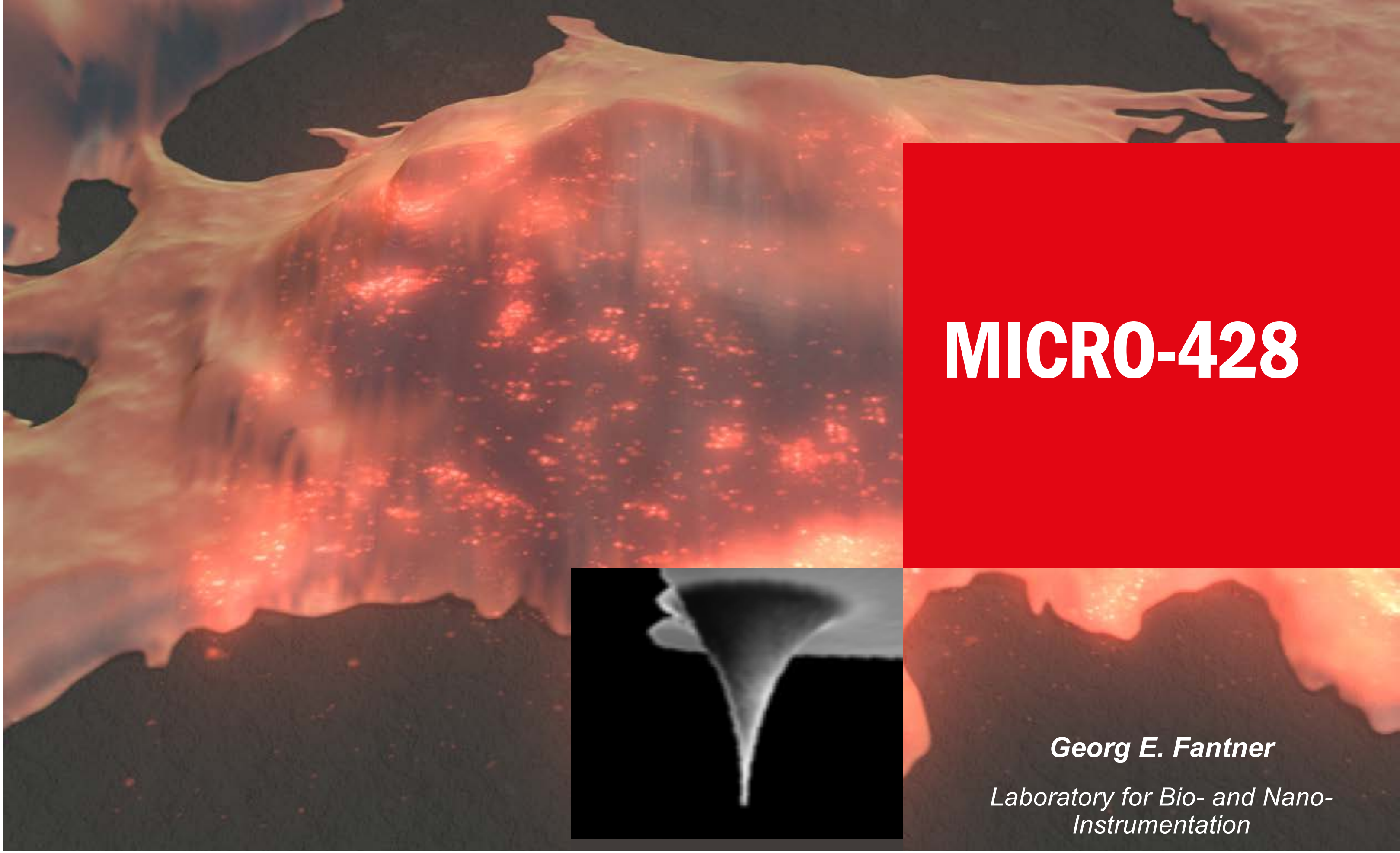
EPFL



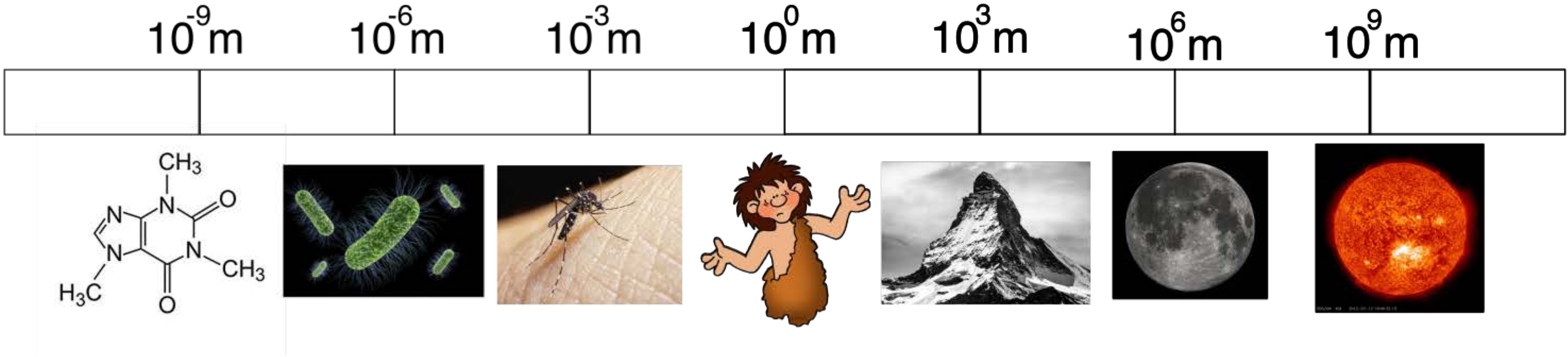
MICRO-428

Georg E. Fantner

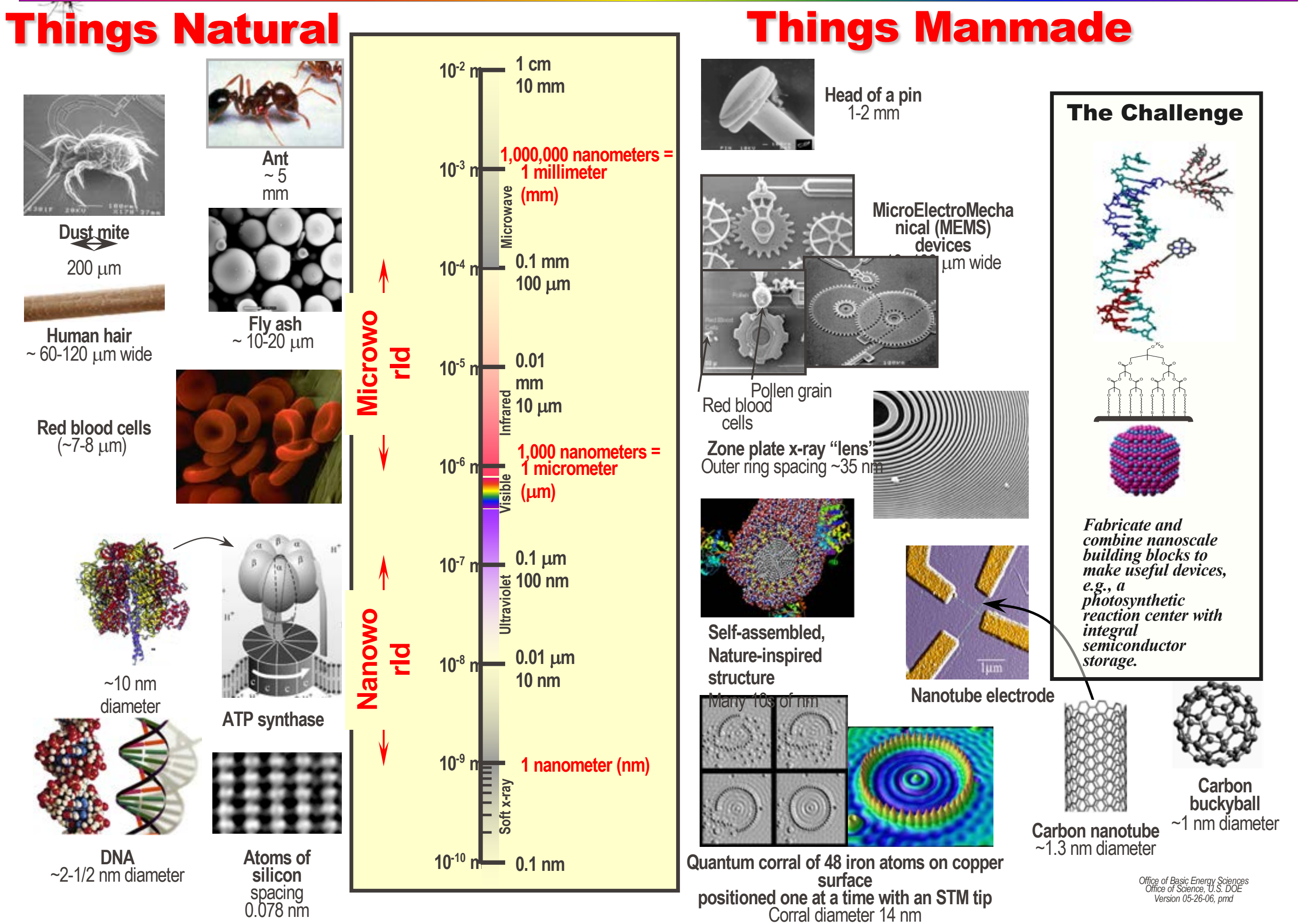
Laboratory for Bio- and Nano-Instrumentation

Nanoscale metrology – Part 1: Atomic force microscopy

Why we need nanoscale microscopy



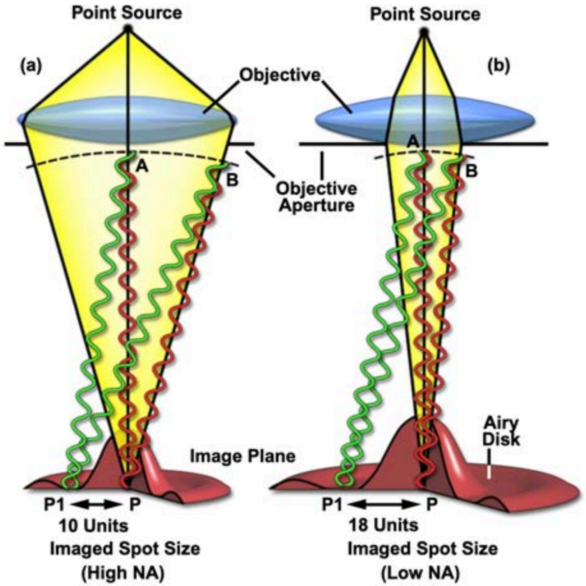
The Scale of Things – Nanometers and More



Why is it difficult to measure small things? The diffraction limit

In any (far field) microscopy system where we create a magnified image of an object via an image projection using diffractive elements (such as lenses) we run into the *diffraction limit:*

Point sources (with zero size) are projected to an Airy disk with a certain size. Two point sources that are close together will result in two Airy disks close together. If the disks are too close together they can no longer be separated based on their intensity. That is then the resolution limit of the microscope.



What determines the achievable resolution

Abbe $\text{Resolution}_{x,y} = \lambda/2NA$

- λ... Wavelength
- NA … Numerical aperture

What can we do to get around this?

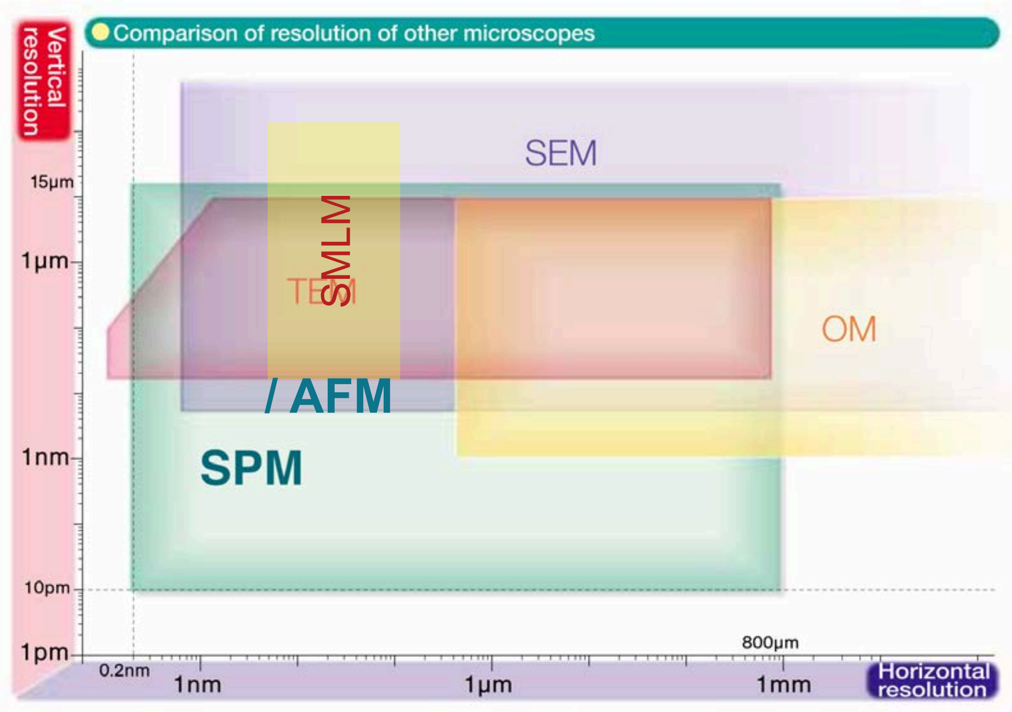
- Work with smaller wavelengths: instead of photons use particles with much smaller wavelength (such as electrons: de Broglie wavelength of an electron with acceleration voltage of 10kV = $1{,}22 \cdot 10^{-11}$m, which is 40’000 times smaller than that of a photon). That is what we use in electron microscopy
- Try to use non far field microscopy techniques (near field techniques or scanning probe techniques). This is wat we do in atomic force microscopy (AFM) or scanning near field optical microscopy (SNOM)

Microscopes we will cover

- Atomic Force Microscope (AFM)
- Scanning Electron Microscope (SEM)
- Transmission Electron Microscope (TEM)

Resolution is NOT everything…

…but it's sure nice to have a good one



Today's Lesson…
Atomic Force Microscopy

- A basic introduction to Atomic Force Microscopy (AFM)
  - What is an AFM?
  - What can we do with it?
  - What are the major components?
- AFM imaging modes
  - Contact mode
  - Force curves
  - Tapping mode
  - Non-contact mode
  - Off resonance mode
- The AFM cantilever
  - Fabrication
  - Actuation
- Resolution and imaging artefacts

What is an AFM?

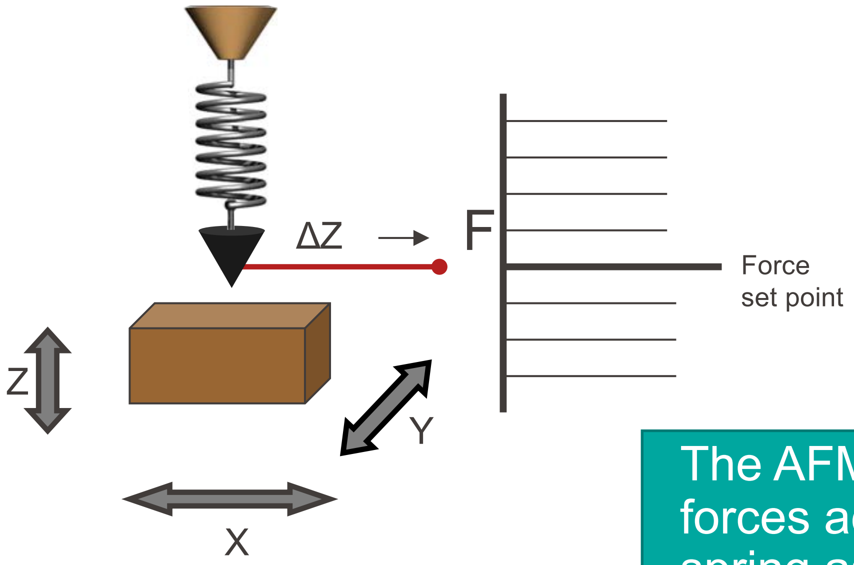
(don’t be fooled by the word *atomic*)

Letter from PhD applicant…

“Dear Professor,

... I found that your research interest are very well aligned with mine. I am fascinated with nuclear energy and atomic engineering. I therefor would like to pursue a PhD in your laboratory…”

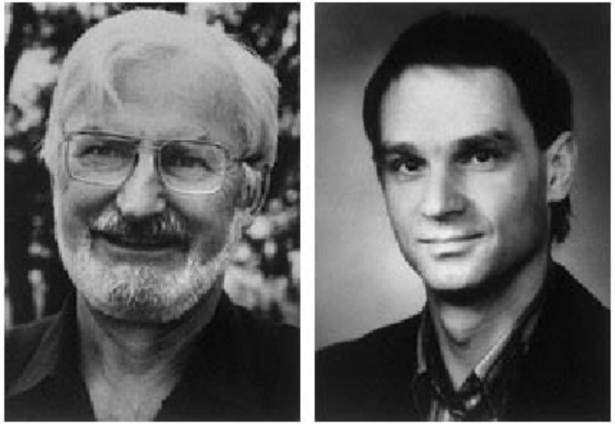
“Scanning Force Microscopy “SFM



The AFM measures the effect of forces acting on the sharp tip on a spring as a function of the position on the surface. – ***sometimes*** these forces are due to topography

It all started with *Tunneling*...

- Binnig, Gerber, Rohrer, Wiebel *Tunneling through a controller vacuum gap.*(Applied Physics Letters **40**, 178 (1982)
- *"This investigation is the first step towards the development of scanning tunneling microscopy, where the surface is scanned by a tunnel current and should open the door to a new area of surface studies."*



Scanning tunnelling microscopy was invented by Gerd Binnig (right) and Heinrich Rohrer (left) in 1981. They were awarded the Nobel Prize in 1986.

…but only for conducting samples!

The AFM…

- *G. Binnig, C. F. Quate and Ch. Gerber, PRL 56, 930 (1986)*

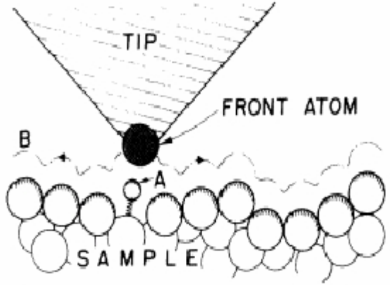


FIG. 1. Description of the principle operation of an STM as well as that of an AFM. The tip follows contour *B*, in one case to keep the tunneling current constant (STM) and in the other to maintain constant force between tip and sample (AFM, sample, and tip either insulating or conducting). The STM itself may probe forces when a periodic force on the adatom *A* varies its position in the gap and modulates the tunneling current in the STM. The force can come from an ac voltage on the tip, or from an externally applied magnetic field for adatoms with a magnetic moment.

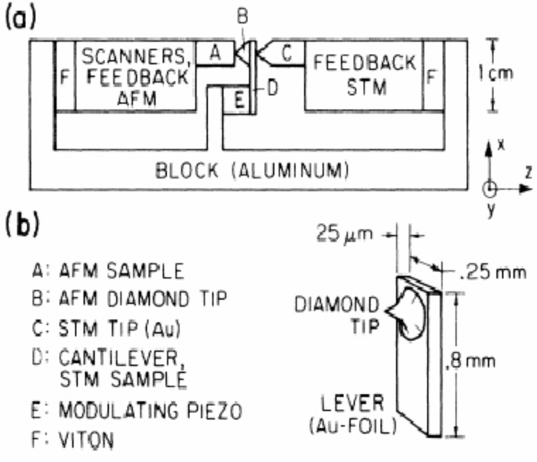
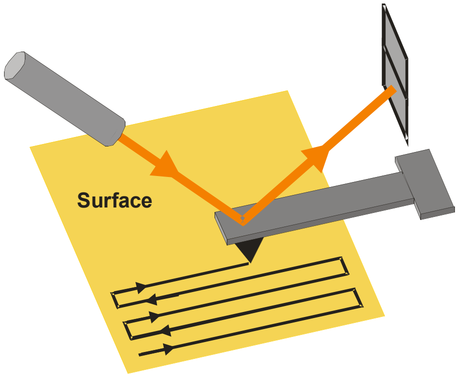
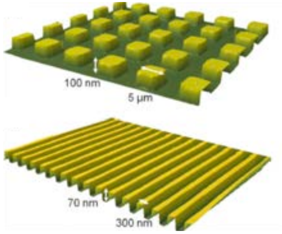


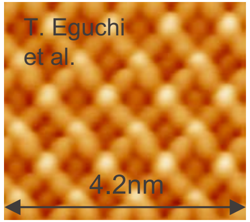
FIG. 2. Experimental setup. The lever is not to scale in (a). Its dimensions are given in (b). The STM and AFM piezoelectric drives are facing each other, sandwiching the diamond tip that is glued to the lever.

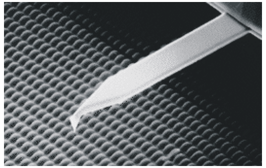
- Binnig invented the AFM in 1986, and while Binnig and Gerber were on a sabbatical in IBM Almaden they collaborated with Calvin Quate (Stanford) to produce the first working prototype

… Became a Versatile Tool for Nanoscale Measurements





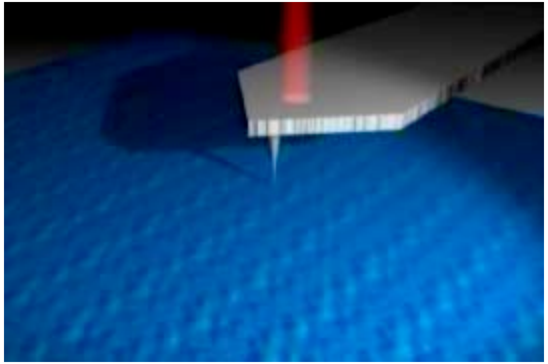




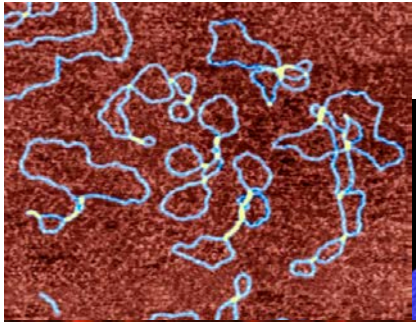
conductivity, surface potential, electrochemical potential, ion currents, magnetism, NMR….and many more

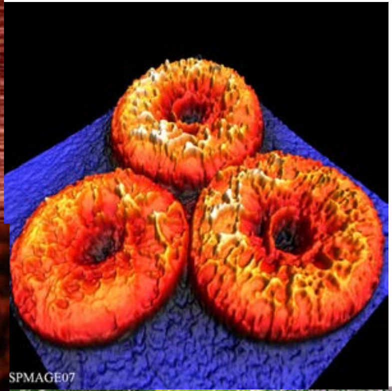
Atomic Force Microscopy

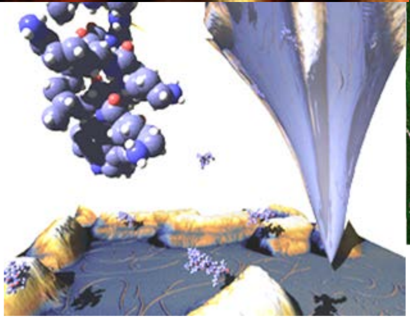
- a Versatile Tool for Nanoscale <u>Biology</u>

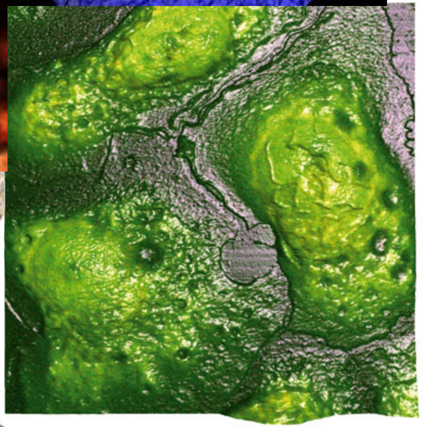


- Single molecule resolution
- High resolution imaging in aqueous solution
- Nanomanipulation
- Single molecule mechanics
- Imaging of living cells



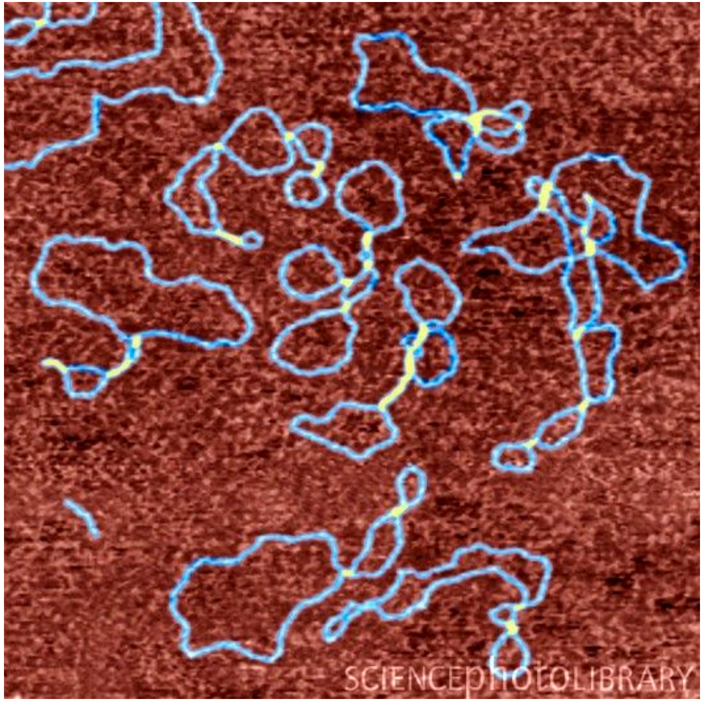






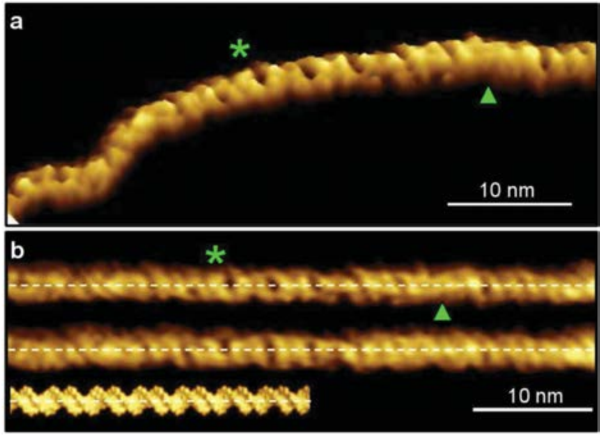
Single molecule resolution

Plasmid DNA on mica



Source: SciencePhotoLibrary

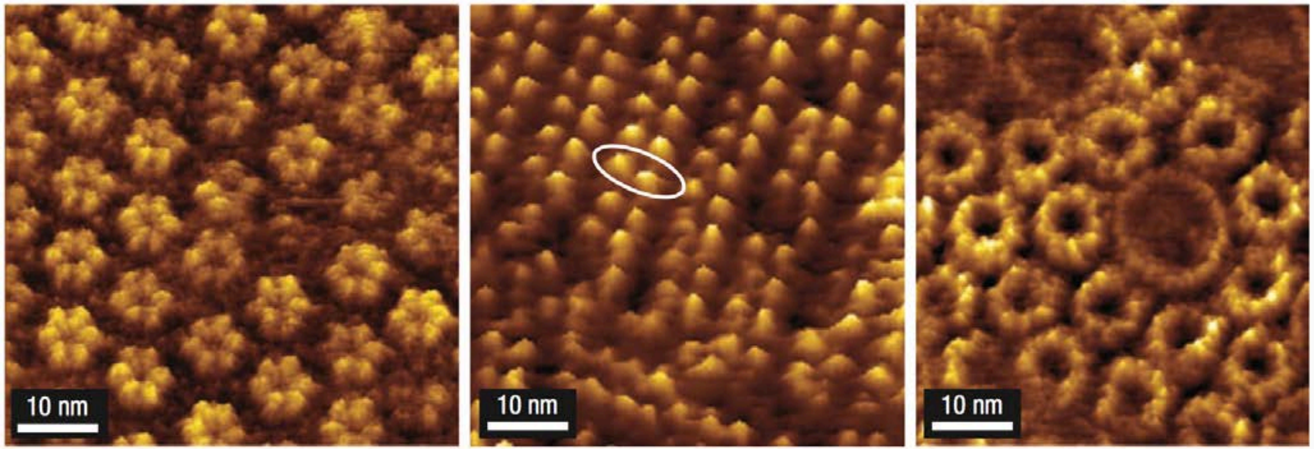
- Single molecules can be easily resolved
- Even the double helix!



Pyne et al. *Small, 10, Nr16, 2014*

High resolution images in fluid of proteins

- Imaging of membranes and membrane bound proteins
- Imaging of live cells



From Review Nature Nanotechnology 2008,D. Müller and I. Dufren,

AFM can be used for Nanomanipulation

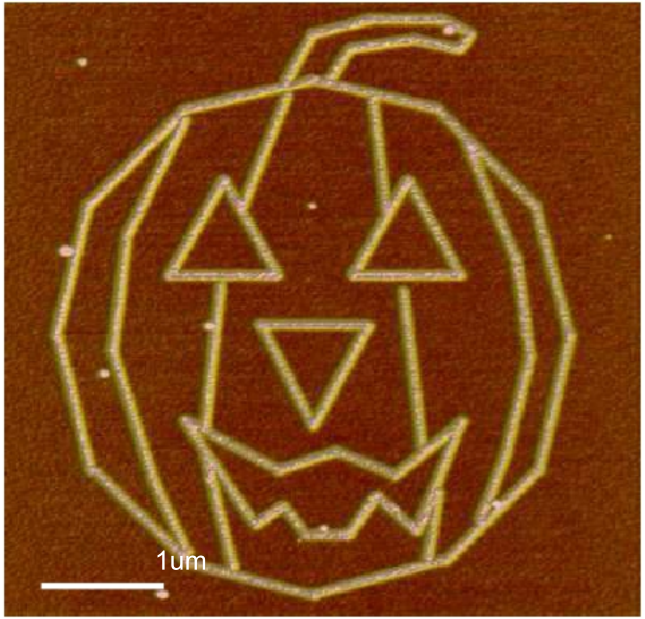


Image from:
http://www.veeco.com/library/nanotheater

- AFM patterning of a silicon surface using anodic oxidation
- Other approaches have been developed such as
  - dip-pen nanolithography and
  - Thermal scanning probe lithography (tSPL)

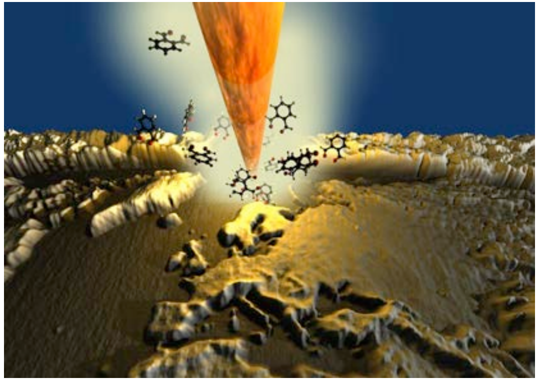


Image from: https://www.swisslitho.com

Different types of scanning probe microscopes

- SPM = scanning probe microscopy
- AFM= Atomic force microscopy (AFM), also known as
- SFM =scanning force microscopy
- STM scanning tunneling microscopy
- …
- SSETM = scanning single-Electron transistor microscopy

Wikipedia lists 41 different SPM modes!

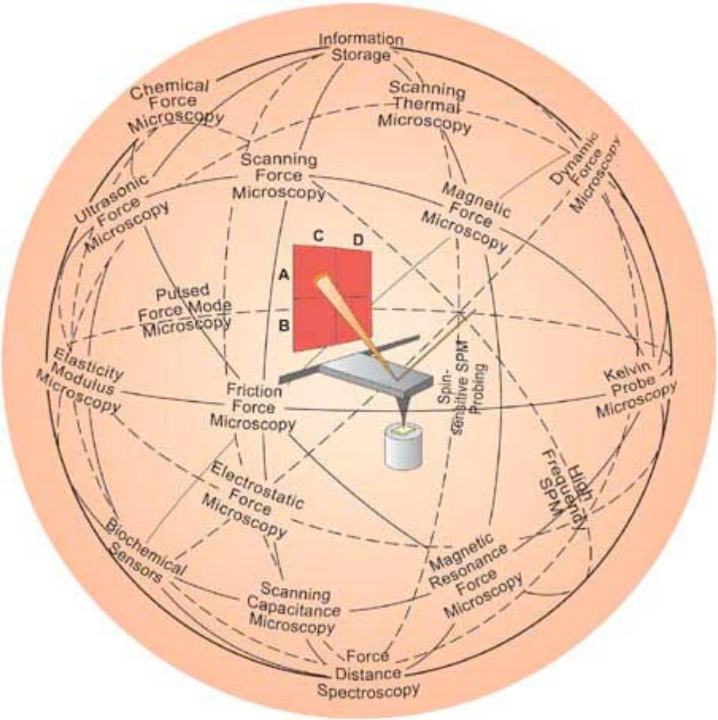
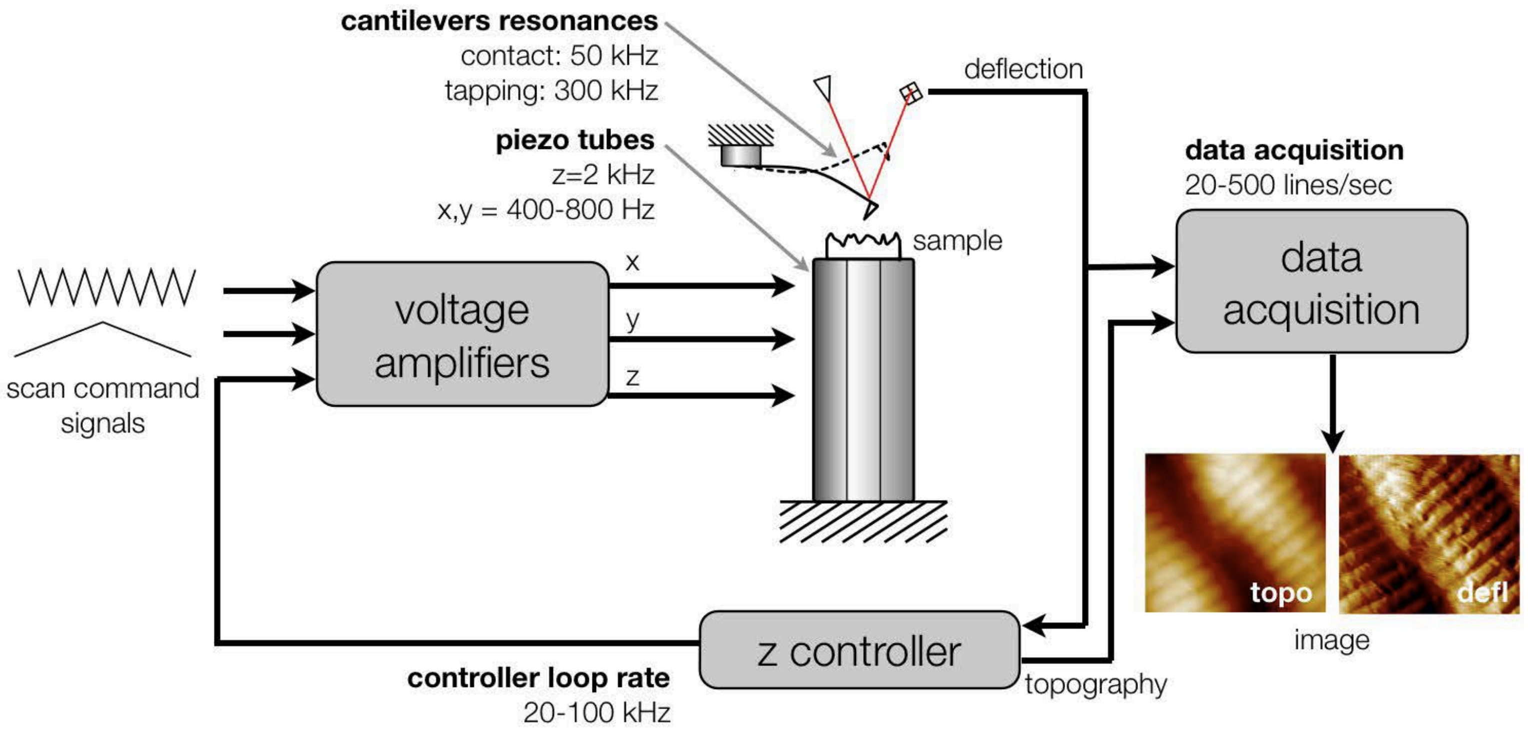


image: Christoph Gerber; copyright Nature Publishing Group

What's in an AFM?



A few principles we should understand

- Optical lever detection
- Piezos
- Feedback
- Setpoint
- Force curves
- Imaging Modes

Optical Lever Detection

Optical lever detection

Transduces cantilever deflection into a voltage

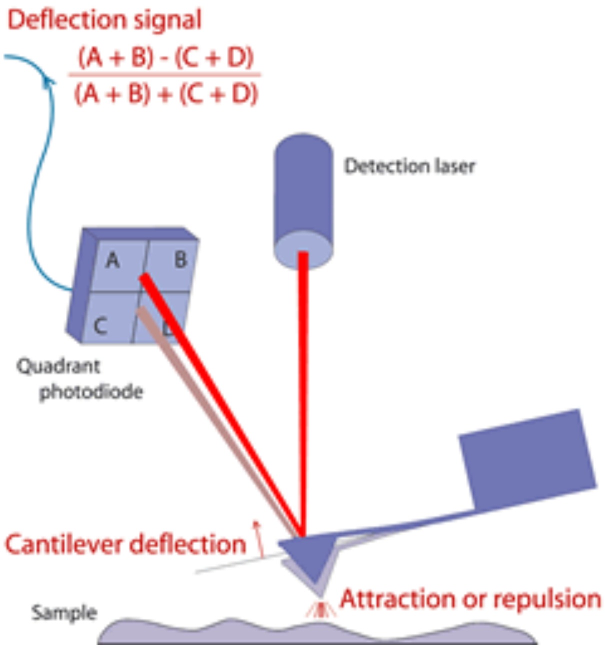
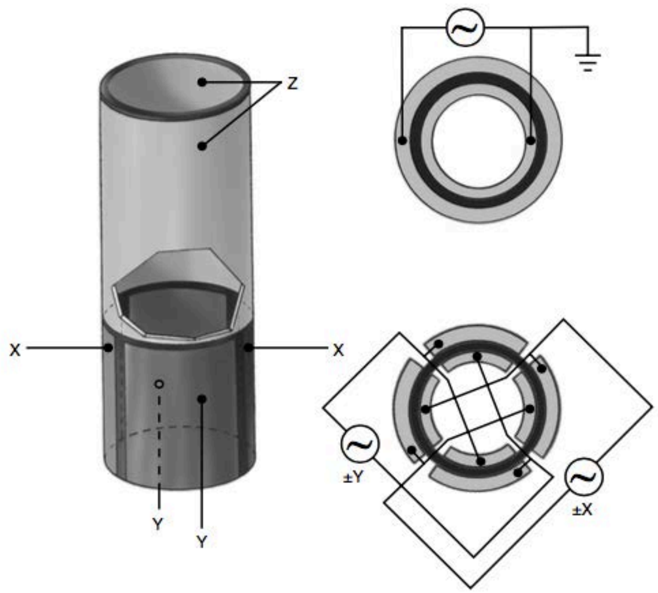


Image source: http://usa.jpk.com

- A very sensitive way to measure cantilever ***angle change***
- The change of angle is amplified by the distance from the cantilever tip to the 4-quadrant photodiode
- Each quadrant creates a current which is turned into a voltage using a transimpedance amplifier (I/V converter)
- The cantilever deflection is the ***normalized*** difference of the top quadrants minus the bottom quadrants

Piezo scanners

Piezo materials expand when a voltage is applied



Piezo scanners can be:

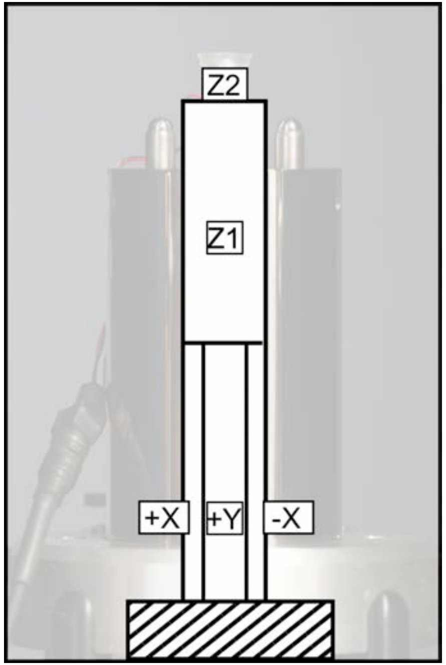
- Tubes
- Stacks
- Plates
- Monolythic piezo blocks

Or other types of actuation can be used:

- Voice coil actuation
- Electrostatic combs
- Linear magnetic motors

Piezo scanners

Piezo materials expand when a voltage is applied



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EPFL

Piezo scanners

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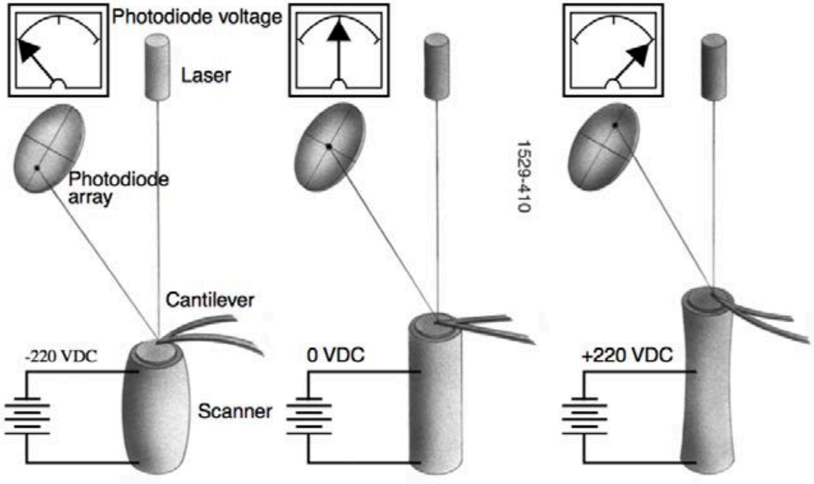
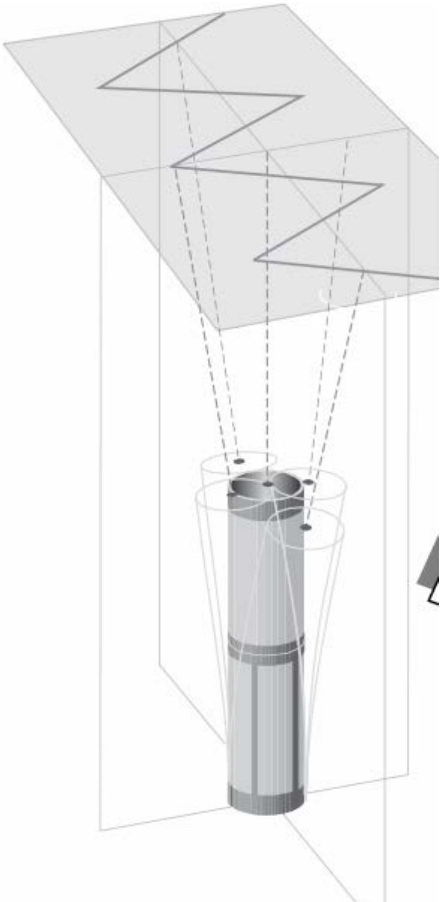


Z actuation

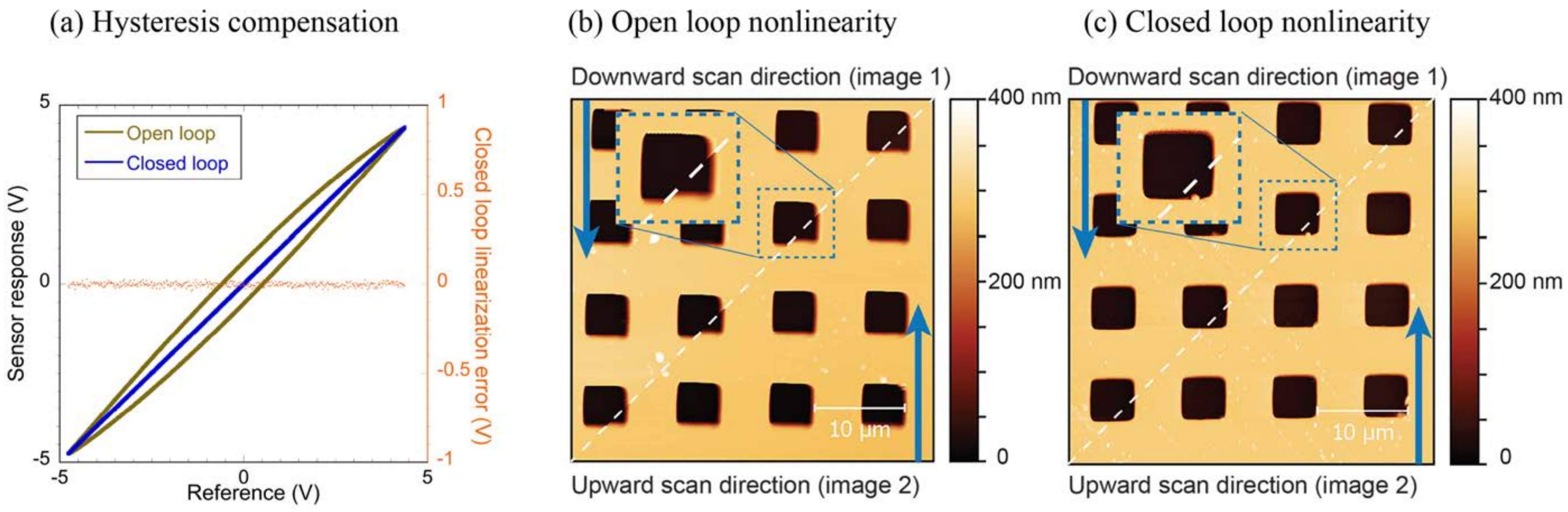


X,Y actuation

Images: Bruker Multimode Manual

Problems with piezo actuators

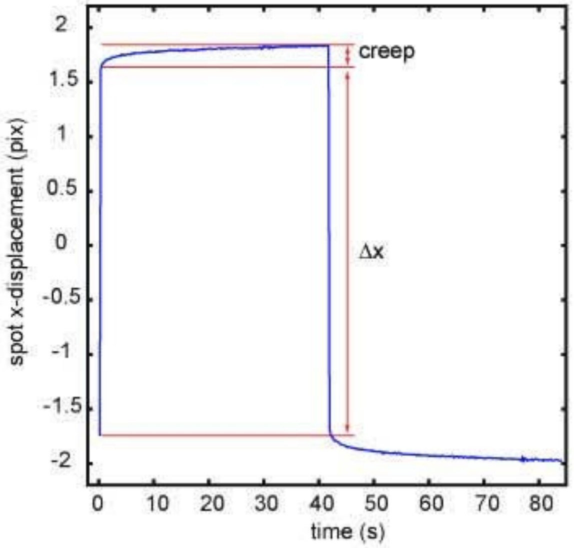
Hysteresis

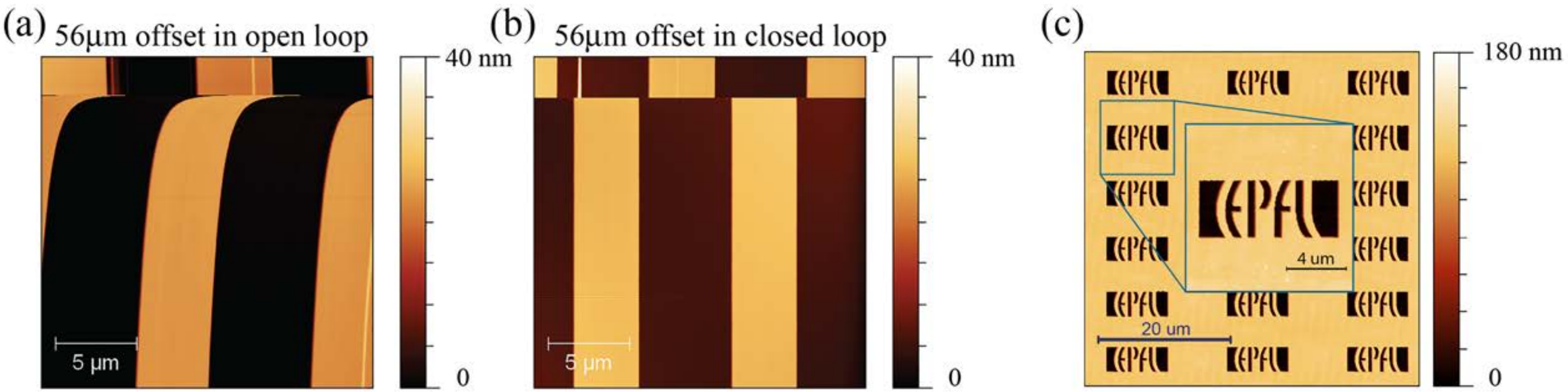


Problems with piezo actuators

Creep

- The piezo only moves ca 90% of the requested distance right away. The rest of the way it creeps very slowly!
- This causes image artefacts like distortion

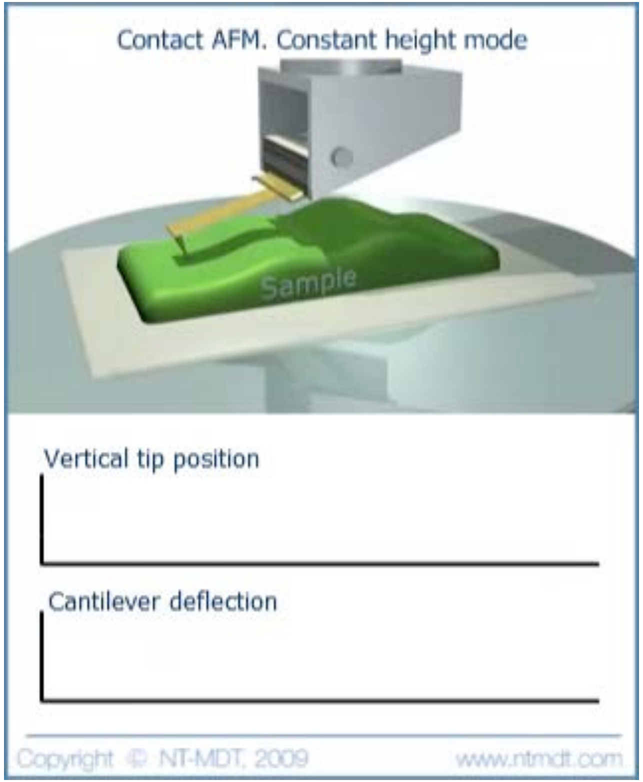




Feedback

Why do we need feedback?

Constant height mode

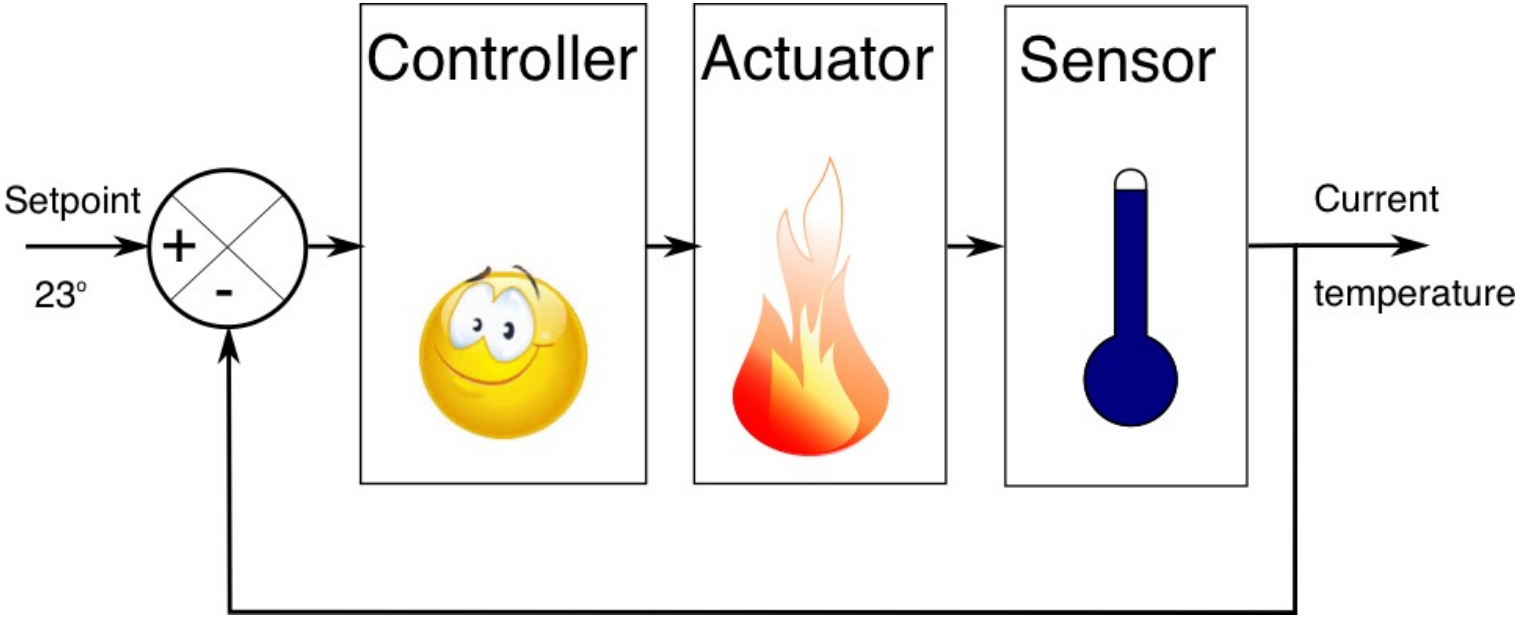


Why don't we just drag the cantilever over the surface?

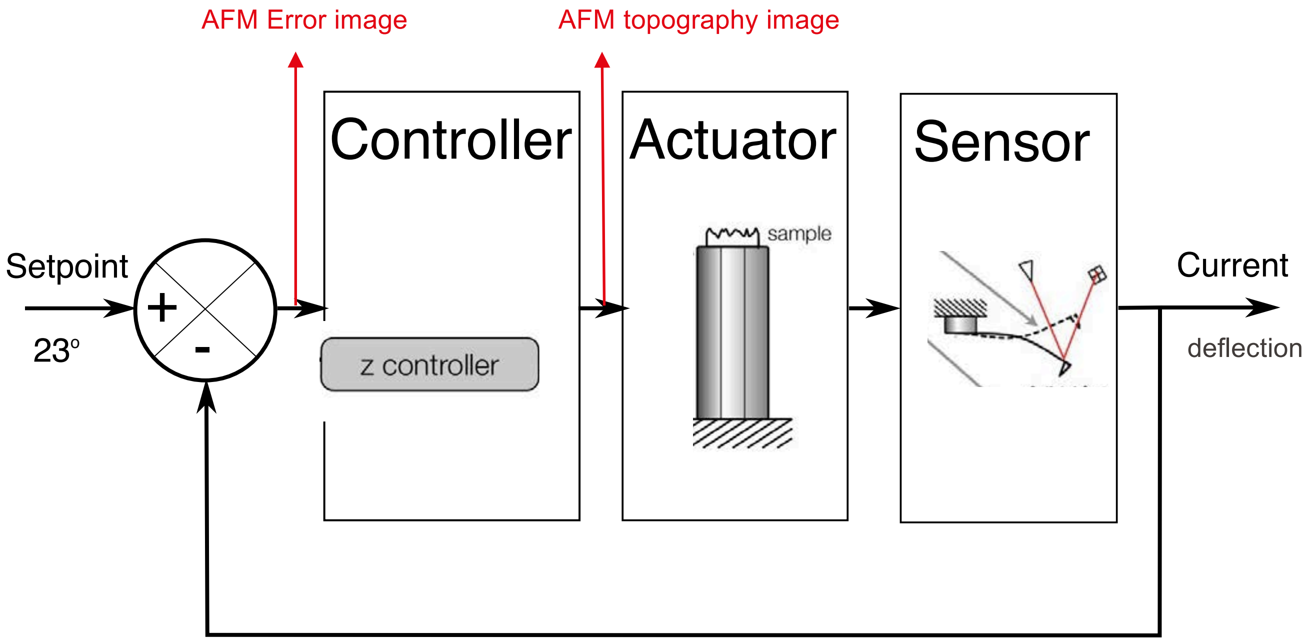
- Cantilever deflection isn't linear → height measurement is distorted
- Force on cantilever is not constant → tip and sample can get damaged

What do you do if you are cold?

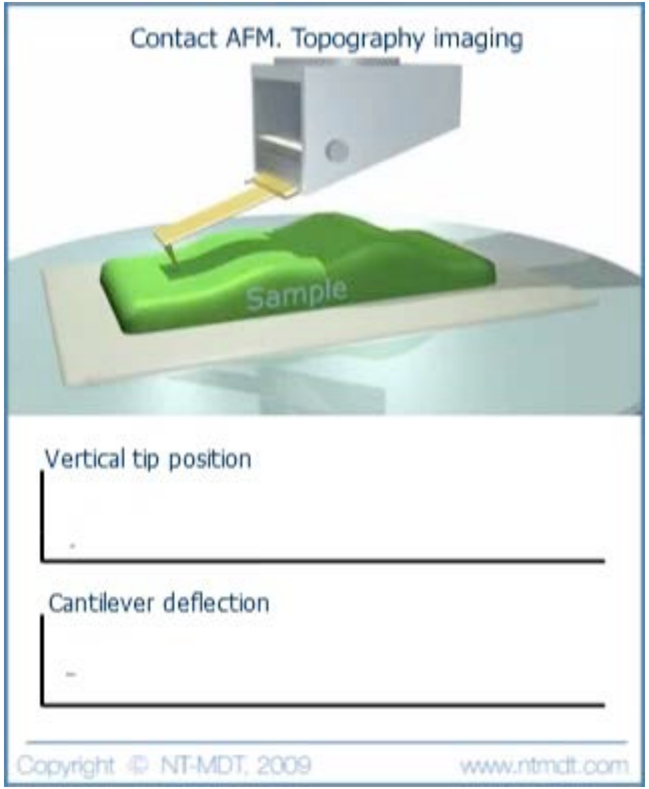
Rearranging into a feedback loop



Rearranging into a feedback loop



Feedback keeps the tip/sample interaction constant

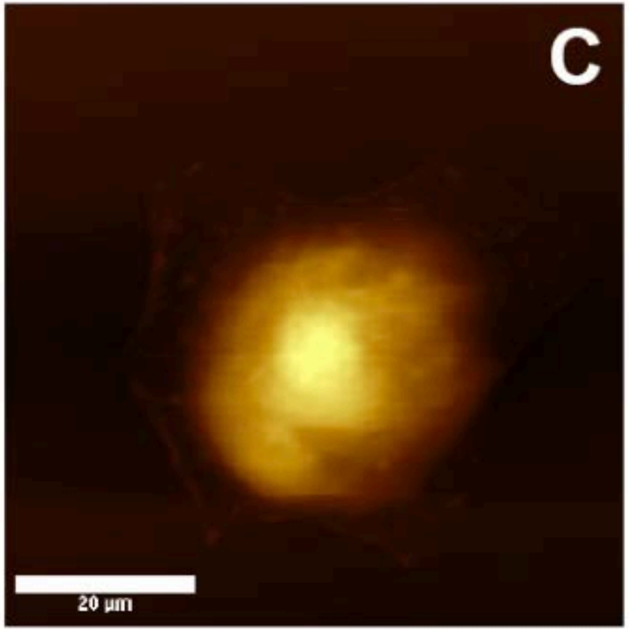


Benefits of operating in feedback:

- Cantilever deflection varies only slightly around setpoint
- The amount that the controller has to move the piezo up or down *approximates* the topography of the sample

Height image vs error image

Height



Error

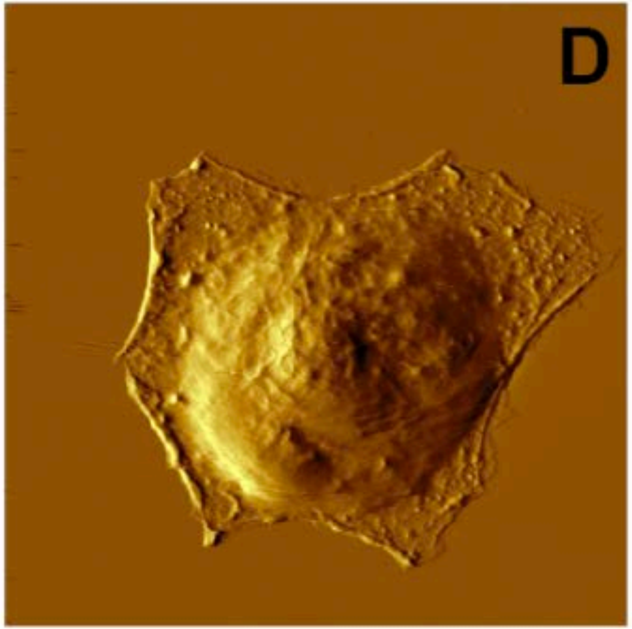
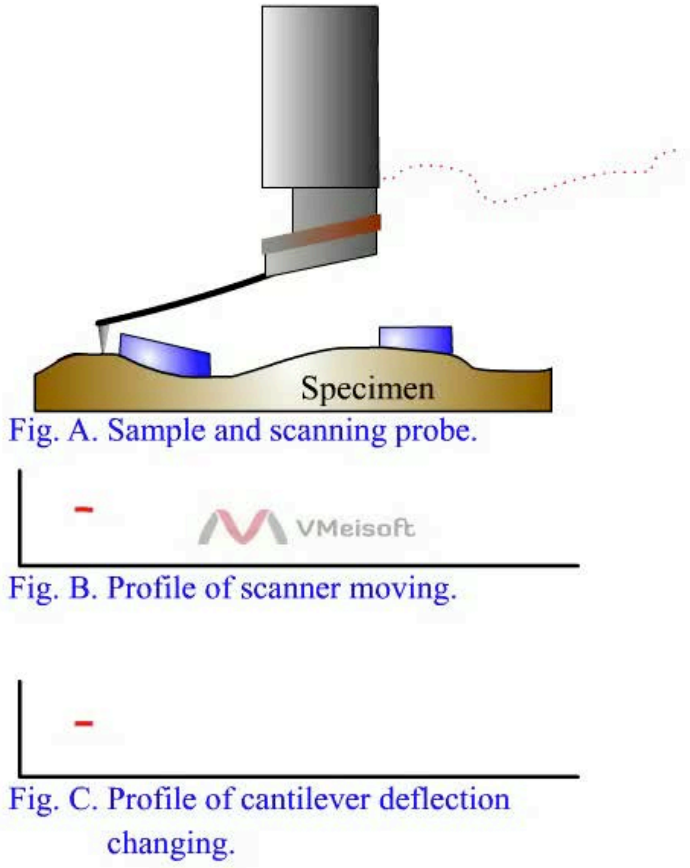


Image source: http://www.ntmdt.com

What is the meaning of the error signal?



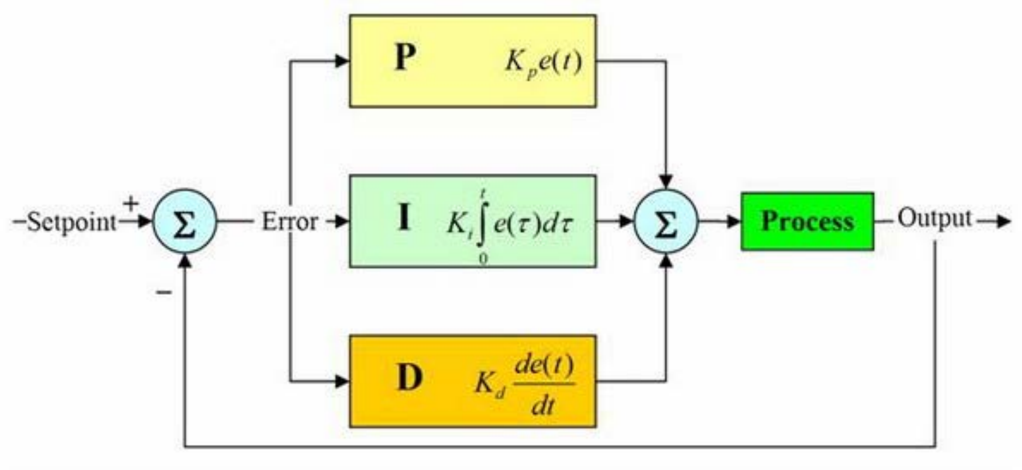
- The deflection/error signal is as much part of the AFM image as the topography image (also called height image)!
- It accentuates edges and features with small spatial frequencies
- The height image combined with the error image represent the “true topography”

AFM simulation

- The faster you scan, the larger is your error signal
- Constant height mode means feedback gain is zero
- If the gains are too high the AFM becomes unstable

PID controller

Proportional, Integral, and Differential controller



- The P gain reacts to the current deflection
- The I gain reacts to all the past deflections
- The D gain tries to predict the future deflection based on the slope

Feedback isn't infinitely fast!

Danger of oscillation

Procedure to tune the feedback gains of an AFM:

1. Set all gains to low values
2. "Increase" the setpoint until the cantilever comes to the surface
3. Increase the I gain until you oscillate – then slightly decrease it
4. Increase the P gain until you oscillate - then slightly decrease it
5. (Increase the P gain until you oscillate - then slightly decrease it)
6. Go back to 3 and iterate until you have a good image

We always want to operate the AFM borderline unstable!

Force Curves

Tip sample interactions

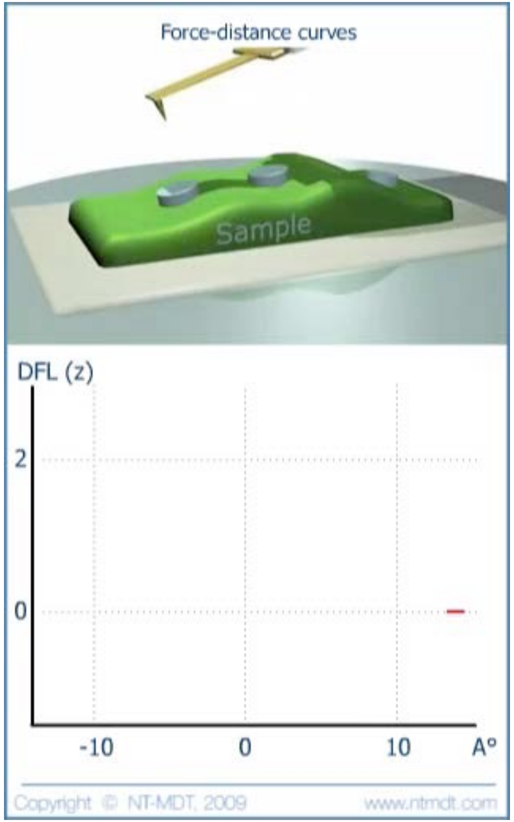
Force curves

There are many forces that can act between the tip and the sample

- Van Der Waals forces (attractive)
- Pauli repulsion (repulsive)
- Electrostatic forces (attractive or repulsive)
- Capillary forces (attractive)
- Magnetic forces (attractive or repulsive)
- …

We can measure what forces act on a cantilever as a function of distance from the surface by measuring a ***Force Curve***

Force curves



Force curves can tell us a lot about the tip sample interaction:

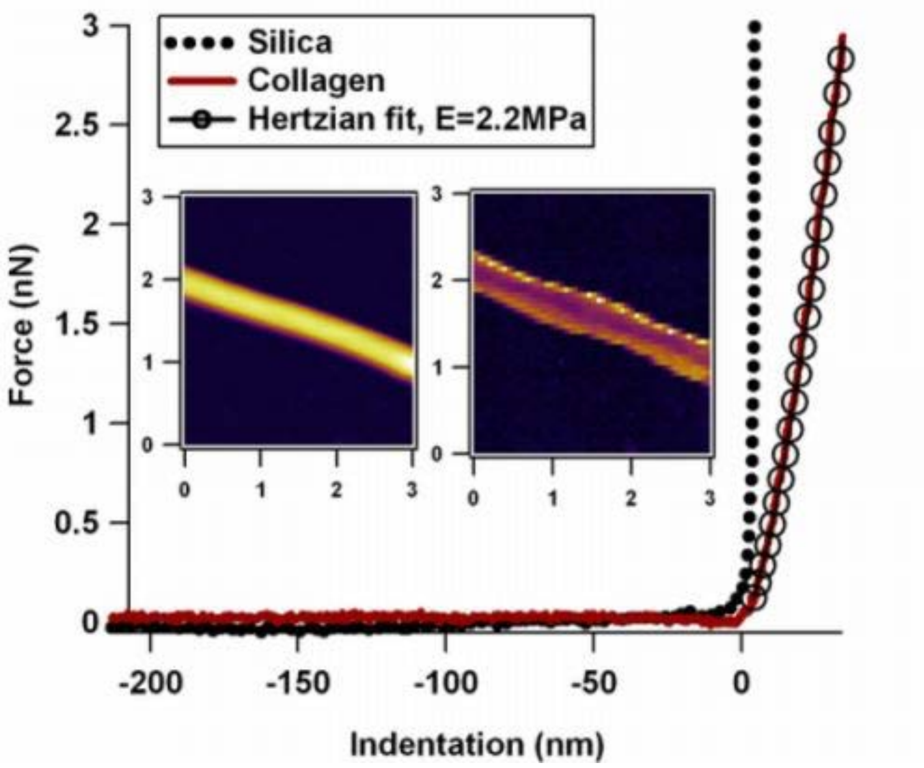
- What is the adhesion of tip to sample
- What is the hardness of the sample
- What is the energy dissipation per cycle

Or about our measurement setup

- What is the deflection sensitivity (how many nm do we have to deflect the cantilever to measure 1V shift in the 4-quadrant photodiode)

Force volume mode

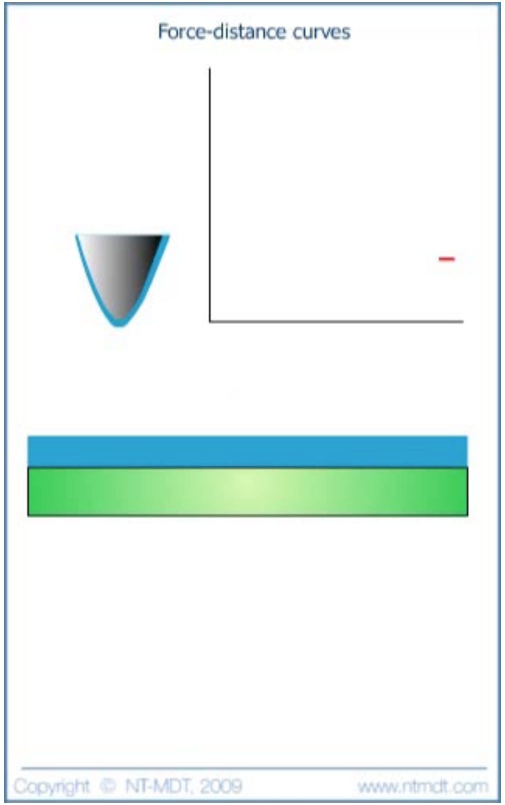
Creating mechanical properties maps



http://www.imaging-git.com

Surface adhesion

Capillary forces are always present when imaging in air!



All surfaces in ambient are covered with a thin water layer.

- Capillary forces act between tip and surface
- When imaging in air they create a “snap-in” as well as adhesion
- They can lead to many artefacts and instabilities

Single molecule force spectroscopy

Force curves as a tool for single molecule mechanics

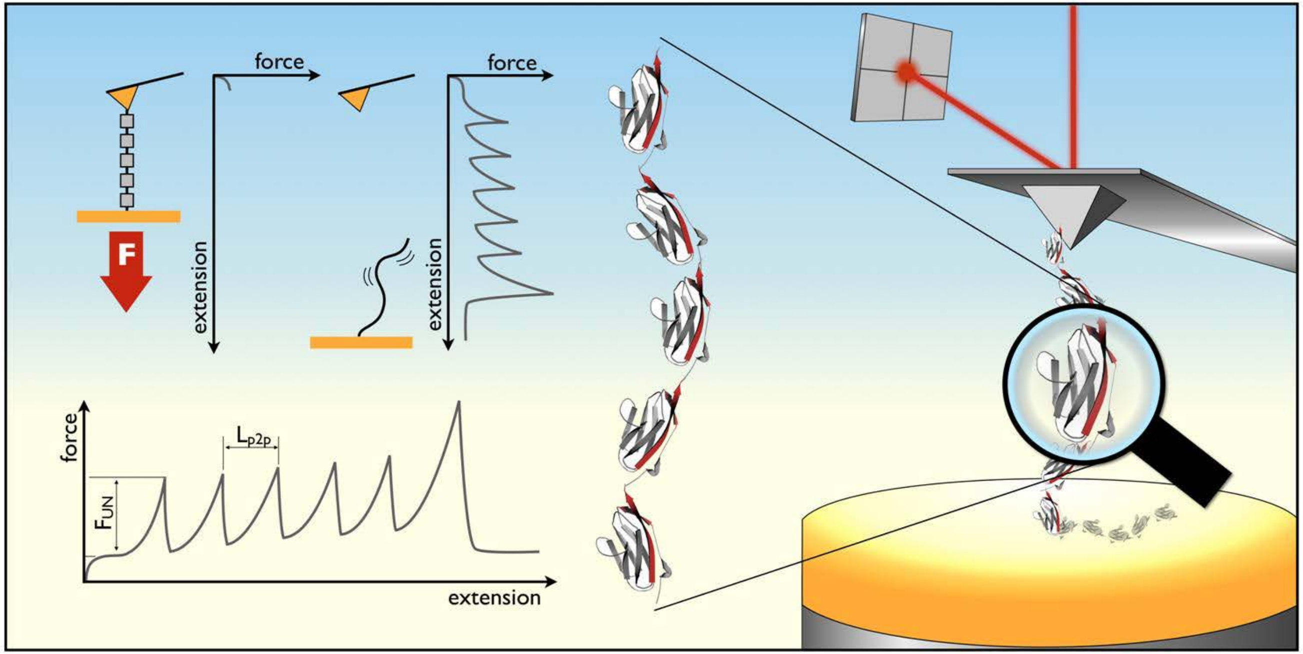
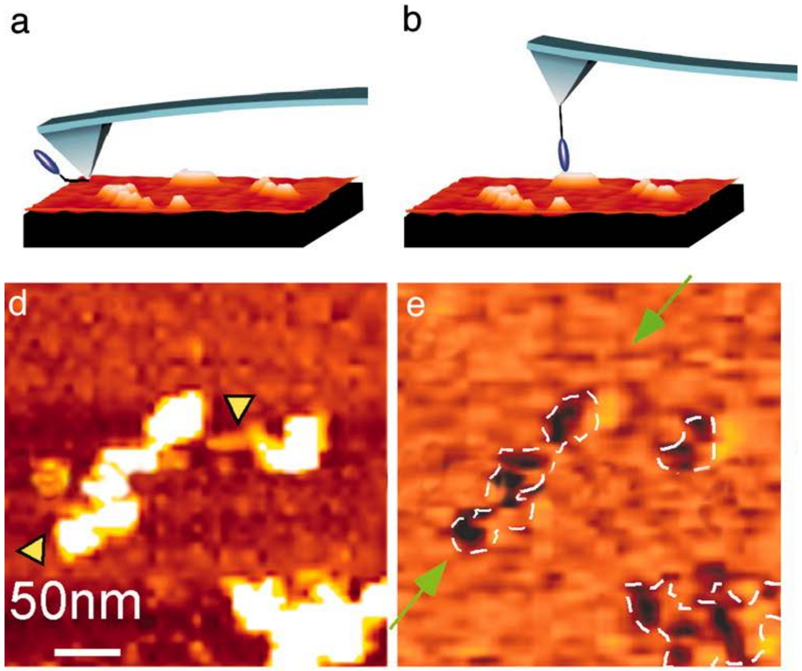


Image source: Dr Lorna Dougan - http://www1.mnp.leeds.ac.uk/ldougan/

Single molecule recognition imaging

Detecting specific antigens on a surface



Adapted from: C. Stroh et al. PNAS 2004;101:12503-12507

Imaging modes (dynamic modes)

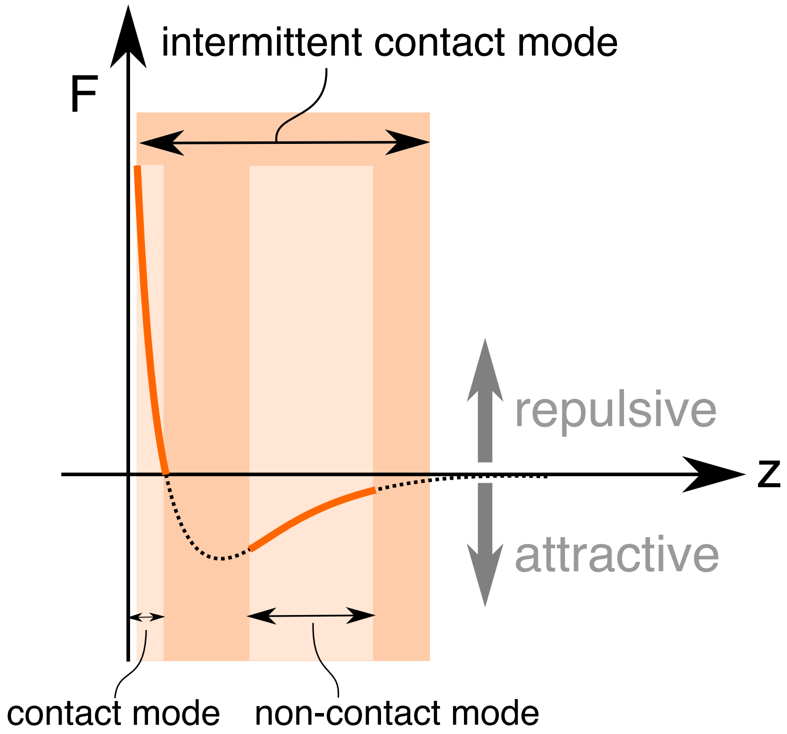
Dynamic modes

Reduces tip sample interactions

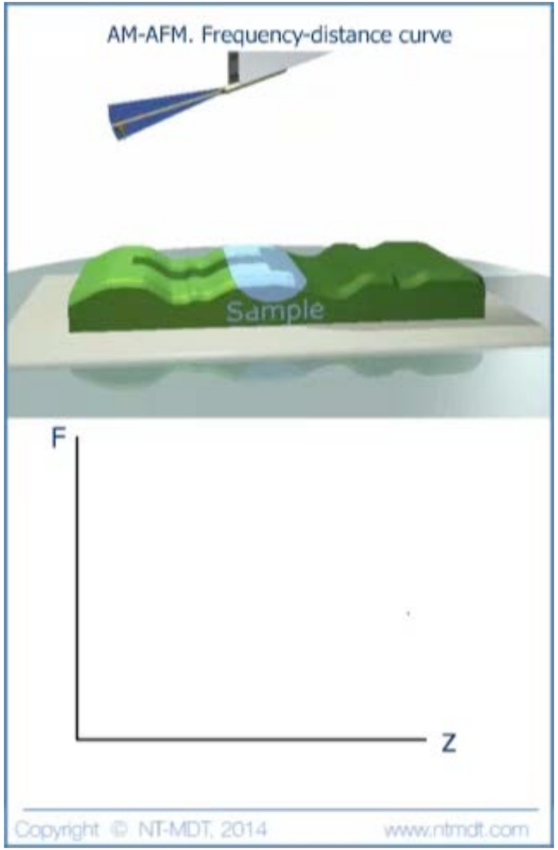
- Tapping mode™ (intermittent contact mode, amplitude modulation mode, dynamic mode,…)
- Non-contact mode
- Off resonance modes (Peak Force Tapping™, QI mode™, hopping mode™, HybriD mode™,…)

Lennard-Jones potential

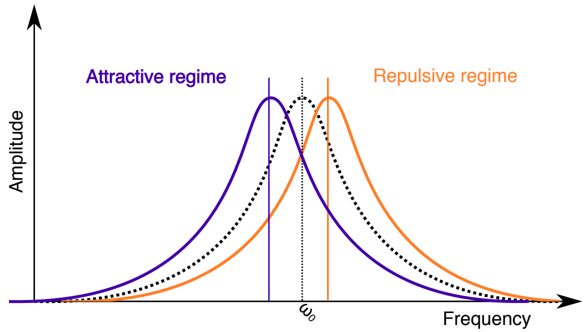
The cantilever feels different force regimes



Oscillating approach curves

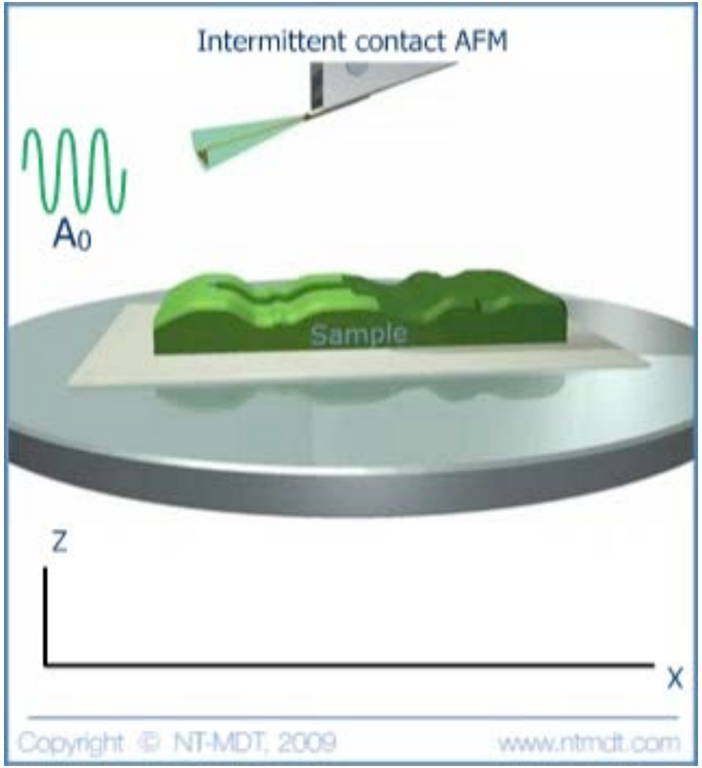


- As the cantilever approaches the surface it feels different forces (due to the Leonard Jones potential)
- When the cantilever is in the attractive regime the resonance frequency decreases
- When the cantilever is in the repulsive regime the resonance frequency increases

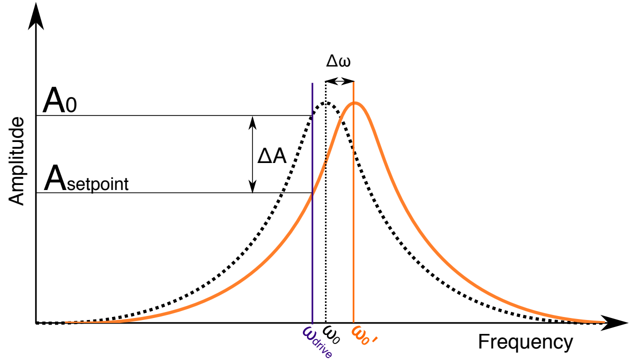


Amplitude modulation

(a.k.a. Tapping mode™)

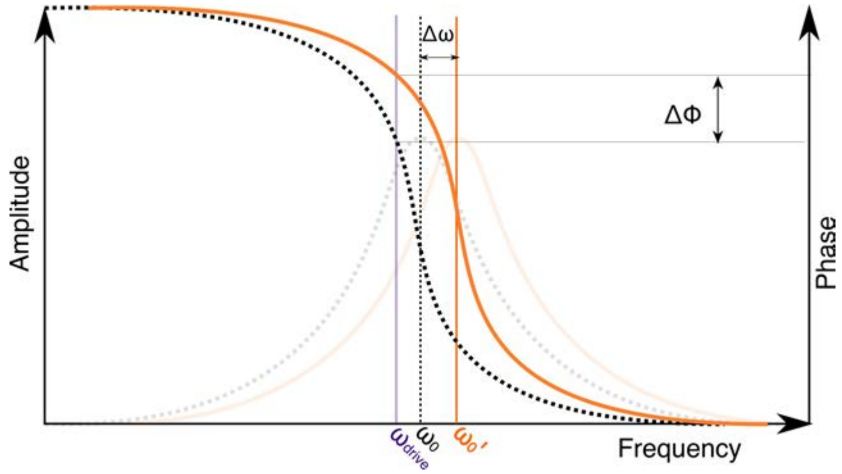


- In tapping mode we excite the cantilever at a fixed frequency ω_{drive} slightly below its resonance frequency
- As the cantilever approaches into the repulsive regime, the resonance frequency (of cantilever + sample force) increases.
- At the fixed frequency ω, the resulting amplitude will therefore drop as we enter the repulsive regime
- The amplitude error is used for the feedback parameter



Phase imaging

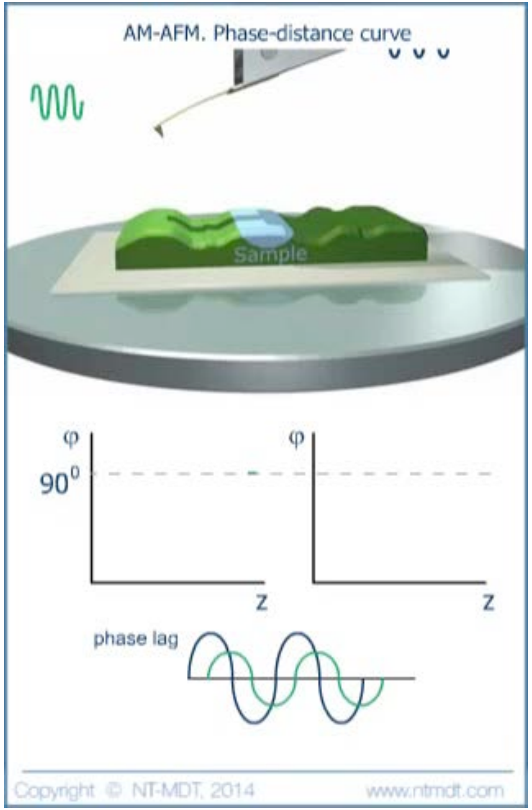
In tapping mode



- The phase difference between the cantilever drive signal and the cantilever oscillation is called the "phase signal"
- The resonance shift Δω also introduces a phase shift Δφ at the driving frequency ω_{drive}
- This shift could also be used for feedback, but…
- … other factors such as materials properties affect phase as well

Phase imaging

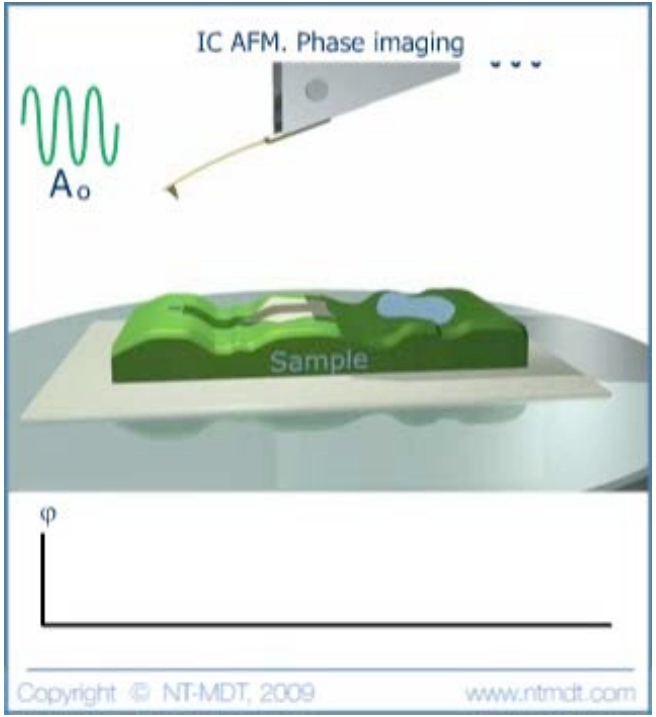
In tapping mode



- The phase signal “represents” the damping that the cantilever feels due to the tip sample interaction
- This damping can be due to topography (especially side walls)
- Or it could be due to damping by the sample material

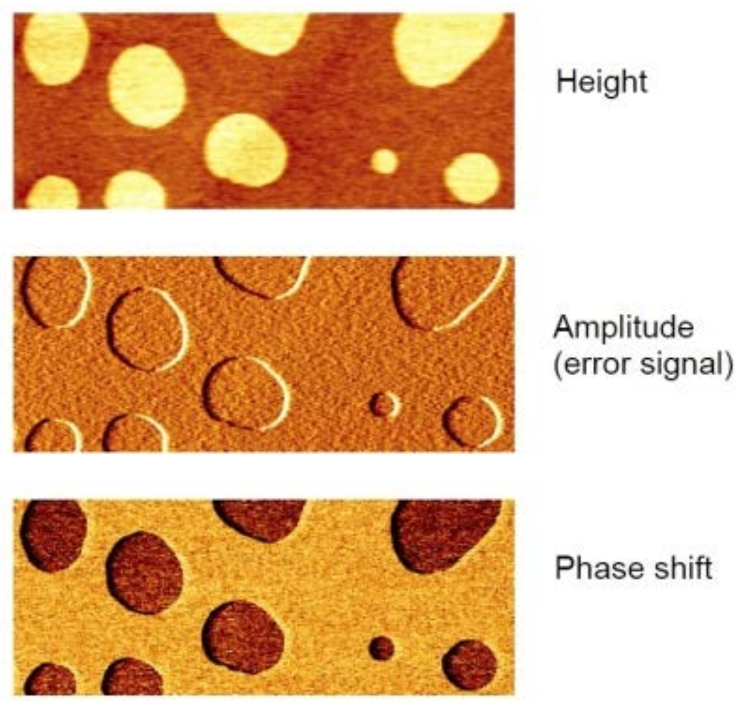
Phase imaging

In tapping mode can give materials contrast



- In tapping mode, the phase channel is an additional observable channel
- If no topography is present, the phase signal can be used to distinguish materials properties
- But beware of interpretation artefacts!

Height vs Amplitude vs Phase

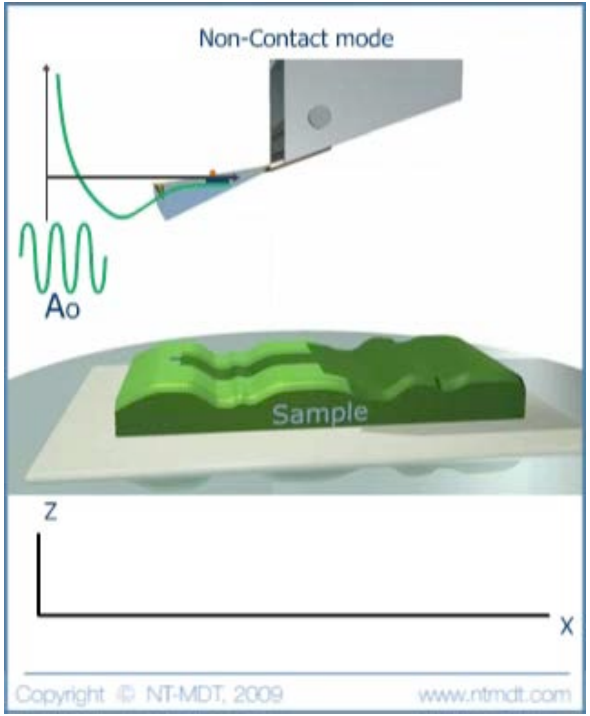


Lipid phase contrast from intermittent contact mode AFM in liquid, 18 x 7 micrometer area. The height (top) and phase (bottom) images show contrast between the lipid phases. The amplitude image (center) shows contrast at the edges of the patches.

http://www.microscopy-analysis.com

Non contact mode

Imaging in the attractive regime

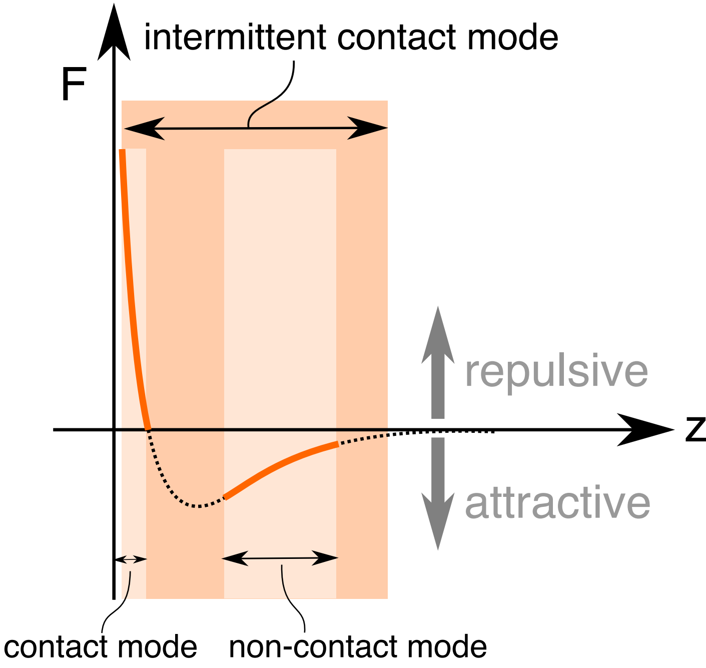


- In non contact mode, the cantilever "never touches" the surface
- Non-contact mode is difficult to maintain due to the low forces and small force gradients
- Primarily used in vacuum

Frequency modulated AFM

However less frequently used…

- The feedback loop controls for the resonance frequency, rather than the amplitude.
  - A self excitation positive feedback loop keeps the cantilever oscillating at the resonance of the cantilever + sample
  - A phase locked-loop detects the resonance frequency
  - The PID gets the resonance frequency shift as the control signal
- Is often used in vacuum to increase imaging speed
- Is more difficult to use since it is bi-stable



Off-resonance modes

Off-resonance modes

Peak Force Tapping, QI mode, hopping mode, HybriD mode,…

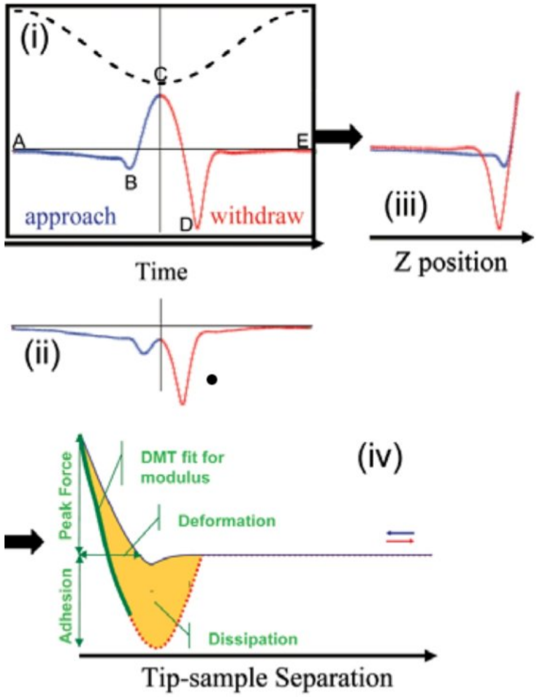
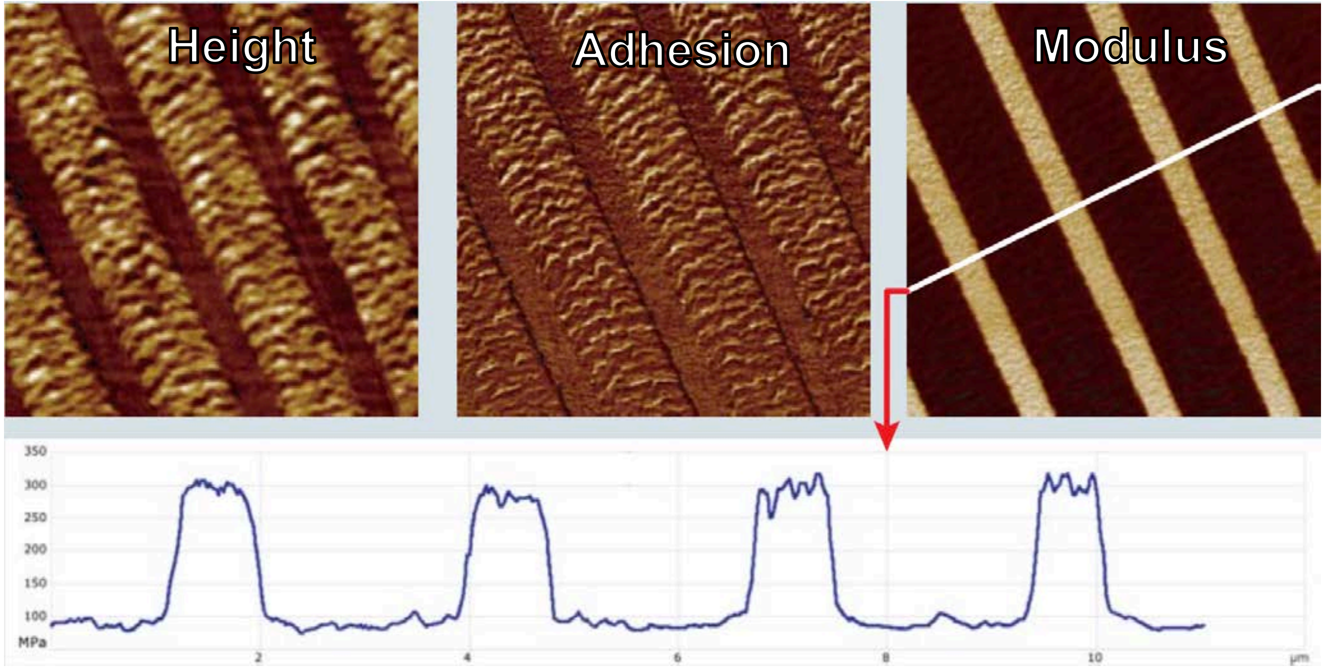


Image adapted from Bruker application note 128

- Like force-volume mode, but faster
- Ramp often done sinusoidally
- After baseline correction a force curve for each pixel is extracted
- Data extracted from the force curve:
  - Elastic modulus
  - Adhesion
  - Indentation
  - Energy dissipation

Measuring multiple sample properties

Multilayer polymer optical film



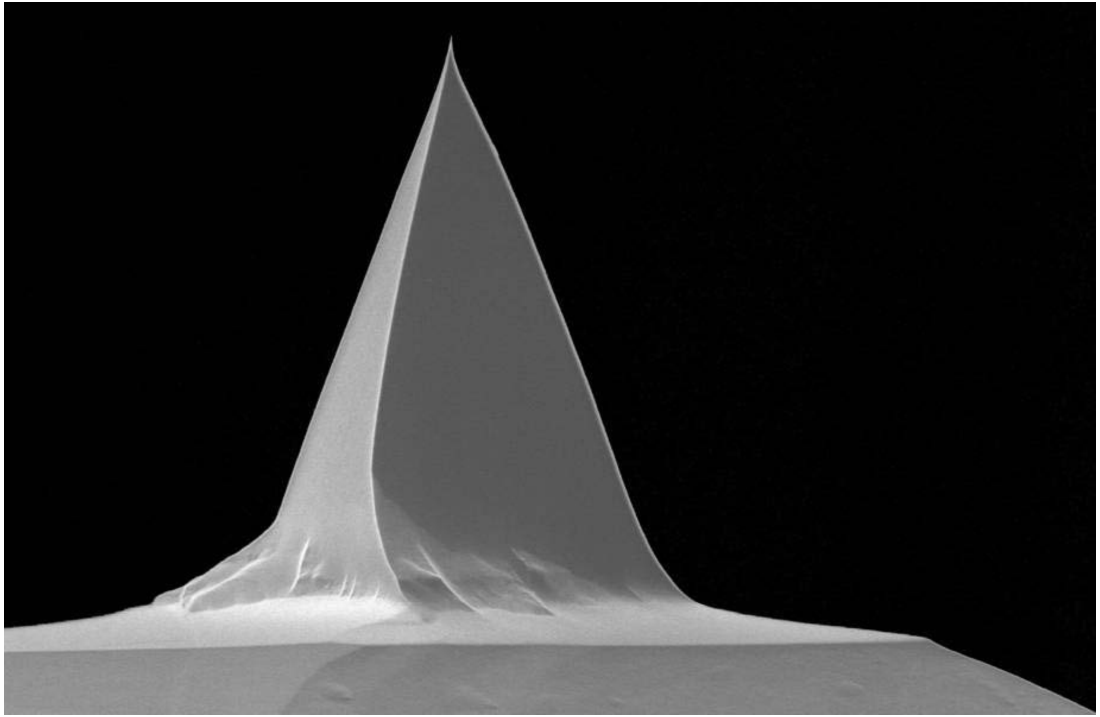
Adapted from Bruker application Note #128

Other scanning probe modes…

… you might come across

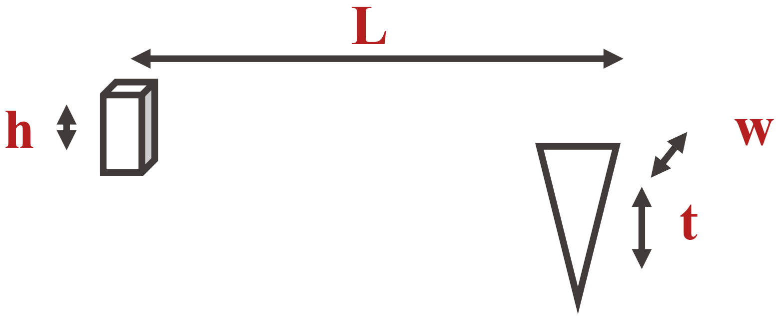
- KPFM (Kelvin probe force microscopy): measures work functions
- C-AFM (conductive force microscopy): measures sample resistance
- EFM (electrostatic force microscopy): measures electrostatic field
- MFM (magnetic force microscopy): measures magnetic properties
- PFM (piezo response microscopy): measures piezoelectric properties
- SCM (scanning capacitance microscopy): measures sample capacitance
- …

The cantilever



Cantilevers

What considerations govern the probe dimensions?



Resonance frequency

$$F_R \cong 0.162\sqrt{\frac{E}{\rho}} \bullet \frac{h}{L^2}$$

Normal spring constant

$$k_N = \frac{Ewh^3}{4L^3}$$

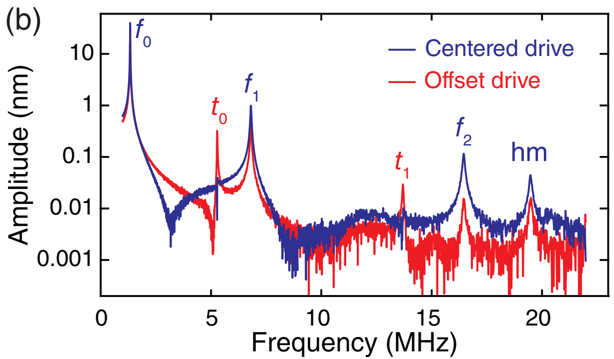
Lateral spring constant

$$k_L = 0.5k_N \frac{L^2}{t^2}$$

E= Young’s modulus, p = density

Cantilever dynamics

Cantilevers have multiple resonance modes



- Flexural resonances
- Torsional resonances
- Multiple Eigenmodes

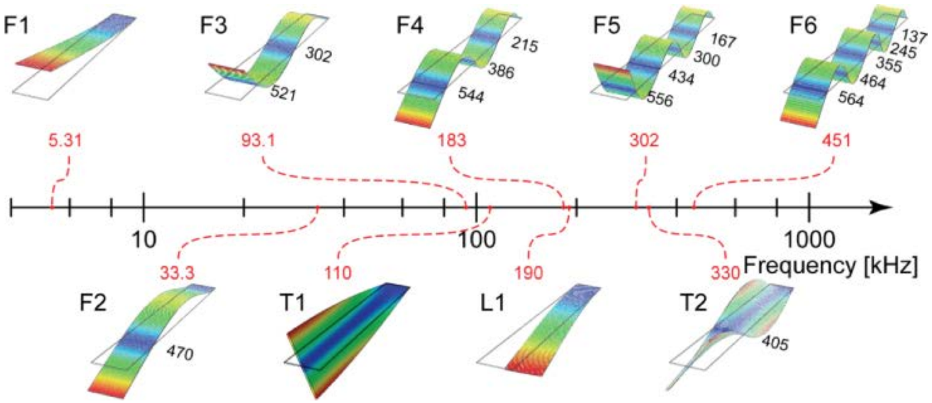
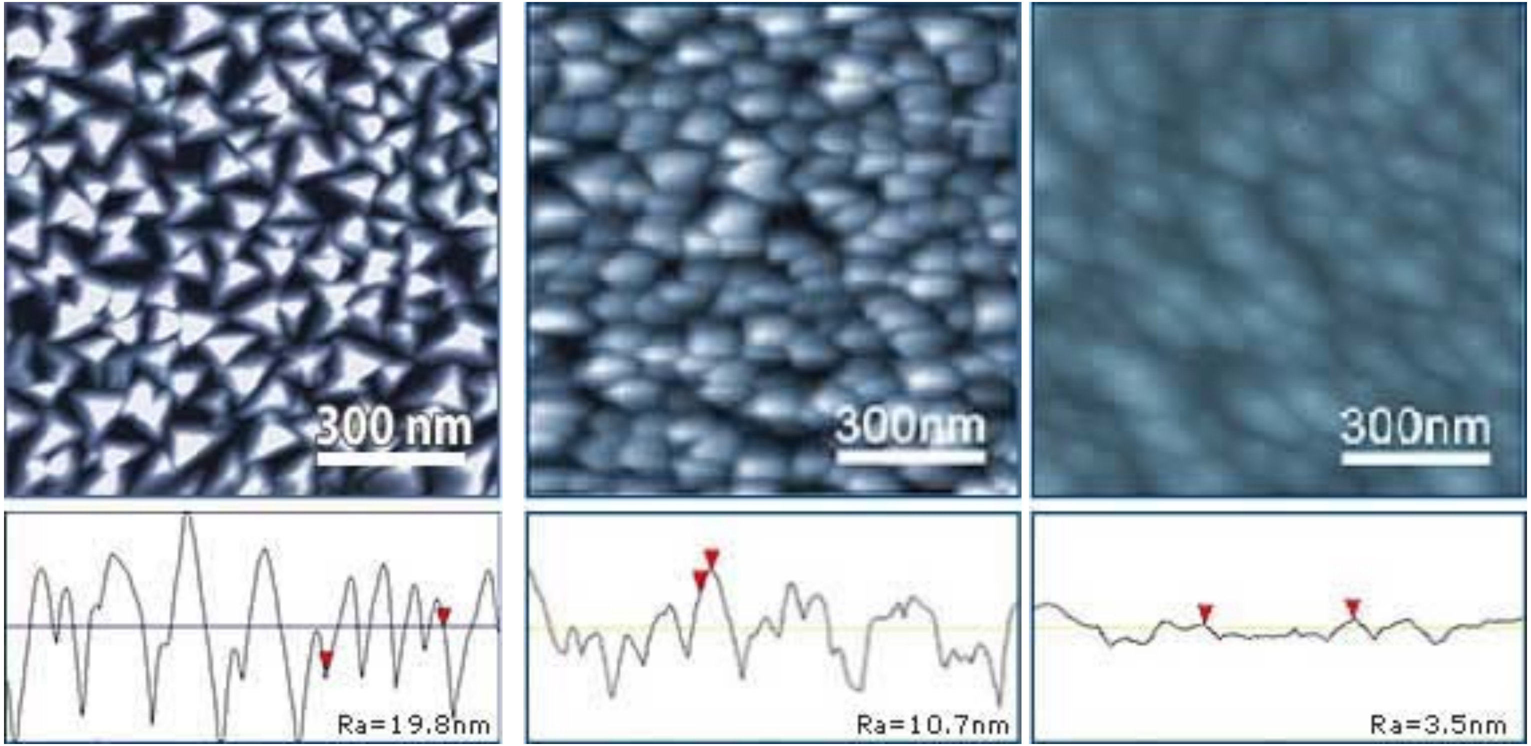


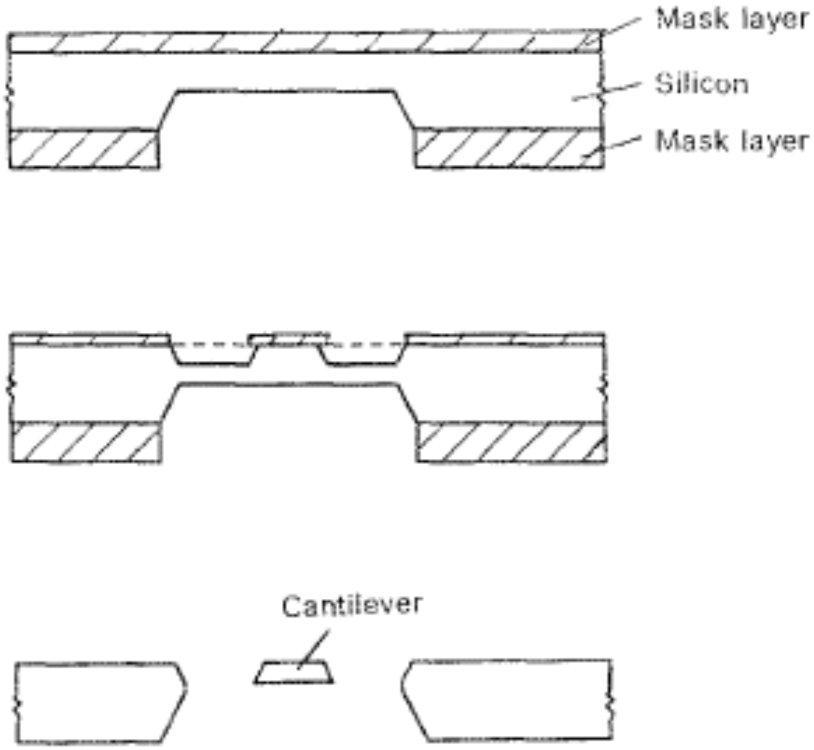
Image resolution and artefacts

Image resolution

Influence of tip sharpness



Cantilevers are made by microfabrication

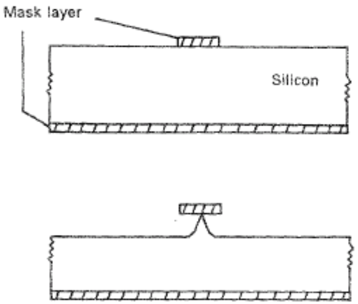


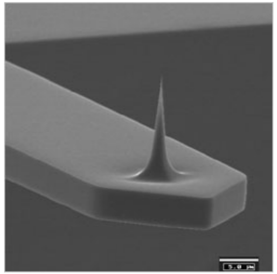
Wolter, Bayer, Greschner, JVST B vol. 9 (1991)

- Cantilevers can be made of a variety of materials: Silicon, silicon nitride, quartz, metals, polymers,…
- Wafer scale micromachining makes them (relatively) inexpensive
- Different methods exist to make sharp tips

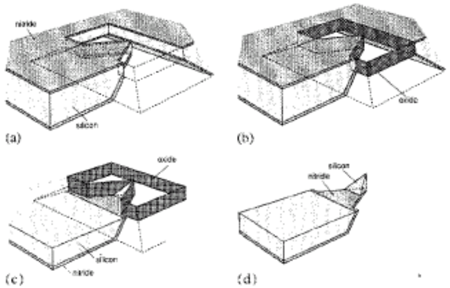
Integration of tips onto the cantilever

Dry etched tips

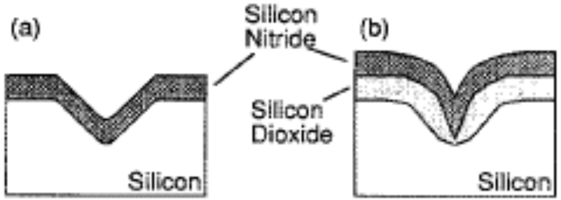




Beaked tips

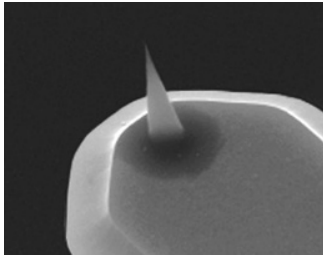


Molded tips



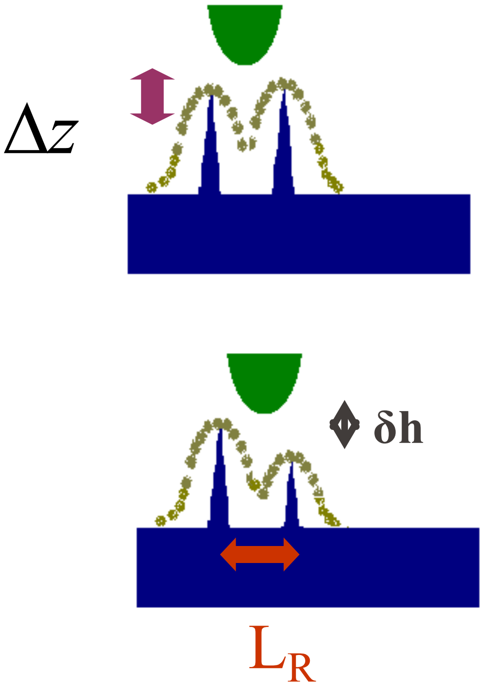
Akamine, Barrett, Quate, APL, vol. 57 (1990)
Akamine and Quate, JVSTB, vol. 10 (1992)

Deposited tips



Nanoworld AG

Resolution depends also on sample



- Resolution depends on:
  - Instrument noise floor (Δz)
  - Tip radius (R)
  - Sample topography (δh)

$$L_R = \sqrt{2R}\left(\sqrt{\Delta z} + \sqrt{\delta h + \Delta z}\right)$$

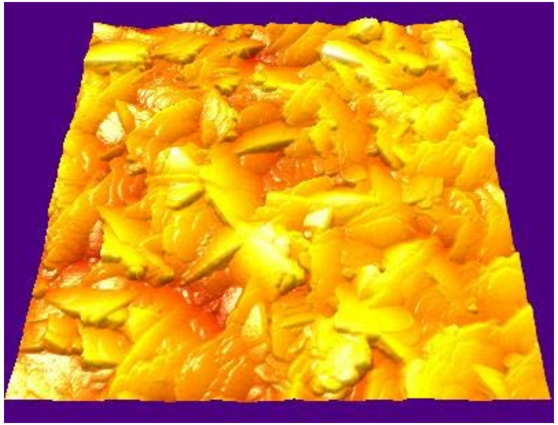
- If the sample is compliant the resolution is even further reduced by the indentation (l):

$$l = 2\left[\frac{3RF}{4E^*}\right]^{1/3}$$

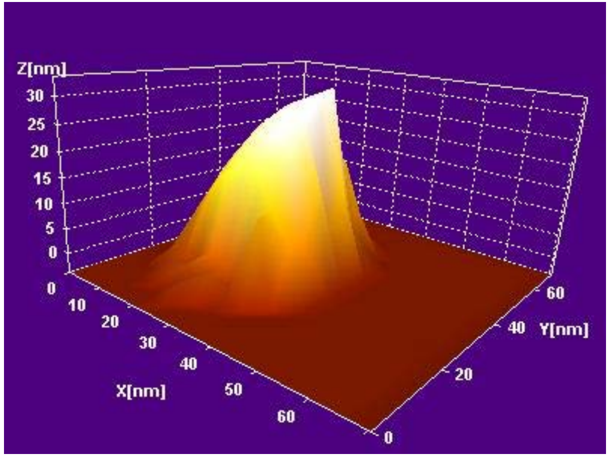
Estimation of tip radius

Blind tip reconstruction

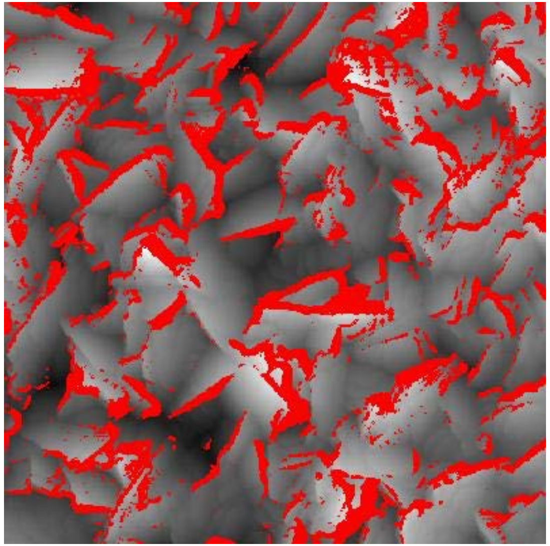
Image sample with sharp features



Calculate “blind tip reconstruction”

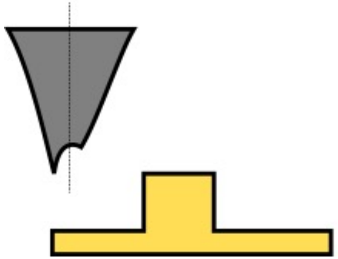


Calculate uncertainty map



Tip artefacts

Draw the height profile the tip would measure

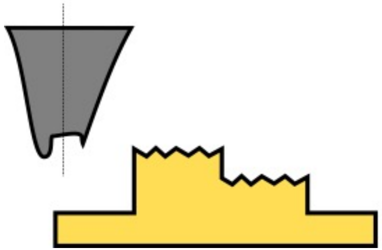


Tip artefacts

Draw the height profile the tip would measure

Tip artefacts

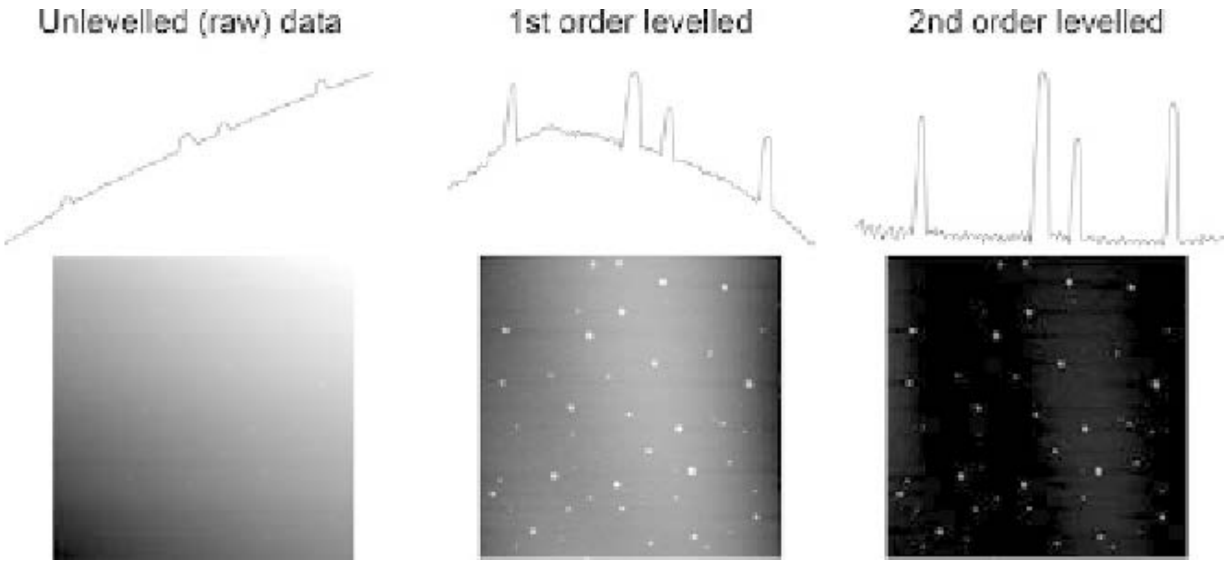
Draw the height profile the tip would measure



AFM image processing

Line flattening and plane subtraction

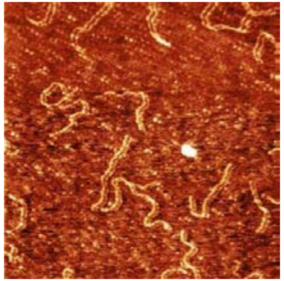
- The raw data that we get from the AFM often needs to be processed before we can see what we want to see.
- One main issue is that if the sample isn't perfectly level, we will have a slant in our image.



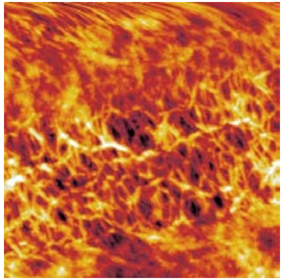
AFM image artefacts

Always be critical when interpreting your AFM images!

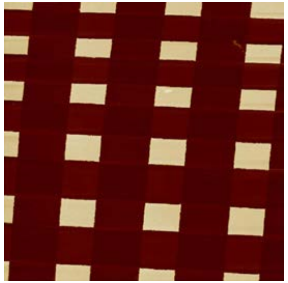
Double tip



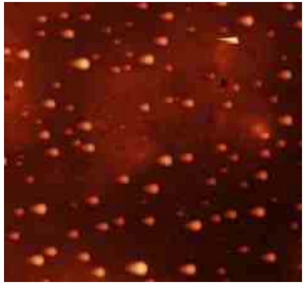
Piezo creep



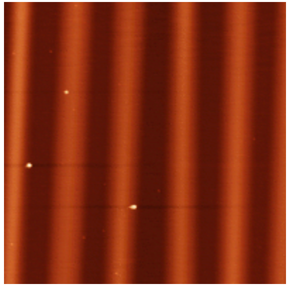
Piezo hysteresis



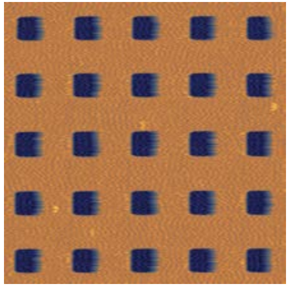
Parashooting



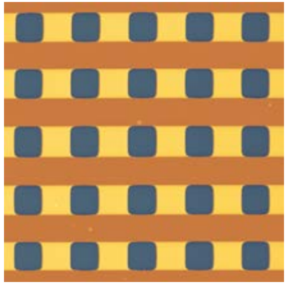
Laser interference



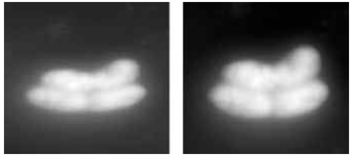
Gains too high



Bad image processing



Sample drift



References & Acknowledgements

- Atomic Force Microscopy, by Peter Eaton and Paul West, Published by Oxford University Press
- Most animations are from the NT-MDT webpage
- Bruker manuals and application notes
- Some material courtesy of:
  - Dr. Sidney Cohen, *Weizmann Institute of Science*
  - Dr. Daniel Burns, *Massachusetts Institute of Technology*
  - *Many other sources from the web, named or unnamed*

Having fun…

Low tech AFM