How Does Atomic Force Microscopy Work and What Can It Do?

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Atomic Force Microscopy
3D Optical Microscopy
Tribology
Automated AFM
Stylus Profilometry
Mechanical Testing, Nanoindentation

Innovation with Integrity
Scanning Probe Microscopy

- **STM-Scanning Tunneling Microscopy**
  (invented 1981, by Binnig and Rohrer at IBM Zurich)

- **AFM-Atomic Force Microscopy**
  While the primary use is imaging, the boundary has been pushed beyond.

**AFM Probes**

**STM Probe**

Si (111) 7x7 by STM
Atomic Force Microscope Modes
Primary Imaging Modes

AFM Revolutions center around force control.

- 1986 Contact Mode, Binnig (IBM), Quate, and Gerber
- 1993 Tapping mode, Digital Instruments
- 2009 PeakForce Tapping Mode, Veeco/Bruker
Beam deflection detection in the MultiMode Head
Contact Mode AFM

- Feedback loop maintains *constant deflection* (force) of AFM cantilever

- Constant contact between the tip and sample surface facilitates:
  - High resolution imaging
  - Fast scanning speeds / fast data acquisition times
Tapping Mode AFM

- AFM cantilever excited at fundamental resonance frequency
- Feedback loop maintains constant amplitude of oscillation of AFM cantilever

Fluid layer 10-100 nm

"Tapping"
PeakForce Tapping Mode

A force-distance curve is obtained in each tapping cycle with controlled peak force.
Fluid Imaging at 50 pN Peak Force
Gentle Touch and High Resolution

OmpG Membrane Pore (Data courtesy of C. Bippes and D. Muller, Dresden Univ.)
Simultaneously obtain quantitative data:

- Topography
- DMT Modulus
  - ~1MPa – 100GPa
- Adhesion
- Energy Dissipation
- Deformation
Multi-component polymer blend (7 µm scan)
Malaria-infected erythrocytes (IE’s, Red blood cell) are misshapen with knob-like structures on surface.

IE’s exhibit cytoadherence.

Prevents IE elimination by the spleen and causes vascular blockages.
PeakForce Tapping

*Resolving the DNA Double Helix*

*FastScan Bio AFM (FastScan-Dx probe, 0.25N/m)*

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**Consecutive images**

![Consecutive images](image)

**Unanchored parts (mobile)**

- **Low [Ni++]**
- **High [Ni++]**

**Movie of DNA on mica ([Ni++]**)

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 Bruker Nano Surfaces Division
ICON EC Setup

Scanner Head

Fluid Probe Holder

EC Cell

ICON EC Chuck w/Heater RT~65°
Lithium Battery: SEI on Si-Anode during Charging

a-Si coated with 200nm Cu with p-Si sputtered islands

Potential Ramp Profile:
1.5 --> 0.36

Potential Ramp Profile:
0.2 --> 1.5 --> 0.05V --> 1.5V
Atom Manipulation with LT-UHV-STM

Donald Eigler and Erhard Schweizer of the IBM Almaden Research Center in San Jose, California, used a scanning tunneling microscope, which can discern individual atoms, to position xenon atoms to form the initials "IBM". They shot a beam of xenon atoms at a chilled crystal of nickel. These were manipulated into patterns using the scanning tunneling microscope. In addition to the IBM initials, they created chains of xenon atoms similar in form to molecules.

Eigler, Schweizer. Nature 1990
Nano-Manipulation
Single Wall Nanotubes

1. Image with TappingMode. 2. Manipulate tubes with AFM tip. 3. Image again.

- All in just a few minutes
- Manipulation Mode: constant height (AFM tip) point-and-click, indicated by arrows

900nm
Secondary Modes
(Piggy Back Modes)
Conductivity Measurement

- Laser Diode
- Photodetector
- Sample
- Scanner
- Conductive AFM Probe
- Current Amplifier
- TUNA Module
- AFM
- Conductive Probe
Carbon Nanotubes with Peak Force TUNA

Sample courtesy of Prof. Hague, Rice University

Current map clearly identifies electrical connectivity of individual carbon nanotubes.
Point & Shoot I-V Curves on V-CNT
SSRM-Scanning Spreading Resistance Microscopy

Resistance Measurements

- Conductive probe
- (10 pA - 0.1 mA)
- \[ R = \frac{\rho}{4 \times \text{radius}} \]
SSRM on Si dopant staircase

- Carrier concentration (atoms/cm³)
- Resistance (Ω)

Graph showing depth (µm) along the x-axis and resistance and carrier concentration on the y-axis.
SSRM Resolution:
100nm PMOS & NMOS transistors
Scanning Capacitance Microscopy (SCM)

$\text{SiO}_2$

$\text{Si}$

=$\text{MIS}$ capacitor

Depletion

High Concentration

Accumulation

$\uparrow$ Low Concentration

$\Delta C$

$\Delta C$

$\text{capacitance}$

$\text{voltage}$

$\text{AC}$

$\text{AC}$
Scanning Capacitance Microscopy 
on LOCOS isolation between 2 transistors
KPFM-Kelvin Probe Force Microscopy

KPFM measures the work function difference of tip/sample.

<table>
<thead>
<tr>
<th>AM</th>
<th>Amplitude-Modulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>FM</td>
<td>Frequency-Modulation</td>
</tr>
<tr>
<td></td>
<td>✓ Better spatial resolution</td>
</tr>
<tr>
<td></td>
<td>✓ Better accuracy</td>
</tr>
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Physical Review B 2005, 71(12) 125424
PeakForce KPFM vs FM-AM
FM detection advantage maintained

FM sees larger and more localized contrast leading to **better accuracy**. AM contrast smaller and more convoluted.

Work functions: Sn 4.42 eV; Pb 4.25 eV

Sn$_{60}$Pb$_{40}$ Alloy
Some Trends

Smaller force (better force control)
Easier to use (ScanAsyst)
More information
Faster Scan
Higher resolution (routinely)
Chemical Identification
Trend: More Orthogonal Information
Simultaneous Morphology, Mechanical and Electrical Mapping (L333-Glue) with PF-TUNA

Li[Ni$_{1/3}$Mn$_{1/3}$Co$_{1/3}$]O$_2$
Trend: Faster for Dynamics and Productivity

Image Specifications:

- Size: 2.2um
- Scan Rate: 100Hz
- Pixels: 256 x 256
- Tip Velocity: 440um/s
- Frame Rate: 2.5s

Real Time Video Duration: ~4min
• Spherulite growth rates are consistent with earlier measurements (Hobbs2003)
  • Front growth rate of ~const for each temperature
• Secondary Nucleation model ignores variation in growth rate of individual lamella
• Rate of nucleation (T) affects spherulite structure
High Resolution AFM
A challenging task for instrumentation and operation

Lattice resolution of Mica, contact mode

Gross, Meyer, 2006
Low Temp UHV

Mica Tapping in liquid

Hoogenboom et al, 2006

Fukuma et al., 2005
Mica Tapping in liquid

Rode et al., 2009
Calcite Tapping in liquid

Atomic resolution with any probe on a flexible sample platform? fluid & ambient?
Trend: Higher Resolution AFM Routinely
Oxygen Atoms of Calcite
Dissolution crystal plane details revealed
Polydiacetylene (PDA) in Air
Multimode 8 Peak Force Tapping

- Peak Force Tapping provides much higher resolution
- Standard probes
- Material property maps

Atom  WdW R, nm
C  0.154
H  0.1
F  0.147
S  0.180
O  0.120

Trend: Nanoscale Chemical Probing
Nanoscale IR + Raman Spectroscopy
Leveraging Vibrational Spectroscopies

1. Use IR/Raman spectroscopy: Label-free, non-destructive chemical ID
2. Use tip as optical antenna: Isolate signal from nanoscale region

RESULT IS NANOSCALE CHEMICAL ID WITH ~ 10nm SPATIAL RESOLUTION
TERS Performance Benchmarking
Malachite Green on Au

- Malachite Green on Au, 633nm excitation, 1s integration, 1mW
- Signal contrast >100 (EF > 10^7) when raising tip 60nm
- High probe to probe consistency
- Innova-IRIS with IRIS TERS probe
Correlated Information on Graphene
Raman, IR-AFM and PeakForce KPFM

- **Raman**: how many layers, defects
- **QNM**: layer heights, mechanical properties
- **KPFM**: work function/fermi energy
- **IR AFM**: plasmonics, # of layers, fermi energy
Summary

- **AFM revolution centers around force control**: Contact mode, Tapping mode, to Peak Force Tapping mode.
- **AFM has opened the door** to the nanoscale world, making accessible a wide range of material properties, including but not limited to mechanical, electrical, magnetic, chemical properties.
- **AFM’s diverse uses** stem from its capability to work in *any environments*: ambient, liquid and vacuum.
- AFM has been widely adopted by the academic and industrial world alike.
- **AFM Trends**: Easier to use, Faster, Higher resolution, More information more quantitatively, and chemical identification (TERS, NanoIR).