With the help of…….

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Outline AFM:

1. Examples, links and homework
2. AFM principle and structure
3. Tip-surface interactions
4. Feedback techniques
5. Force-distance curves and modes of operation
6. The scanner
7. Tip convolution and resolution
8. Cantilevers and tips
Books and Internet Sites

“Scanning Probe Microscopy and Spectroscopy”, R. Wiesendanger
“Scanning Force Microscopy”, D. Sarid

http://www.embl-heidelberg.de/~altmann/

http://www.eng.tau.ac.il/~yosish/courses.html
http://www.eng.tau.ac.il/~yossir/course/
http://www.chembio.uoguelph.ca/educmat/chm729/STMpage/stmtutor.htm
http://www.weizmann.ac.il/surflab/peter/afmworks/index.html
http://www.cmmp.ucl.ac.uk/~asf/physics/Ncafm.html
http://spm.phy.bris.ac.uk/welcome.html
http://www.physik.uni-ulm.de/en_wissensch.htm
http://chemistry.jcu.edu/mwaner/research/afm/
http://spm.aif.ncsu.edu/tutorial.htm

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Homework 6

1. Read the paper:
   “The millipede – more than one thousand tips for future AFM data storage”
   - Emphasize the “lithography” part.

2. Find on the web, in a paper or in a book the 3 most impressive SPM (not STM) images:
   a. 1 - Technically
   b. 1 - Scientifically
   c. 1 - Aesthetically
   Explain your choice. If needed compare with additional images.

3. For “Maskianim” – Read the “practical Guide for SPM” (in my notes or in: http://www.topometrix.com/spmguide/contents.htm)
The Principle of Operation
Schematic of Generalized AFM

- Means of sensing the vertical position of the tip
- A feedback system to control the vertical position of the tip
- A piezoelectric scanner which moves the sample under the tip (or the tip over the sample) in a raster pattern
- A coarse positioning system to bring the tip into the general vicinity of the sample
- A computer system that drives the scanner, measures data and converts the data into an image.
Contact to Surface...
Monitored by Laser...
Obtaining Surface Profiles

The tip is attached to a piezoelectric and scans the surface.
SFM Block Diagram

- SPM tip
- Piezoelectric scanner
- HV Amplifiers and signal conditioning
- Signal detector
- SPM Signals
- SFM 3 dimensional image of a tumor cell HeLa (37x37µm²)
- Personal Computer
- Digital Signal Processor
SFM Head

Vibration isolation + Stiffness

- Laser diode
- Beam adjustment
- Coarse approach
- Window for optical microscope
- Photodiode
- Photodiode adjustment system
- Piezoelectric scanner

1cm
Vibration Isolation

\[ \omega \propto k^2 \]

\[ \omega_d \text{ as small as possible (2Hz)} \]
\[ \omega_m \text{ as high as possible (2kHz)} \]

Stiffness scales with size. SPM as small as possible.
Binnig, Quate, and Gerber invented the AFM in 1986 mainly due to the limitations of the STM.

The first AFM image was $\text{Al}_2\text{O}_3$ imaged with a diamond tip mounted on a gold cantilever, and tunneling was used for force detection.
AFM Images

Magnetic bits of a zip disk

G4-DNA

Nanotube between electrodes

DNA-Nanotube
The Microcantilever and Hook’s Law

\[ F = -kz, \quad k = \frac{E}{4} \cdot \frac{W}{T/L^3} \]

- \( F \) is the force
- \( k \) is the spring constant
- \( E \) is the Young modulus
- \( W \) is the width
- \( T \) is the thickness
- \( L \) is the length

\( k \) depends on the geometry and material

\( E \) - Young modulus, \( W \) - width, \( T \) - thickness, \( L \) - length
More about springs

The resonance frequency of the cantilever also depends on the geometry and material, for a rectangular cantilever:

\[ \omega_0 = 0.162 \cdot \frac{(E/\rho)^{1/2}}{T/L^2} \]

\[ D(\omega) = \frac{A}{\sqrt{\left(\omega_0^2 - \omega^2\right)^2 + 4 \cdot \beta^2 \cdot \omega^2}} \]

The graph shows the normalized displacement versus frequency for different values of Q:
- Q=100 (in air)
- Q=50
- Q=10 (in liquids)

The driving force is given by \( F_0 \cos(\omega t) \).
**Tip-Surface Interaction**

- **Potential**
  - $U(z)$
  - Tip-sample distance
  - $F > 0$ Repulsive
  - $F < 0$ Attractive

- **Force**
  - $|F_{vdw}| = |F_{ion}| + |F_{el}|$
  - Intermittent contact
  - Distance (tip-to-sample separation)
  - Contact
  - Non-contact
  - Attractive force
  - Repulsive force
Forces During Approach

- Force
- Repulsion
- Attraction
- Contact
- Snap-in
- No force action
- Cantilever-sample distance
- Pull-off
AFM Principle and Tip-Surface Interaction

\[ w(r) = \frac{B}{r^{12}} - \frac{A}{r^6} \Rightarrow F(r) \equiv -\frac{dW}{dr} = 12B/r^{13} - 6A/r^7 \]
Tip-Surface Interaction

- **Repulsion forces**: incomplete screening of nuclear charge, Pauli exclusion principle
- Due to the sharpness and size of the tip, long range forces (like electrostatic) and hence many-body-interactions play the dominant role.
- **Electrostatic forces** (attractive) play the dominant role at distances greater than 10 nm (simple parallel-plate capacitor model)
The Two Springs Model

The distances represent:
- z-tip-sample distance, d-cantilever deflection, $\Delta$-cantilever-sample distance (=piezo movement)
- $\Delta_z=d+z$
Equilibrium Situation

The total energy:

\[ V_{\text{tot}}(z, \Delta) = V_{\text{surf}}(z) + \frac{1}{2} C_{\text{lev}}(\Delta - z)^2 \]

\( C_{\text{lev}} \) – Cantilever spring constant

The position of the tip for an equilibrium situation is determined by the force balance:

\[ F(z, \Delta) \equiv \frac{\partial V_{\text{tot}}}{\partial z} = \frac{\partial V_{\text{surf}}}{\partial z} + C_{\text{lev}}(\Delta - z) \]

The stability condition can be obtained from:

\[ C_{\text{eff}}(z, \Delta) \equiv \frac{\partial^2 V_{\text{tot}}}{\partial z^2} = + \frac{\partial^2 V_{\text{surf}}}{\partial z^2} + C_{\text{lev}} \geq 0 \]
Dynamic SFM

The cantilever is oscillated at its resonance frequency:

Two terms must be added:

- External excitation force
- Damping term (proportional to the tip velocity)

The equation of motion:

\[ m \frac{\partial^2 z}{\partial t^2} = \frac{\partial V_{\text{surf}}}{\partial z} + C_{\text{lev}}(\Delta - z) + F_{\text{ext}}(t) - \frac{m \omega_{00}}{Q} \frac{\partial z}{\partial t} \]

Q – quality factor, \( \omega_{00} \) – resonance frequency

The external force:

\[ F_{\text{ext}}(t) = F_0 \cos(\omega_{00} t) \]
Dynamic SFM

The resonance frequency for a small free oscillation:

$$\omega_{00} = \sqrt{\frac{C_{\text{lev}}}{m_{\text{eff}}}}$$  \hspace{1cm} m_{\text{eff}} \approx 0.24 \ m_{\text{lever}}$$

A quadratic interaction potential changes the effective spring constant:

$$V(z) = C_{\text{pot}} z^2 \quad \rightarrow \quad C_{\text{eff}} = C_{\text{lev}} + V''$$

The resonance frequency shifts:

$$\omega_0 = \sqrt{\frac{C_{\text{eff}}(z)}{m_{\text{eff}}}} = \omega_{00} \sqrt{1 + \frac{C_{\text{pot}}}{C_{\text{lev}}}}$$
Amplitude and Phase Curves for a Damped Harmonic Oscillator

However .... In reality the potential is not quadratic and the oscillation is not small .... 😞

So .... more complicated non-linear models have to be applied... 😊
Relevant Interactions in SFM

The vibration frequency of atoms, $\omega$, at room temperature $\sim 10^{15}$ Hz

The mass, $m$, of an atom $\sim 10^{-30}$ kg

The effective spring constant, $k$, between atoms is:

$$k = \omega^2 m \approx 1 \text{N/m}$$
Relevant Interactions in SFM

Repulsive interaction and contact force:

- At very small tip-sample distances (a few angstroms) a very strong repulsive force appears between the tip and sample atoms. Its origin is the so-called exchange interactions due to the overlap of the electronic orbitals at interatomic distances.

- When this repulsive force is predominant, the tip and sample are considered to be in “contact”.
Relevant Interactions in SFM

Van der Waals:

- A polarization interaction between atoms: An instantaneous polarization of an atom induces a polarization in nearby atoms - and therefore an attractive interaction.
Relevant Interactions in SFM

**Magnetic interaction:**
- Caused by magnetic dipoles both on the tip and the sample. This interaction is used for Magnetic Force Microscopy to study magnetic domains on the sample surface.

**Electrostatic interaction:**
- Caused by both the localized charges and the polarization of the substrate due to the potential difference between the tip and the sample.
- It has been used to study the electrostatic properties of samples such as microelectronic structures, charges on insulator surfaces, or ferroelectric domains.
Relevant Interactions in SFM

Friction and adhesion:

- The SFM cantilever bends laterally due to a friction force between the tip and the sample surfaces.

- Adhesion can be defined as “the free energy change to separate unit areas of two media from contact to infinity in vacuum or in a third medium”.

- In general, care has to be taken with the term adhesion since it is also used to define a force - the adhesion force, as for example in SFM.

- In SFM at ambient conditions in addition to the intrinsic adhesion between tip and sample there is another one from the capillary neck condensing between them. Then, the pull-off force is considered as the adhesion force, which is in the range of a few nanonewton to tens of nanonewton.
Figure 2.6 (a) Complete wetting. Partial wetting (b, c) of a droplet on a surface. Case (c) is nearer to the non-wetting case, being the contact angle larger than in (b).
The snap-in distance increases depending on the relative humidity, up to 10-15 nm.

\[ F_{\text{adh}} = 4\pi R(\gamma_{\text{lg}} \cos \theta + \gamma_{\text{sl}}) = 4\pi R \gamma_{\text{lg}} \]

\( \theta \) - contact angle, \( \lambda_{\text{sl}}, \lambda_{\text{lg}}, \lambda_{\text{lg}} \) - surface energies at solid/liquid, liquid/gas and solid/gas interfaces.
Feedback Techniques
Detecting Cantilever Deflection

- a) STM tip
- b) Capacitance
- c) Beam deflection
Cantilever Deflection Measurement: Tunneling

Disadvantages:
- Difficult alignment
- Sensitivity of ~0.01 Å, but extremely sensitive to surface conditions
- Thermal drifts, local changes in barrier height affect force measurements

Figure 2.12. Early contact AFM which allowed imaging non-conductive samples. In this scheme, a contact AFM tip was monitored using the STM tip directly above it.
The Beam Deflection Method

(a) Normal force
- UP: A + B = UP
- Down: C + D = DOWN

(b) Lateral Force
- Left: A + C = LEFT
- Right: B + D = RIGHT

Photodiode
Laser
The Feedback in SFM

Piezoelectric material: changes its shape when an electric potential is applied

The normal force is kept constant using the feedback

Advantages:

- Negligible force on cantilever
- Not very sensitive to cantilever surface
- Disadvantage: Sample illumination
Force-Distance Measurements and modes of Operation
This technique is used to measure the normal force vs. distance.

It can be used to measure similarly any other quantity: Lateral force, electrostatic force etc.

Can be used to study contaminations, viscosity.....
Forces vs. Distance

- This technique is used to measure the normal force vs. distance.

- A force vs. distance curve is a plot of the deflection of the cantilever versus the extension of the piezoelectric scanner, measured using a position-sensitive photodetector.

- It can be used to measure similarly any other quantity: Lateral force, amplitude, phase, current etc.

- Can be used to study contaminations, viscosity, lubrication thickness, and local variations in the elastic properties of the surface.

- Contact AFM can be operated anywhere along the linear portion of the force vs. distance curves.
Forces-Distance at Various Environments

Water

Air

Air+contamination
In contact mode the deflection of the cantilever is kept constant.

In dynamic mode the tip is oscillated at the resonance frequency and the amplitude of the oscillation is kept constant.
AFM: Non-contact and Tapping

- Non-Contact and Tapping: frequency of operation
- Feedback Mechanism
- Tapping: provides high resolution with less sample damage
AFM: Dynamic Mode
Figure 3.5 Force vs. distance curve for an oscillating cantilever. In region $A$ the cantilever oscillates freely, while in $B$ the interaction between the tip and the sample produces an amplitude reduction. The tip snaps into the sample at point $C$. In region $D$ the tip and the sample are in contact, the interaction is repulsive and the oscillation amplitude is zero. Line $n$ corresponds to the surface position while line $m$ represents the minimum position of the cantilever oscillation. Since $m$ and $n$ are not coincident the tip is not touching the surface at the interaction region and the SFM is operating in the non-contact mode.
Contact mode imaging (left) is heavily influenced by frictional and adhesive forces which can damage samples and distort image data. Non-contact imaging (center) generally provides low resolution and can also be hampered by the contaminant layer which can interfere with oscillation. Tapping Mode imaging (right) eliminates frictional forces by intermittently contacting the surface and oscillating with sufficient amplitude to prevent the tip from being trapped by adhesive meniscus forces from the contaminant layer. The graphs under the images represent likely image data resulting from the three techniques.
Contact vs. Tapping – Si (100)

- **Tapping Mode** images show no surface alteration and better resolution.

- **Contact imaging** shows clear surface damage. Material has been removed by the scanning tip, while in other cases, additional oxide growth or more subtle changes may occur.

- This type of surface alteration often goes undetected since not all researchers check for damage by rescanning the affected area at lower magnification.
Non-Contact vs. Contact Through Water

Non-Contact

Contact
**Constant Height and Constant Force**

**Constant Height:**

- In constant-height mode, the spatial variation of the cantilever deflection can be used directly to generate the topographic data set because the height of the scanner is fixed as it scans.

- Constant-height mode is often used for taking atomic-scale images of atomically flat surfaces, where the cantilever deflections and thus variations in applied force are small. Constant-height mode is also essential for recording real-time images of changing surfaces, where high scan speed is essential.

**Constant Force:**

- In constant-force mode, the deflection of the cantilever can be used as input to a feedback circuit that moves the scanner up and down in z, responding to the topography by keeping the cantilever deflection constant. In this case, the image is generated from the scanner’s motion. With the cantilever deflection held constant, the total force applied to the sample is constant.

- In constant-force mode, the speed of scanning is limited by the response time of the feedback circuit, but the total force exerted on the sample by the tip is well controlled. Constant-force mode is generally preferred for most applications.
Magnetic force microscopy (MFM) images the spatial variation of magnetic forces on a sample surface.

MFM operates in non-contact mode, detecting changes in the resonant frequency of the cantilever induced by the magnetic field’s dependence on tip-to-sample separation.
Lateral Force Microscopy (LFM)

LFM measures lateral deflections (twisting) of the cantilever that arise from forces on the cantilever parallel to the plane of the sample surface.

LFM images variations in surface friction, arising from inhomogeneity in surface material, obtains edge-enhanced (slope variations) images of any surface.

To separate the effects AFM and LFM should be used simultaneously.
Force Modulation Microscopy (FMM)

- In FMM mode, the tip is scanned in contact with the sample, and the z feedback loop maintains a constant cantilever deflection (as for constant-force mode AFM).
- A periodic signal is applied to either the tip or the sample. The amplitude of cantilever modulation that results from this applied signal varies according to the elastic properties of the sample.
- The system generates a force modulation image, which is a map of the sample's elastic properties, from the changes in the amplitude of cantilever modulation.
- The frequency of the applied signal is on the order of hundreds of kHz, which is faster than the z feedback loop is set up to track. Thus, topographic information can be separated from local variations in the sample's elastic properties, and the two types of images can be collected simultaneously.
FMM of Carbon fiber/polymer Composite Collected Simultaneously (5µm)

Contact AFM  

FMM
Phase detection monitors the phase lag between the signal that drives the cantilever to oscillate and the cantilever oscillation output signal.

Phase imaging is used to map variations in surface properties such as elasticity, adhesion and friction.

Phase detection images can be produced while an instrument is operating in any vibrating cantilever mode.

The phase lag is monitored while the topographic image is being taken so that images of topography and material properties can be collected simultaneously.
Phase Imaging of an Adhesive Label (3 µm)

Non-contact AFM

Phase image
In EFM voltage is applied between the tip and the sample while the cantilever hovers above the surface, not touching it.

The cantilever deflects when it scans over static charges. The magnitude of the deflection, proportional to the charge density, can be measured with the standard beam-bounce system.

EFM plots the locally charged domains of the sample surface.

EFM is used to study the spatial variation of surface charge carrier density. For instance, EFM can map the electrostatic fields of an electronic circuit as the device is turned on and off.
Figure 2.3 Electrostatic force vs. tip-sample distance. The thick solid curve corresponds to the total electrostatic force, while the other three represent separately each of the contributions form the cantilever (dash line), tip cone (short dash) and tip apex (thin solid). These curves have been calculated for $U=1$ volt, and a probe with dimensions of 100 µm long and 25 µm wide cantilever, 3µm long tip and a radius of 20 nm for the tip apex. The angle for the tip is $\pi/4$ and an angle of $\pi/8$ was assumed between the cantilever and the sample. These curves were obtained following the model proposed in [Colcherio submitted].
Additional Force Microscopy Techniques

- Scanning capacitance microscopy (SCM)
- Scanning thermal microscopy (SThM)
- Near field scanning optical microscopy (NSOM)
- Kelvin probe microscopy
- Scanning spreading resistance microscopy
- Contact Potential Difference (CPD)
- Pulsed force mode (PFM)
- 3D mode
- Jumping mode
The Scanner
Ideally, a piezoelectric scanner varies linearly with applied voltage.

\[ S = dE \]

- \( S \) - Strain [Å/m], \( d \) - Strain coefficient [Å/V], \( E \) - Electric field [V/m]
Ideally, the intrinsic nonlinearity is the ratio $\Delta y/y$ of the maximum deviation $\Delta y$ from the linear behavior to the ideal linear extension $y$ at that voltage.

It is in the range 2-25%.
The **hysteresis** of a piezoelectric scanner is the ratio of the maximum divergence between the two curves to the maximum extension that a voltage can create in the scanner: $\Delta Y/Y_{\text{max}}$. Hysteresis can be as high as 20% in piezoelectric materials.
When an abrupt change in voltage is applied, the piezoelectric reacts in two steps: the first step takes place in less than a millisecond, the second on a much longer time scale. The second step, $\Delta x_c$, is known as creep.

Creep is the ratio of the second dimensional change to the first: $\Delta x_c / \Delta x$. It ranges from 1% to 20%, over times of 10 to 100 sec.
The *aging* rate is the change in strain coefficient per decade of time.

The piezoelectric coefficient, $d$, changes exponentially with time: increase with regular use, decrease with no use.
**Scanner Cross Coupling**

- **Geometric effect:** The x-y motion of a scanner tube is produced when one side shrinks and the other expands.
- Can be corrected with image-processing software, checked using a sample with a known curvature (lens).
Software Correction
Hardware Correction

A sensor “reads” the scanner actual position, and a feedback system applies voltage to drive the scanner to the desired position, the total nonlinearity can be reduced to 1%.
Tip Convolution and Resolution
Tip Convolution

This profile... can be made with this monster...

or with this bug!
Tip Convolution - example

AFM image
Nominal width 180 nm

Cross section
Measured width 350 nm
Resolution

- Tip convolution is not linear: results do not add up!!!
- The resolution depends on tip AND sample.
Resolution: apparent width

\[ x^2 = (R_{\text{tip}} + R_{\text{sample}})^2 - (R_{\text{tip}} - R_{\text{sample}})^2 \]
\[ x^2 = \sqrt{R_{\text{tip}}^2 + 2R_{\text{tip}}R_{\text{sample}} + R_{\text{sample}}^2} - \sqrt{R_{\text{tip}}^2 + 2R_{\text{tip}}R_{\text{sample}} - R_{\text{sample}}^2} \]
\[ x = 2\sqrt{R_{\text{tip}}R_{\text{sample}}} \]
\[ w = 2x = 4\sqrt{R_{\text{tip}}R_{\text{sample}}} \]

DNA: 2 nm, \[ \text{tip} \sim 20 \text{ nm} \Rightarrow w = 25 \text{ nm} \]
\[ \text{tip} \sim 10 \text{ nm} \Rightarrow w = 18 \text{ nm} \]
Convolution Test by Rotation

- **Tip imaging**: the image does not change upon rotation.
- **True imaging**: the image rotates upon scan direction rotation.
Additional Artifacts

- Convolution with other “physics” (sample charging, stiffness, contamination etc.)
- Feedback artifacts
- External noise and fields
- ....
Tests for Artifacts

- Repeat the scan to ensure that it looks the same.
- Change the scan direction and take a new image.
- Change the scan size and take an image to ensure that the features scale properly.
- Rotate the sample and take an image to identify tip imaging.
- Change the scan speed and take another image (especially if you see suspicious periodic or quasi-periodic features).
Cantilevers and Tips
The Force Sensor – The Tip

(a)

(b)

(c)

(d)

\[ \alpha, \alpha' \]
The Force Sensor – The Tip

\[ k = \frac{Ewt^3}{4l^3}, \]

\[ E \quad \text{- Young Module} \]

\[ w \quad \text{- width,} \quad l \quad \text{- length,} \quad t \quad \text{- thickness} \]

Comercial cantiliver \( s = SiO_2 \) and \( Si_3N_4 \)

Tip radii \( - 300 \) \( \AA \)
The Force Sensor – The Tip
The Force Sensor – The Tip

Diamond-coated tip

FIB-sharpened tip

Gold-coated Si$_3$N$_4$ tip
Tip Fabrication

- Pit etching in Si
- SiN coating
- Si underetching
Tip Resolution

1. Tip is rastered across surface
2. Surface atoms
3. Missing atom
4. Signal traces (signal vs. time) for tip atoms 1, 2, and 3
5. Sum signal trace:
Sample Wear Over Time

Conventional Probe (new)
- Initially
- After 1 hour
- After 15 hours

ProbeMAX Nanotube Probe
- Initially
- After 1 hour
- After 9 hours

All scans 1µm, intermittent-contact mode, SiGe QDs on Si(001), some drift

www.piezomax.com
Summary

- SFM is a powerful tool for surface imaging.
- SFM covers various techniques that enable many physical measurements of interactions with the surface.
- SFM interpretation is not straightforward.