# Scanning Probe Microscopy

(SPM)

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PH.D2.397



#### **Outline:**

- Introduction: What is SPM, history
- STM
- AFM
- Image treatment
- Advanced SPM techniques
- Applications in semiconductor research and industry

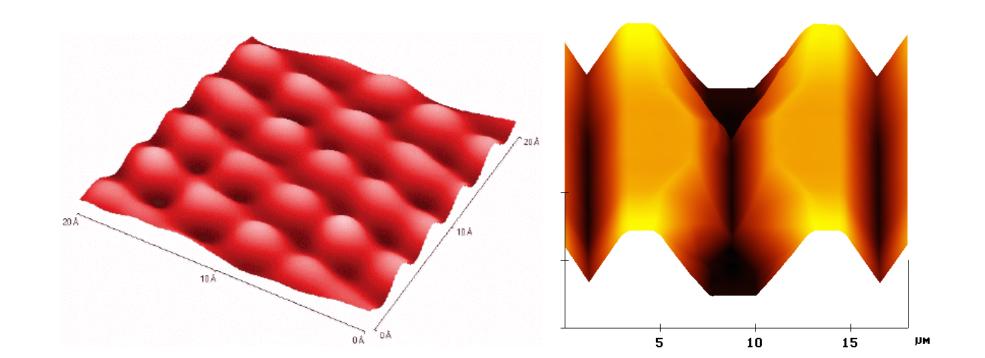


#### What is SPM?

#### Scanning Probe Microscopy:

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

Usually, only surface properties are observable





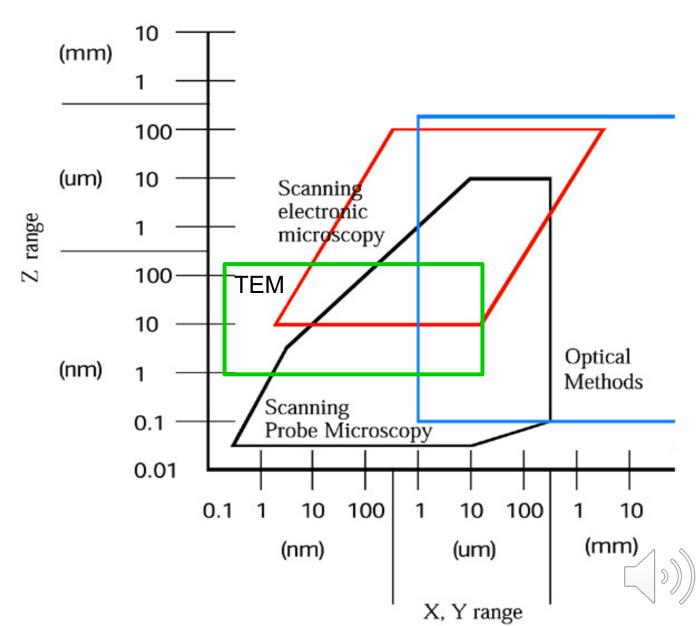
# How does SPM compare with other microscopy techniques?

Microscope	Optical	Confocal	Interferen ce	SEM	TEM	STM	AFM	SNOM
XY resolution	400 nm	150 nm	250 nm	1 nm	0.05 nm	0.1 nm	(<)1-10 nm	<50 nm
Z resolution	-	1 nm	0.1 nm	-	-	0.01 nm	0.01 nm	(0.01nm)
Ambience	air (liquid)	air (liquid)	air (liquid)	vacuum	vacuum	vacuum (air,liq.)	air (liquid)	air (liquid)
Sample preparation	none	none	none		polishing, ion milling	none / UHV cleaving	none	none
Damage to sample	none	none	none	/\		none	none (scratch)	none
Price (kFr)	5-30	50-200	50-200	200-700	500-5000	70-300	50-300	70-30)

## **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)



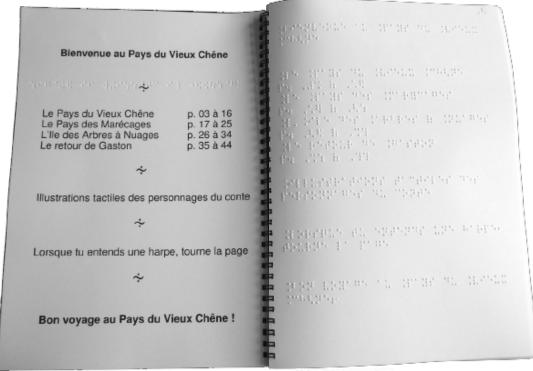
#### A (short) history of SPM

To get an idea of the surface of a sample, we can:

Observe it (optical imaging)

Or:

Touch it (mechanical scanning)

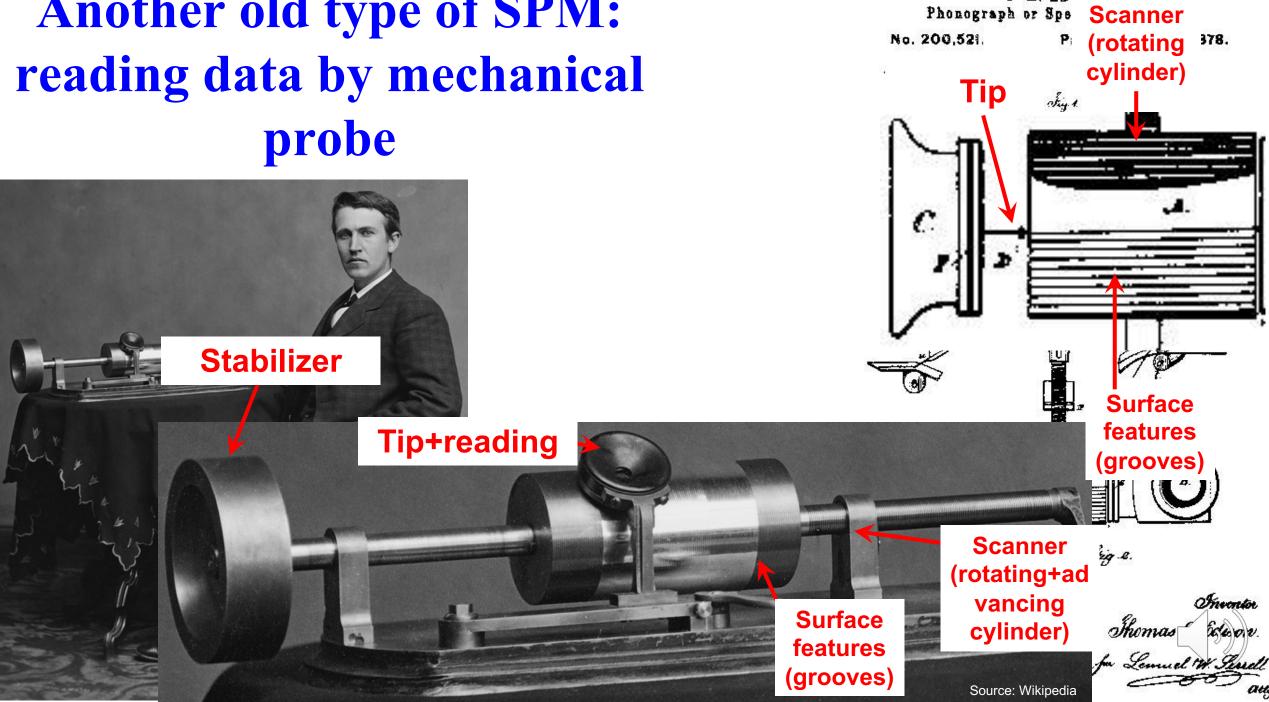


- Parallel reading
- (High resolution)

- Serial reading
- (Low resolution)



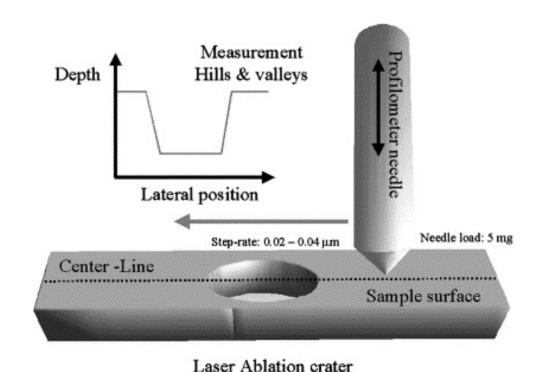
# Another old type of SPM: probe



T A. EDVON

# A more modern instrument: The Stylus Profilometer ("Alpha-step")

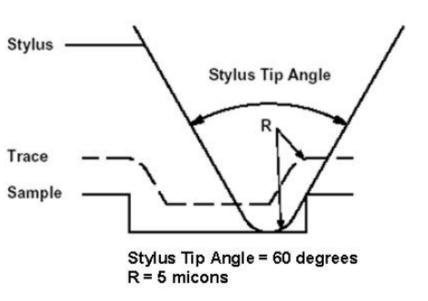
- Scans a line profile of the surface with a mechanical tip at a low force
- Measures the height profile across the line scan

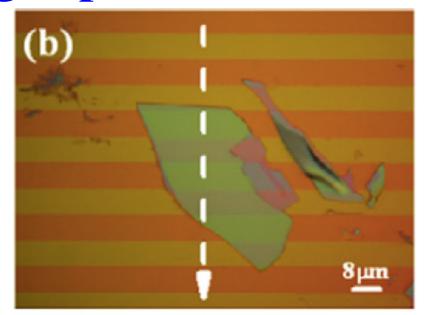


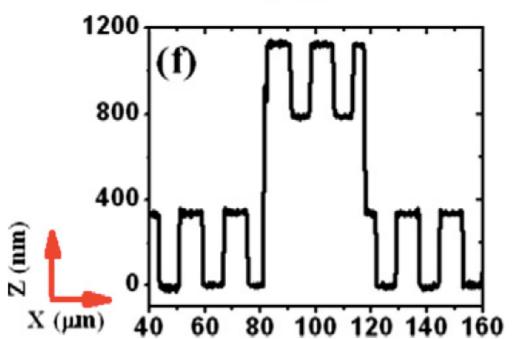


#### Typical height profiles

- Scan length: up to 100 mm
- Z-resolution: 5-10 nm
- X-resolution: ~10 μm (due to tip size)
- Scan time: 10-100 s

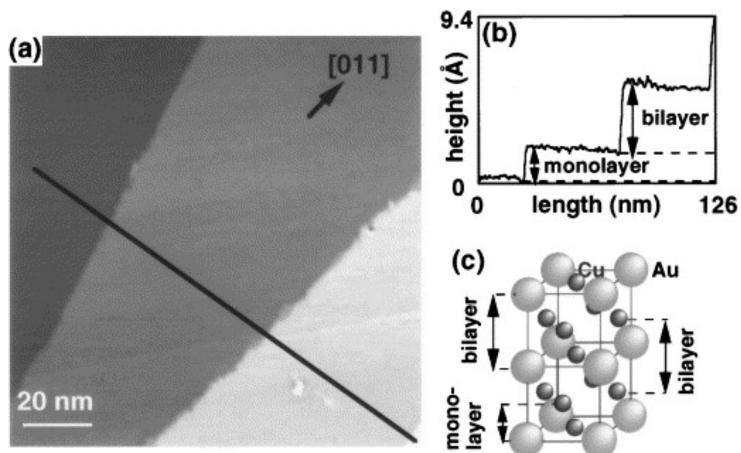








# The challenge of modern SPM: How to get nm resolution in all dimensions (X,Y,Z)?





#### First attempt: The Topografiner (1972)

#### Student presentation:

R. Young, J. ward, F. Scirer, Rev. Sci. Inst. **43**, 999 (1972) "A noncontacting instrument for measuring the microtopography of metallic surfaces"



# Let's think: How to get nm resolution?

#### Potential problems:

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

#### Solutions:

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



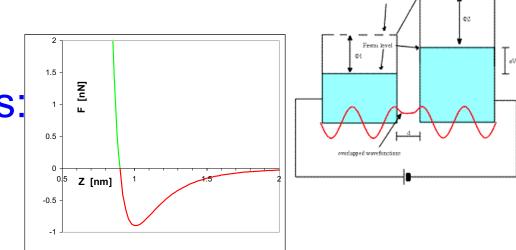
#### How to get nm resolution:

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

Nuclear (strong) forces
 But range is too short!

Quantum-mechanical electron tunneling: STM

Van-der-Waals forces:
 AFM



## First working STM: Binnig & Rohrer, 1982

#### Student presentation:

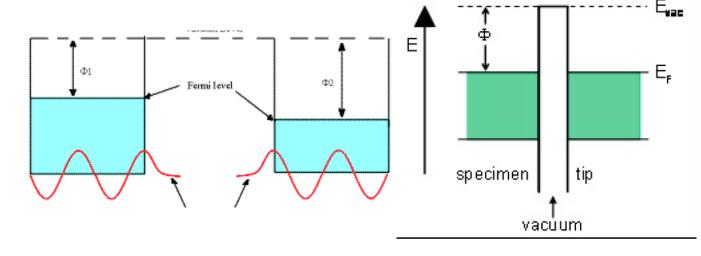


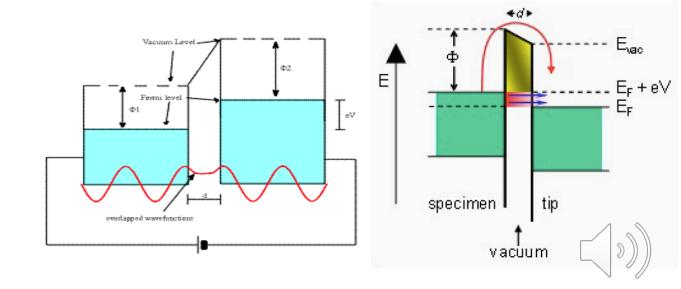
G. Binning, H. Rohrer, Ch. Gerber, and E. Weibel, Phys. Rev. Lett. **49**, 57 (1982) "Surface Studies by Scanning Tunneling Microscopy"



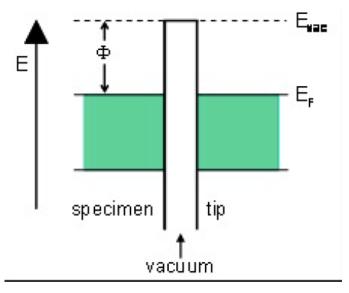
#### Quantum Tunneling between metals

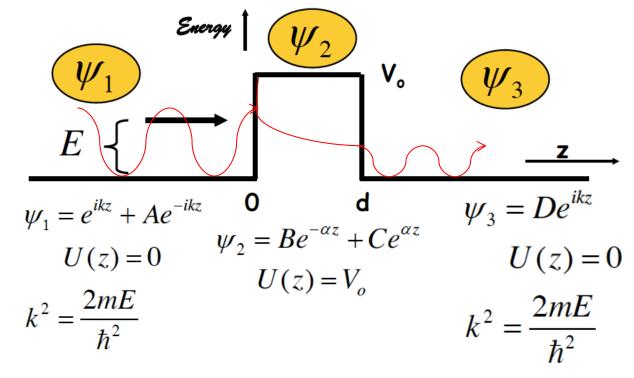
- Electrons in metals fill up energy levels to the Fermi level, which is about 5 eV (the work function) below the Vacuum level Φ.
- External potential between metals shifts the relative Fermi levels, so that electrons could pass from full (left) to empty (right) levels, but... the energy barrier is too high!
- When the metals are close enough, electron wavefunctions can overlap and tunneling current flows





#### Quantum Tunneling: wavefunctions





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



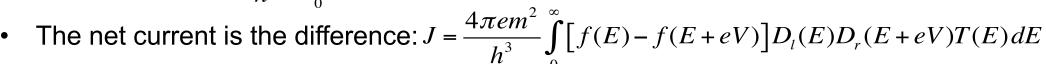
#### Quantum Tunneling: current flow

- 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):

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

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

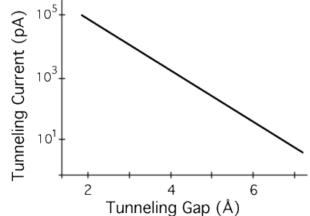
$$J_{r\to l} = \frac{4\pi em^2}{h^3} \int_{0}^{\infty} f(E+eV)D_l(E)D_r(E+eV)T(E)dE$$

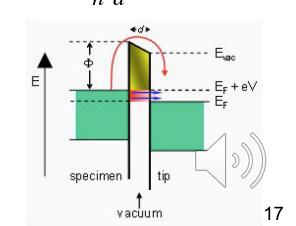




Fermi distribution function and get: 
$$J = \sigma_0 V e^{-2d\sqrt{2m\Phi}/\hbar} \propto V e^{-2\alpha d}$$
, with:  $\alpha = \frac{\sqrt{2m\Phi}}{\hbar}$   $\sigma_0 = \frac{e^2\sqrt{2m\Phi}}{\hbar^2 d}D_l(E_F)D_r(E_F)$ 

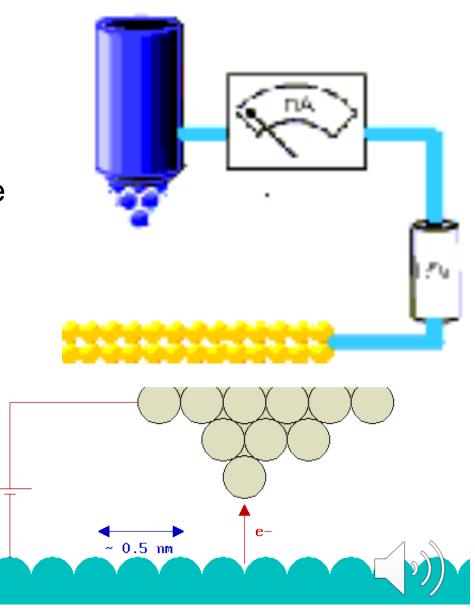
- The I-V curve is linear, as in an Ohmic contact
- Wavefunction decay length is very short:  $\ell = \frac{\hbar}{\sqrt{2m\Phi}}$  ~ 0.1 nm!
- This makes the current very sensitive to the distance: a change of distance of one atomic monolayer = 0.3 nm, gives change of current by x1000!





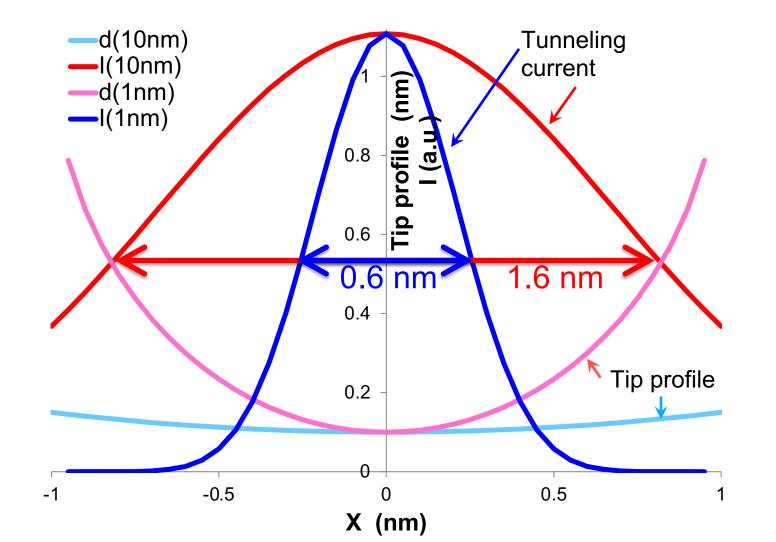
#### Tunneling as surface probe:

- 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 a tip-sample distance of 1Å



#### Tunneling between plane and tip

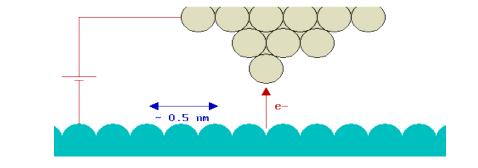
Calculation of tunneling current between plane and half-spherical tip of identical metals, for tip radii of 1 and 10 nm: apparent width is much smaller!

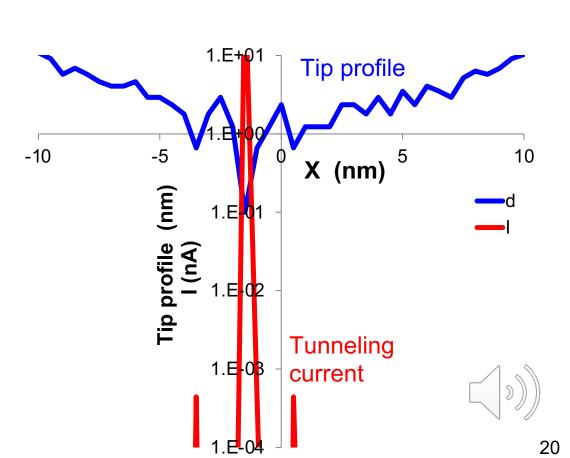




#### Tunneling between plane and tip

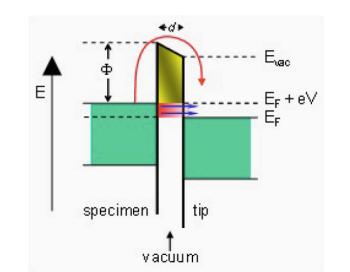
- In a more realistic case, the random atomic nature of the tip will "promote" one atom to produce most tunneling current. Here the tip R=10 nm, h=0.1 nm, lattice constant 0.56 nm
- The current through the lowest atom is bigger by 10<sup>4</sup> than the current from the next one!
- The real "tip" (atom) is not at the position X=0, but it doesn't matter! It's only a small fixed shift in the image





#### Tunneling between non-identical materials

- The tunneling current is given by:  $J = \frac{4\pi e m^2}{h^3} \int_0^\infty [f(E) f(E + eV)] D_l(E) D_r(E + eV) T(E) dE$
- In tunneling between different materials, the electron transmission probability depends on the electron density of states (DOS) at the Fermi level  $D(E_F)$ . This gives tunneling current:  $J = \propto VD(E_F)e^{-2\alpha d}$
- There is still exponential dependence on tip-sample distance
- The DOS of the sample can be measured

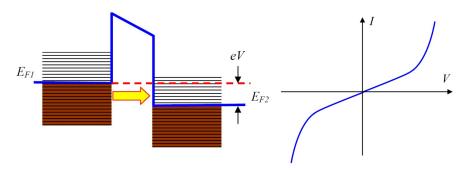


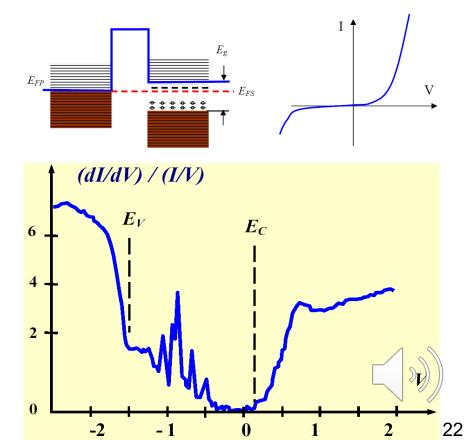


#### STS: Scanning Tunneling Spectroscopy

- Tunneling between the metallic STM tip and a metal shows a gapless (Ohmic) I/V curve
- Tunneling between the metallic STM tip and a semiconductor shows the energy gap in the I/V curve (diode-like)
- Similar behavior in superconductors, where pair tunneling can be measured

- 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
- These measurements are often done at low temperatures (reduce phonons)





# The main problem: How to get nm resolution?

Potential problems: Solutions:

Tip size
 Short-range interactions

2. High-resolution XY scanning 2. Piezo scanner

3. Non-destructive 3. Non-contact

4. Keep distance from sample 4. Height feedback

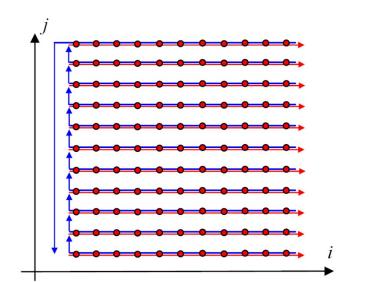
5. Vibrations 5. Rigid structure, isolation

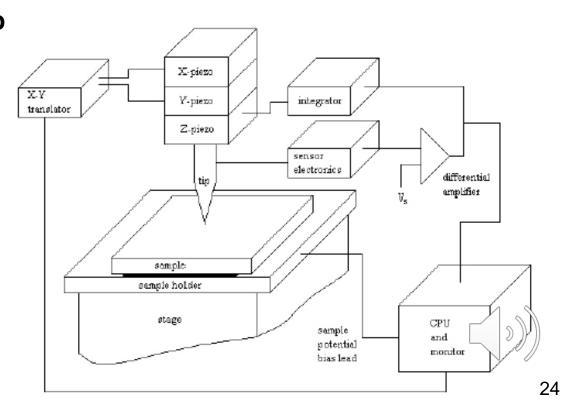
6. Thermal stability 6. Compensation



## High resolution: the piezo scanner

- The X-Y-Z relative movement between tip and sample is controlled by a Piezoelectric Scanner with < 0.1nm precision</li>
- 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

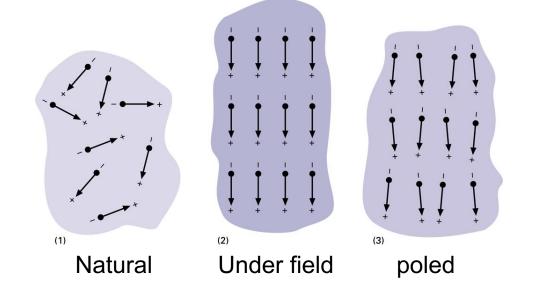


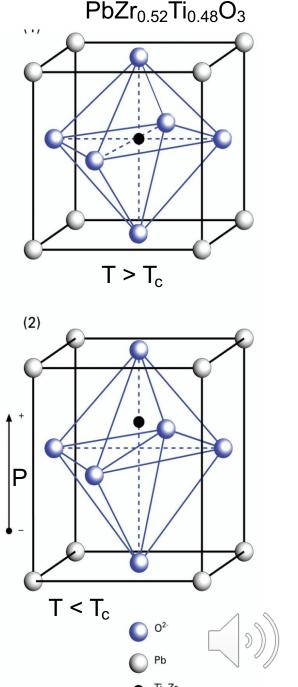


## The piezoelectric effect

What is the piezoelectric effect?

- Change in length induced by electric field (also the reverse)
- Atomic structure of PZT: cubic crystal becomes tetragonal at low temperatures, producing a dipole (Ti<sup>+</sup>/Zr<sup>+</sup> ion is above the O<sup>-2</sup> ions).
- Dipoles in domains can be ordered by an external field, applied during cooling (poling)





#### The piezo scanner

The displacement of the piezo element:  $\frac{\Delta L_i}{L_i} = d_{ij}E_i$  i,j=1,2,3

#### The most interesting directions:

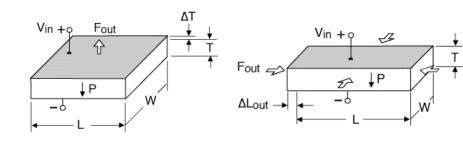
- i=j=3 (z), used in linear motion
- i=1, j=3, used in flexing motion

Typical values: 
$$d_{33} = 5.10^{-10} \text{ m/V (Small!)}$$

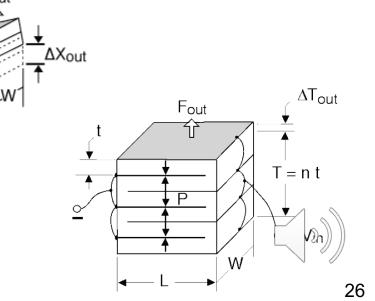
$$d_{13} = -2.10^{-10} \text{ m/V}$$

#### Ways to extend the motion:

- Levering system
- Multi-layer stack, same voltage applied to many piezo elements in series
- Can reach 100μm movement with a voltage of 100-1000V

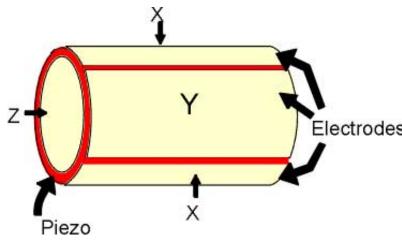


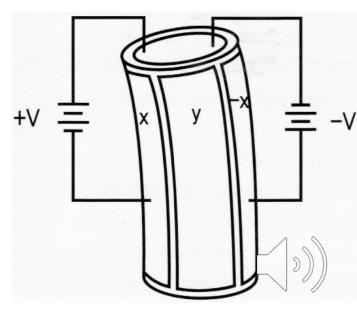
Source: http://piezo.com



#### Types of piezo scanners: Tube

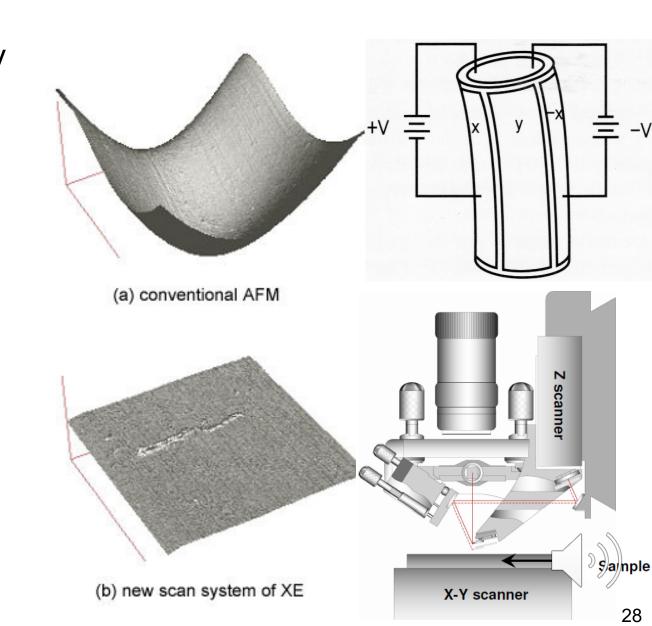
- All movements (X-Y-Z) can be achieved by a single tube scanner:
  - Applying opposite voltages to pairs of electrodes bends the tube in one direction (X,Y)
  - Applying voltage to the inner electrode makes the tube contract/expand (Z)
- Advantages of tube scanner:
  - Simple, small, rigid, cheap (single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





## Types of piezo scanners: XY/Z

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

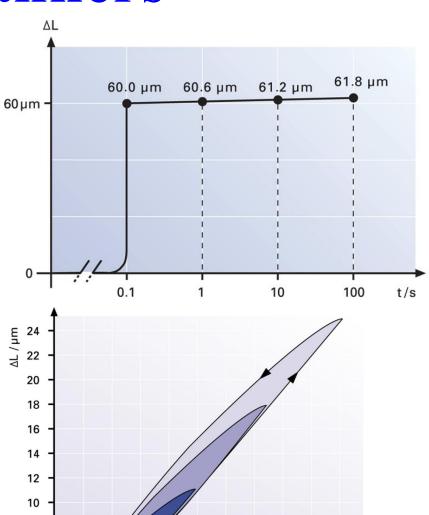


#### Problems of piezo scanners

• Creep: The displacement of the piezo element continues slowly after a (large) step:

$$\Delta L(t) = \Delta L(t_0) \left[ 1 + \gamma \ln(t/t_0) \right] \qquad \gamma = 1-2\%$$

- Hysteresis: The displacement does not follow the same curve when moving back and forth
- Non-linearity: The displacement is non-linear with applied voltage

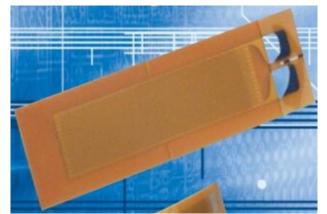


#### Improving the piezo scanner

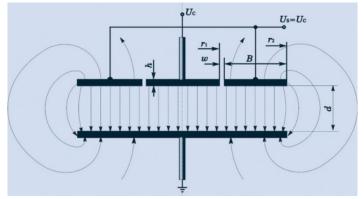
The piezo does not give precise positioning. We need sensors to get precise position readout / correction

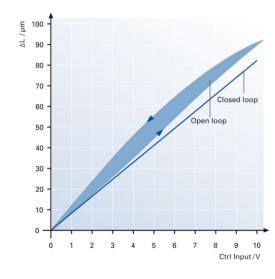
Types of sensors:

Strain

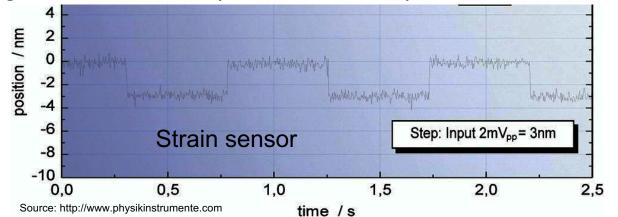


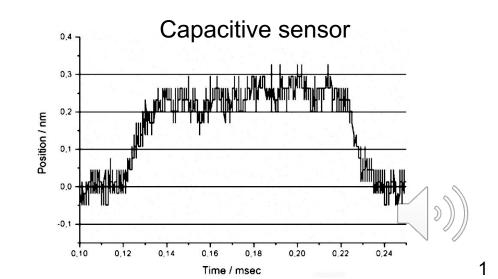
Capacitive





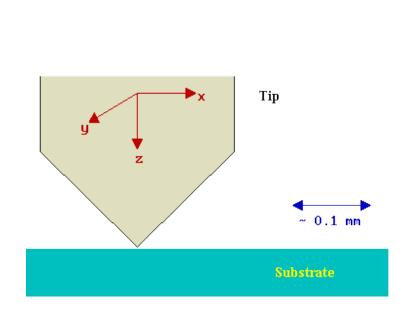
The limiting factor: noise (also in driver)!

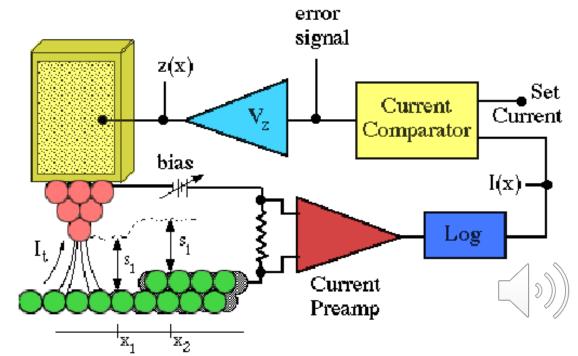




#### The use of feedback:

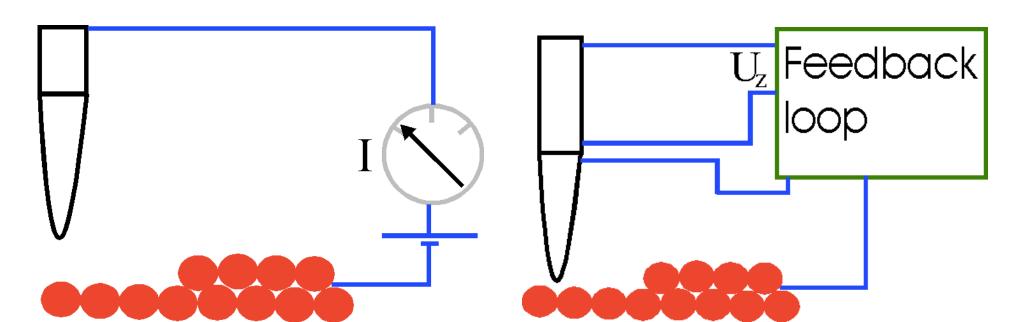
- In STM, current varies exponentially with tip-sample distance
- A log amp gives signal inversely proportional to distance
- The signal is fed back to a Piezoelectric actuator, to keep the same current = same distance
- The piezo tracks the surface of the sample, giving a high-resolution height map





#### Two ways to scan:

- Constant tip height: Imaging the different surface atoms (due to their different work functions), revealing the surface composition or defects.
- Constant current: Imaging the surface topography at atomic resolution if the surface is composed of the same atoms.





## The main problem: How to get nm resolution?

Potential problems:

Solutions:

1. Tip size

1. Short-range interactions

2. High-resolution XY scanning 2. Piezo scanner

3. Non-destructive

3. Non-contact

4. Keep distance from sample

4. Height feedback

5. Vibrations

5. Rigid structure, isolation

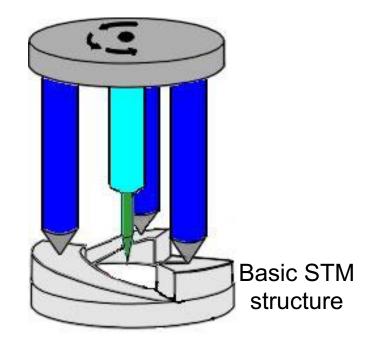
6. Thermal stability

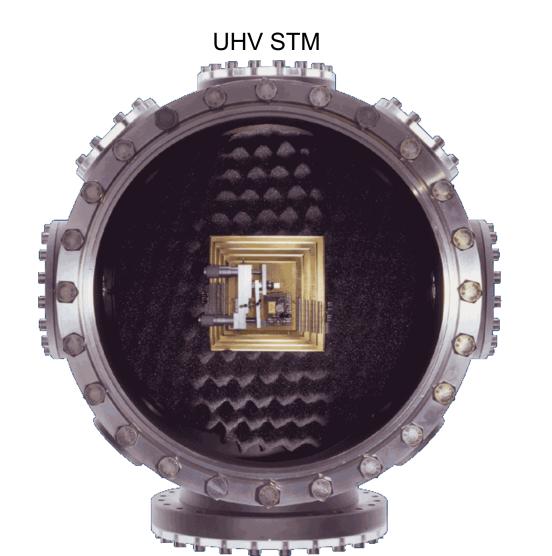
6. Compensation



#### STM construction

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

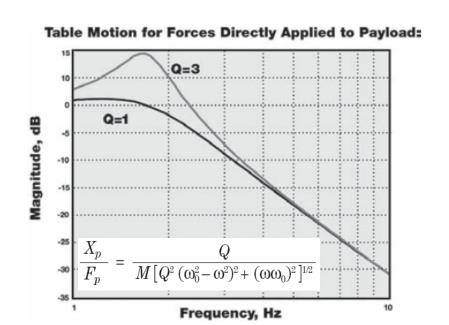


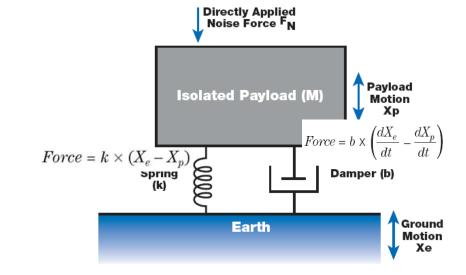


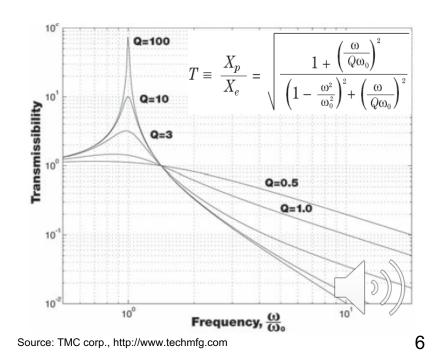


#### **Vibrations**

- Sources of vibrations:
  - Ground vibrations
  - Directly coupled vibrations (acoustic)
- Model of vibration isolation: mass, spring, damper
- Resonant system with characteristic frequency ω and quality factor Q

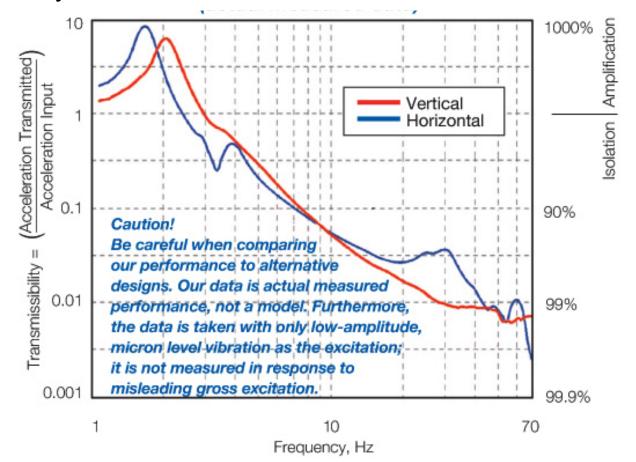


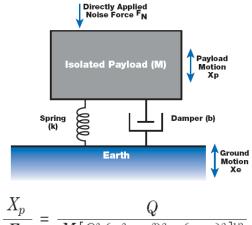




#### **Vibrations**

- Performance of a real passive table:
  - No damping (even amplification...) below 2-3 Hz
  - Performance increases with frequency T  $\sim \omega^{-2}$
  - Secondary resonances at 3.2, 30, 53, 70 Hz



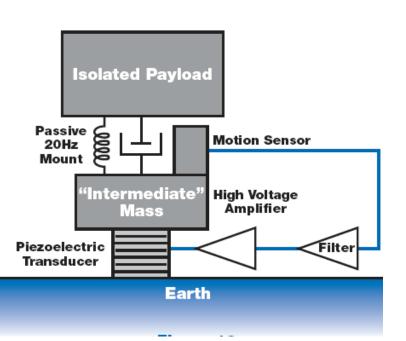


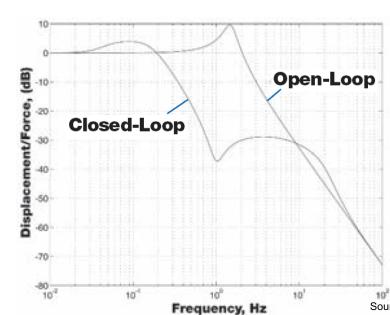
$$\frac{X_p}{Y_p} = \frac{Q}{M[Q^2(\omega_0^2 - \omega^2)^2 + (\omega\omega_0)^2]^{1/2}}$$

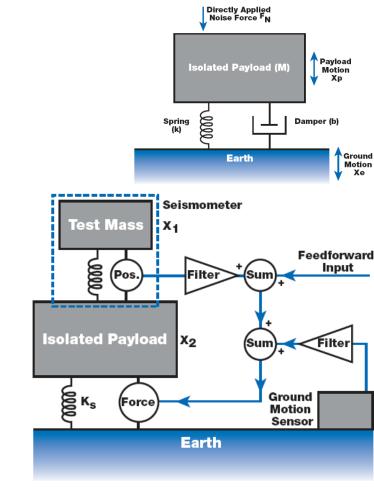


## **Vibrations**

- Active tables: feedback system
  - Motion sensor
  - Piezo or motor to counteract external forces
- Combination: active+passive



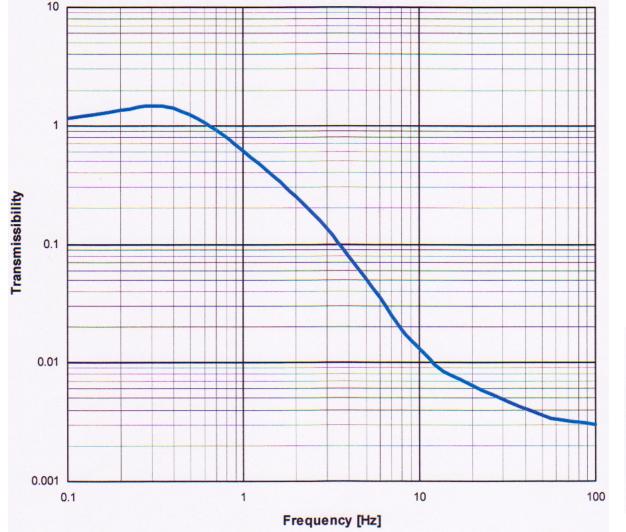


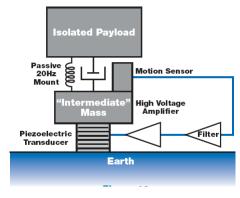




## **Vibrations**

- Active tables:
  - Small size, light weight
  - (almost) no amplification at low frequencies



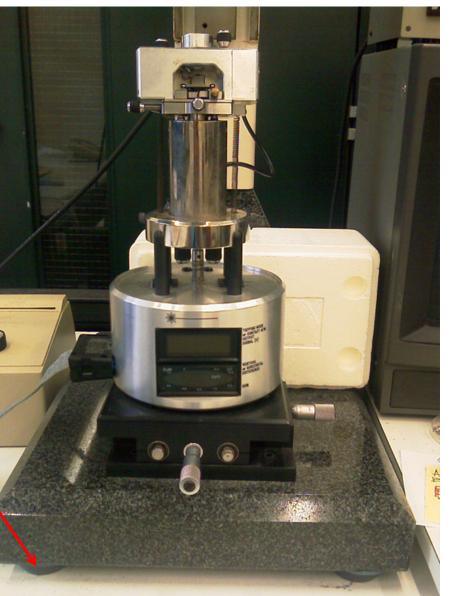




# Vibrations: examples

- Passive isolation
  - Floating table
  - AFM mounted on granite slab with rubber feet (secondary passive isolation)

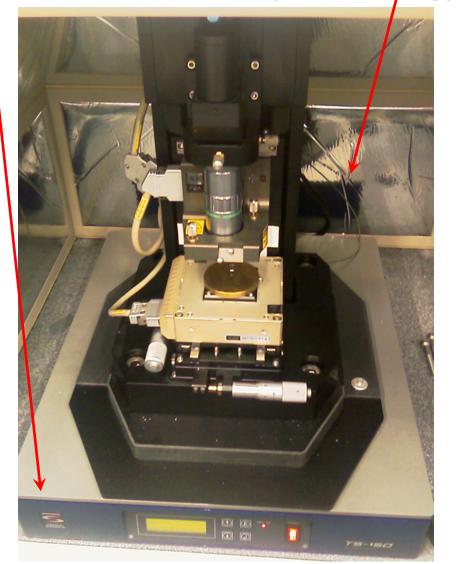






# Vibrations: examples • Active isolation table inside passive metal enclosure

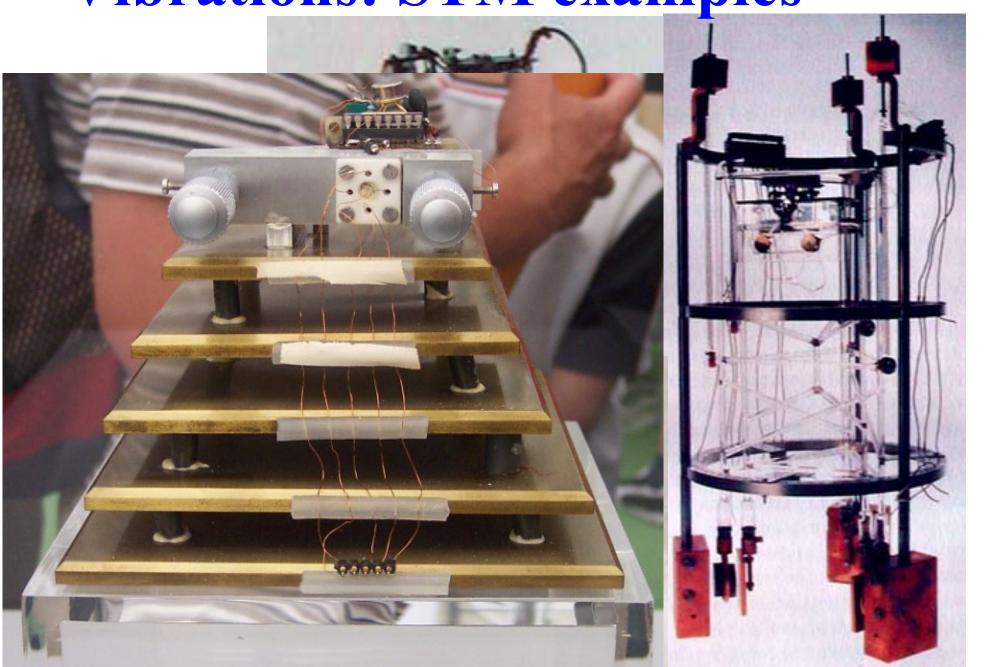
Acoustic isolation (inside lining)







Vibrations: STM examples



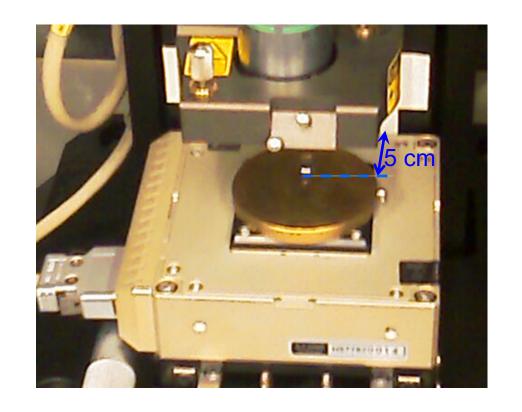


## Thermal stability

#### Example 1:

- AFM structure made of AI (thermal expansion coeff. 23·10<sup>-6</sup> °C<sup>-1</sup>)
- Distance between sample and support: 5 cm
- Temperature change of 1°C
- By how much would the sample move?

Answer: by  $1.06 \mu m$ !



Not very good for measuring atoms...



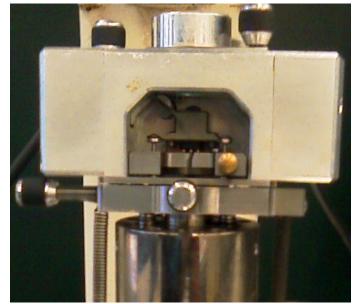
# Thermal stability

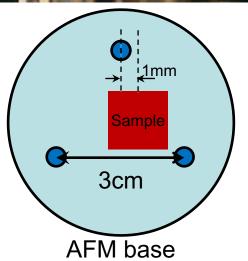
#### Example 2:

- AFM head made of Fe (thermal expansion coeff. 12·10-6 °C-1)
- Distance between supporting screws: 3 cm
- Sample is offset by 1mm from center of support
- Temperature change of 1°C
- By how much would the sample move?

Answer: by 12 nm!

- We need to stabilize room temperature, but also:
  - To use materials with small expansion coeff. (Invar)
  - To construct the AFM in a self-compensating, symmetrical way

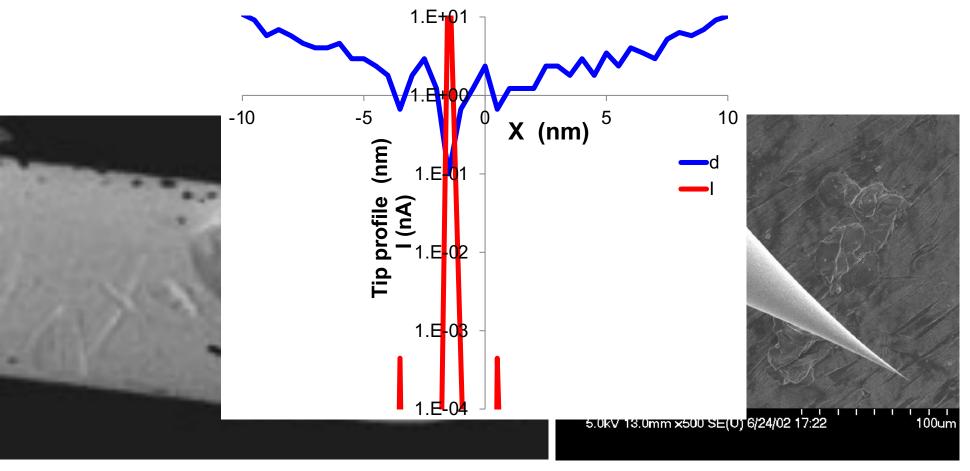






# STM tips

#### Hint: remember this curve?



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

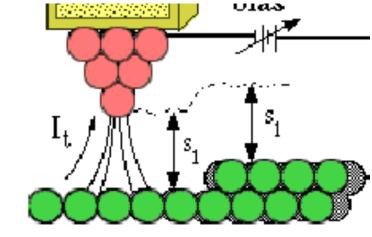
W wire, diameter 0.2 mm, electroetched in KOH solution

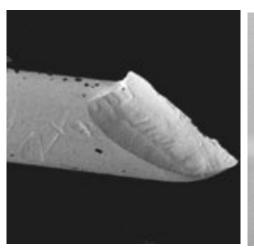


# STM tips

#### Answer: They give the same performance!

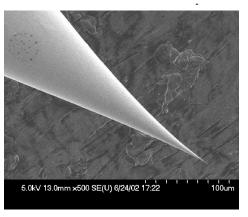
- Tip radius is not very important: rigidity is more important than tip radius!
- Exponential tunneling current dependence "selects" that only the very last atoms of the tip participate in the imaging











Etched STM tip



Cut STM tip

"Swiss" STM tip

(Same, enlarged x108)

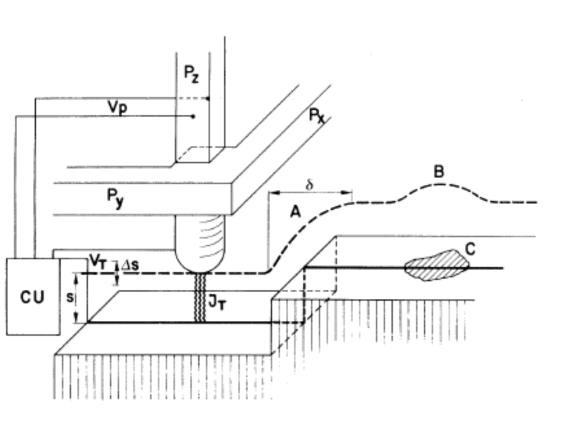
# The first STM (1982)

#### Surface Studies by Scanning Tunneling Microscopy

G. Binning, H. Rohrer, Ch. Gerber, and E. Weibel

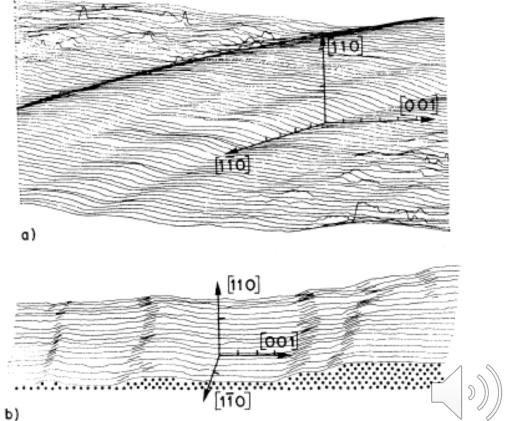
IBM Zurich Research Laboratory, 8803 Rüschlikon-ZH, Switzerland

(Received 30 April 1982)



Schematic structure of the STM

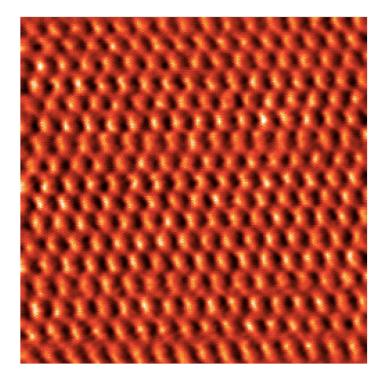
emission microscopy. However, since suppression of vibrations is evidently more vital for the STM, long and narrow field-emission tips might not be satisfactory. Instead, we used solid metal rods of 1 mm diameter, and ground  $90^{\circ}$  tips with a conventional grinding machine. This yielded overall tip radii of only some thousand angstroms to  $1\,\mu$  m, but with some rather sharp minitips. The extreme sensitivity of the tunnel current on gap width then selects the longest of the minitips for operation of the STM. The lateral resolution



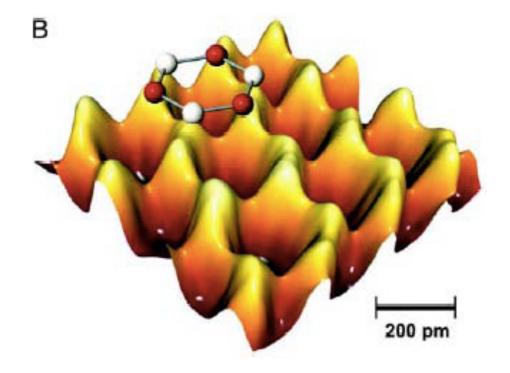
STM Image of Au surface (b: after annealing)

## **STM Images**

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



STM Image of pyrolitic graphite atoms

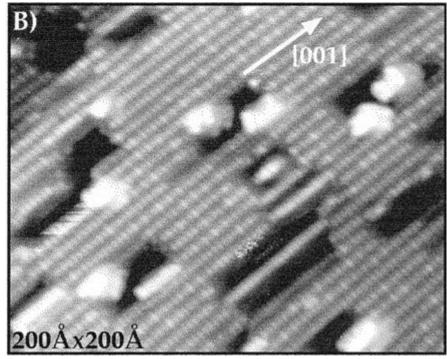


STM Image of Graphite, and the carbon ring underlying (top)

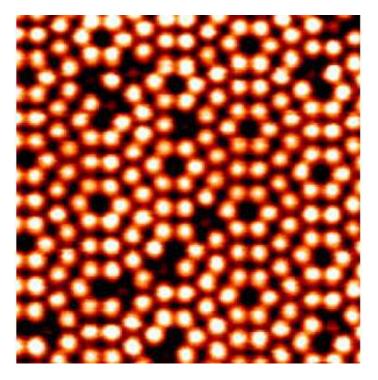


## **STM Images**

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



STM Image of CO molecules on RuO<sub>2</sub> crystal

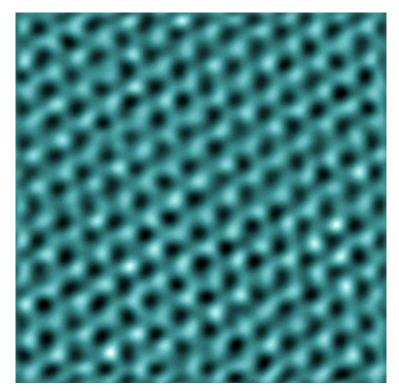


STM Image of Si atoms

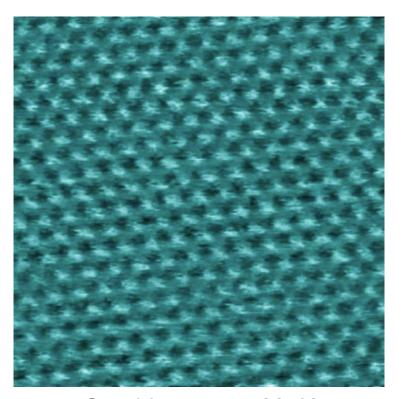


## The effect of temperature

• STM images are sensitive to thermal atomic distance fluctuations, so low-T STM is essential for atomic studies



Graphite atoms, 77K



Graphite atoms, 295K



More STM Images later ...

### **Outline:**

- Introduction: What is SPM, history
- STM
- AFM
- Image treatment
- Advanced SPM techniques
- Applications in semiconductor research and industry



#### Back to our solutions:

#### The main problem: How to get nm resolution?

#### Potential problems:

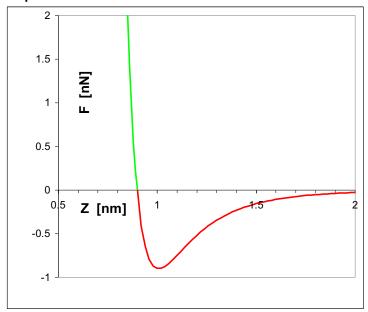
- 1. Tip size
- 2. High-resolution XY scanning
- 3. Non-destructive
- 4. Keep distance from sample
- 5. Vibrations
- 6. Thermal stability

#### Short-range interactions:

Van-der-Waals forces

#### Solutions:

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



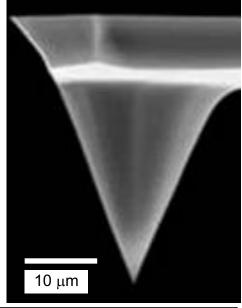


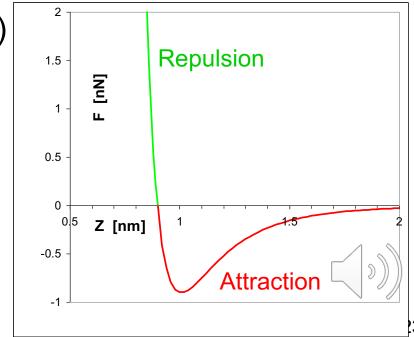
# Atomic Force Microscope: AFM

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

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

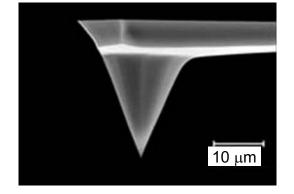
- But: Van-der-Waals force are small: order of nN
- We need a sensitive force sensor

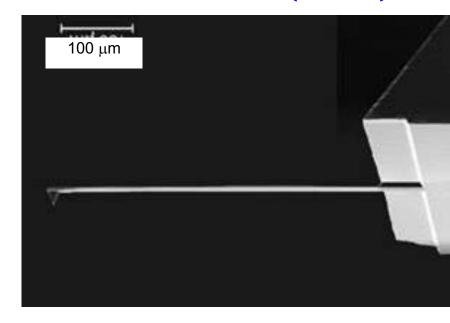


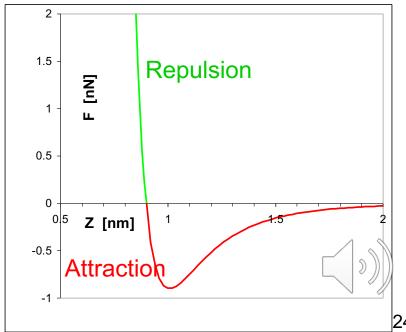


## AFM: How do we measure small forces (nN)?

- Solution: use the flexibility of the tip-carrying cantilever
- Material: Si
  - Tough, flexible E= 2.10<sup>5</sup> N/cm<sup>2</sup>
  - Easy to structure by photo-lithography and chemical etching
- The force on the tip moves (flexes) the cantilever.



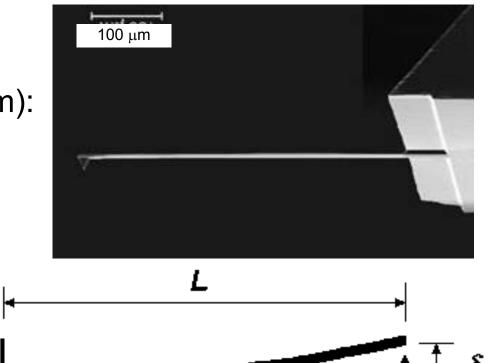




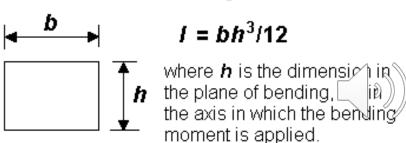
## How do we measure small forces (nN)?

- Deflection of a beam:  $\delta = FL^3/3EI$
- For a typical cantilever (L=400  $\mu$ m, b=20  $\mu$ m, h=2  $\mu$ m):
  - $\delta = 0.2 \,\mu\text{m/nN}$
- The angular deflection is:
- $\alpha = F/Lk$ , where  $k = 3EI/L^3$
- Typical value: 0.6 °/nN
- Next question:

How do we measure a small deflection?

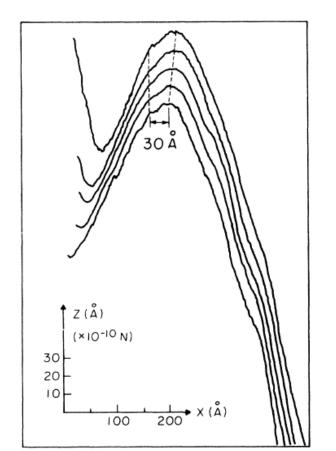


Moment of inertia for rectangular section



#### How do we measure small deflections?

- First solution (1986): By an STM!
- Use the stm tip to sense the z-position of the AFM tip on the PHYSICAL REVIEW LETTERS cantilever (used as a spring)
- Complicated!





#### Atomic Force Microscope

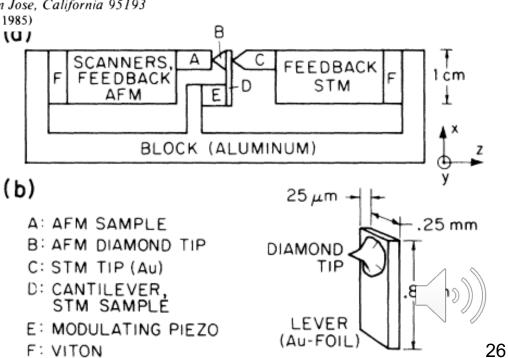
G. Binnig<sup>(a)</sup> and C. F. Quate<sup>(b)</sup>

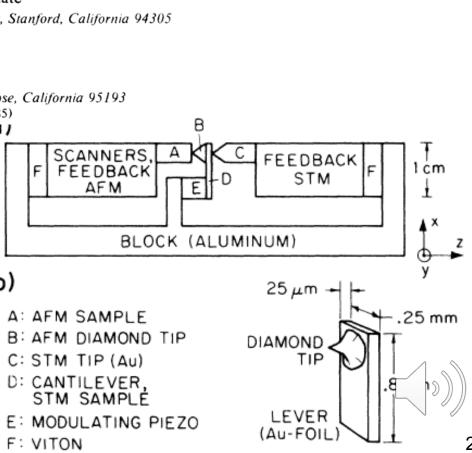
Edward L. Ginzton Laboratory, Stanford University, Stanford, California 94305

and

Ch. Gerber(c)

IBM San Jose Research Laboratory, San Jose, California 95193 (Received 5 December 1985)

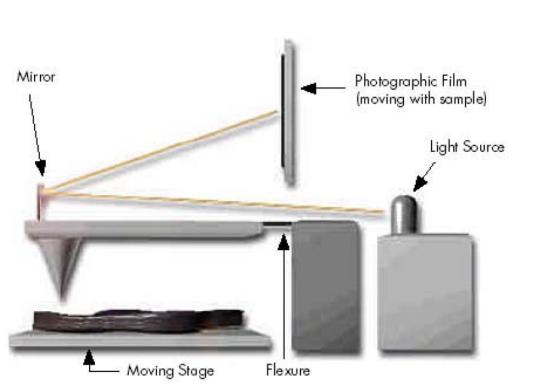


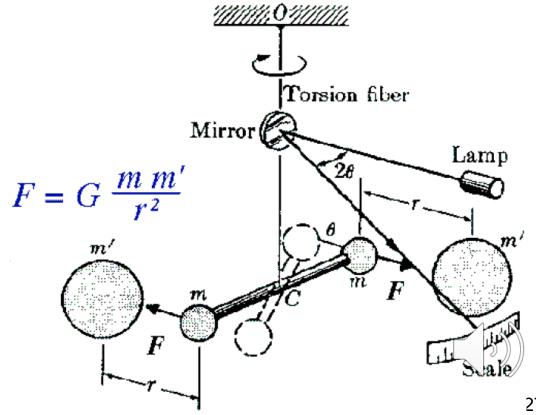


100 μm

# How do we measure small deflections (nm)? With light!

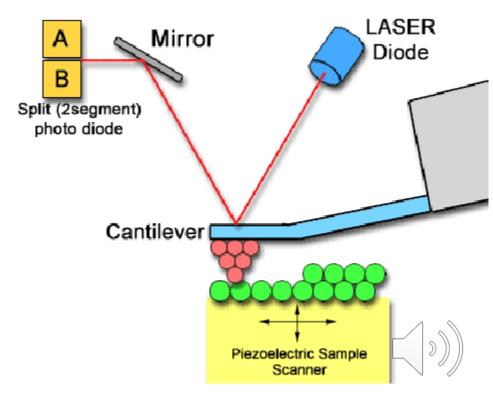
- Light beams have been used to measure small deflection, e.g. by Cavendish in his measurement of G (1798) and surface profilometer of Schmaltz (1929)
- The light beam serves as a long weightless lever





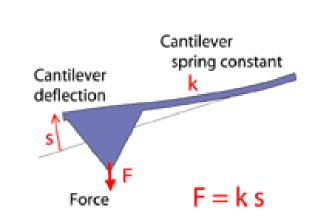
# How do we measure small deflections (nm)? With a laser!

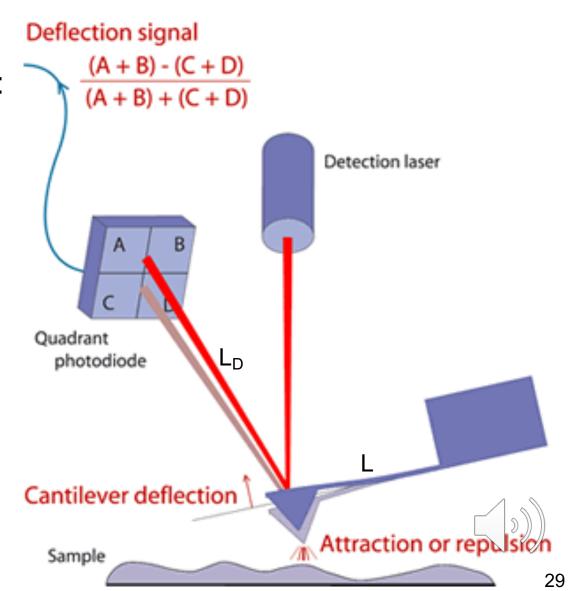
- A laser beam is reflected from the cantilever to a position-sensitive photodetector
- The detector signal is proportional to the tip displacement, which is proportional to the force
- The tip is scanned over the sample (as in STM) to produce the image



## **Operation of contact AFM**

- We saw that the deflection of the cantilever is:
  - $\delta$  = FL<sup>3</sup>/3EI, typical 0.2  $\mu$ m/nN
- The reflected laser beam moves by a distance:  $\delta L_D = \delta L/L_D = FL^2/3EIL_D$
- Typical value ( $L_D \sim 20 \text{ mm}$ ):  $\delta L_D = 10 \mu \text{m/nN}$

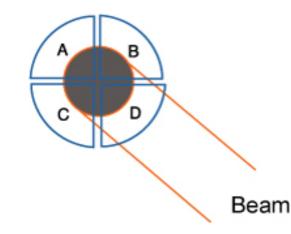




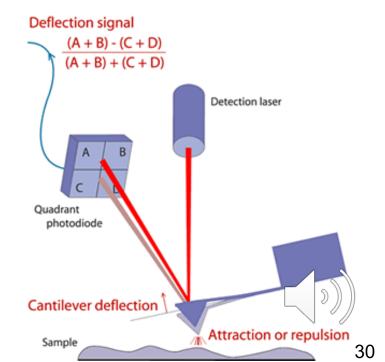
## **Operation of contact AFM**

• The laser beam movement (10  $\mu$ m/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 laser beam alignment on the cantilever
- 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,  $P_{refl}$ =10 $\mu$ W, gives a SUM signal of 10V. Difference of 10  $\mu$ m (1%, corresponding to a force of 1 nN) gives a difference signal of 0.1 V



## **AFM feedback**

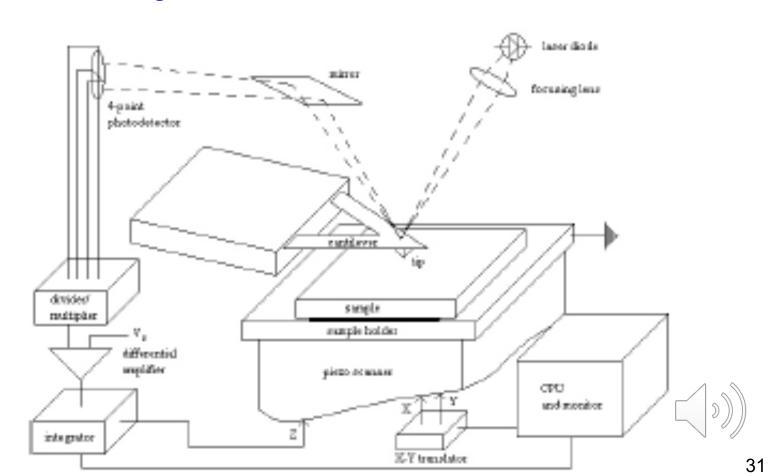
• As in STM, feedback is used to keep the tip at a constant force from the sample, the height is plotted as image.

On flat surfaces (to a few nm), constant height can be maintained, and the force is

displayed

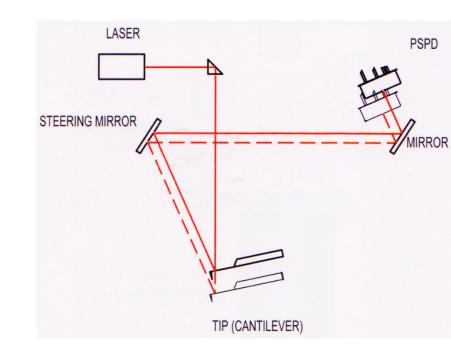
#### Solutions:

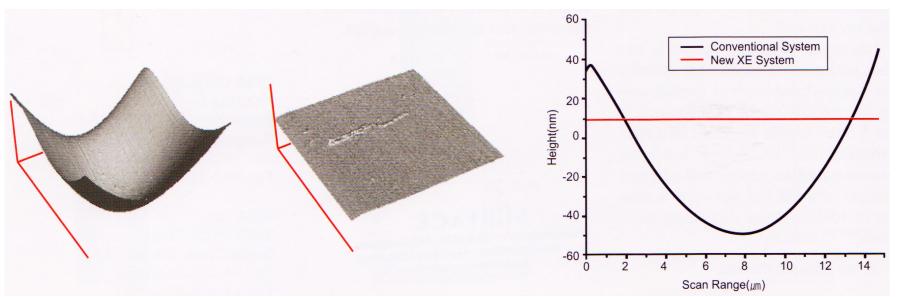
- 1. Short-term interactions
- 2. Piezo scanner
- 3. Non-contact
- 4. Height feedback
- 5. Rigid structure, isolation
- 6. Compensation

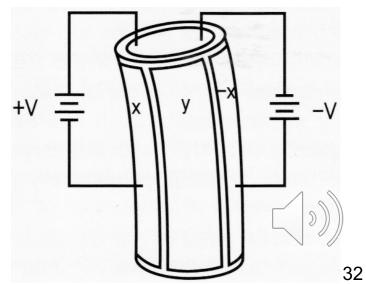


# Scanner types

- As in STM, there are two scanner types: tube and separate (XY-Z)
- The separate XY-Z scanner is more linear, and has no Z-distortion
- Linearization is provided by sensors

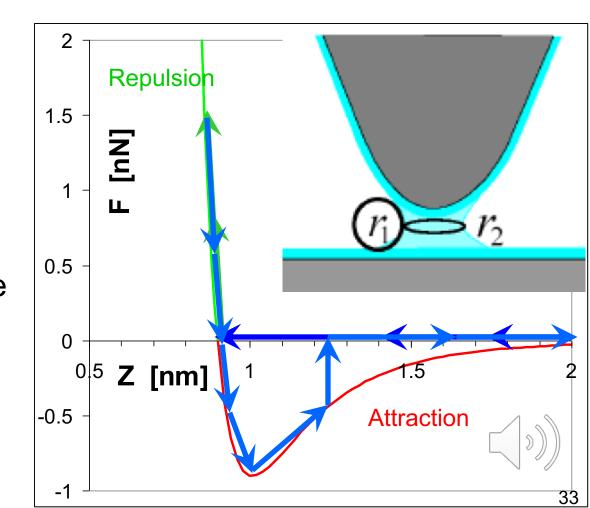






## The other side of the force ...

- 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 the sample surface.
  - 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.



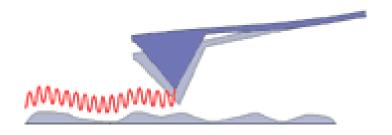
# How to avoid contact sticking in AFM?

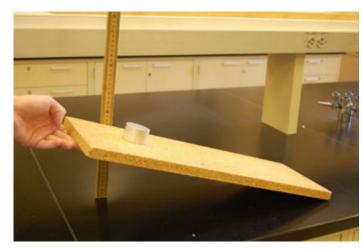
• Let's get inspired by the study of friction:

We know that dynamic friction is smaller than static friction

Let's try to make the AFM tip dynamic!

In AFM: vibrate the tip to avoid sticking

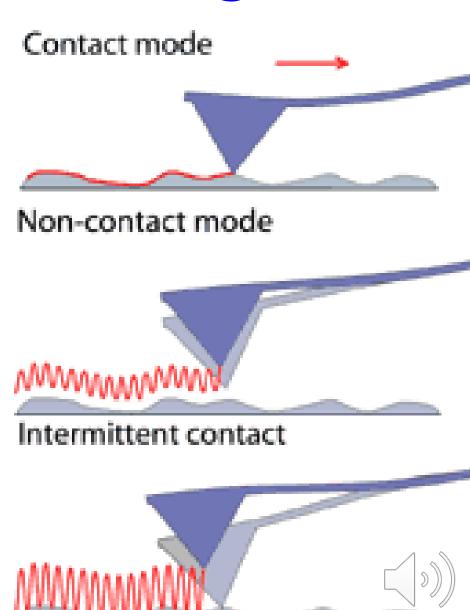






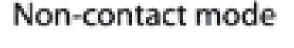
# Non-contact AFM: no sticking!

- "Classical" contact-mode AFM is prone to tip sticking problems.
- To avoid the problems of tip sticking, non-contact AFM modes are 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.



## Operation of non-contact AFM

- The cantilever is vibrated by a special piezo at its resonance frequency:  $f_0 = \sqrt{K/\mu}$ . where K = force constant,  $\mu$  = cantilever mass, both per unit length.
- PZT
- The laser beam reflected onto the position-sensitive detector vibrates 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.





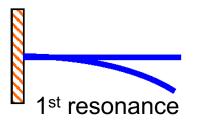


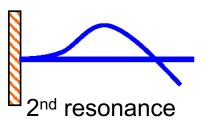
### Resonances of the cantilever

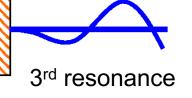
- The cantilever can have several resonances:
- The resonance frequency of the i<sup>th</sup> mode of the cantilever is:  $f_i = \frac{\lambda_i}{L^2} \sqrt{\frac{EJ}{\rho S}}$ , where E = Young's modulus,  $\rho$  = material

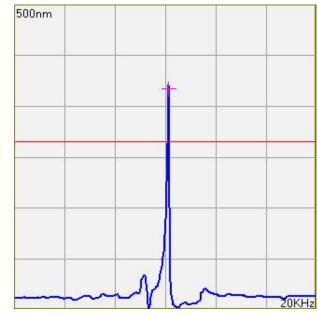
density, L = cantilever length, S = cantilever cross-section, I = cantilever moment of inertia,  $\lambda_i$  = numerical coefficient dependent on the mode number:  $\lambda_1 \sim 0.5$ ,  $\lambda_2 \sim 3$ , etc.)

• Usually the first (highest-Q) resonance is used

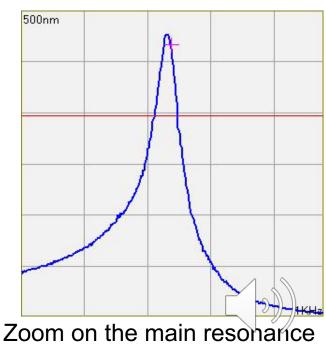








Scan over a large frequency range

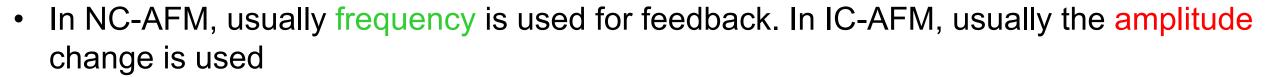


First three cantilever resonances

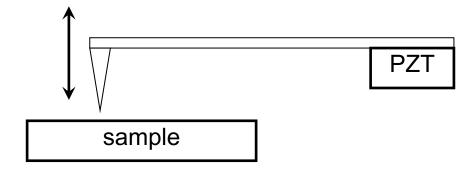
\_

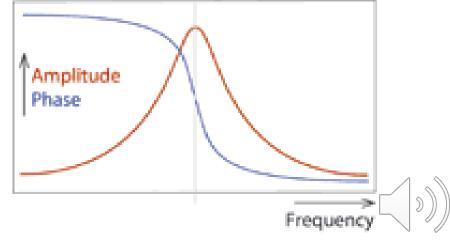
### Feedback in non-contact AFM

- What changes by tip-sample forces?
- Changes in the vibrations'
  - Frequency
  - Phase
  - Amplitude



Sometimes lock-in (phase) detection is used, e.g. to plot height + phase images





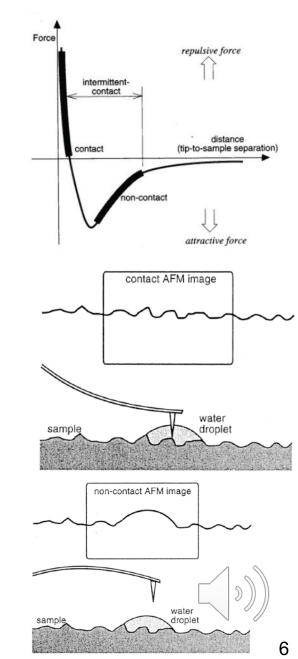
#### Force, resolution in non-contact AFM

#### NC and IC AFM use different force regimes:

- Contact AFM works with repulsive forces, at close distance (<0.5 nm)</li>
- 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). However the force is applied only for a short time during the cycle

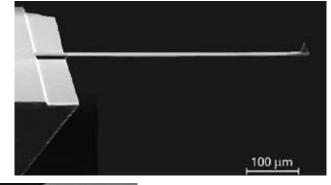
#### Results:

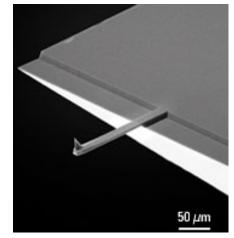
- Contact AFM has the highest resolution (atomic, like STM), but uses high forces
   (1 nN 1 μN) which can scratch the surface
- NC-AFM uses less force (1 pN 1 nN), good for delicate surfaces (polymers), but has lower resolution
- IC-AFM uses higher force (0.1 100 nN), but for a short time in the cycle. good for all surfaces, has higher resolution



## Types of AFM cantilevers

- For contact mode, usually a long cantilever is used to increase sensitivity. Remember: δ = FL<sup>3</sup>/3EI
  - 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:  $f = \frac{\lambda}{L^2} \sqrt{\frac{EJ}{\rho S}}$ 
  - Typical values: K=10 N/m, L = 100  $\mu$ m
  - $f_0 = 200-400 \text{ kHz}$
- Sometimes, two-beam cantilevers are used,
   e.g. to measure lateral forces.

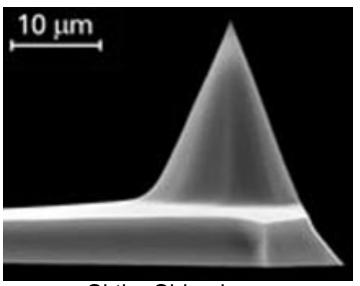




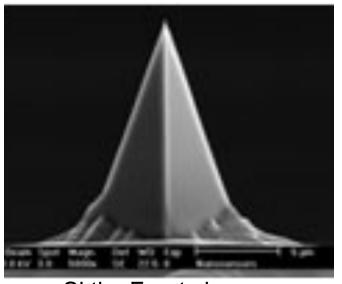


## Standard AFM tips

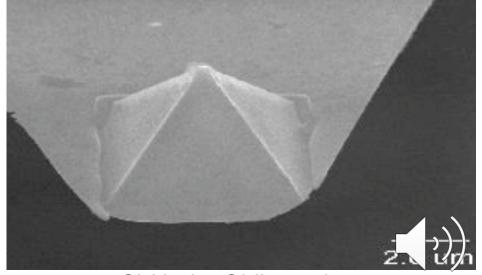
- Most AFM tips are made from Si, by photolithography and directional etching.
- The tip is pyramidal in shape, with height ~20  $\mu$ m and sidewall angles of ~20° to the normal
- The standard tip radius is on the order of 5-10 nm
- In some cases Si<sub>3</sub>N<sub>4</sub> is used: has longer life but larger tip radius



Si tip: Side view



Si tip: Front view



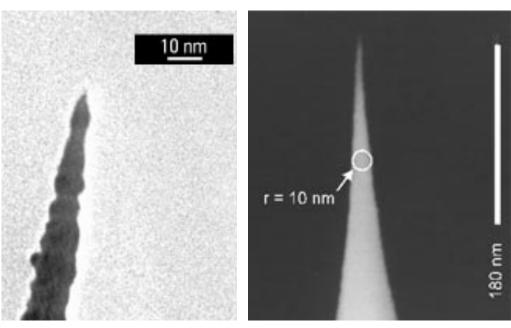
Si<sub>3</sub>N<sub>4</sub> tip: Oblique view

## **Special AFM tips**

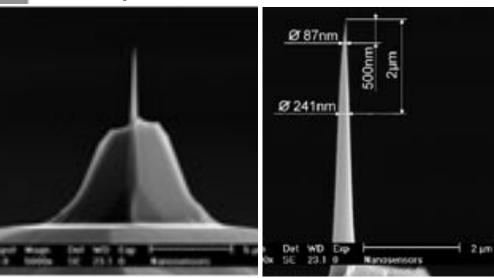
For metrology: "elephant foot" to measure sidewalls:

Inclined tips to probe edges:





 High aspect-ratio tips to probe trenches and holes



Diamond tips for hardness testing and long life (low resolution!)

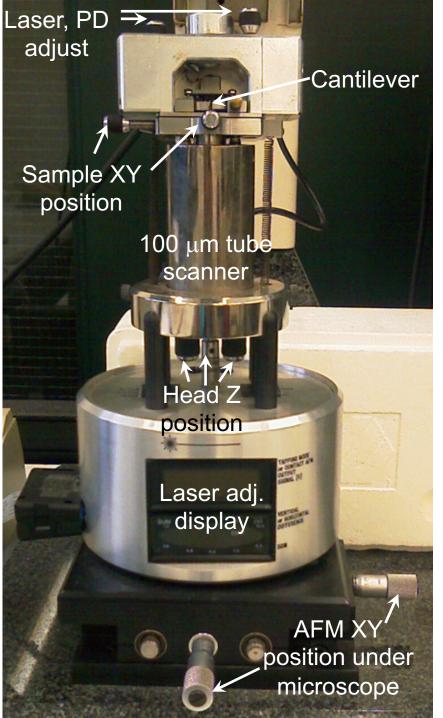
• Different coating on tips: conducting, magnetic etc.



## **AFM** construction

Small-sample AFM, tube scanner:

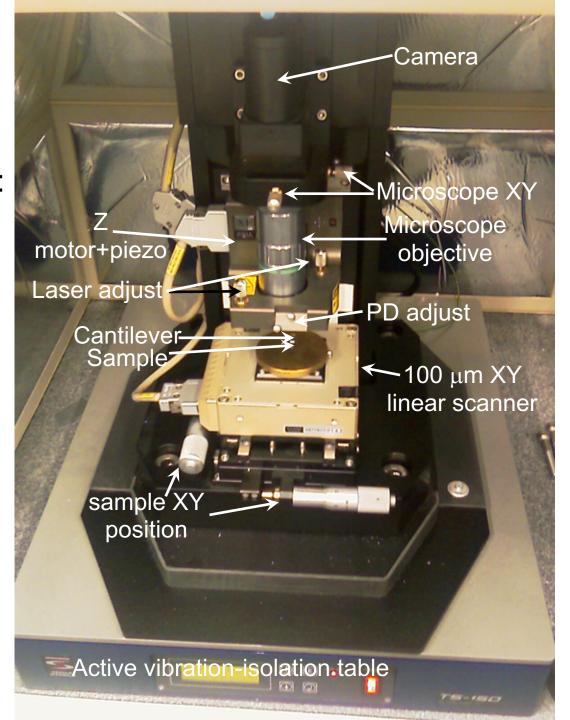






#### **AFM** construction

Big-sample AFM, XY/Z linear scanners:





#### **Outline:**

- Introduction: What is SPM, history
- STM
- AFM
- Image treatment
- Advanced SPM techniques
- Applications in semiconductor research and industry



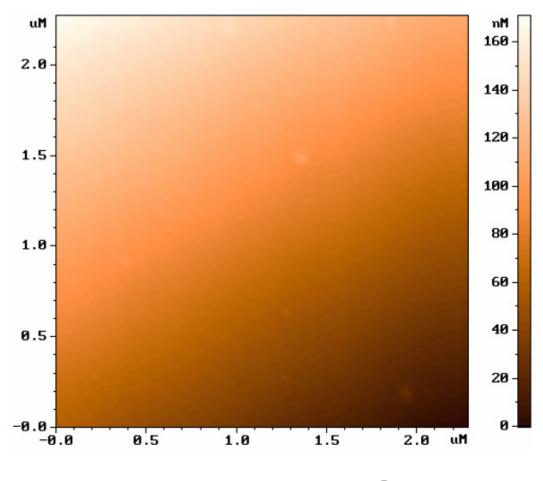
### Image processing

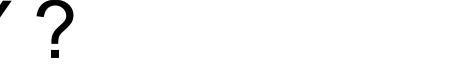
- SPM images are rarely perfect "as taken"
- "Basic" image processing:
  - Removing sample tilt, scanner non-linearity, tip jumps, noise
- "Advanced" image processing:
  - Filtering, deconvolution, finding & characterizing objects
- Measurements:
  - Size, distance of features
  - Line profiles & their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



# **Basic Image Treatment**

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



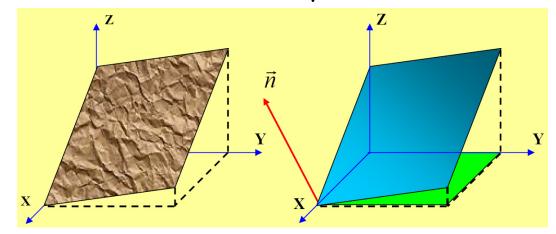




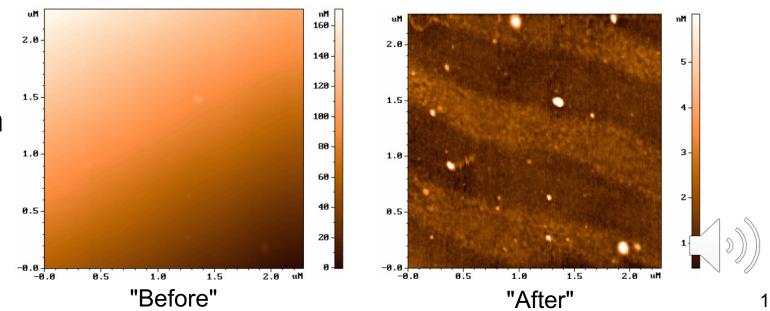
# **Basic Image Treatment: plane-fit**

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  $\mu$ m scan!

The plane-fit correction: An average plane is calculated by LSQ fit of the image to: ax+by+cZ=0

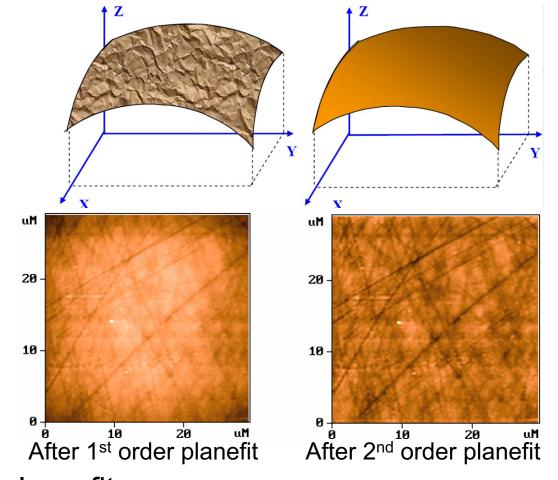


This plane is subtracted from the image, to "planarize" it:



### 2<sup>nd</sup> 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.

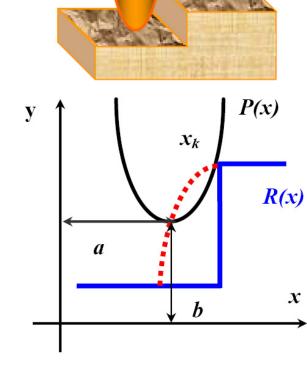


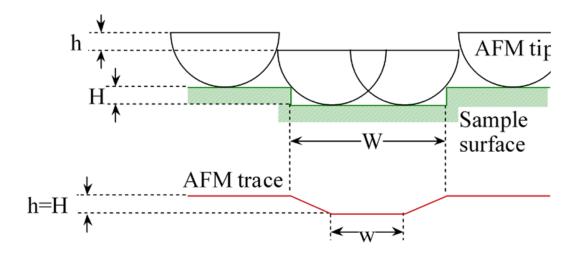
The 2<sup>nd</sup>-order (and sometimes even higher order) plane-fit correction subtracts from the image a LSQ-fitted curved plane Caution: in some cases, high-order plane-fit correction can remove real image features (e.g. sample undulations)

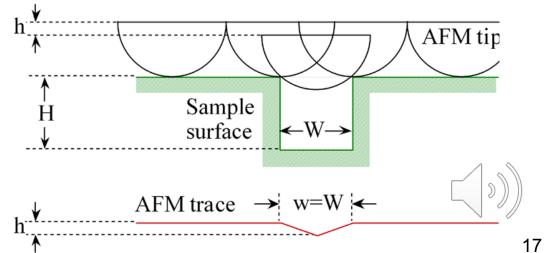


# Effect of the tip size on the Image

- 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
- A blunt tip will not penetrate a deep trench a triangular image will result, shallower than the real depth. The width is correct (at the top)!

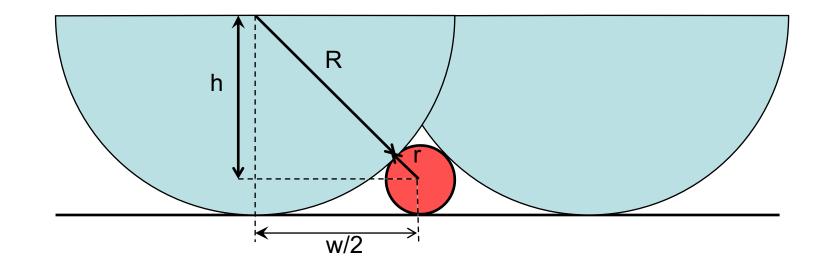






## Effect of the tip size on the Image

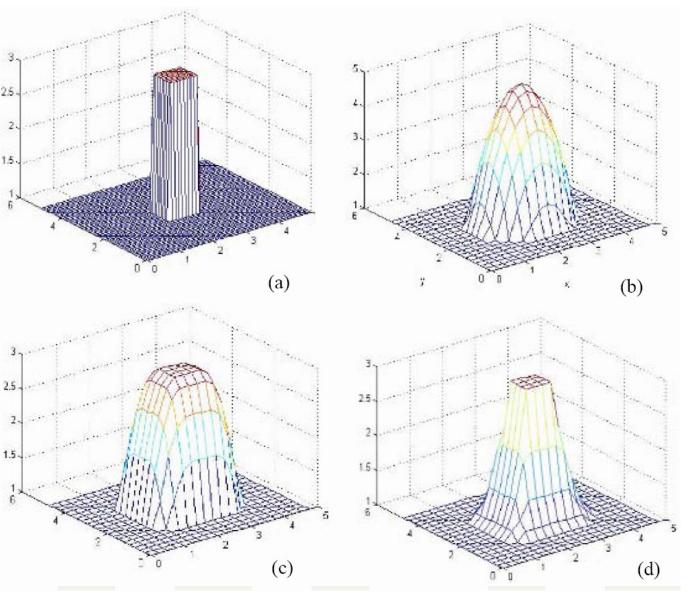
- Example: Semi-circular tip, radius R, measures a molecule (or particle), radius r < R</li>
- The apparent width is measures between the points where the tip touches the molecule from each side
- Geometrical calculation shows:  $w = 2\sqrt{(R+r)^2 (R-r)^2} = 4\sqrt{Rr}$
- Typical values: r = 1 nm, R=10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





# Example of tip deonvolution

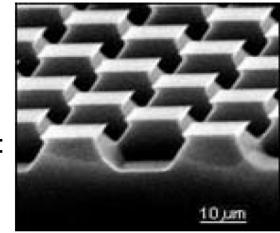
- A square object (a) is scanned with a similar-size rounded tip (b).
- The resulting image (c) combines features of both tip and sample
- The object can be partially restored by deconvolution (d)



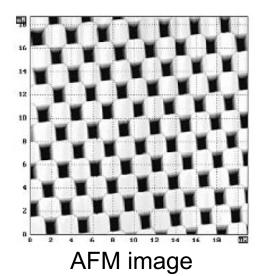


#### **AFM** calibration

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

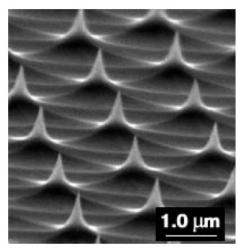


SEM picture of sample

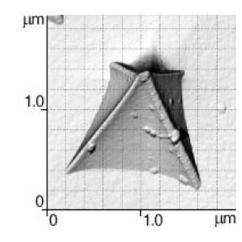


– Sharp "tips" to calibrate tip shape:

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



SEM picture of sample



reconstructed tip shape



#### **Outline:**

- Introduction: What is SPM, history
- STM
- AFM
- Image treatment
- Advanced SPM techniques
- Applications in semiconductor research and industry



#### **Advanced modes - SPM**

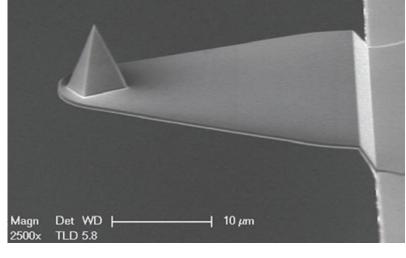
- The AFM can measure small forces (< 1 nN), so any phenomenon that can be translated to force can thus be measured by the AFM
- The tip is very close to the sample, so it can be used to interact with the sample while scanning
- The general name : Scanning probe microscopy SPM

- 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



#### **Faster AFM**

- Usually, AFM is slow! >5 mim/image
- Speed limits of AFM:
  - Cantilever resonance
  - Electronic feedback speed
  - Z piezo speed (resonance)
  - XY scanner speed (resonance)
- To get higher speed, we need to improve all parameters:
  - Short, wide cantilever: resonance 1.3 MHz in air
  - Faster scanner: 2.5 mm/s
  - High speed electronics
- Results: scan speed of 6-1200 Hz!



High resonance-frequency cantilever

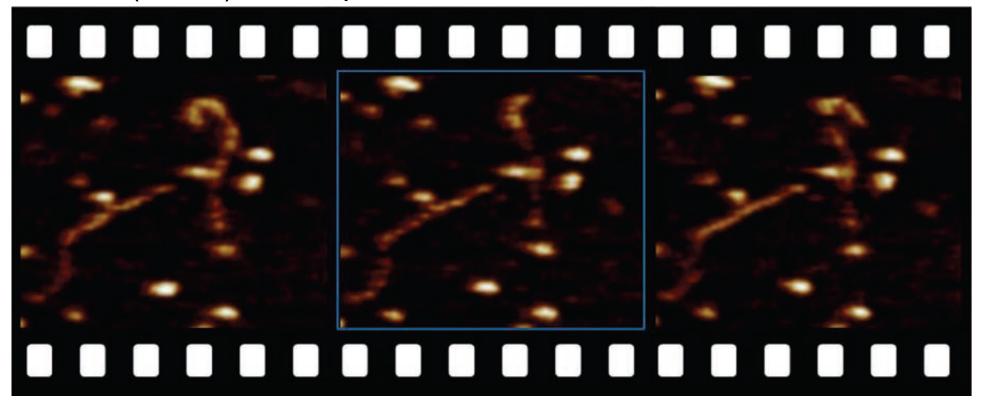


High-speed scanner



# Faster AFM: example of use

- Sample: DNA on Mica in fluid
- Scan speed: 1 sec / image
- Result: "film" showing DNA movement
- Real-time video (10 Hz) is now possible



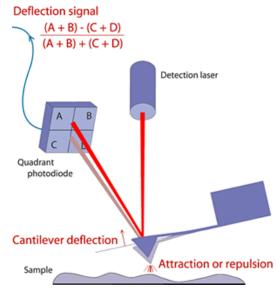


#### **Lateral Force AFM**

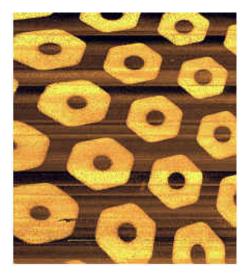
 Lateral movements of the cantilever are measured by the photodetector:

$$\frac{(A+C)-(B+D)}{(A+C)+(B+D)}$$

Useful to measure friction and material contrast on planar structures.

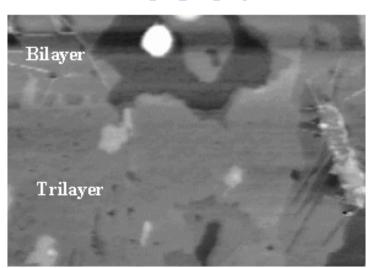




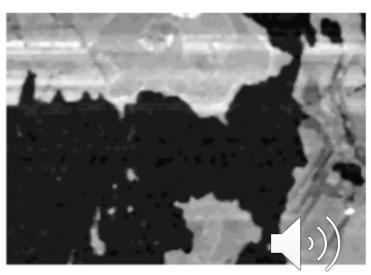


Patterned alkanethiol on Au (topography and LFM)

#### Topography



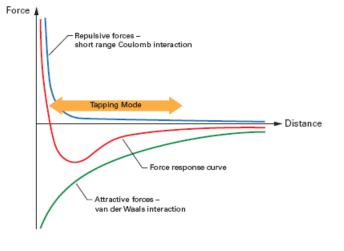
#### Friction force image

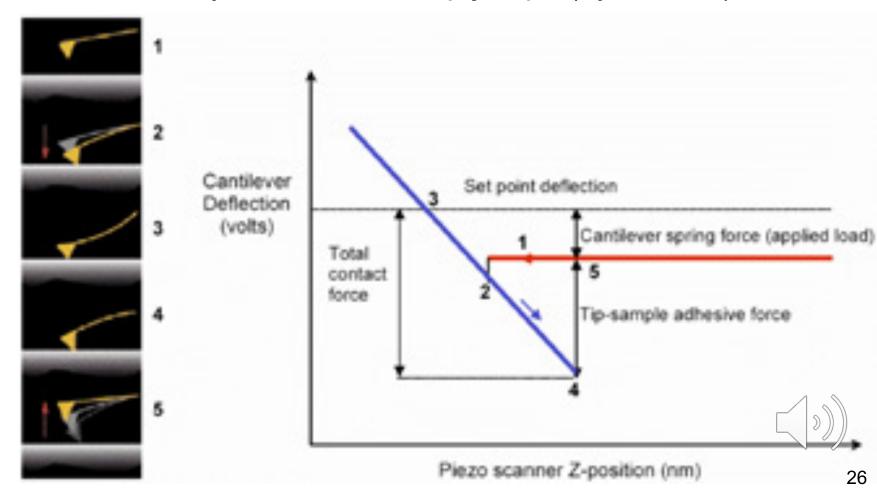


Si tip distinguishes hydrophilic and hydrophobic layers

#### Force curves: contact mode

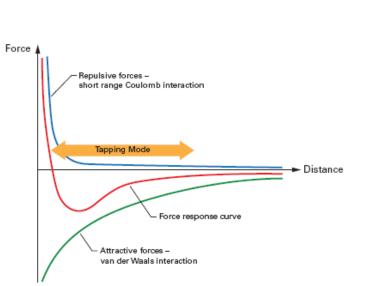
- In contact mode: Sweeping the tip up-down can show the force distance curve, useful for analyzing mechanical properties (polymers, bio samples...)
- The van-der-Waals curve can not be fully traced due to tip jumps (hysteresis)

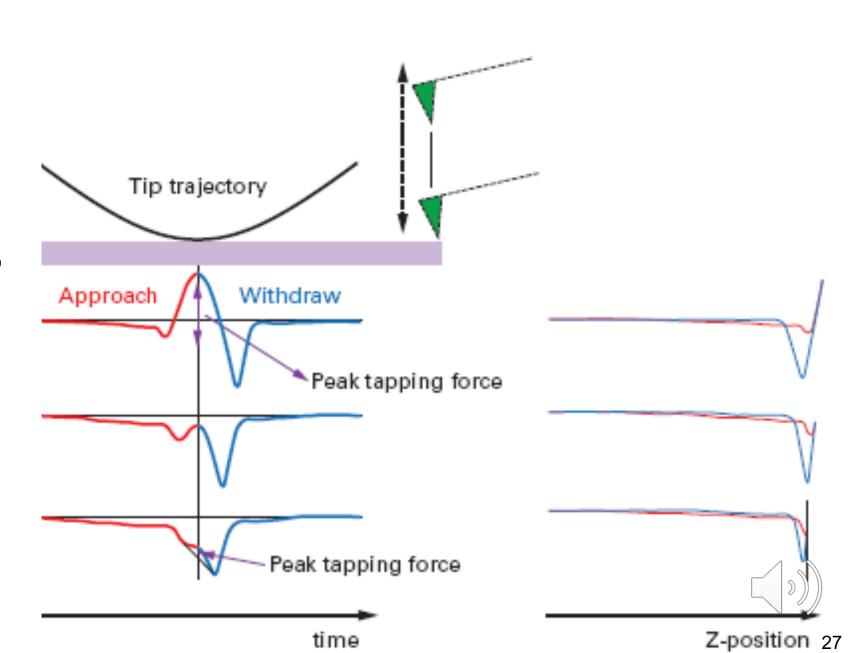




#### Force curves: intermittent contact

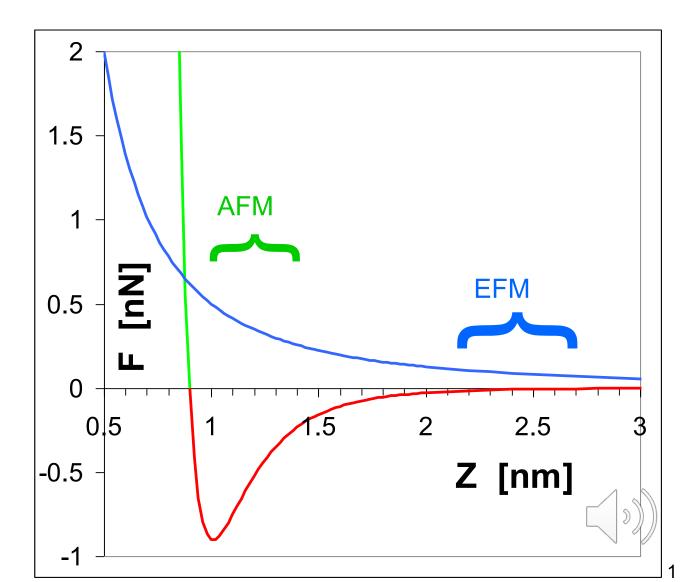
- New fast AFM can
  measure force-distance
  curve during each
  oscillation cycle of the tip
- Good control of peak force, reconstruction of curve





# Electric Field – EFM: know your force ...

- When a voltage is applied between sample and tip, electric (Coulomb) forces are present
- Electric forces are long-range (1/r²):
   can be separated from the short-range
   Van der Waals force (1/r²) by
   scanning at a higher Z position



#### Electric Field – EFM: DC

- Drawback of scanning at great distance: lower resolution
- A better solution :Taking differential image at 2 distances ("lift mode")

1.5

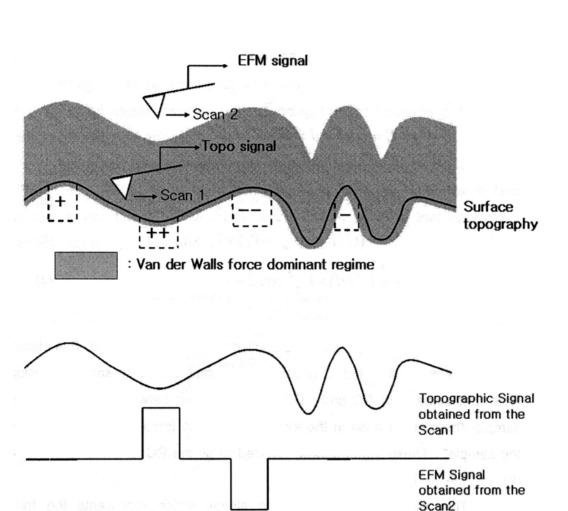
0.5

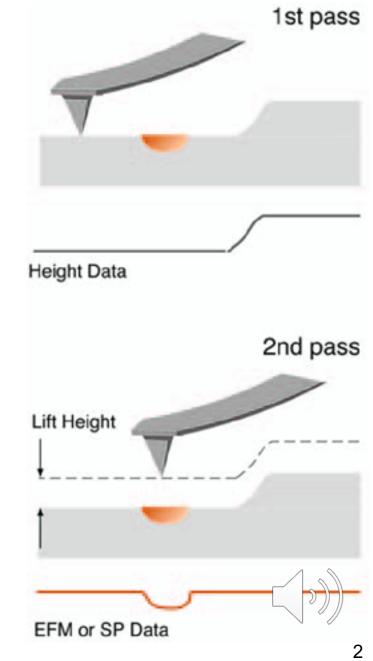
-0.5

E N

2.5

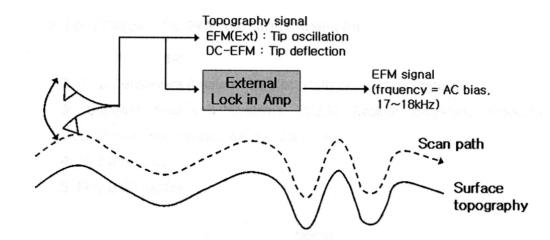
**Z** [nm]





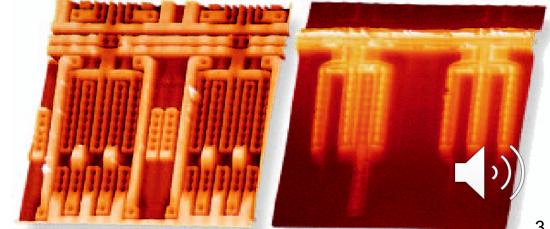
# EFM: AC for high sensitivity

- In a parallel-plate geometry, the electric force is:  $F_e = CV^2/d$ ; C = tip-sample capacitance, d = tipsample distance
- The tip-sample voltage is :  $V = V_{dc} + V_{ac} \sin(\omega t)$



• The force is: 
$$F_e = \frac{C}{d} \left[ V_{dc}^2 + \frac{1}{2} V_{ac}^2 + 2 V_{dc} V_{ac} \sin(\omega t) - \frac{1}{2} V_{ac}^2 \cos(2\omega t) \right]$$

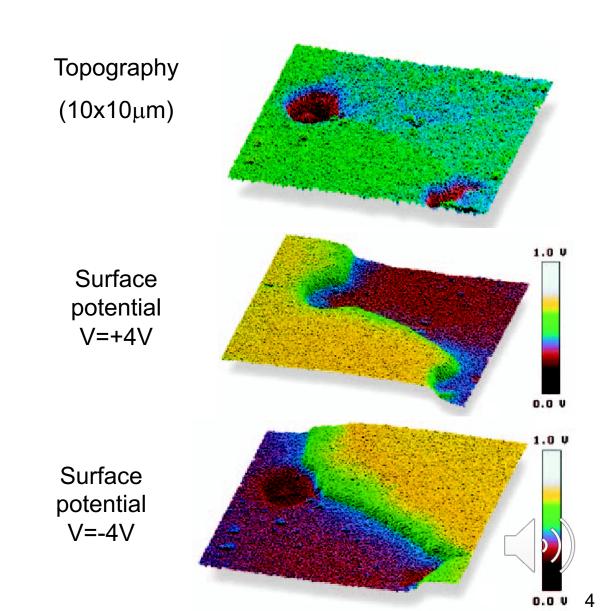
We can use the AC component at ω by lock-in detection



Si transistors: Topography and EFM

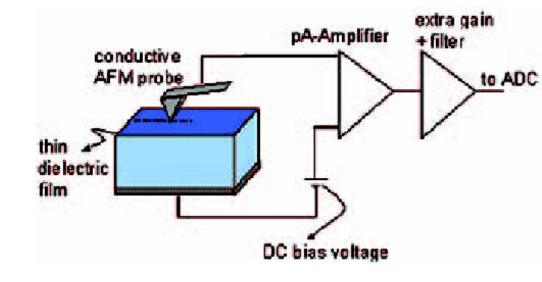
# Voltage – Kelvin Force (KF)-AFM

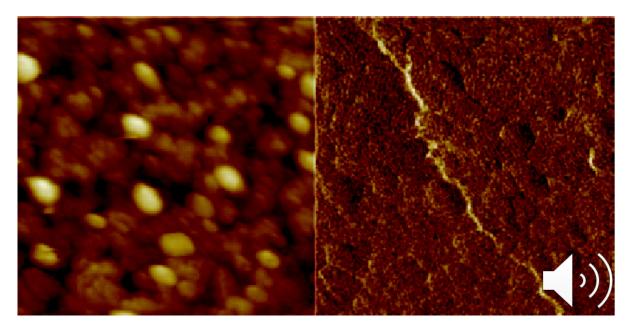
- AC-EFM can be used to measure the voltage at the surface of the sample
- A feedback loop is used to keep the EFM signal constant by changing the sample bias
- The resulting signal is the surface potential at the sample



# Current – I-AFM, CAFM

- Contact AFM with a metal-coated tip can be used to pass current between tip and sample
- For semiconductors and insulators a sensitive current preamplifier should be used
- Low currents (<1 pA) can be measured</li>

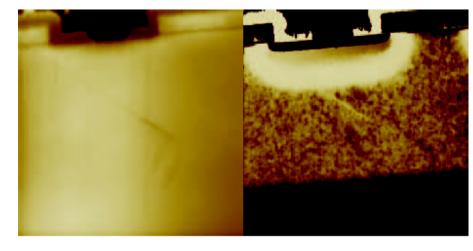




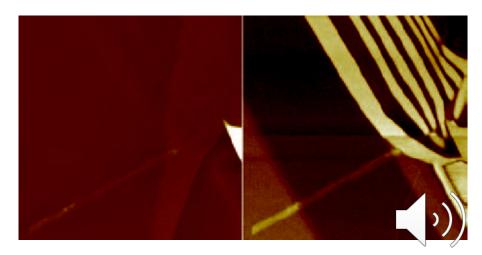
BaTiO<sub>3</sub> film, 2x2 μm, 2 pA (topography and current)

# Spreading-resistance – SSRM

- I-AFM can be used to measure resistivity:
   supposing a circular tip-sample contact of radius
   r, the local resistivity is given by:
  - $\rho = 4rV/I$
- Local resistivity is linked to doping concentration, leakage
- In air, high forces (μN) are needed diamond tips



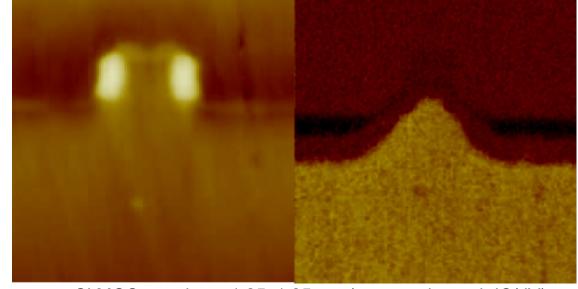
Si DMOS transistor, 12x12 μm (topography and resistance)



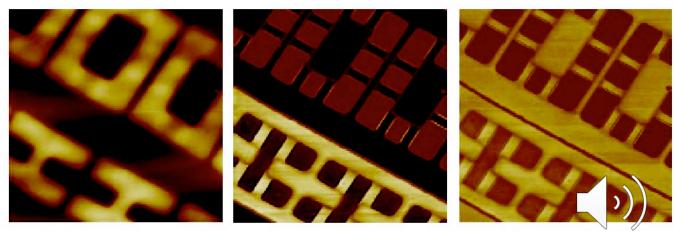
InP transistor, 7x7 μm (topography and resistance)

# Capacitance – SCM

- An AC voltage applied between tip and sample leads to the flow of AC current
- Using high frequency (~1 GHz), small capacitance (<1 aF) can be measured</li>
- SCM can be useful for measuring doping in semiconductors



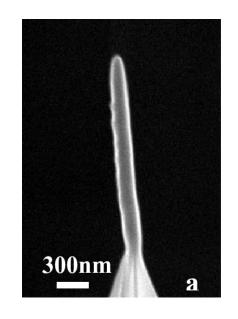
Si MOS transistor, 1.25x1.25  $\mu m$  (topography and dC/dV)

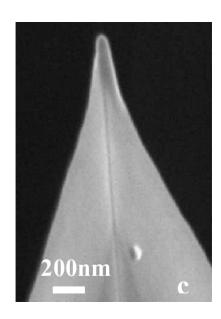


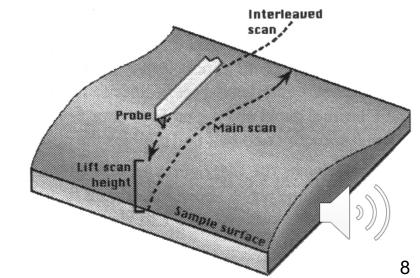
Si DRAM cell (topography and dC/dV amplitude and phase)

# Magnetic Force - MFM

- AFM tip made from or coated with magnetic materials (Co, Ni)
- Magnetic forces are long-range: can be separated from Van der Waals force (1/r<sup>6</sup>) by:
  - Scanning at greater distance only magnetic forces remain
  - Taking differential image at 2 distances ("lift mode")
  - Looking at phase in non-contact mode



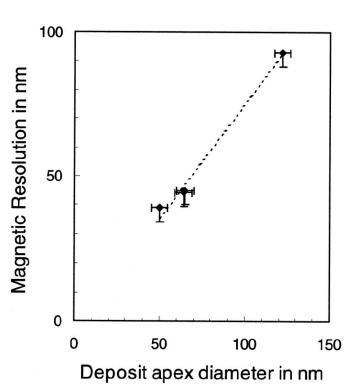


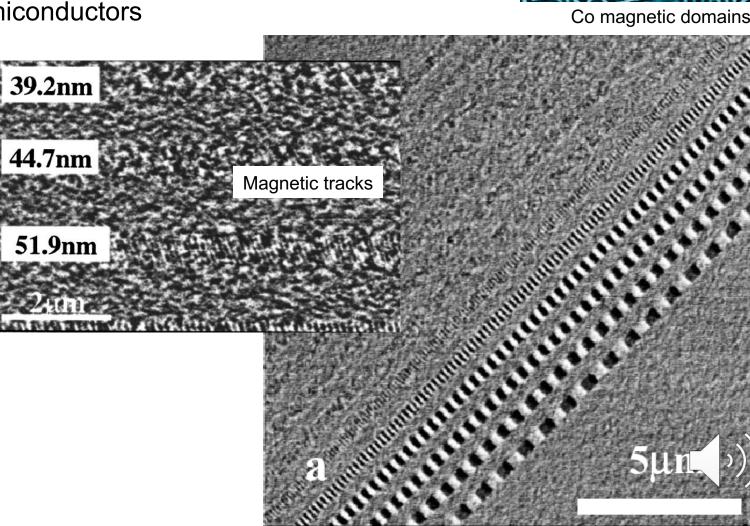


# **Magnetic Force - MFM**

#### Uses of MFM:

- Characterization of magnetic materials and nanostructures, e.g. magnetic semiconductors
- Hard-disk testing
- Future magnetic storage





Co magnetic domains

### Hall Effect

- A miniature Hall cross (0.1  $\mu$ m) is evaporated on the AFM tip
- Local magnetic fields can be measured with sub-μm resolution
- Resolution is lower than MFM, but sensitivity is higher





# Temperature – SThM

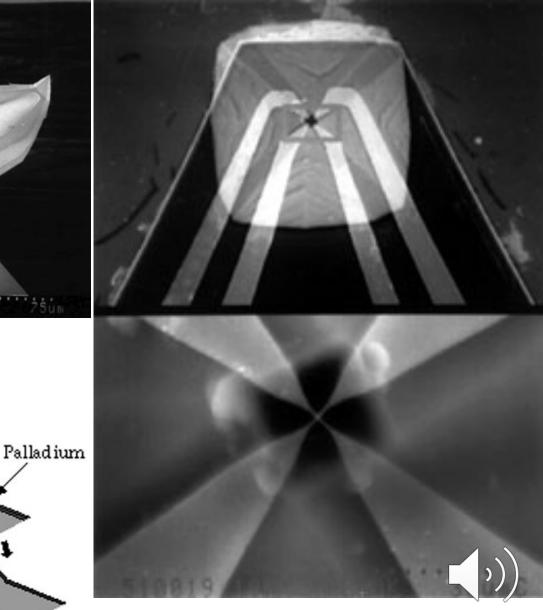
SiN Cantilever

Gold

Thermocouple

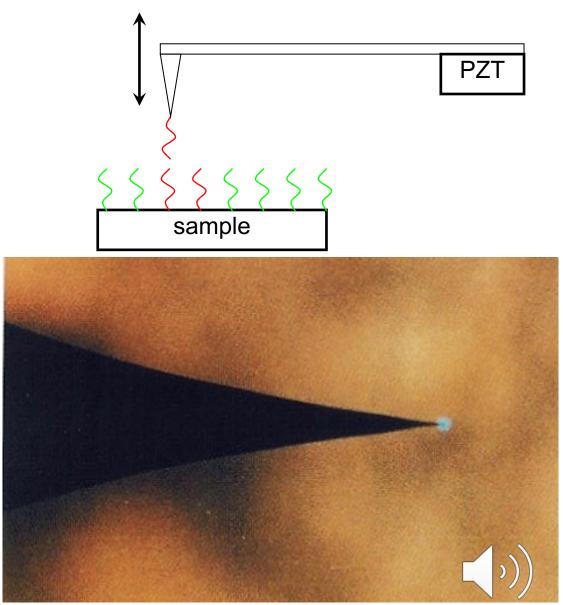
- To sense temperature at the AFM tip, two methods are used:
  - A thermocouple is evaporated on the cantilever, with the junction at the tip
  - The (temperature-sensitive) resistance of the Si cantilever is monitored
- Local defects in circuits can thus be found





# Chemically sensitive AFM

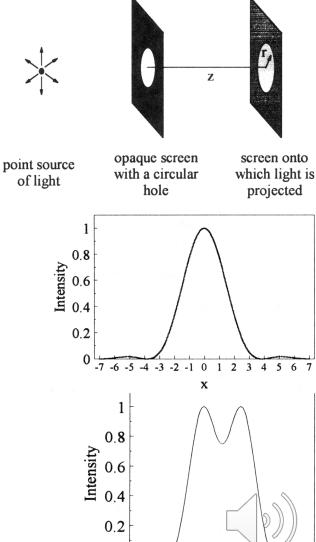
- The high force sensitivity of AFM can be used to measure chemical interactions
- The AFM tip is coated by a chemically active molecule
- This tip is then sensitive to molecules which interact with the tip's coating
- When the right molecule is sensed, chemical interaction causes a change in the force



Phosphorescence at the chemically modified tip edge 12

# Scanning Near-field Optical Microscopy – SNOM

- Optical microscopy is normally limited in resolution by the diffraction of light to about 0.5  $\mu$ m :  $I(r) = 4I_0J_1^2(x)/x^2$   $x = \pi dr/\lambda z$ 
  - d=hole diameter in mask,
  - z=distance mask-wafer,
  - r=distance from center
- Close to a sub-wavelength aperture, the optical near field has the extent of the aperture, but it diverges quickly
- The AFM scanning technique can be used to scan a small aperture close to a surface, thus providing sub-wavelength resolution
- Several SNOM techniques exist, mostly using metal-coated optical fibers with a small aperture (< 100 nm) at their end</li>

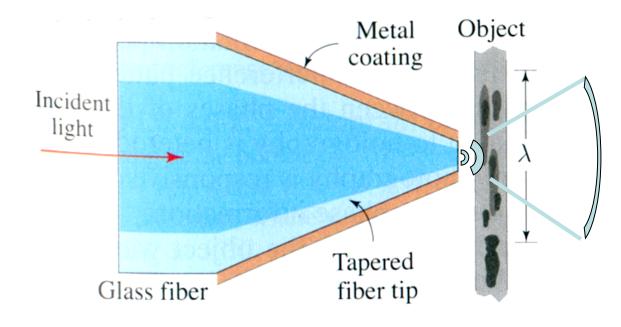


13

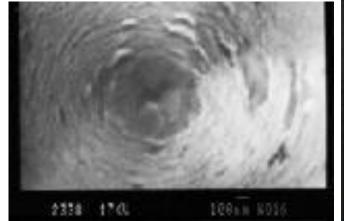
X

# Principle of operation

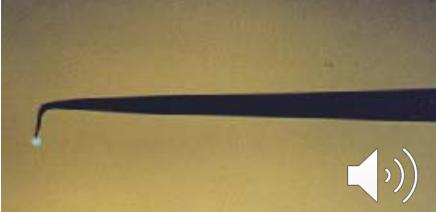
- A conical-end (drawn or etched) optical fiber is coated by a reflecting metal (AI)
- The conical end of the tip is exposed to let the light come out through a small hole



• The tip is scanned close (< 50 nm) to the sample, so that near-field light goes through and can be detected, giving high resolution







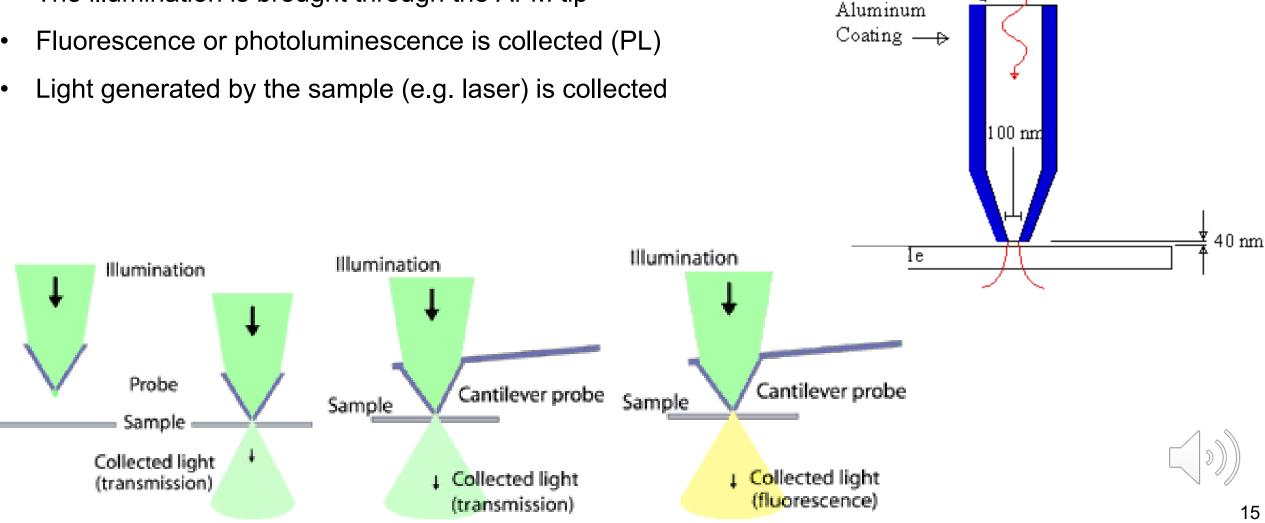
# Types of SNOM (1)

Near-field Optical Excitation

Fiber optic core

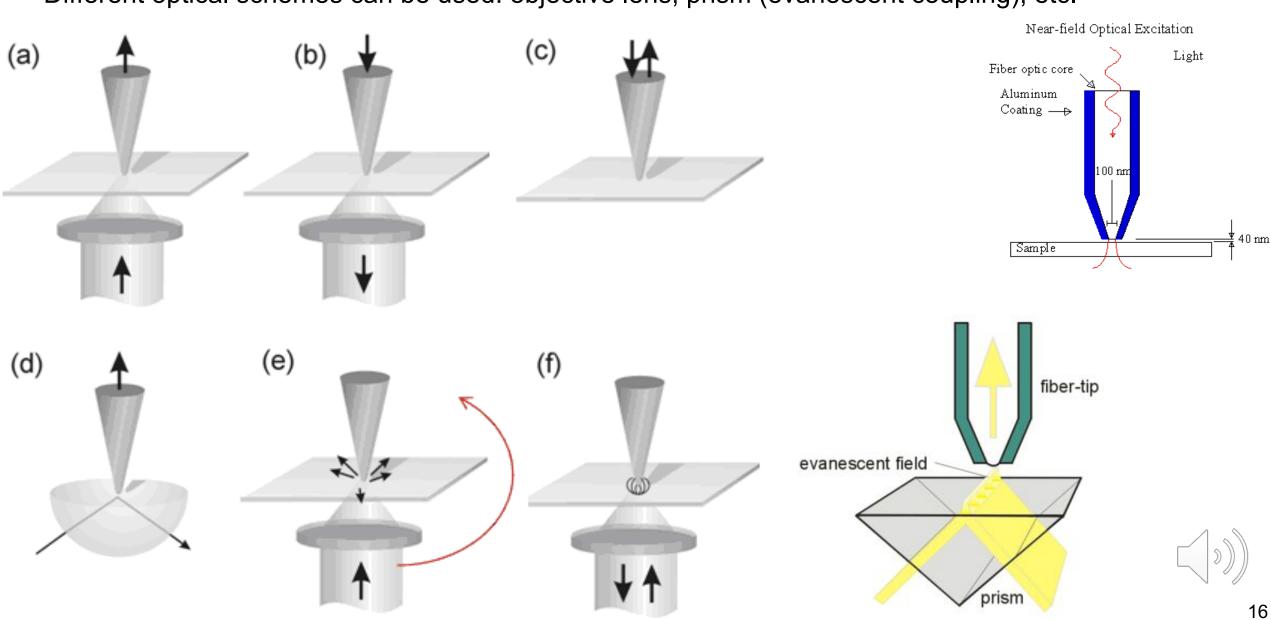
Light

- The illumination is brought to the tip of a fiber, transmitted light is collected through the sample (Transmission)
- The illumination is brought through the AFM tip



# Types of SNOM (2)

• Different optical schemes can be used: objective lens, prism (evanescent coupling), etc.



#### Interferenzfilter (488 nm) Structure of the SNOM Faser Ar'-Laser Kamerabeleuchtung Dither-Piezo Quarzstimmgabel Polarisationsfilter Probe xyz-Piezo xyz-Piezo Objektiv Interferenzfilter (FITC) Drehbarer CCD - Kamera Spiegel

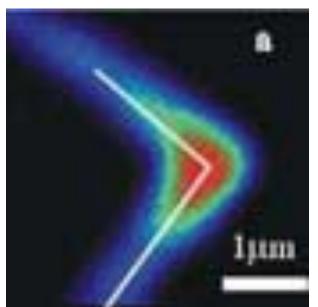
Polarisationsfilter

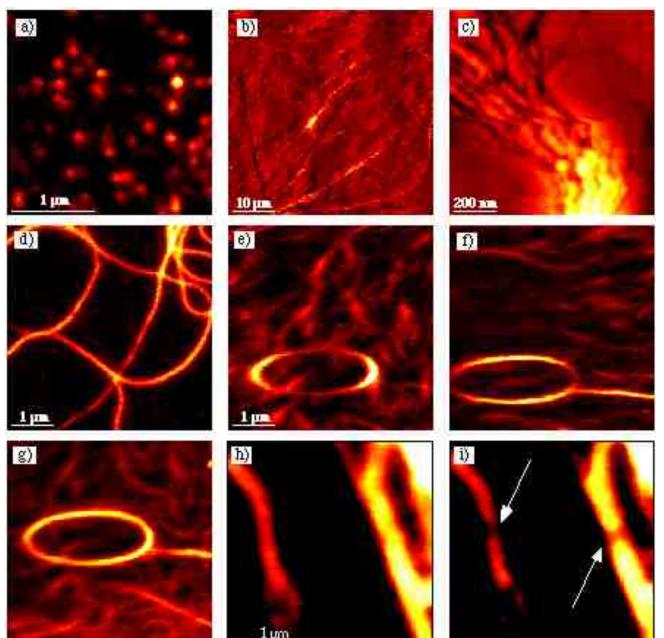
APD

# Some SNOM images

Fluorescent molecules:

 V-shaped laser (cross-section)

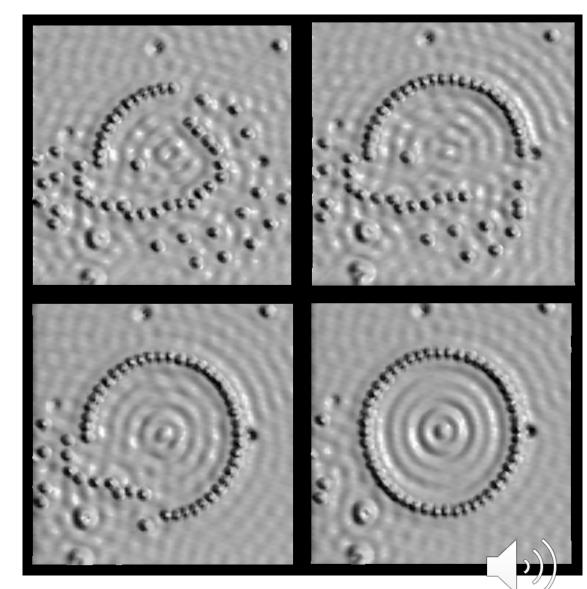






# STM and AFM lithography

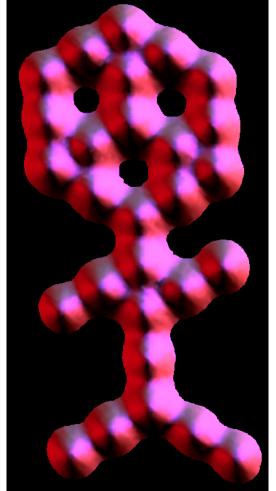
- The small forces between tip and sample can be used to move atoms and molecules
- At low temperatures, Ar or metal atoms can be adsorbed on an atomically flat metallic surface (Ag, Cu) and manipulated by STM
- Artificial structures can be constructed ... vary slowly

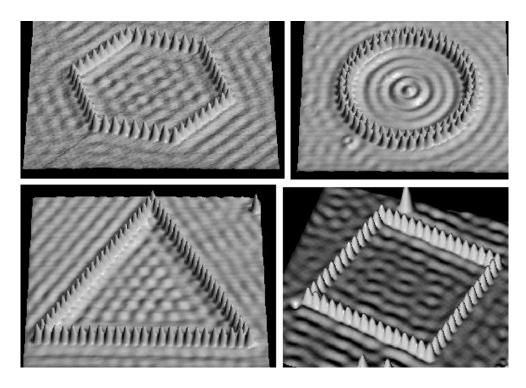


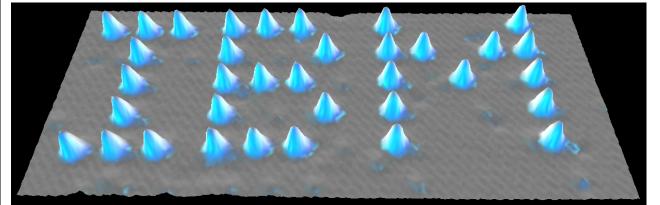
Note the quantum-mechanical interference!

## **Examples of STM lithography**

From the labs of you-know-who...



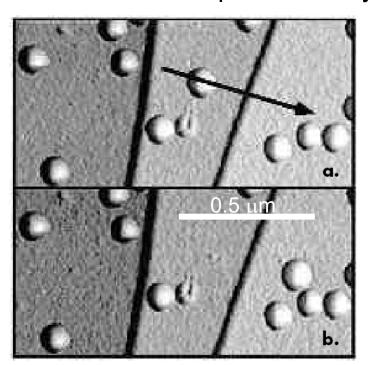


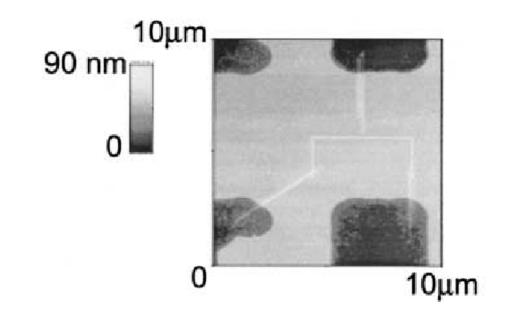


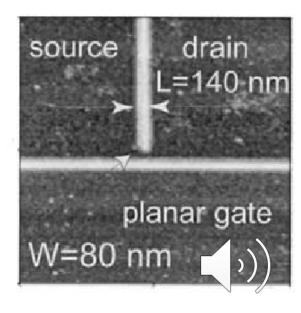


#### **AFM** lithography

- 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







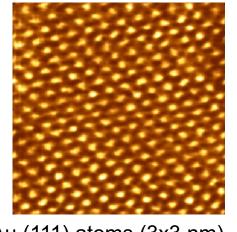
#### **Outline:**

- Introduction: What is SPM, history
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- AFM
- Image treatment
- Advanced SPM techniques
- Applications in semiconductor research and industry

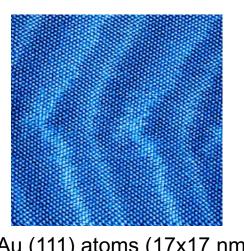


### **STM Applications**

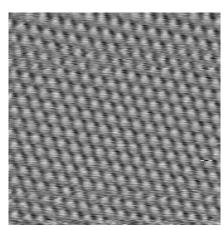
- **STM** is most useful in vacuum:
  - Naturally forming oxide layer distorts surface states
  - Water layer interferes with tunneling current
- Many MBE growth system have a UHV STM (sometimes low-T STM) coupled to the growth chamber
- STM can show growth morphology, electrical properties, doping on the atomic scale



Au (111) atoms (3x3 nm)



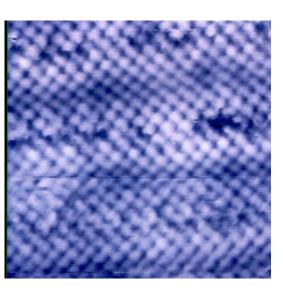
Au (111) atoms (17x17 nm)



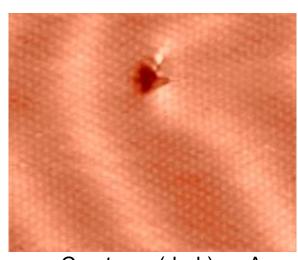
Ag (111) atoms



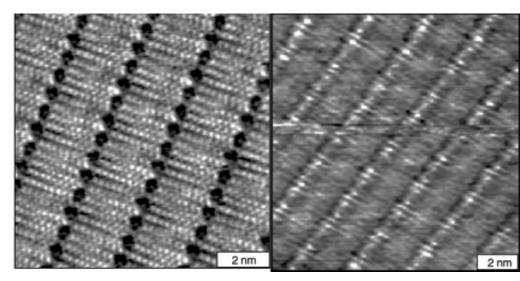
## Some STM Images (1)



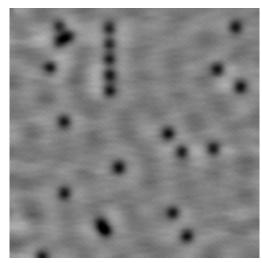
BSCO HTSC superconductor at 4K



Co atoms (dark) on Au (111) surface



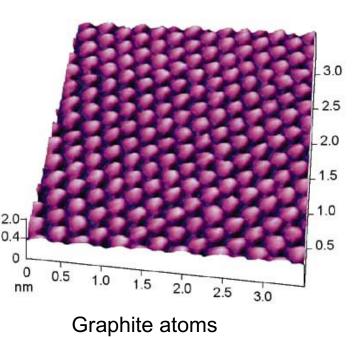
STM Images of molecules. Right – tip is modified to interact with specific part of molecule

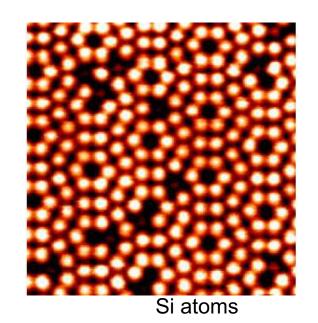


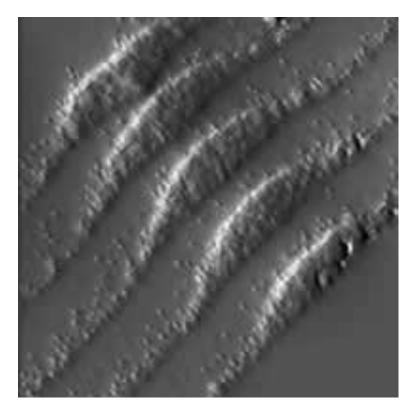
Co atoms (dark) on Ag surface



# Some STM Images (2)



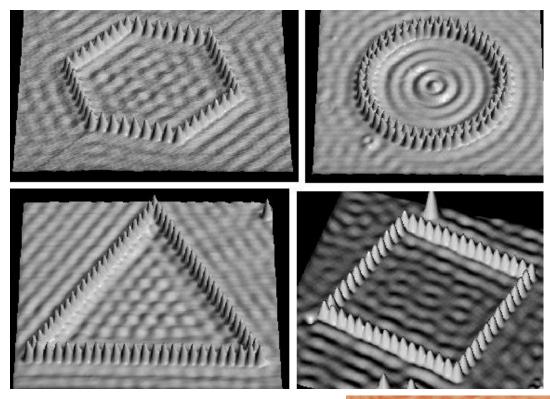


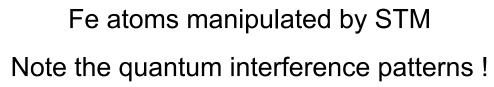


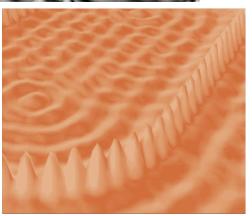
Stacked InGaAs quantum dots

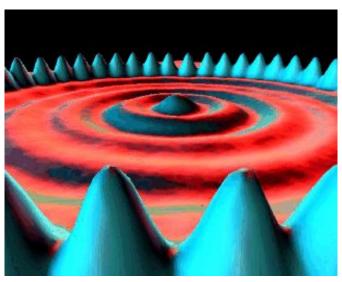


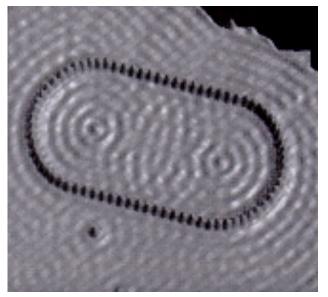
### Some STM Images (3)











The rat race ...



### Detailed example of X-STM atomic imaging

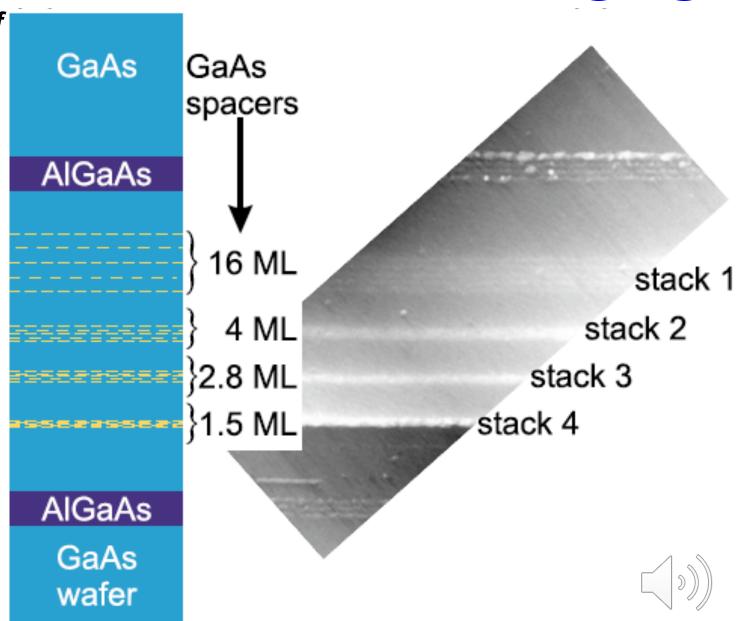
Atomic structure and optical properties of InAs submonolayer depositions in GaAs,

A. Lenz, et al.

J. Vac. Sci. Tech. B 29, 04D104 (2011)

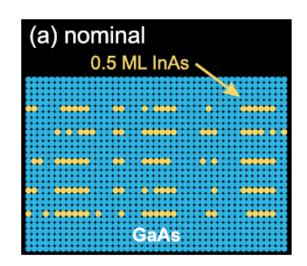
#### Sample structure:

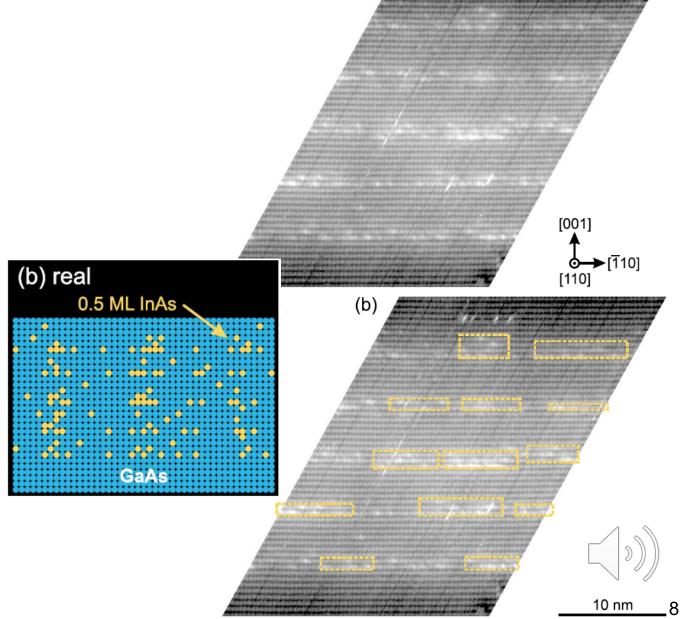
- AlGaAs barriers at extremities ("garbage collection")
- Four "stacks", each of five 0.5 ML InAs layers in GaAs
- Different spacing between InAs layers in stack: 1.5-16 ML.
- Sample cleaved in UHV, STM of crosssection (XSTM)
- InAs brighter than GaAs



XSTM images of one stack

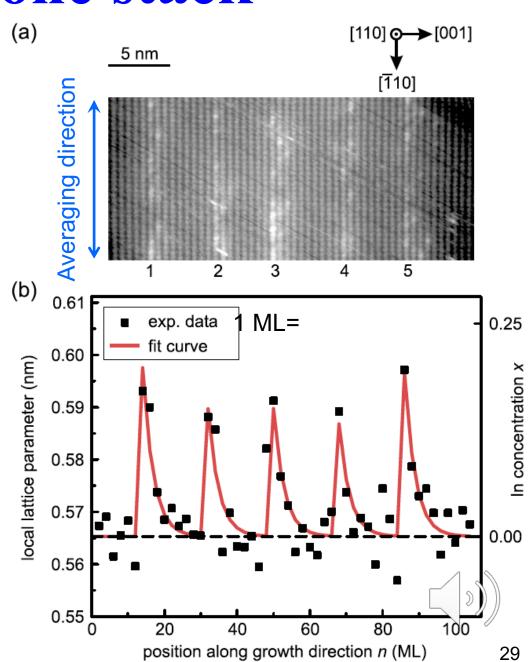
- We can distinguish single atoms
- InAs atoms are brighter than GaAs:
  - Higher topography due to strain release
  - Electron state density higher
- The InAs atoms tend to "cluster" (fig.b) in height





#### XSTM images of one stack

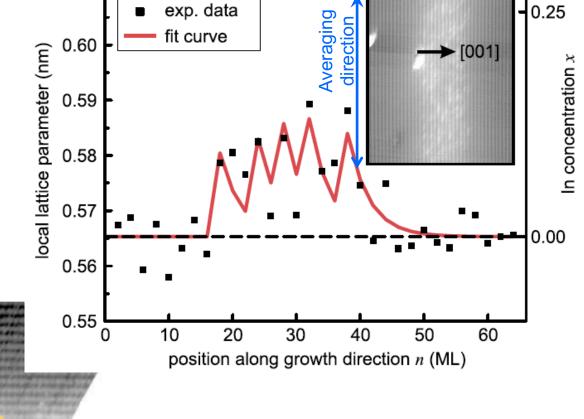
- We can quantify the In contents by measuring local lattice parameter between rows (averaged along the rows)
- Accuracy: ~1 pm!

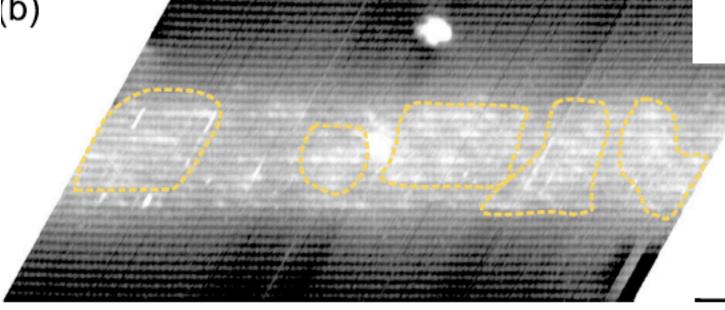


### XSTM images of one stack

0.61

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







1 ML=

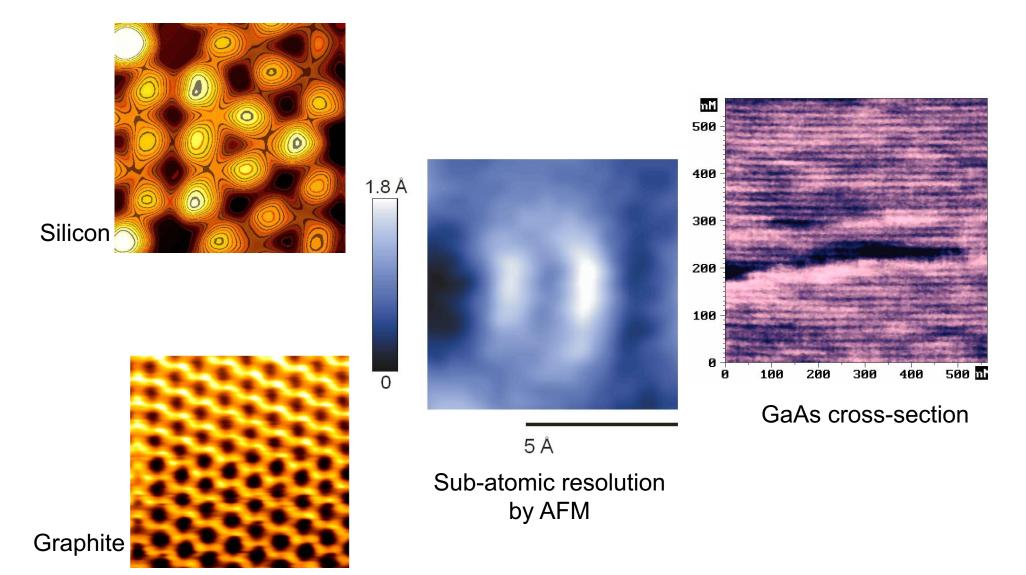
### **AFM Applications**

- AFM is more used than STM:
  - Easier to use and to obtain an image
  - Can work at ambient air
- 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 :

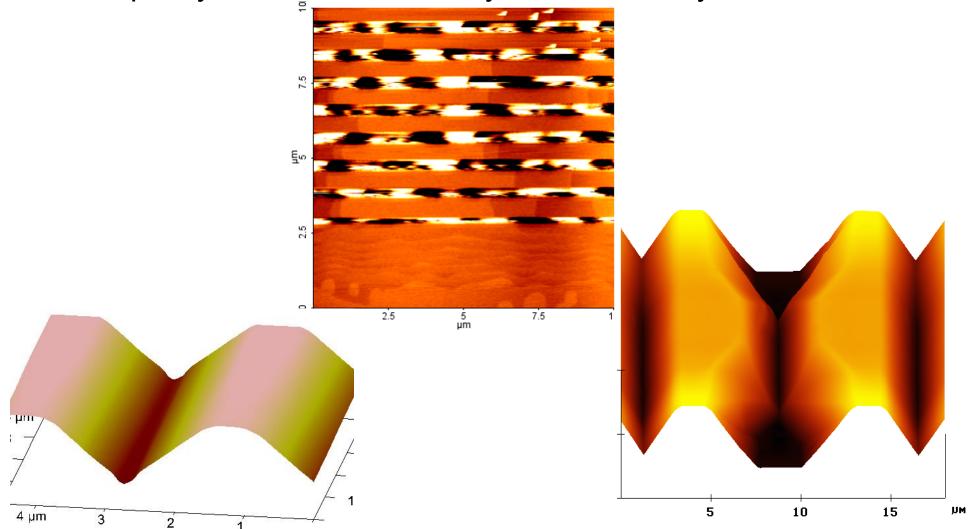




#### Substrate characterization

Example: etched V-groove substrate

The quality of surfaces is easily determined by AFM

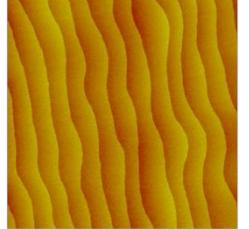


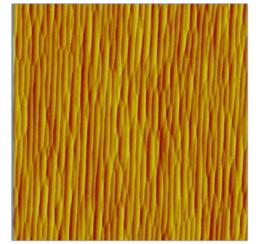


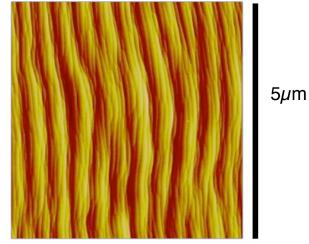
#### Surface characterization

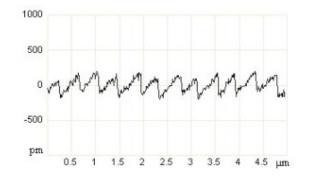
- Example: monolayer steps on GaAs structures

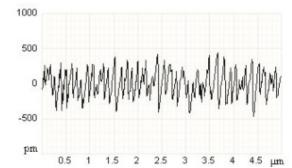
The substrate misorientation angle determines the growth mode: step-flow, step-bunching, multi-step bunching **Exact** 0.3°B 0.6°B

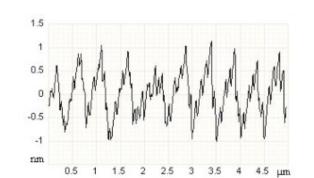








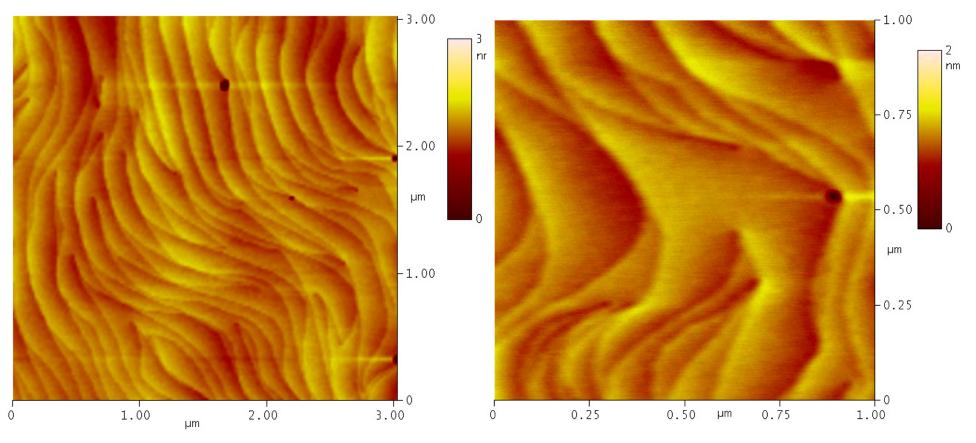






#### Surface characterization

Example: monolayer steps and dislocations on GaN layers



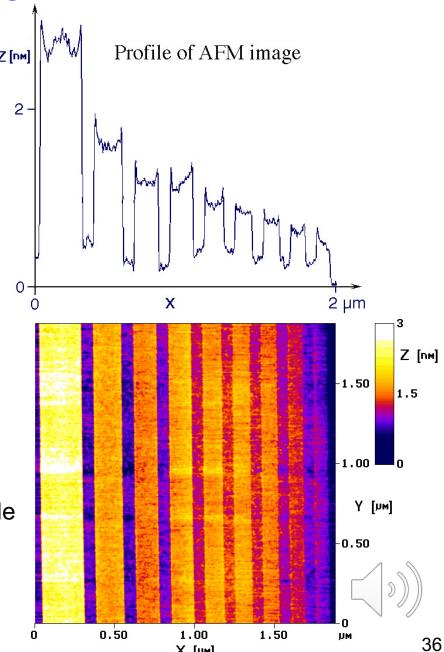
Images of GaN layer on Sapphire by AFM



#### **Cross-sectional characterization**

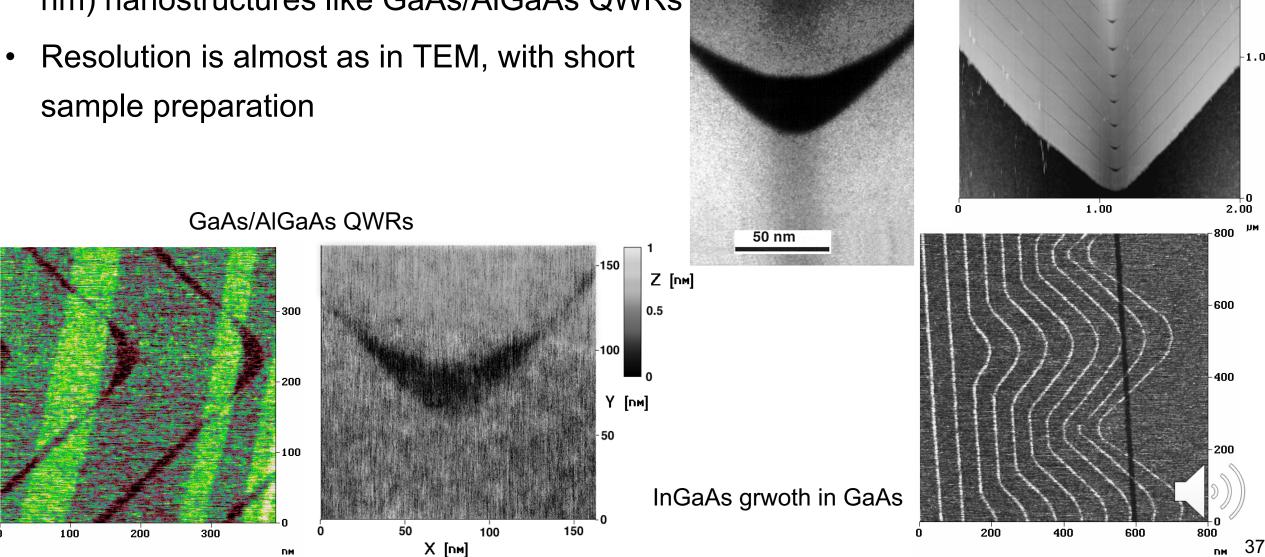
- Oxidation rate in air of GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As, depends on Al contents x.
- After 1 hr the oxide reaches finite thickness, depending only on composition
- GaAs is darker than AlAs
- InAs and InP oxidize even less than GaAs

AFM image of multi-layer AlGaAs sample



### Cross-sectional imaging of QWRs

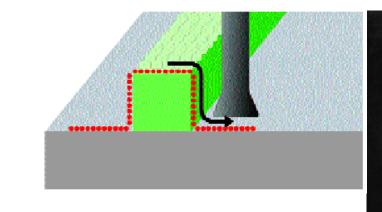
 High-resolution AFM can show small (a few nm) nanostructures like GaAs/AlGaAs QWRs

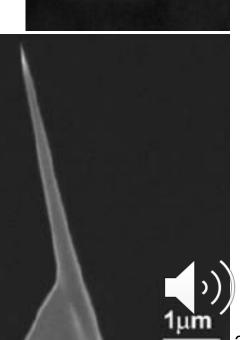


TEM of single QWR

### Industrial applications

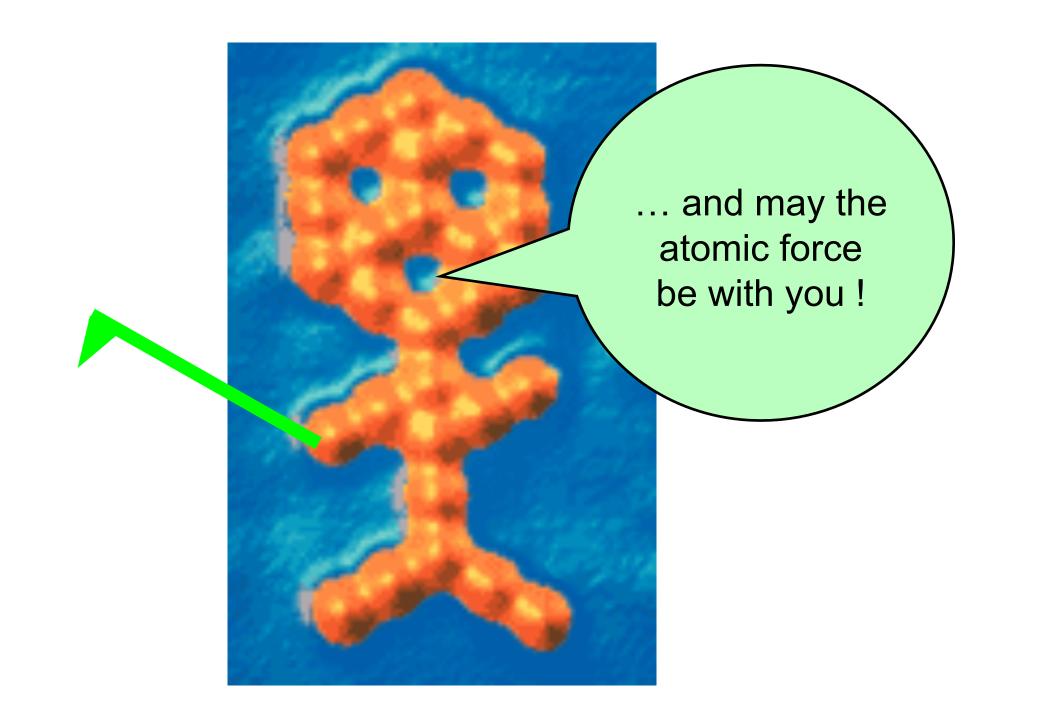
- Metrology: assessment of CDs, calibration
  - Tip characterization is very important
- Measurement of doping concentration
- Detection of defects in structures
  - Structural defects
  - Electrical defects
- The main problem with AFM in industry: Speed!
  - Typical AFM scan : 1-5 min/image, max. size 100 μm
  - To scan a 4" wafer will take 1000 hrs!
  - New high-speed AFM is better (0.1-1s/image)
  - Only surface sampling can be used on production wafers, and no cross-sections!





#### 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



#### References

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