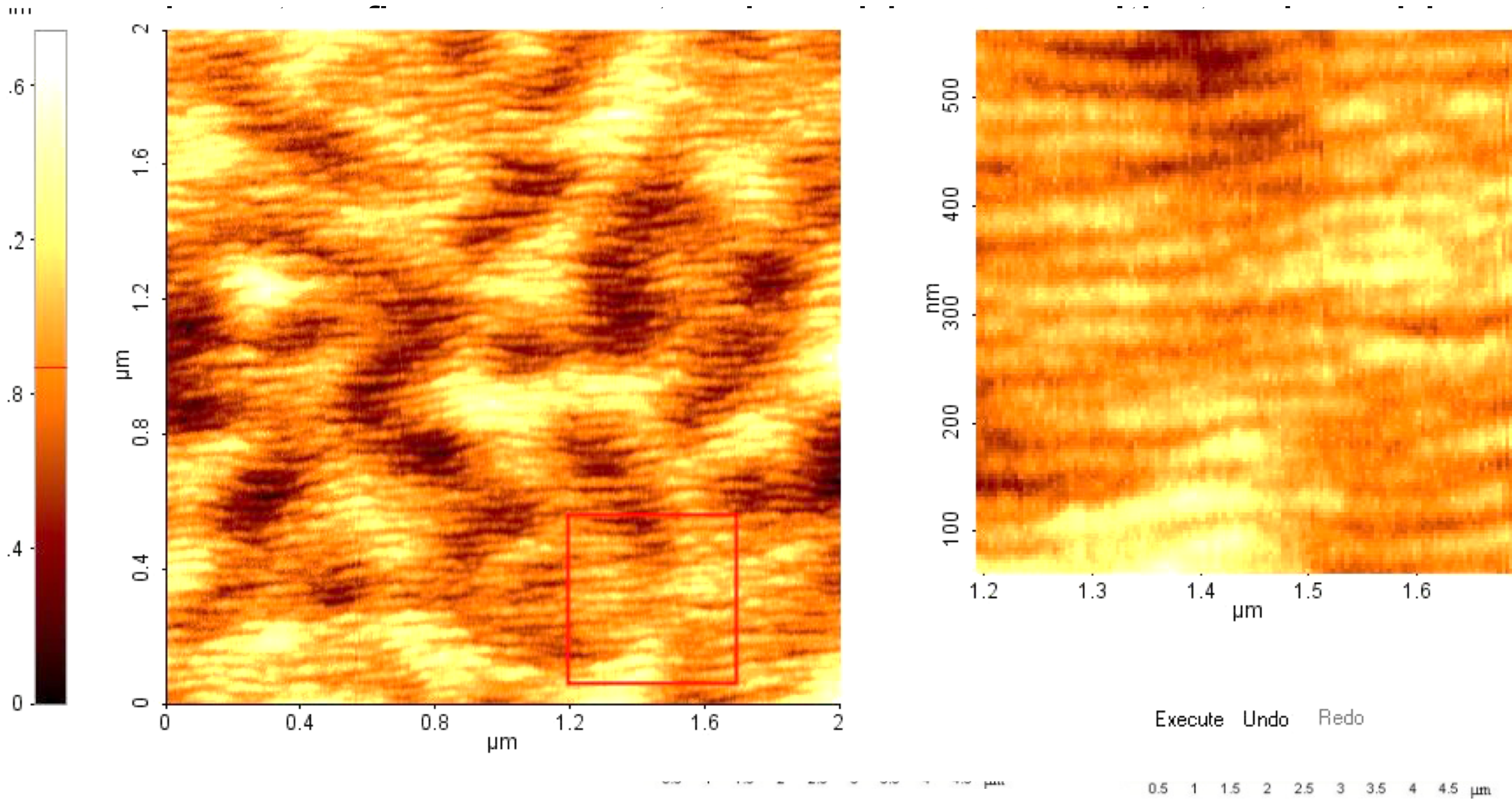






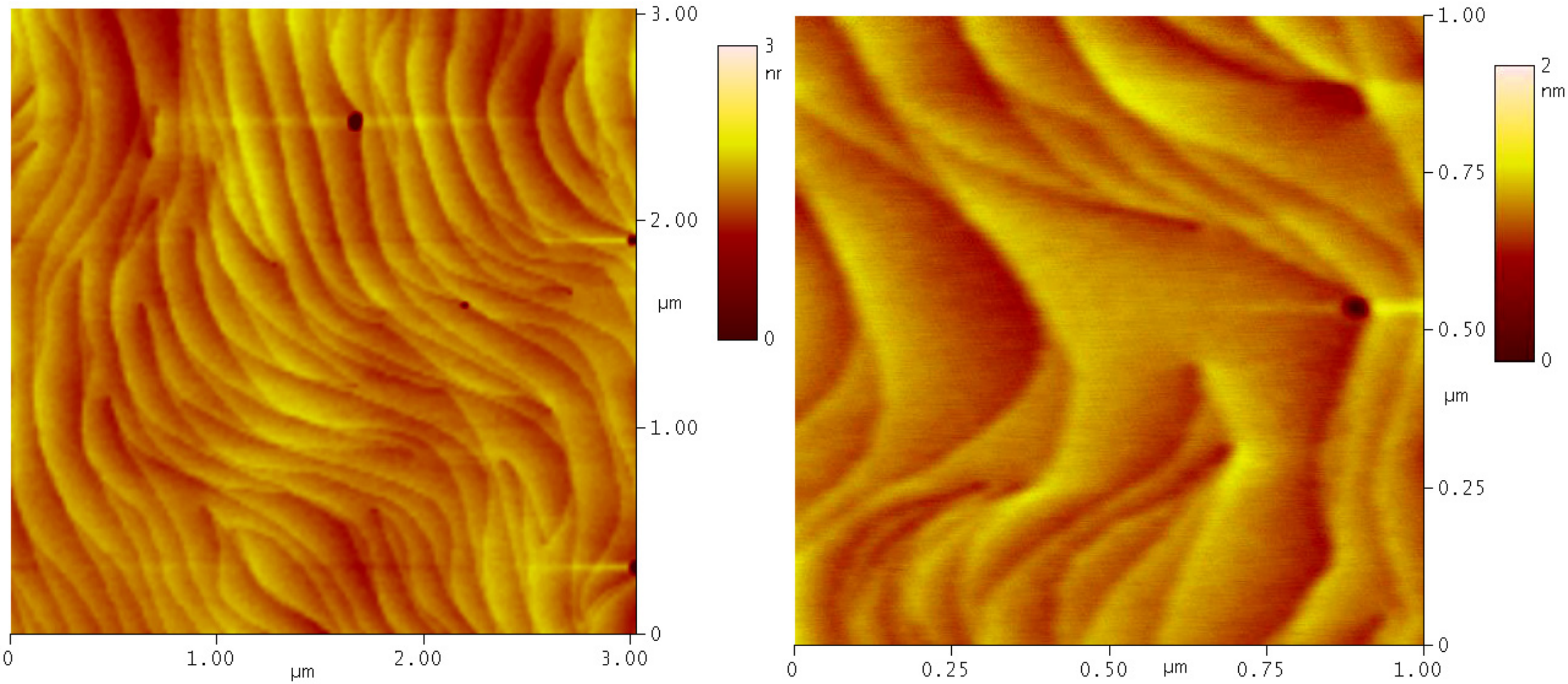
Surface characterization

- Example: monolayer steps on GaAs structures
- The substrate misorientation angle determines the growth



Surface characterization

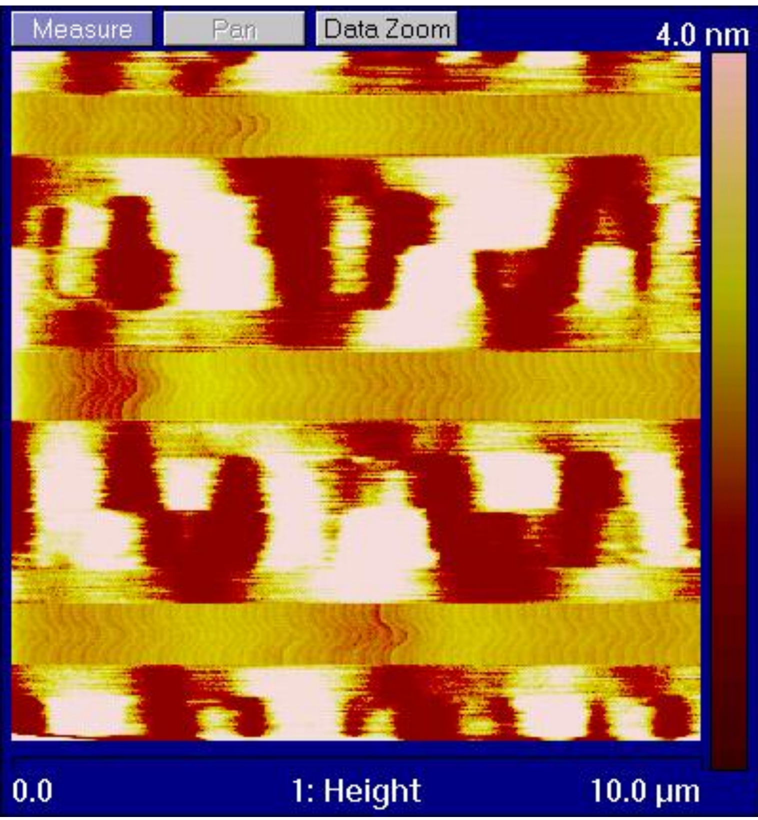
- Example: monolayer steps and dislocations on GaN layers

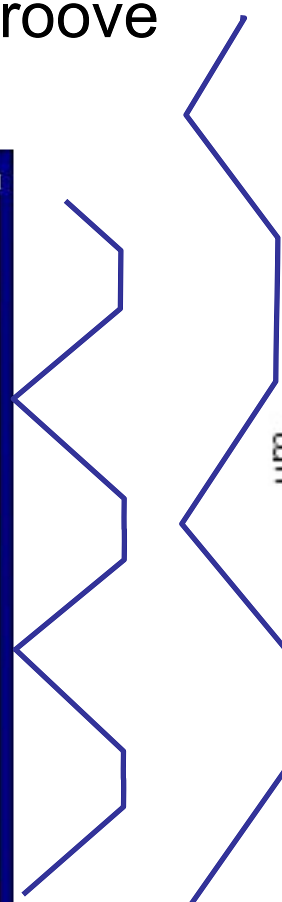


Images of GaN layer on Sapphire by AFM

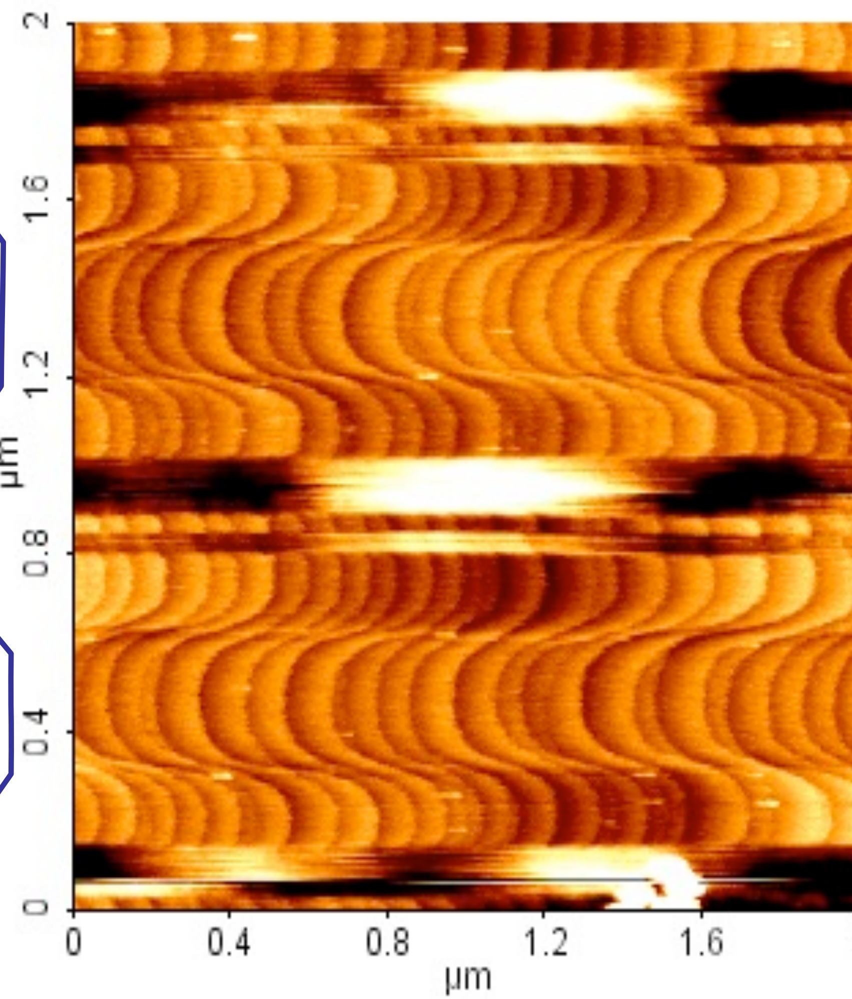
Surface characterization

- Example: monolayer step orientation on GaAs (misoriented 0.3°B) near a deep V-groove





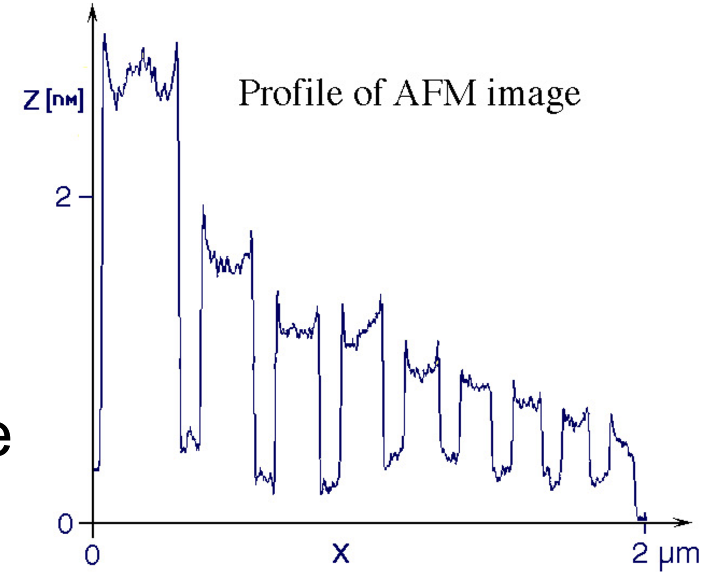
Surface profiles

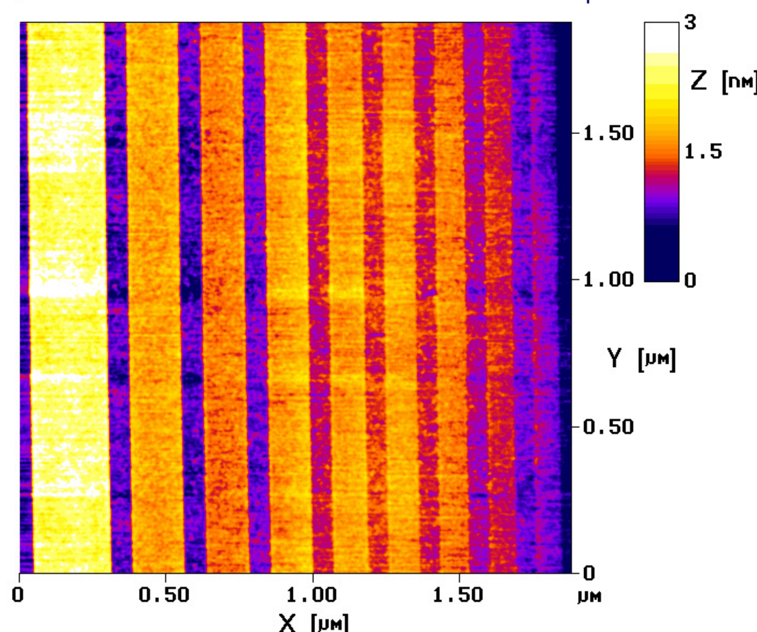


GaAs step orientation by AFM

Cross-sectional characterization

- Oxidation rate in air of $GaAs/Al_xGa_{1-x}As$, depends on Al contents x.
- After 1 hr the oxide reaches finite thickness, depending only on composition
- GaAs is darker than AlAs
- InAs and InP oxidize even less than GaAs



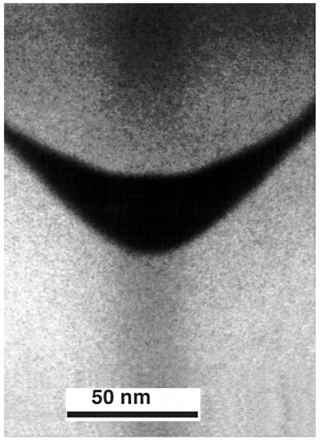


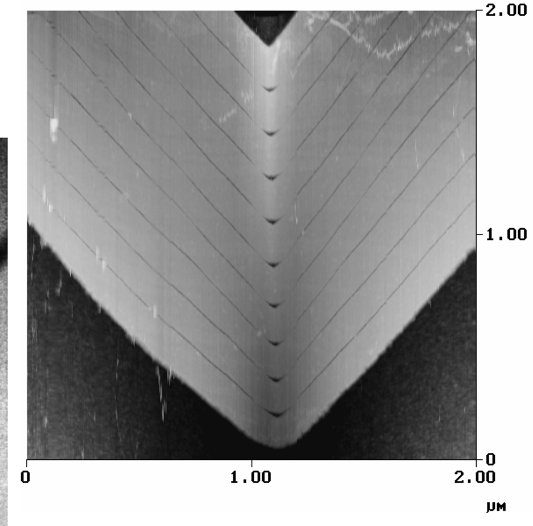
AFM image of multi-layer AlGaAs sample

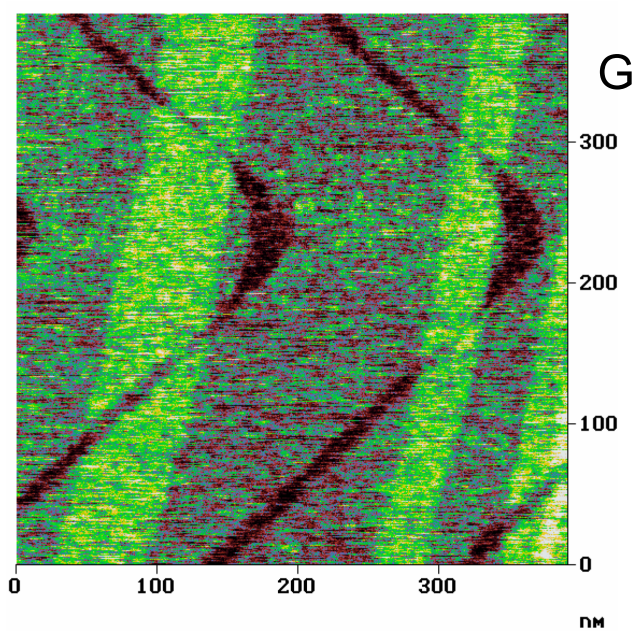
Cross-sectional imaging of QWRs

- High-resolution AFM can show small (a few nm) nanostructures like GaAs/AlGaAs QWRs
- Resolution is almost as in TEM, with short sample preparation

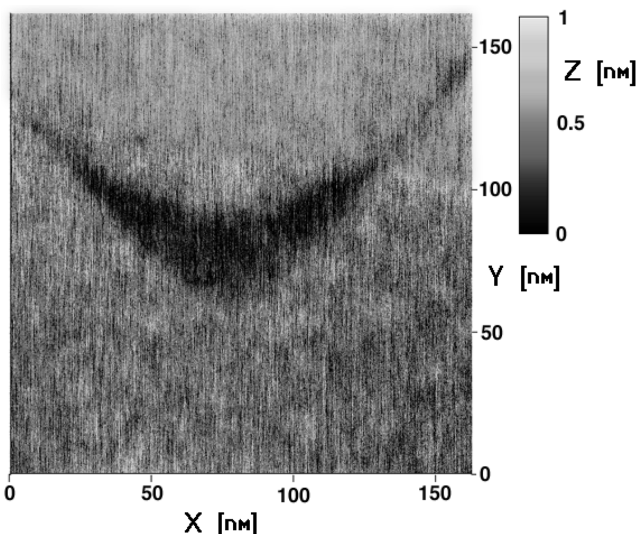
TEM of single QWR

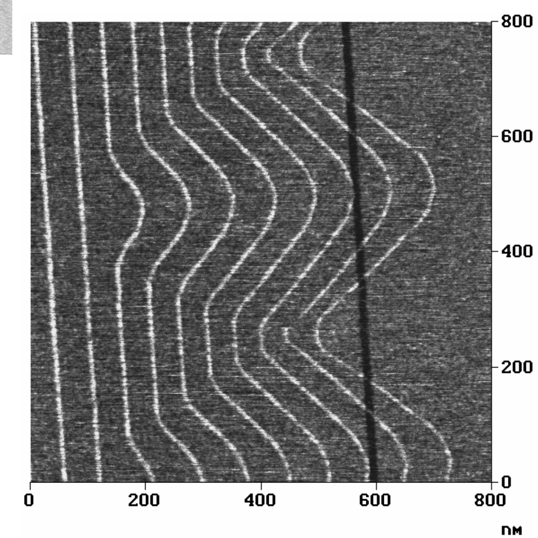






GaAs/AlGaAs QWRs

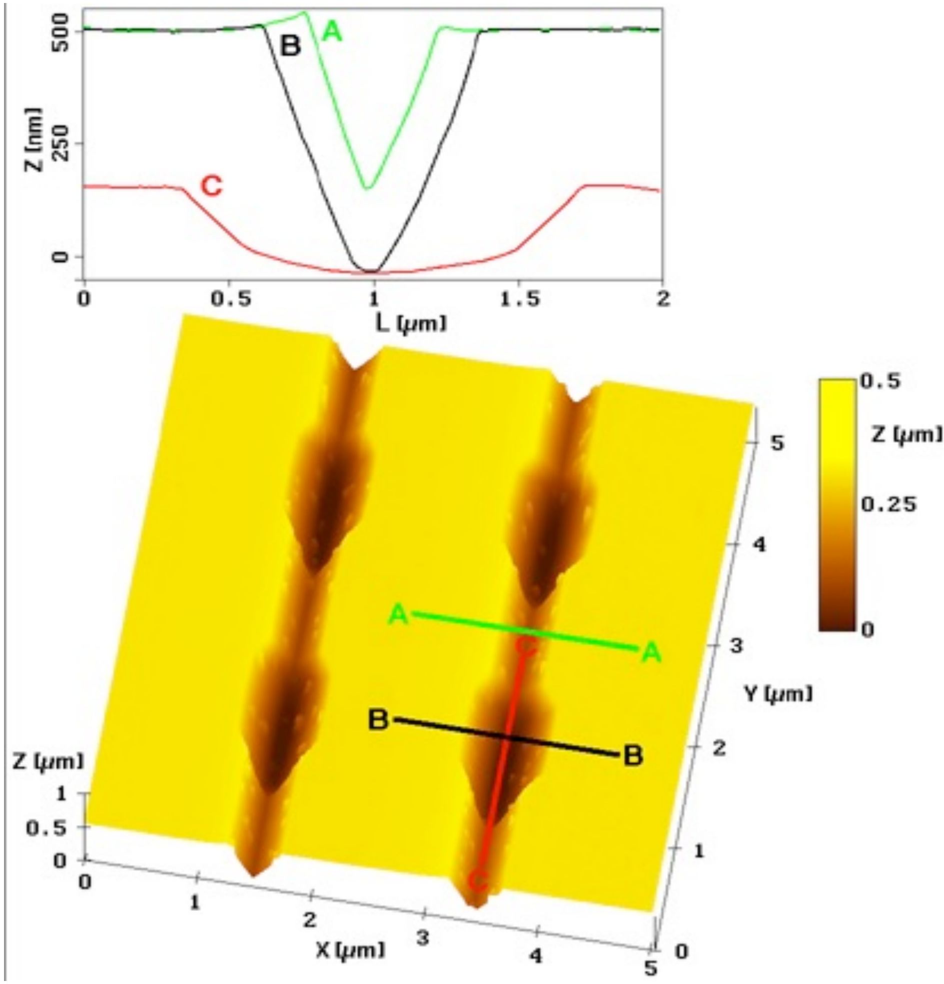




InGaAs grwoth in GaAs

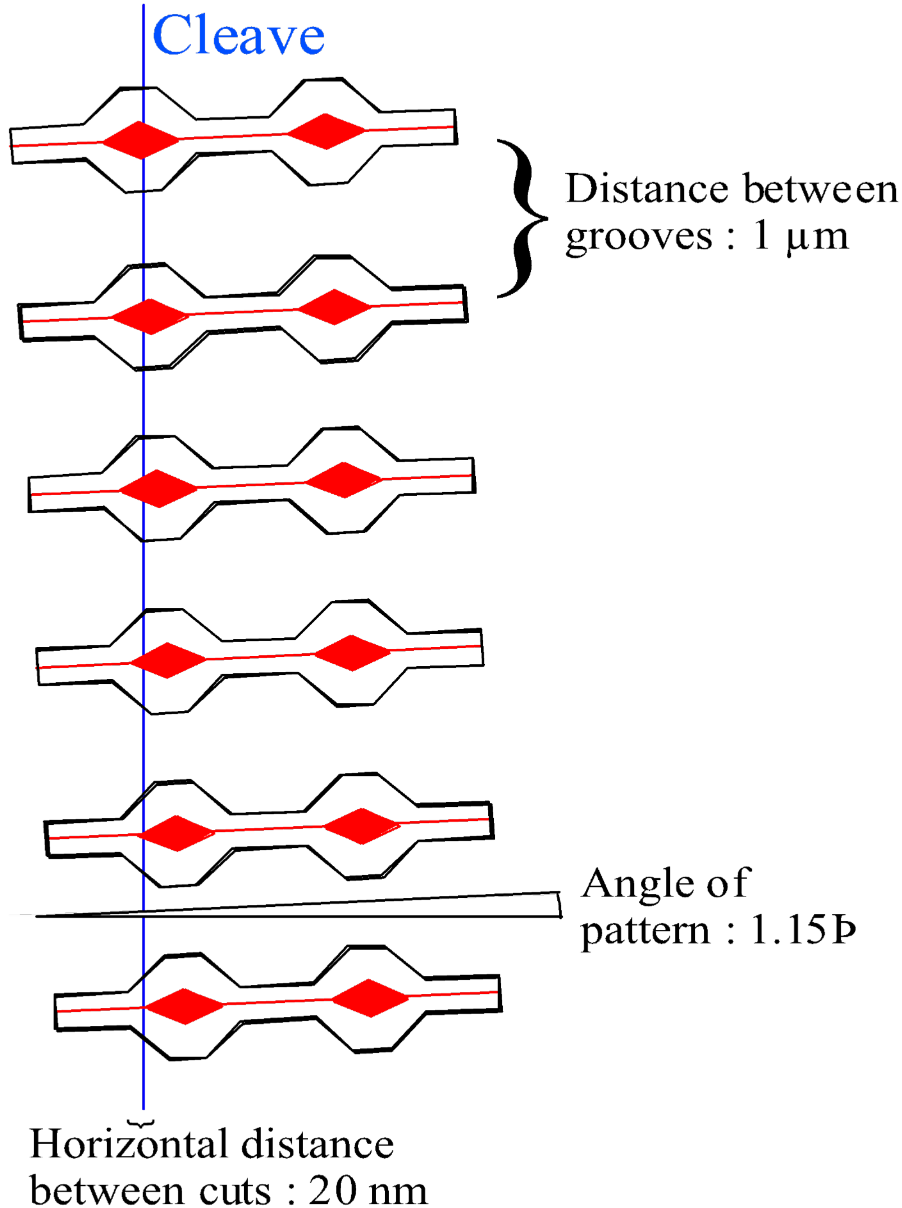
Exemple : cross-sectional growth study in V/U grooves

- We study the growth of GaAs/AlGaAs layers in a variable-shape V/U groove
- SEM and AFM images of the substrate show the profile.

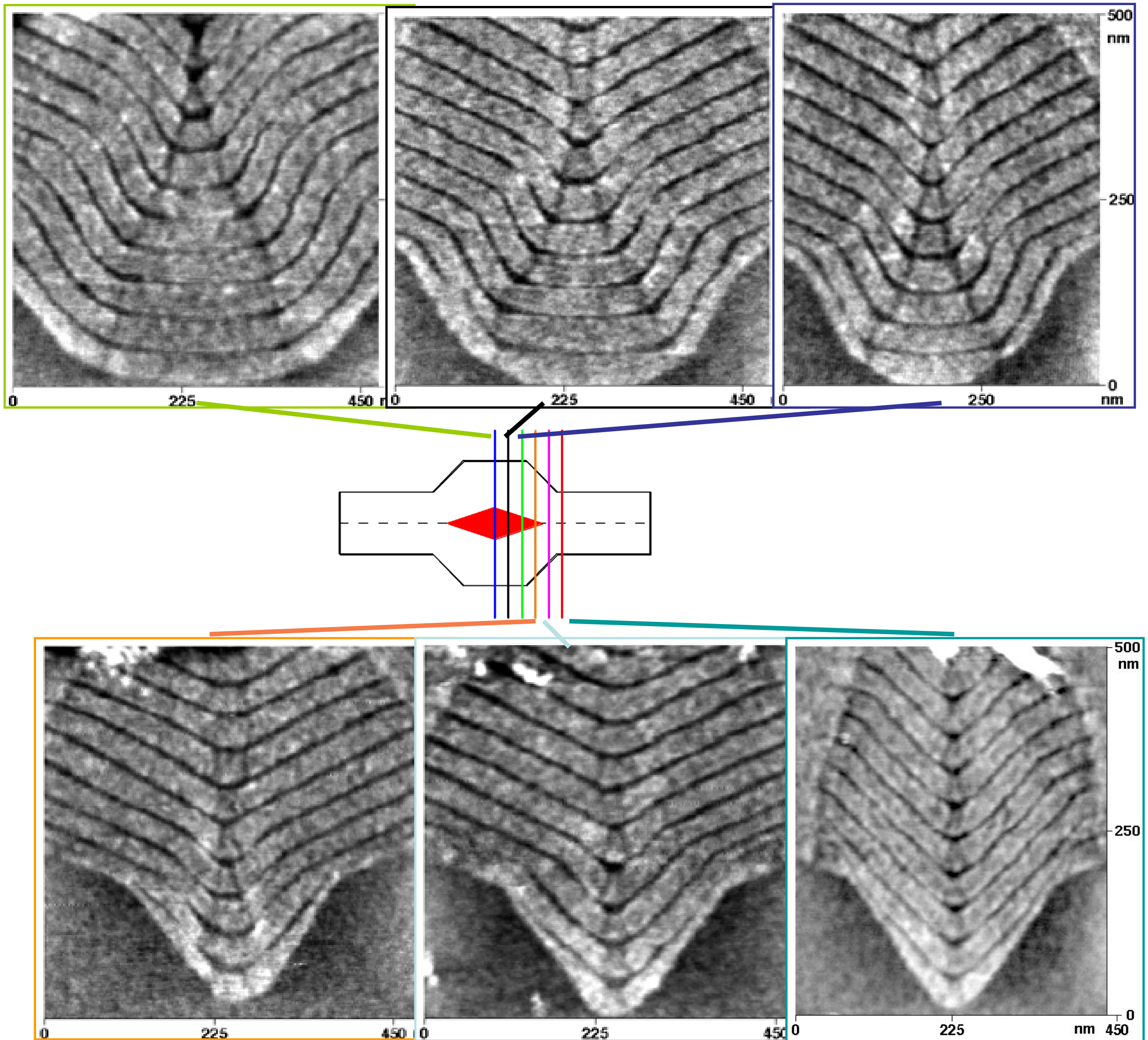


Sequential cross-section

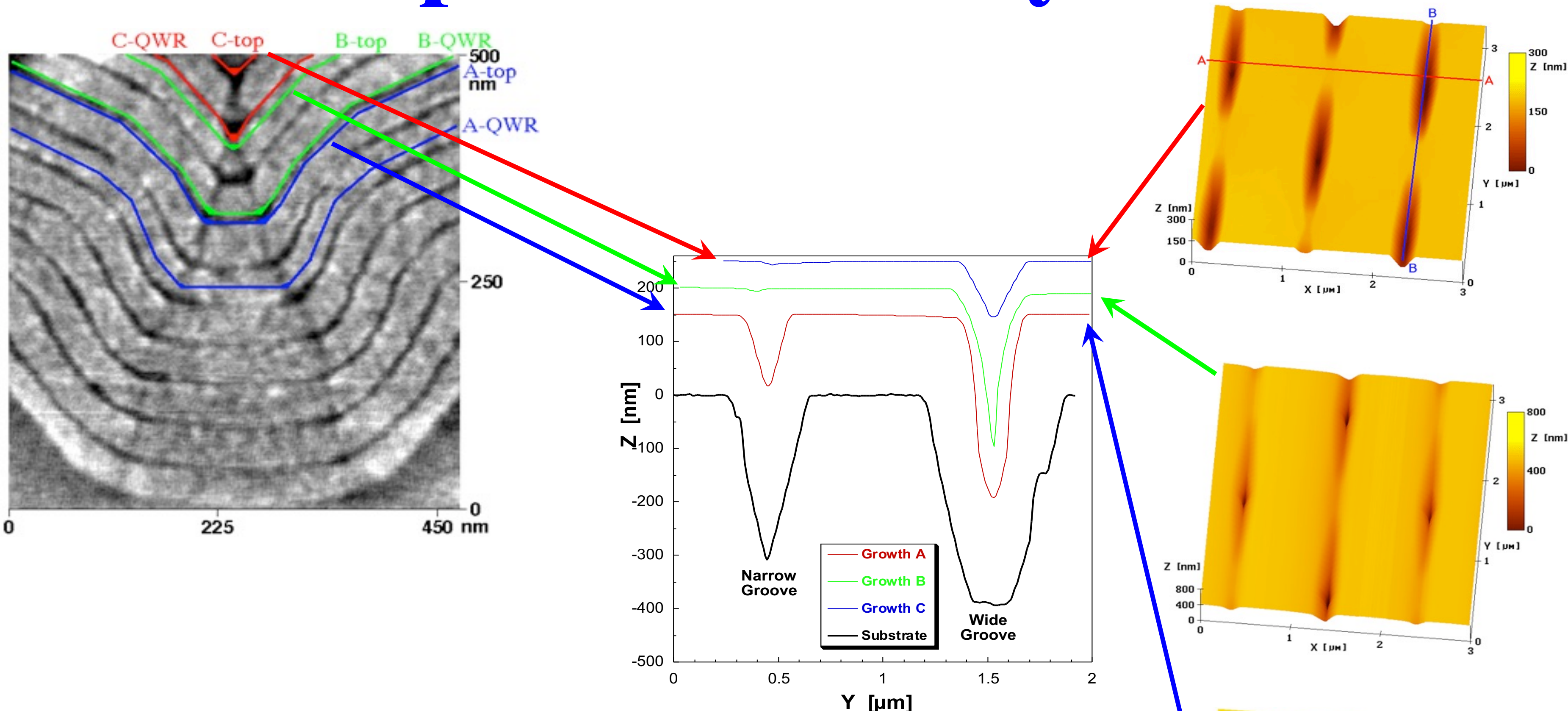
- The groove pattern is written at an angle (1.15°) to the crystal plane
- Result : a single cleave (at a crystal plane) moves gradually between successive grooves
- Successive grooves are 20 nm apart – this is the resolution along the structure

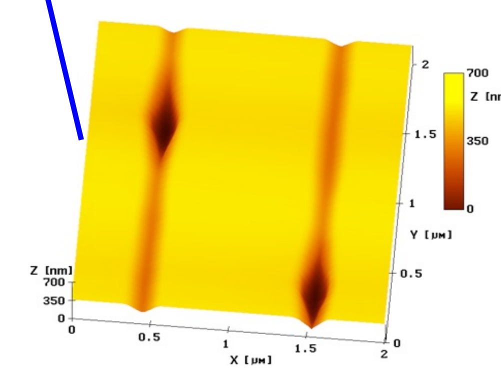


Sequential cross-section results



Top view of 3 layers

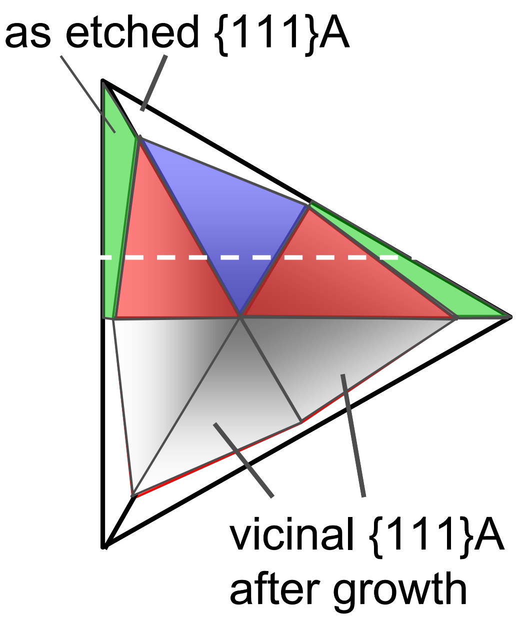


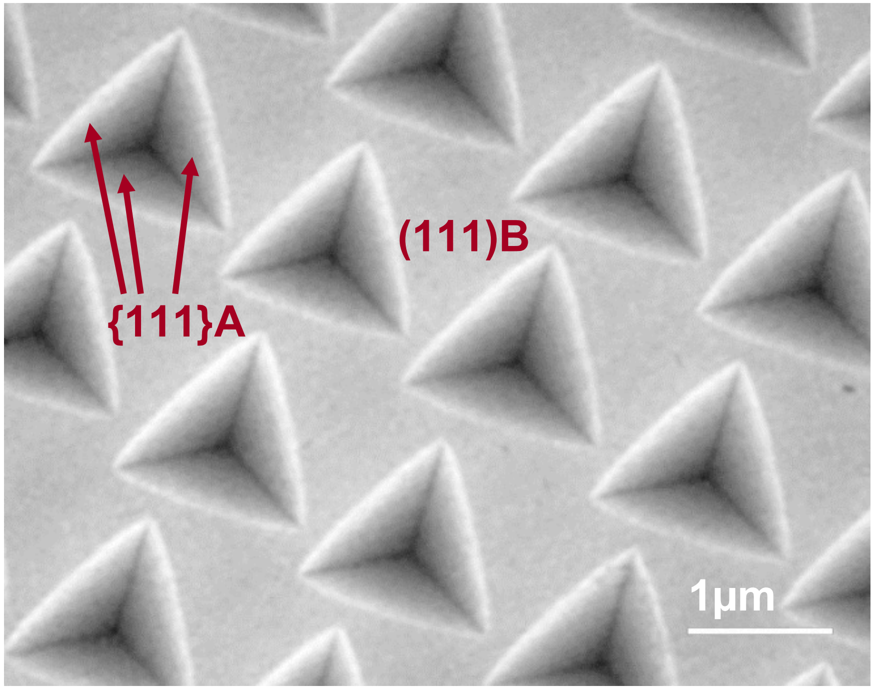


- Following to the cross-sectional measurements, growth of the corresponding thickness gives a similar top view image.

Cross-sectional imaging of QDs

- We grow QDs in pyramidal pits etched in (111)B GaAs substrates :
- After growth, the pits show vicinal {111}A planes

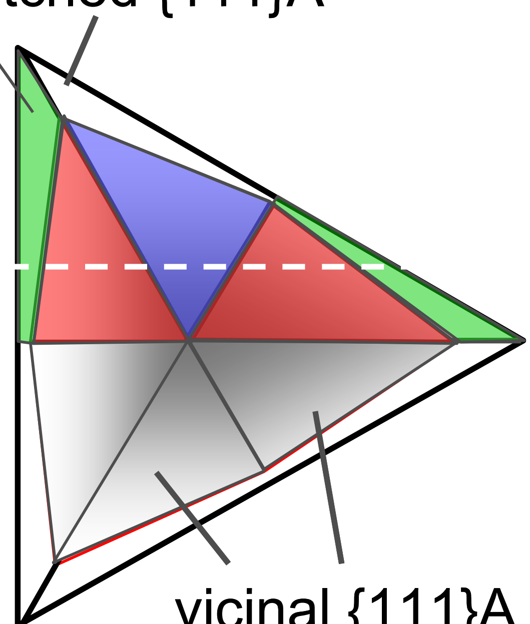




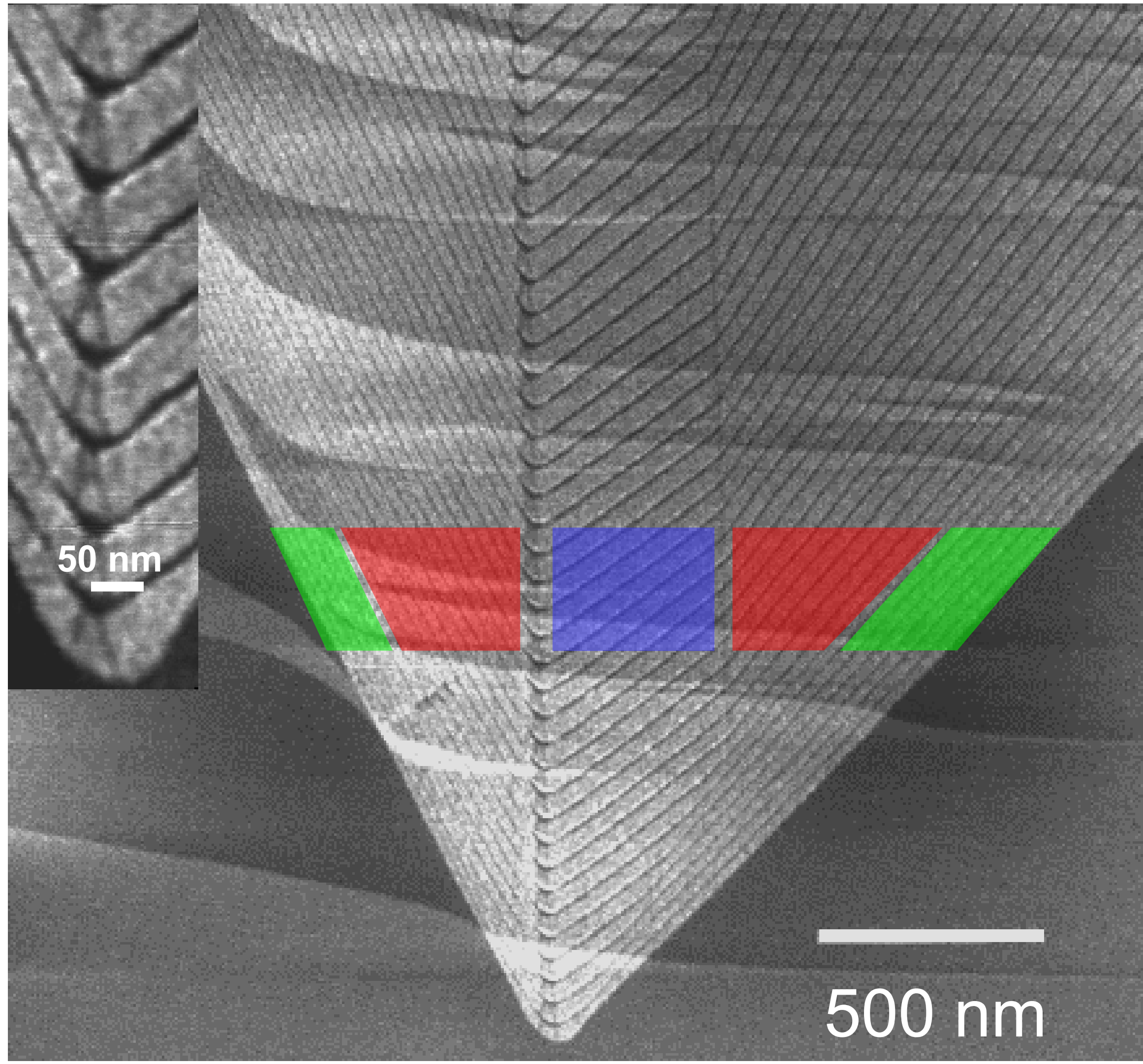
Cross-sectional imaging of QDs

- An AFM cross-sectional images of a series of AlGaAs/GaAs layers grown in a large (5 μm) pit.

as etched {111}A

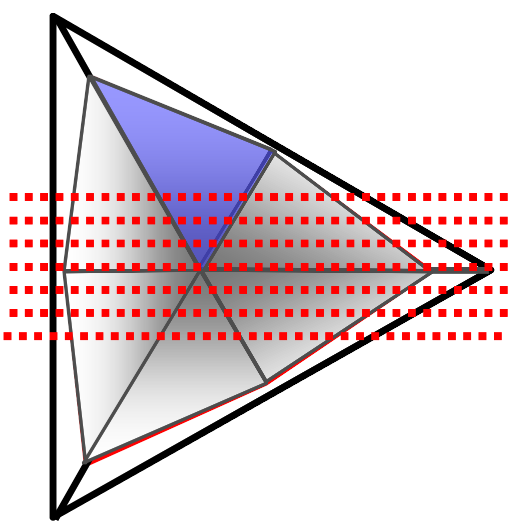


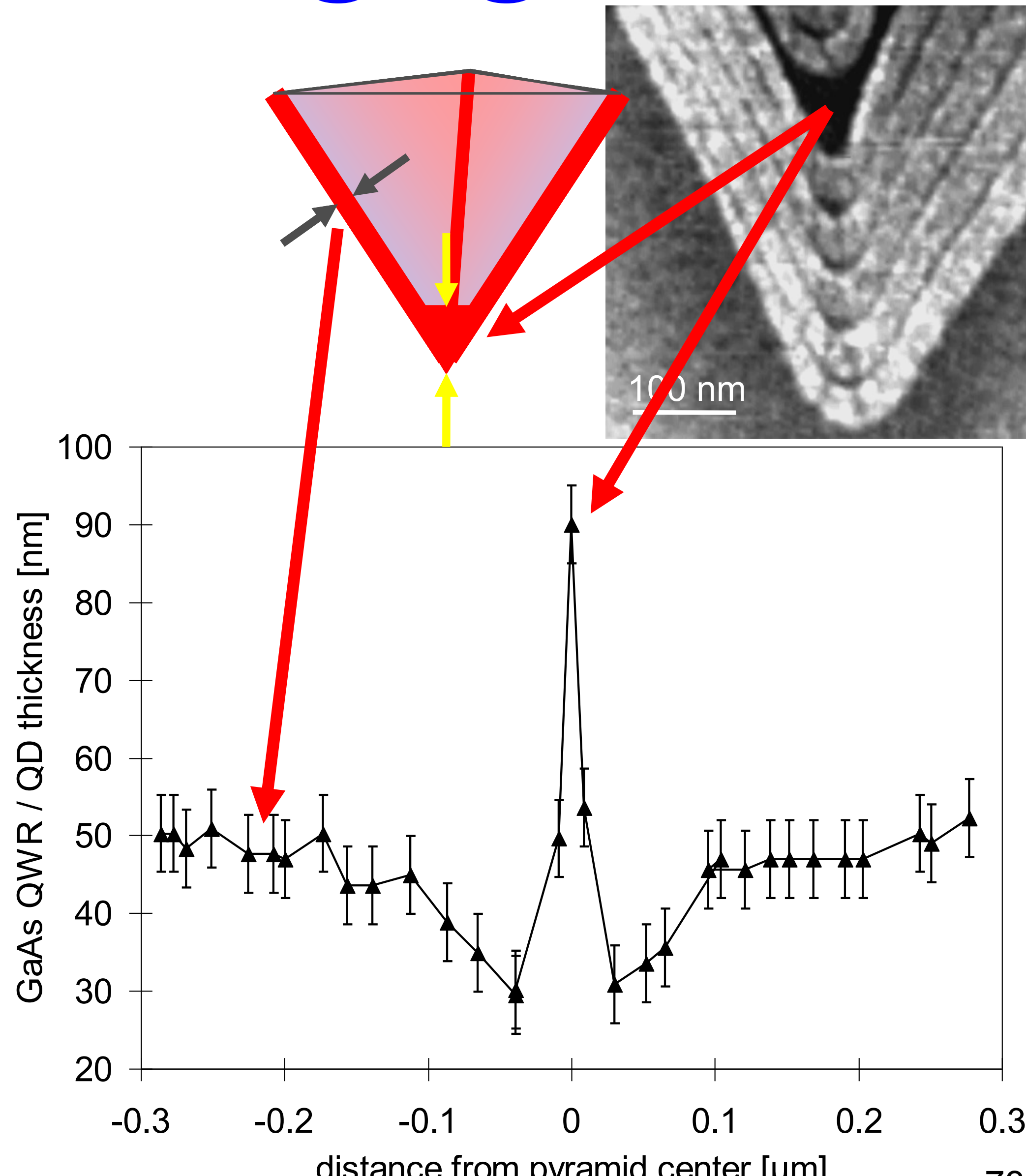
vicinal {111}A after growth



Cross-sectional imaging of QDs

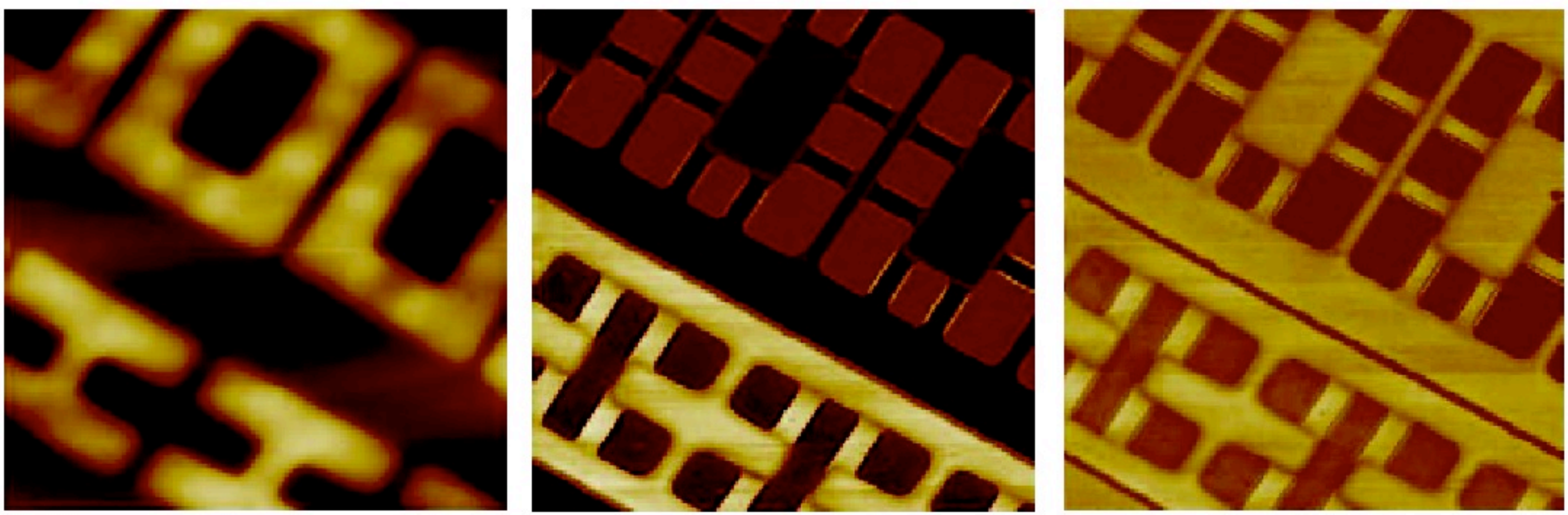
- A series of identical QDs are grown in line at a small angle to the crystal axis
- A single cleave enables a series of cuts into the dot structure
- The 3D structure can be reconstructed





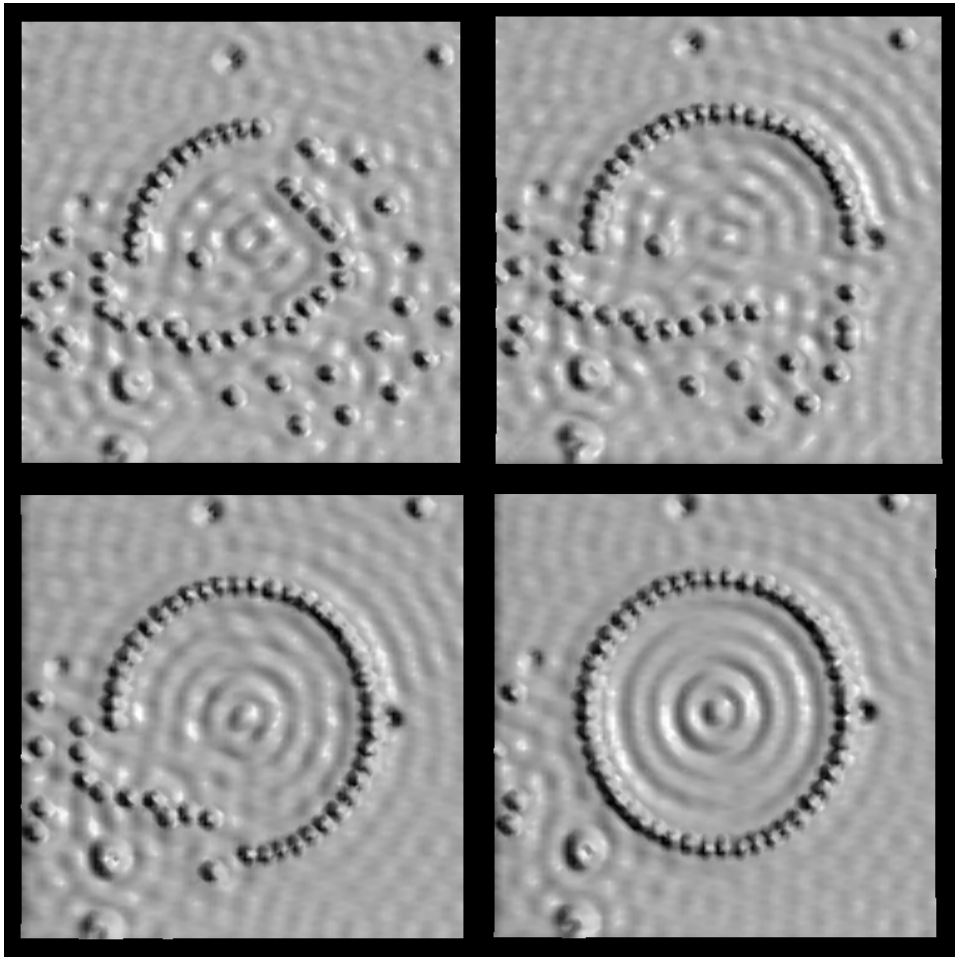
Advanced AFM modes

- EFM and SCM can show doping structures
- SSRM can be used to measure doping
- Special KF-EFM can be used to measure surface states



STM and AFM lithography

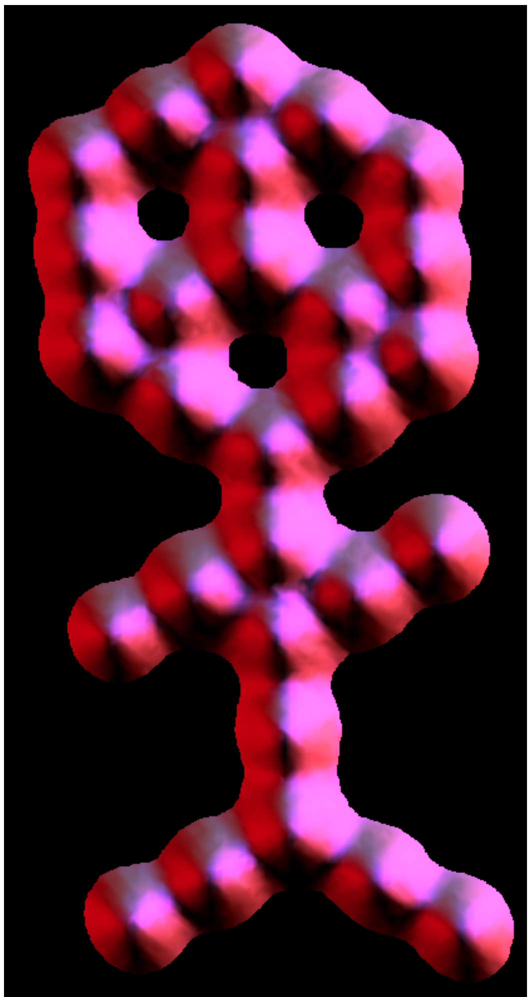
- The small forces between tip and sample can be used to move atoms and molecules
- At low temperatures, Ar or metal atoms can be adsorbed on an atomically flat metallic surface (Ag, Cu) and manipulated by STM
- Artificial structures can be constructed … vary slowly

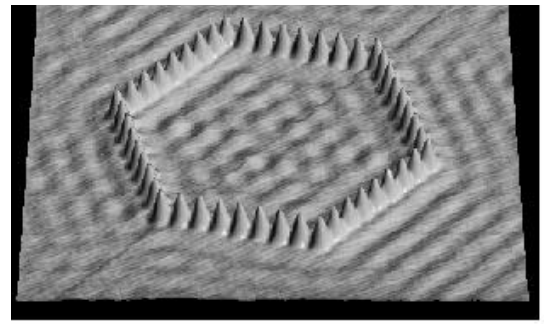


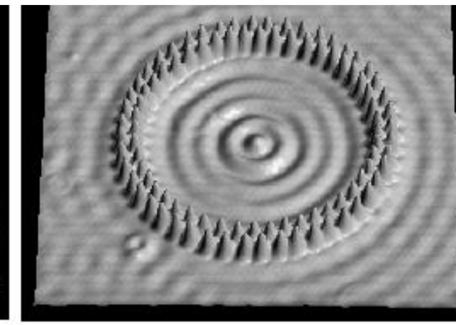
- Note the quantum-mechanical interference !

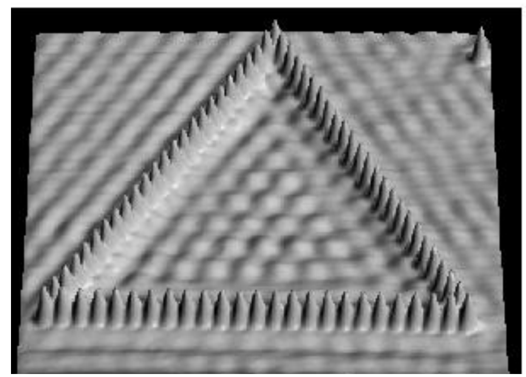
Examples of STM lithography

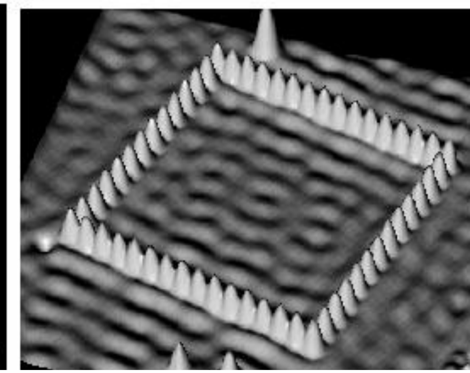
- From the labs of you-know-who…

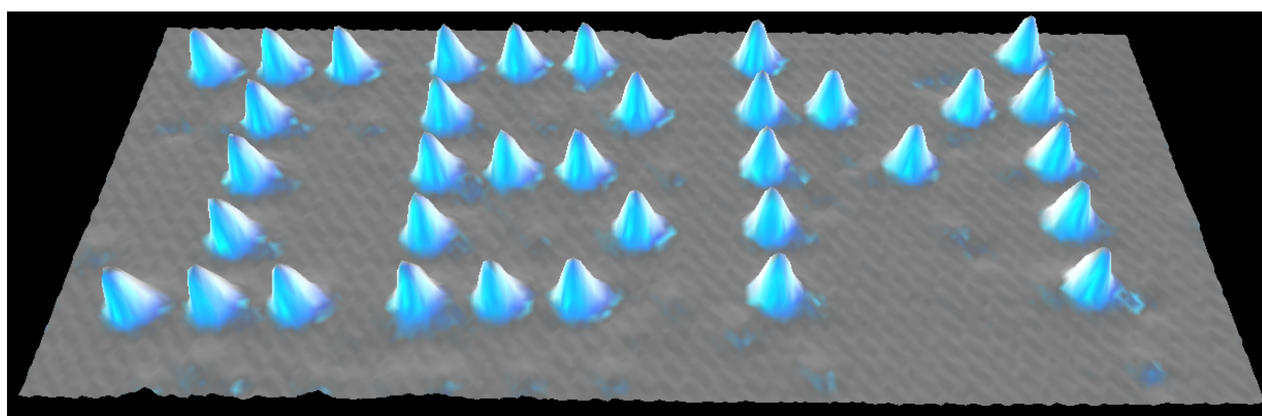






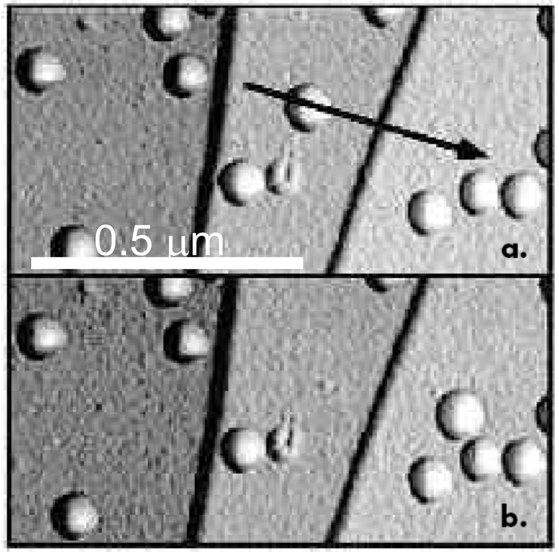


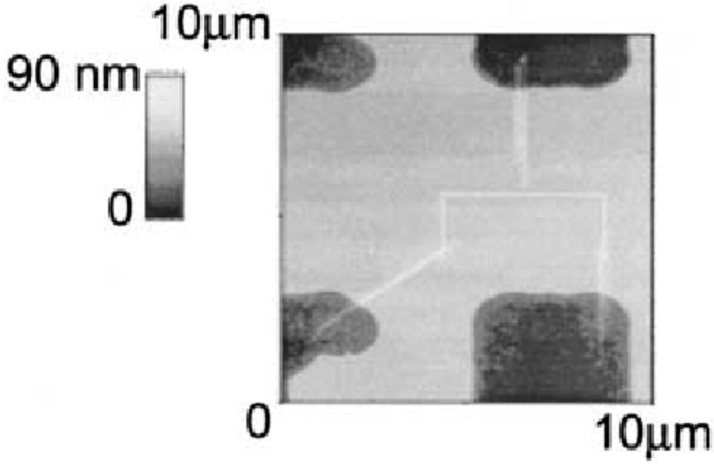


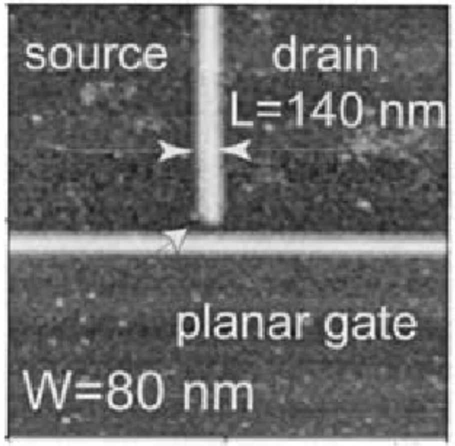


AFM lithography

- AFM can be used for nano-lithography in several ways:
  - The AFM tip can scratch or indent soft surfaces, e.g. a PMMA layer on Si, which is then used as template for etching, deposition, etc.
  - A voltage applied between tip and sample can oxidize a metallic (Ti) layer or the surface of a semiconductor (GaAs), forming isolated regions
  - The AFM tip can directly move molecules, nanoparticles

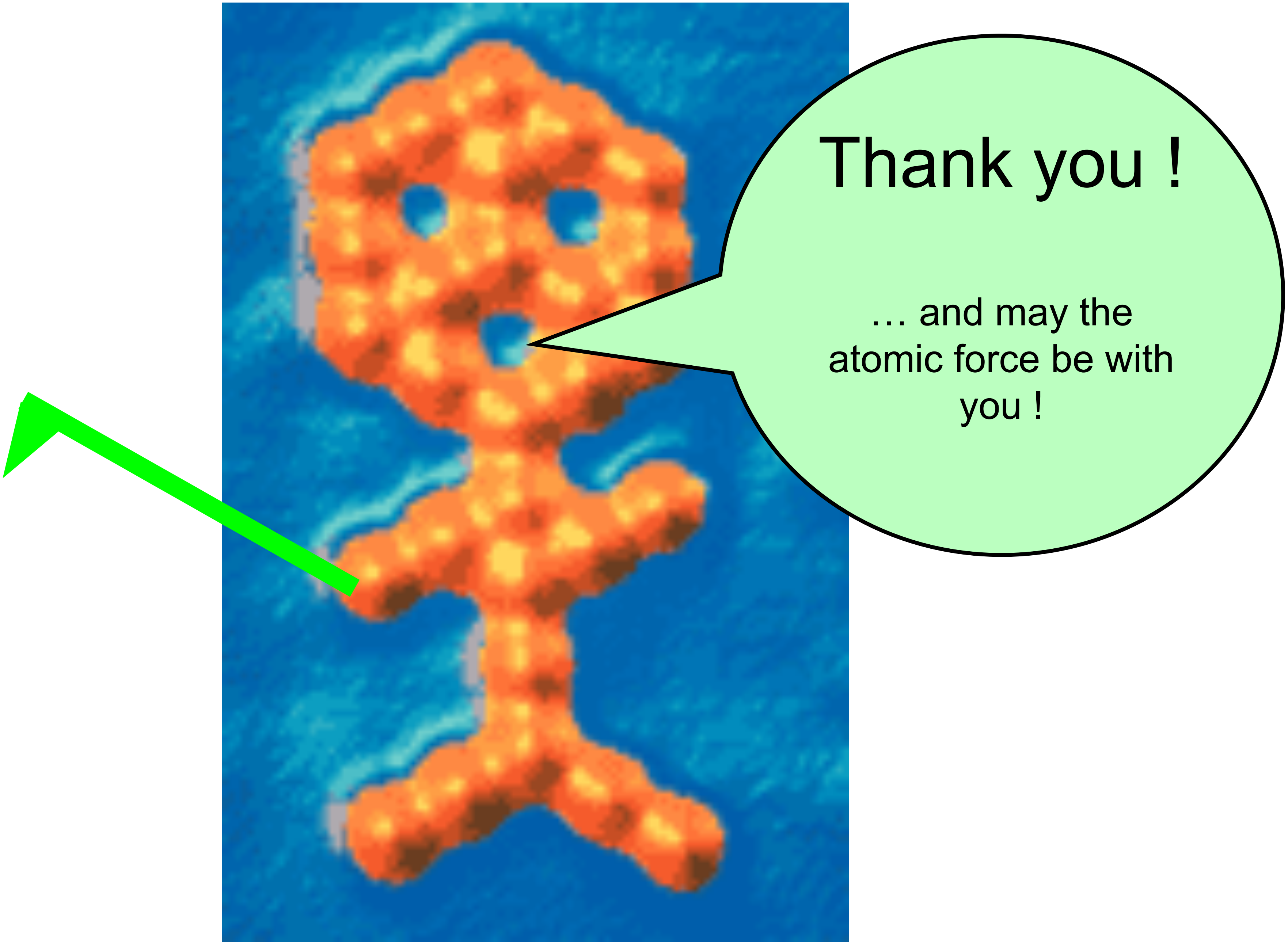






Summary

- The field of SPM has developed in 20 years into a multitude of surface characterization techniques
- AFM and its derivatives are simple and useful for characterization of semiconductors
- Cross-sectional imaging can reveal the insides of structures
- Industrial applications are growing, limited by AFM speed


Thank you !
… and may the atomic force be with you !

References

- NCSU tutorial : www.ncsu.edu/aif/SPM/AFM%20Tutorial.pdf
- NT-MDT application notes : http://www.ntmdt.com/page/primer
- Pacific Nanotech tutorial : http://www.pacificnanotech.com
- Nanosensors tips : http://www.nanosensors.com
- Nanonics SNOM : http://www.nanonics.co.il/learn-nsom.html
- LPN-SB, EPFL : http://lpn.epfl.ch