Advanced Materials Characterization workshop

Atomic Force Microscopy

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go.illinois.edu/AMC2023
• How AFM works

• (A few) featured applications

• What can go wrong?
What's an Atomic Force Microscope?

"Atomic Force" Microscopy—forces between atoms in the tip and atoms in the sample

detector | laser source

cantilever

tip

(sample) (side view)

false-color surface topographs
What's an Atomic Force Microscope?

• "Atomic Force"—interactions between tip and sample
  • Sub-angstrom vertical resolution
  • Laterally, not quite atomic resolution (usually)
  • Nanoscale lateral resolution (depends on tip and sample)

• "Microscope"—surface topograph

measured step: 330 pm

HOPG (highly oriented pyrolytic graphite)
What’s an Atomic Force Microscope?

• “Atomic Force”—interactions between tip and sample
  • Sub-angstrom vertical resolution
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  • Nanoscale lateral resolution (depends on tip and sample)

• “Microscope”—surface topograph
  • Tip at the end of a cantilever
  • Raster tip over surface to build up an image

• Also sensitive to sample stiffness, adhesion, other properties depending on tip choice and instrument mode

Turquoise, 1μm x 1μm color overlay: mechanical phase
What is Atomic Force Microscopy Used For?

- 3D surface imaging
  - XYZ coordinates of sample surfaces (with excellent resolution)
  - Quantitative height maps of surfaces
  - Step height/thickness measurements
  - Roughness measurements
- Nanomechanical characterization
- Nanoscale electromagnetic characterization
- Nanoscale optical spectroscopy
- Nanomanipulation
Typical AFM Scales

(only what’s most common, not all of what’s possible)

- Sample sizes — mm to cm
- Image sizes — few to few tens of μm²
- Feature peak-to-valley — Å to μm (within scan area/field of view)
Typical AFM Scales

(only what’s most common, not all of what’s possible)

- Sample sizes — mm to cm
- Image sizes — few to few tens of $\mu$m$^2$ verbal shorthand 10$\mu$m, 10x10 (usually square)
Typical AFM Scales

(only what’s most common, not all of what’s possible)

- Sample sizes — mm to cm
- Image sizes — few to few tens of $\mu$m$^2$
- Feature peak-to-valley — Å to µm (within scan area/field of view)
  - Overall sample thickness — sub-mm to many cm
  - Z range — ~2 µm to ~40 µm (max feature height + sample tilt)
AFM Schematic

- Laser detector
- Laser source
- Tapping piezo
- Tip
- Sample
- Z motion
- XY motion

Free

Interacting
AFM in the Lab

- Laser detector
- Laser source

Tip
Sample
Z motion

XY motion

(laser source & detector)

(side view)

Asylum Research Cypher S AFM

The rest of the instrument
AFM in the Lab

top view
sample is down below
cantilever
tip (underneath)
laser spot
(support chip)

laser source & detector

(the rest of the instrument)
tip
sample

XYZ motion

samples are mounted flat on steel disks, glass slides, etc. depending on the AFM model

side view
Asylum Research Cypher S AFM
AFM Measurement Process

- Load sample and tip
- Scan
- Process image
- Extract numerical results (application-dependent)
Sample Mounting

- Sample mounted for conductive AFM
- 1 inch
- 3 inches
- In a petri dish lid for fluid

- In the AFM
- In a fluid heater (sample is a pink gel)
AFM Schematic

Laser detector
Laser source

Tip
Sample

Z motion

XY motion

Free

Interacting

Laboratory

(the rest of the instrument)
Deflection Detection

(exaggerated schematic)

non-interacting

laser spot
(reflected from back of cantilever)

segmented photodetector

normal direction
(topography)

lateral direction
(friction)

(side view)
Deflection Detection

(exaggerated schematic)

sample pushing up

(laser spot (reflected from back of cantilever))

segmented photodetector

(normal direction (topography))

(lateral direction (friction))

(side view)
Deflection Detection

(sample pulling down)

(exaggerated schematic)

laser spot (reflected from back of cantilever)

lateral direction (friction)

normal direction (topography)

segmented photodetector
Deflection Detection: A Metaphor

- photodetector
- laser spot
- reflective coating
- cantilever
- tip
AFM tips are consumables, not instrument components.
Tip Terminology

top view

side view

scanning probe microscopy

SEM images taken using MRL's JEOL 6060LV
A Closer Look at the Tip

one (of many) common tip for imaging
- silicon tip, radius of curvature < 10 nm
- cantilever stiffness ~25-40 N/m

SEM images taken using MRL's JEOL 6060LV
Types of Tips

- Different cantilever back side ("reflex") coatings (e.g., Al, gold)
- Different tip coatings (wear-resistant, conductive, magnetic)
- Ultrasharp or high aspect ratio tips — preserve with careful handling
- Colloidal probes, specialty coated tips, made-to-order probes
- Functionalized tips
  - Make your own coatings
Types of Tips

- Different cantilever back side ("reflex") coatings (e.g., Al, gold)
- Different tip coatings (wear-resistant, conductive, magnetic)

- Ultrasharp or high aspect ratio tips — preserve with careful handling

- Colloidal probes, specialty coated tips, made-to-order probes
  - Basically anything (tiny) you want to stick on the end of the cantilever
  - Make your own using "tipless cantilevers"

- Functionalized tips
  - Adhesion, coefficient of friction between materials
  - Make your own coatings
“How long does a tip last?”

- Tips are consumables
  - Contamination from samples
  - Wear from samples
  - Dropping them

- Typical tapping mode tip ~$21
  - When your tip goes bad, just throw it out!

- Generally purchased in 10-packs
  - 50-packs to stock up
  - 5-packs for pricey tips
AFM Measurement Process

- Load sample and tip
- Scan
- Process image
- Extract numerical results (application-dependent)
Raster Scanning on the AFM

**height**

**phase**
(stay tuned)

**camera**
current value

action

setpoint value
Feedback on the AFM

• Cantilever position adjusted to keep feedback signal equal to setpoint
  • too much force—move away
  • too little force—move closer

atomic force microscopy

• Distance extended or retracted describes the height of the feature
Contact Mode Imaging

- Drag tip along surface like a stylus profilometer
- Feed back on the deflection (proportional to force)
- Adjust tip-sample separation to keep cantilever deflection constant
Tapping Mode Imaging

- Standard mode for AFM topography
- Tip is not constantly in contact with the surface
- Driven, oscillating cantilever
- Tip—sample interaction forces affect cantilever oscillation
- Tapping, AC, TappingMode™, amplitude modulation*, intermittent contact*, non-contact*
  * specific meaning
Driven, Damped Harmonic Oscillator

Cantilever is driven; feedback maintains constant amplitude

cantilever oscillation amplitude

resonant frequency

drive frequency

phase

Interactions with the sample damp the cantilever oscillation
Cell on gold electrode on SiO2
Complementary Techniques: Imaging

- 3D Optical Profilometry
- Scanning Electron Microscopy
- Atomic Force Microscopy

- blue glitter crayon tip
- Optical characterization tutorial earlier this morning
- Roughness measurements by 3D optical profilometry today, 1pm

- AFM tip
- (quantitative height measurements are trickier)
- SEM tutorial tomorrow, 9am

- pencil “lead”
- Advanced AFM applications today, 3pm

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Similar applications to 3D optical profilometry, but...

- Far better resolution
- Samples can be truly transparent
- No index of refraction effects
- Can’t “zoom out” to get low-magnification images

Turquoise, 1μm x 1μm AFM topograph
(false color, true Z:XY ratio in 3D)
Similar applications to SEM, but...

- Easily measure true depths/heights
- Samples do not need to be electrically conductive
- Can be done in air, in liquids, in vacuum/inert gas
- Can’t “zoom out” to get low-magnification images

Turquoise, 1μm x 1μm AFM topograph (false color, true Z:XY ratio in 3D)
Interpreting Height Colorscales

- Range of colors in the scale, not necessarily heights in the image
  - Some colors may not have corresponding heights
  - Some heights may not have corresponding colors

Turquoise, 1µm x 1µm AFM topography (false color, correct apparent height)
Interpreting Height Colorscales

same image, different color ranges

color range of the displayed image, not necessarily all heights on the surface

polymer blend, 10µm x 10µm AFM topograph (false color) processed using Gwyddion data analysis software
Interpreting 3D Images

microbe's-eye view

photomask
not necessarily 1:1:1 Z:X:Y
Z often exaggerated compared to XY to convey texture information
<table>
<thead>
<tr>
<th>Measuring Surface Topography: Step Heights</th>
</tr>
</thead>
<tbody>
<tr>
<td>• AFM measures relative heights</td>
</tr>
<tr>
<td>• Need a height difference</td>
</tr>
<tr>
<td>• There is no “sea level” equivalent</td>
</tr>
<tr>
<td>• Can only see the tops of surfaces</td>
</tr>
<tr>
<td>• Film thickness is measured by step height</td>
</tr>
<tr>
<td>• Measure a height difference</td>
</tr>
<tr>
<td>• Leave some bare substrate (patches are OK)</td>
</tr>
<tr>
<td>• Scratch down to the substrate</td>
</tr>
<tr>
<td>• Multilayer material—exposed underlayer</td>
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</tbody>
</table>
HOPG (highly oriented pyrolytic graphite)

measured step: 330 pm

(should be 3.4 Å)
Complementary Techniques: Step Height/Film Thickness

If your step’s too broad for the AFM (continuous film or with a step edge width >~80um), try...

- Stylus profilometry
- 3D optical profilometry
- X-ray Reflectivity (XRR)
- Rutherford Backscattering Spectrometry (RBS)

X-ray analysis tutorial **today, 4pm**
RBS tutorial **tomorrow, 8:30am**
Step Heights and Widths

Full width at half maximum (minimum)

Tip
Step Heights and Widths

- As depth increases, tips get broader
- Steep drop-offs look less sharp
- High aspect ratio tips are available
Application: Roughness

- “The roughness” depends on the scale

- Choose measurement technique to match the feature scale of interest
  - AFM
  - Stylus profilometry
  - 3D optical profilometry

- AFM probes nanoscale roughness
  - Not always a predictor of macroscale roughness
  - Peak-to-valley height must be within the instrument-to-valley range (typically < a few μm)

What is the roughness of this landscape?
Beyond Surface XYZ Coordinates

- Tip-sample interaction forces come from various sources, not just contact
  - Electrostatic
  - Magnetic
  - Chemical
  - Optical

- AFM-level resolution of sample properties besides topography
  - Often in the couple 10s of nm lateral resolution

- Correlative measurements: in the same location, AFM topography + ...
Application: Electromagnetic Characterization

- Conductive AFM
  - Scan at fixed or varying bias, measure current
  - I–V curves
- Magnetic Force Microscopy
- Piezoresponse Force Microscopy
- Electrostatic Force Microscopy
- Kelvin Probe Force Microscopy
- Scanning Microwave Impedance Microscopy
- and more, many more...
Application: Conductive AFM

- Measure electrical conductivity of sample
  - Use conductively coated tip as an electrical probe
  - Caveat: contact resistance

- Transverse conductivity through a sample
  - Bias sample substrate
  - Scan in contact mode
  - Conductive/insulating areas

- Need a conductive pathway
  - OK – insulators in conductors
  - Won’t work – conductors in insulators (conductors on insulators may work)
$I-V$ curves on carbon tape
Asylum Cypher AFM
(carbon tape’s pretty conductive)
Conductive AFM

- Longitudinal (two-point) conductivity along sample
  - I—V curves
  - Sample (e.g., nanowire) on insulating surface
  - One end of sample on a biased electrode
  - Conductive AFM tip as movable other electrode
- Caveat: contact resistance
Two-Pass Techniques

- Interleaved—topography, other signal
- Image a line, then repeat that line raised by a few tens/hundreds of nm
- Long-range (non-topographic) forces between the tip and sample affect the cantilever

![](image)

- Forces from topography dominate
- Longer-range forces only
- Lift height
Magnetic Force Microscopy

- Tip coated with a magnetic material, then magnetized
- Magnetic forces from the sample attract or repel the tip
Magnetic Force Microscopy

MFM phase (colors) overlaid on height
BOPP/PE toothbrush package

phase color scale overlaid on 3D topography
redder areas are more dissipative
Application: Mechanical Characterization

- Tapping mode phase: easy qualitative characterization of mechanical differences within a sample

- Image of mechanical differences
  - Phase (tapping mode)
  - Maps of quantitative measurement results
  - Force modulation, AM-FM, contact resonance, etc.
• **Tip**—surface interactions affect cantilever oscillation
  • Cantilever driven at a specific frequency
  • Dissipative interactions cause a phase lag
    • Compliant areas
    • Sticky areas

• **Contrast in phase image shows differences in mechanical properties**
  • Qualitative, not quantitative
  • Great for mixtures
  • Great for soft materials deposited on hard surfaces
Phase (Qualitative)

• Can help locate and identify small sample components
• Can point out contaminants in sample
  • If phase isn’t homogeneous, neither is the material
  • Can’t do explicit chemical identification (stay tuned)

proteins on silicon deposited from PBS

topography phase

Dimension 3100 AFM
Quantitative Nanomechanical Measurements

- Force curves (pointwise or mapping)
  - Force curves are good for ~few kPa to 10s of GPa
  - For higher-modulus materials, consider nanoindentation

- Contact resonance, force modulation, AM-FM, other mapping modes

Nanoindentation tutorial tomorrow, 10:50am
• Force—distance curves at one point

• Quasistatic (most common)
  • Cantilever is not oscillating
  • Force is proportional to deflection

• Dynamic
  • Cantilever is oscillating
  • Damping force decreases oscillation amplitude
Tip pushes into surface, deflecting cantilever

Force is proportional to Deflection
Tip pushes into surface, deflecting cantilever
Tip pushes into surface, deflecting cantilever

- Force curves
- Hard surface
- Towards surface
- Away from surface

Force vs. Distance graph
Tip pushes into surface, deflecting cantilever

-force curve showing force vs distance
-hard surface
-towards surface
-away from surface
-pull off

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Tip pushes into surface, deflecting cantilever

- Hard surface
- Towards surface
- Away from surface

Distance

Force

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**Force Curves**

- **Slope:** sample stiffness
  - Can be converted to modulus with some care

- **Easier to compare stiffnesses**
  - Determining moduli requires careful fitting, modeling, assumptions
  - Overlaying force curves is easy
- Depth of well in retraction curve: adhesion
  - Depends on tip material
    - Silicon tip, metal-coated tip
    - Functionalized tip
  - Depends on the weather
    - Humidity and capillary forces
  - Depends on contact area between tip and sample
    - Colloidal (bead) probes
    - Varies with surface roughness
• Models for AFM
  • Sneddon > Hertz
  • JKR—strong adhesion
  • DMT—sharp tips

• Unless high spatial resolution is needed, consider colloidal probes

• Tricky to know the exact contact area with ordinary tips
  • Only a few nm displacement into the sample (indentation depth)
  • Quoted radius of curvature is an assumption
    • Tip-to-tip variation
    • Tip damage
    • Not actually a sphere
How to Choose a Cantilever

Cantilever stiffness should be similar to sample stiffness for force curves

- **Cantilever too compliant**: most observed bending is due to cantilever itself, not to sample

- **Cantilever too stiff**: cantilever isn’t deflected much by interaction with the sample

- **Calibrate to account for cantilever compliance**
How to Choose a Cantilever

Cantilever stiffness should be similar to sample stiffness for force curves.

- Cantilever too compliant: bending due to cantilever
- Cantilever too stiff: hard to measure

Easiest: try a couple different cantilevers.
Beyond a Single Force Curve

- Force volume maps
  - Grid of force curves
  - Build an “image” out of the results (height, stiffness, adhesion)
  - ~1 second/force curve
- Fast force mapping
Beyond a Single Force Curve

- Force volume maps
  - Grid of force curves
  - Build an "image" out of the results (height, stiffness, adhesion)
    - ~1 second/force curve
- Fast force mapping
  - Speed depends on cantilever and on desired loading rate

Nail polish adhesion map
10 μm x 10 μm
512 x 512 force curves
Asylum Research Cypher VRS in Fast Force Mapping mode
Application: Sticky Samples

- Tip can stick to sample
  - Can’t scan, or can’t scan nicely
  - Can damage the tip
  - Can contaminate the tip
Application: Sticky Samples

- Tip can stick to sample
  - One strategy: image with a higher tapping amplitude
  - One strategy: different tip material

- Static discharger

- Work in fluid
Application: Fluid

- Can image and do some mechanical measurements in fluid
- Different setups
  - Droplet of fluid on sample
  - Submerged sample in open dish
  - Closed fluid cell
- Fluid is trickier
  - Setup (need to be more careful)
  - Hydrodynamics (partial solution: photothermal cantilever excitation)
Droplet on Sample

- Easiest to set up

- Good for samples which
  - Don’t swell much when wet
  - Aren’t porous
  - Use fluid which doesn’t evaporate quickly

- Cantilever is also completely immersed in fluid

  (ring drawn with hydrophobic pen) allows small sample to be inside droplet
• Instrument setups for fluid
  • droplet of fluid on sample
  • submerged sample in open or covered dish
  • closed fluid cell
  • some AFMs need a different cantilever holder for fluid
  • some AFMs can’t do fluid at all

• Fluid is trickier
  • setup (be more cautious)
  • hydrodynamics (partial solution: photothermal cantilever excitation)
What Fluids are OK?

Unless using a closed fluid cell...

- Must not evaporate too quickly
- Must not produce toxic vapors or vapors which can get into the electronics

Side note: some closed cells are gas-tight and can be used for air-sensitive samples
BioHeater for Asylum MFP-3D heated, sealable fluid cell

(sample is a pink gel)

Some closed cells are gas-tight and can be used for air-sensitive samples
What Fluids are OK?

- Must allow the laser to pass through and be detected
  - Transparent to the laser wavelength (usually infrared or red)
  - No particulates floating in fluid which could block the laser

If your samples are suspended...
Samples Shouldn’t Float or Flex
Samples Shouldn’t Move
(unless you’re trying to move them)

Scanning downwards... ... then scanning upwards

dehydrating chewing gum in air

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Use the tip to alter the sample

- Force
- Magnetization
- Local surface charge
- Piezoelectric domains

Charge lithography on quartz, imaged with DART PFM on the Asylum Cypher AFM. Colorscale: 2nd phase, topography slightly exaggerated in z.
Application: Chemistry

- Phase shows material contrast... but which material is which?
  - Force curves can differentiate mechanically (A is stiffer than B, etc.)
  - Mechanical mapping techniques (contact resonance, etc.) can differentiate based on modulus
  - Functionalized tips can differentiate based on strength of interactions with the tip coating

- If you know what is in your sample, AFM can tell you where that is... ...but what if you don’t know what is in your sample?
Scanning Probe Optical Spectroscopy

- Optical techniques (IR, Raman) combined with AFM
- Chemical information with tens of nm lateral and nanoscale vertical resolution (better than confocal)
- AFM-scale lateral resolution (~ few or ~10s of nm)
- AFM + optical spectroscopy
- Tip-enhanced optical spectroscopy
- AFM + infrared (various types)
- Generally separate instruments rather than instrument accessories
Tip-enhanced Raman spectroscopy (TERS)

- Side view of tip
- Gold- or silver-coated cantilever
- Sample (HOPG)
- Raman laser aimed here
- Raman shift [cm⁻¹]
Scanning Probe Optical Spectroscopy

AFM + infrared microscopy/spectroscopy

• Shine an IR laser at the tip-sample interface

• Detect
  • scattered light
  • thermomechanical changes to the sample
  • tip-sample force changes as a function of IR wavenumber

• Map an area at a specific wavenumber

• Sweep the laser frequency to get spectra
Artifacts

Double tip!
What Can Go Wrong in AFM?

- Sample
  - Moving too quickly (setup and scanning)
  - Drift (sample mounting, thermal)

- Tip
  - Multiple tip
  - Tip contamination/breaking/wear

- Image processing
  - Flattening artifacts
Navigating the Sample Too Quickly

- Sample
  - Moving too quickly (setup and scanning)

Be cautious when moving around on rough or tilted or transparent samples.
Sample Drift

- Sample
  - Moving too quickly (setup and scanning)
  - Drift (sample mounting, thermal)

gradually dehydrating chewing gum
Tip Artifacts

- Sample
  - Moving too quickly (setup and scanning)
  - Drift (sample mounting, thermal)

- Tip
  - Multiple tip
  - Tip contamination/breaking/wear
- Tip contamination
- Tip breaking
- Tip-sample forces occur between the sample and tip 1, tip 2, tip...
Multiple Tip Artifact

Really, really multiple tip

Cypher STM (+ 0.5 V, 0.08 nA)

500 nm x 500 nm

125 nm x 125 nm

SEM of an STM tip (not this tip)

artist’s conception of this tip’s shape
Contaminated Tip

10 μm partial scan

Neaspec IR-AFM
Contaminated Tip looks like
Tip Artifacts

- Sample
  - Moving too quickly (setup and scanning)
  - Drift (sample mounting, thermal)

- Tip
  - Multiple tip
  - Tip contamination/breaking/wear
What Can Go Wrong in AFM?

- **Sample**
  - Moving too quickly (setup and scanning)
  - Drift (sample mounting, thermal)

- **Tip**
  - Multiple tip
  - Tip contamination/breaking/wear

- **Image processing**
  - Flattening artifacts
Image Processing

- Image processing generally needs to be done
- Practically every sample is tilted on the nanoscale with respect to the tip
- Either a plane or line-by-line subtraction is generally needed

- Image processing can introduce artifacts or misleading appearances
  - Flattening artifacts
  - Confusing colorscale choices
  - Watch out for these in papers
Tilt Correction

Original

After subtracting substrate tilt

photomask sample courtesy of Jeff Grau
Line-by-Line Correction

- Difference from line to line, abrupt or gradual
- Rastering has a fast scan and slow scan direction
- Changes often happen along the slow scan direction
  - Drift
  - Periodic noise
  - Tip condition changes
- Polynomial subtraction
masking: ignore outliers when processing
Masking

line subtraction

artificially lowered areas
Masking

- line subtraction
- mask outlier areas
- areas to ignore when processing
line subtraction
masked line subtraction
(polynomial of order 1)

no more streaks
Image Processing

- Image processing will affect the image appearance
- Image processing will affect step height results
- Image processing will affect roughness results
- But XYZ coordinates are not all AFM can give...
So Many Applications

• EFM (conductors in insulators)
• KPFM (surface electrical potential)
• MFM (magnetic domain mapping)
• LFM (friction)
• PFM (piezoelectric domain mapping)
• Nanolithography/nanomanipulation

• ... and these are just a few of the ones that generally don’t need extra gear (except different tips)
Accessories for the MRL AFMs

- ORCA Conductive AFM
- Scanning Microwave Impedance Microscopy (sMIM)
- Environmental Controller
- BioHeater
- PolyHeater (up to 300°C)
- Petri Dish Heater
- Inverted Optical Fluorescence Microscope
- MFP-3D Leg Extenders

- Fast Force Mapping
- Dual-Gain ORCA Conductive AFM
- Contact Resonance Viscoelastic Mapping Mode
- AM-FM Viscoelastic Mapping Mode
- blueDrive Photothermal Excitation
- Piezoresponse Force Microscopy (HV-PFM)
- Scanning Tunneling Microscopy (STM)
- Air Temperature Controller (ATC)
- Droplet Cantilever Holder Kit

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Lab tour tomorrow 4-5pm (don’t miss it!)

B12: main MRL lab (Cypher, MFP-3D-SAs)

0024: scanning probe optical spectroscopy lab
(PiFM/Raman, TERS/TEPL, sSNOM)

0026: bio AFM lab (MFP-3D on inverted optical
fluorescence microscope)

B80: stylus and optical profilometry lab
(affiliated techniques)
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