Scanning Probe Microscopy
AFM and SNOM

**Introduction to SPM**
- Scanning Tunneling Microscopy (STM)
- Atomic Force Microscopy (AFM, SFM)
  - Primary operation modes
  - Artifacts
  - Primary and Secondary imaging
  - Scanning Near Field Microscopy (SNOM)

**Application example**
- Piezoelectric writing and imaging of a polymer

**Force spectroscopy**
- Interpretation of force curves
- Examples from literature
X = Tunneling current  STM (1981)
X = Force interaction  AFM (1986)
X = Near Field Optical Interaction  SNOM
Scanning Tunneling Microscopy (STM)

Current increase approx. one order of magnitude for each Å of approach.

Ideal for high resolution mapping!

$I \propto \exp(-2\kappa d)$

$\kappa = \sqrt{\frac{2m\Phi_B}{h^2}} \approx 1.1Å^{-1}$

d is tip - surface separation

$\Phi_B$ : Barrier height

$\kappa$ : Inverse decay length

Constant current image of Si(111)7×7
Inherent advantages of SPM techniques:

- Can be used to manipulate nano-size objects
- Can often operate in vacuum, air and liquid

Extreme example of manipulation. Atom corrals presented by Don Eigler and coworkers, IBM Almaden Research Center.
Atomic Force Microscope, AFM (SFM)

Force interaction is more complex than tunneling current, and have both repulsive and attractive components.
<table>
<thead>
<tr>
<th>Interaction</th>
<th>Sign</th>
<th>Range</th>
<th>Sphere/flat expression</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Born force</td>
<td>repulsive</td>
<td>~1Å</td>
<td>Complex</td>
<td>Always present</td>
</tr>
<tr>
<td>Valence interaction (&quot;chemical forces&quot;)</td>
<td>repulsive</td>
<td>~3Å</td>
<td>Complex</td>
<td>Quantum mechanic</td>
</tr>
<tr>
<td>Van der Waals (dispersion forces)</td>
<td>normally attractive</td>
<td>~100Å</td>
<td>Dipole interaction always present</td>
<td></td>
</tr>
<tr>
<td>Electrostatic</td>
<td>normally attractive</td>
<td>long range</td>
<td>Contact potential often present</td>
<td></td>
</tr>
<tr>
<td>Magnetic</td>
<td>repulsive</td>
<td>long range</td>
<td>Complex</td>
<td>If tip and sample are magnetized</td>
</tr>
<tr>
<td>Capillary force</td>
<td>attractive</td>
<td>~100Å</td>
<td>In humid atmosphere</td>
<td></td>
</tr>
<tr>
<td>Double layer force</td>
<td>normally repulsive</td>
<td>~1000Å</td>
<td>In liquid when surfaces charged</td>
<td></td>
</tr>
<tr>
<td>Hydrophobic force</td>
<td>repulsive</td>
<td>long range</td>
<td>Complex</td>
<td>Water mediated</td>
</tr>
<tr>
<td>Steric force</td>
<td>repulsive</td>
<td>~50Å</td>
<td>Complex</td>
<td>In liquids</td>
</tr>
</tbody>
</table>
AFM cantilevers

The cantilever-tip units are normally micro-machined of Si or Si$_3$N$_4$.

Characterized by:
- Tip radius (typ. 10 nm)
- Spring constant (0.1-100 N/m)
- Resonance frequency: (5-500 kHz)

Different coatings available
Magnetic cantilevers available
Tip shape and resolution

If the interaction is short ranged like tunneling current, the resolution is determined by a micro-tip. This makes it often easy to make STM tips that give atomic resolution on atomically flat terraces.

If the sample structure is larger, the macroscopic tip geometry (radius an cone angle) is critical.
Artifacts to look out for in AFM

Important classes of artifacts:

- Tip geometry dependent artifacts

- Force induced artifacts

- In ambient conditions, the sample surface is covered by a liquid layer, causing capillary forces.
Examples of artifacts

TEM image

AFM image
bad tip

AFM image
god tip

Palladium islands on SiO₂

AFM image of plasmid DNA. The width of the double-helix is approx. 2 nm. In the image the width is approx. 15 nm.
Primary operation modes

Contact Mode
- dc operation
- $F = k \times X$, where $X$ is lever deflection and $k$ spring constant.
- $z = f(x,y)$ at const. $F$

Non-contact Mode
- ac operation
- small amplitude
- $z = f(x,y)$ at const. $F'$
- Used for long-range forces like electrostatic and magnetic

Intermittent-contact Mode ("Tapping Mode")
- ac operation
- large amplitude
Block diagram of AFM

DEFLECTION DETECTOR → LOCK-IN AMPLIFIER

PIEZO → FREQUENCY GENERATOR

Tip oscillates 5-15 Å

PIEZO SCANNER

Vertical positioning

Horizontal positioning

FEEDBACK AND DATA TREATMENT → HORIZONTAL SCAN GENERATION

IMAGES

RESONANCE INFORMATION

Amplitude [Amp] vs. Frequency shift [Hz]

Phase vs. Frequency [Hz]
Scanner configurations for AFM

Piezoelectric tube-scanner
Primary/Secondary image
Example: Topography and Phase information

![Diagram showing the components of a primary/secondary image system with labels for PZT, Scanner, Amplitude detector, Feedback, Δφ, Phase image, and z, topography.]
Lift mode

Two stage measurement:

- First the topography is recorded using tapping mode or contact mode.
- System makes a retrace without feedback control at a pre-defined height during which the long-range force is measured using an ac non-contact method.

-Suitable for long range forces like electrostatic and magnetic
Scanning Near-Filed Optical Microscopy, SNOM (NSOM)

In a SNOM, the tip is made from a tapered optical fiber with a small aperture at the end.

The surface is traced using an ac mode AFM method.

As a "secondary image", the transmitted or reflected light can be recorded. The probe can also be used for light collection.
As the optical near-field is generating the image, the Abbe limit that restricts the resolution of a normal optical microscope to $\lambda/2$ is defeated.

Comparison between AFM and SNOM images of 30 nm gold spheres.

(Nanonics Imaging Ltd)
### Acronyms

- **SPM** Scanning Probe Microscopy
- **STM** Scanning Tunneling Microscopy
- **STS** Scanning Tunneling Spectroscopy
- **AFM** Atomic Force Microscopy
- **SFM** Scanning Force Microscopy
- **LFM** Lateral Force Microscopy
- **FFM** Frictional Force Microscopy
- **FMM** Force Modulation Microscopy
- **EFM** Electrostatic Force Microscopy
- **MFM** Magnetic Force Microscopy
- **SCM** Scanning Capacitance Microscopy
- **SNOM** Scanning Near-field Optical Microscopy
- **NSOM** Near-field Scanning Optical Microscopy
- **SSRM** Spreading Resistance Microscopy
- **CAFMS** Conductive AFM
- **TUNA** Tunneling AFM
- **ECAFM** Electro Chemical AFM
- **AFAM** Acoustic AFM
Application example

Using the AFM to **create and image** ferroelectric domains in a polymer

![Diagram showing AFM tip, ferroelectric polymer, and conducting substrate with field distribution around the tip](image)
Piezoelectric force microscopy

5 kHz amplitude give piezo-respons signal
ac signal extracted by lockin

Piezoresponse

Topography (primary) contact mode

Dc for poling

5 kHz 1 V

5 kHz

Scanner

Feedback

Piezoelectric sample

Conducting substrate

Laser

Detector

Lock-in
The raw "Force curve" that is measured by an AFM is really a Force vs. Sample position curve. See figure to the left.

1: Sample presses on the tip. If the contact is stiff a straight line results.

2: The tip is pulled back but sticks to the surface. Adhesion.

3: The adhesion decrease stepwise until the tip finally is free.
Interpretation of force curves

True Force vs. separation curve

AFM ”Force curve”
Force vs. Sample pos.

Corrected exp. Force curve
Force vs. separation

Due to the snap-to-contact instability, it is normally not possible to trace out the complete interaction curve. The local pressure at the micro-tip will also make it impossible to obtain atomic resolution in contact mode.
Measurement in liquid

Cantilever mount plate
Glass window
Membrane
Force head
Cell frame
Baseplate
O-ring
Sample holder
Sample holder mount
Tube scanner
Biotin-avidin interaction studied by AFM

Florin, Moy and Gaub Science, 264, p 415 (1994)

A: Avidin on tip, biotin on surface
B: Most interaction sites blocked by excess of avidin
C: As B but other scale

Agarose bead attached to AFM cantilever

Histogram of height of last step in retract curve. A force quantum of $160 \pm 20$ nN was found from the statistics.
Force measurement in biology: Example from literature

AFM image of purple membrane

Force data

AFM image after rhodopsin molecule had been extracted

Principle side view of purple membrane

F. Oesterhelt, D. Oesterhelt, M. Pfeiffer, A. Engel, H. E. Gaub, D. J. Muller
Science 288 (2000)
Points are superposition of 17 force spectra.

Grey lines are from theoretical modelling.

F. Oesterhelt, D. Oesterhelt, M. Pfeiffer, A. Engel, H. E. Gaub, D. J. Muller
Science 288 (2000)
True atomic resolution in AFM

AFM, ac-mode

STM, empty states
STM, filled states

Lever deflection curve and Frequency spectra vs separation
Force-feedback

\[ F = C(\Delta U + U_0)^2 \]

Force feedback

Interferometer

Fiber

Metal coating

Sample

Piezo tube

Frame

W tip against Si(111) in UHV with feedback stabilized cantilever
Conclusions

- Versatile techniques due to the multitude of interactions that can be probed
- Operates in most environments
- Can image various properties and manipulate the sample on the nano-scale
- High resolution force measurements are important in many scientific fields