



**Grainger College
of Engineering**

UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

Atomic Force Microscopy

Kathy Walsh

Senior Research Scientist

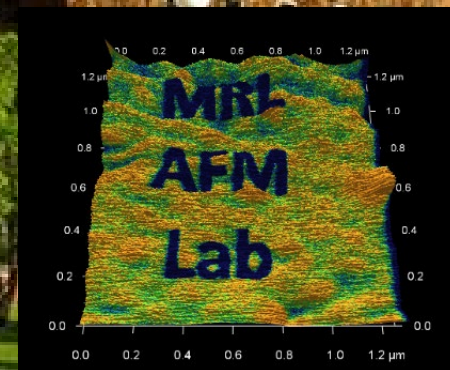
Scanning Probe Microscopy

Materials Research Laboratory

Central Research Facilities

Physics 403

3/26/24





Illinois Materials Research Lab Central Research Facilities

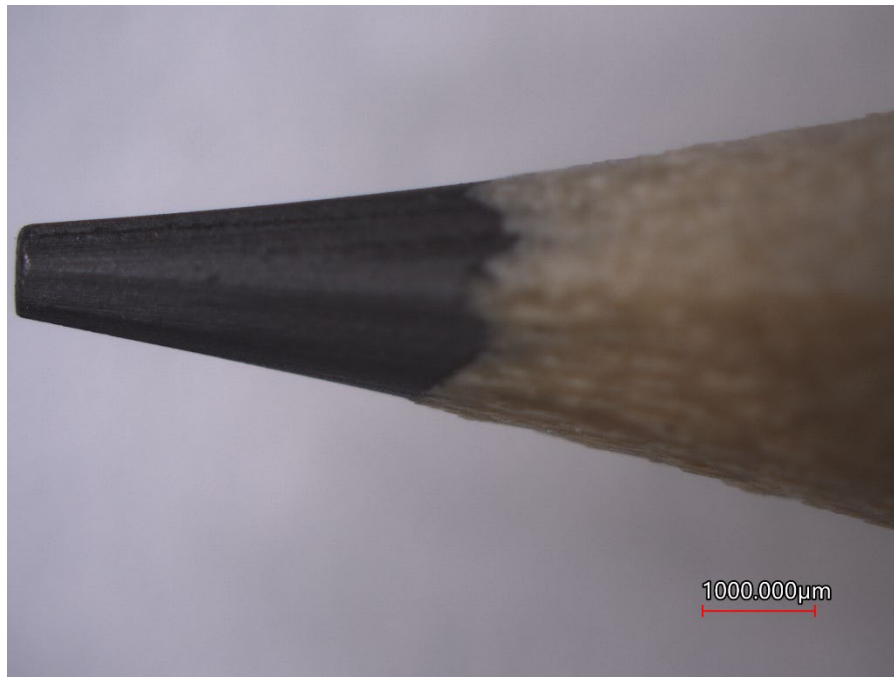
- User facility—anyone can be trained
 - UIUC and non-UIUC researchers welcome
 - Undergraduate researchers welcome
 - Staff collaboration or analysis available
- mrl.illinois.edu/facilities
- mrl-facilities@illinois.edu



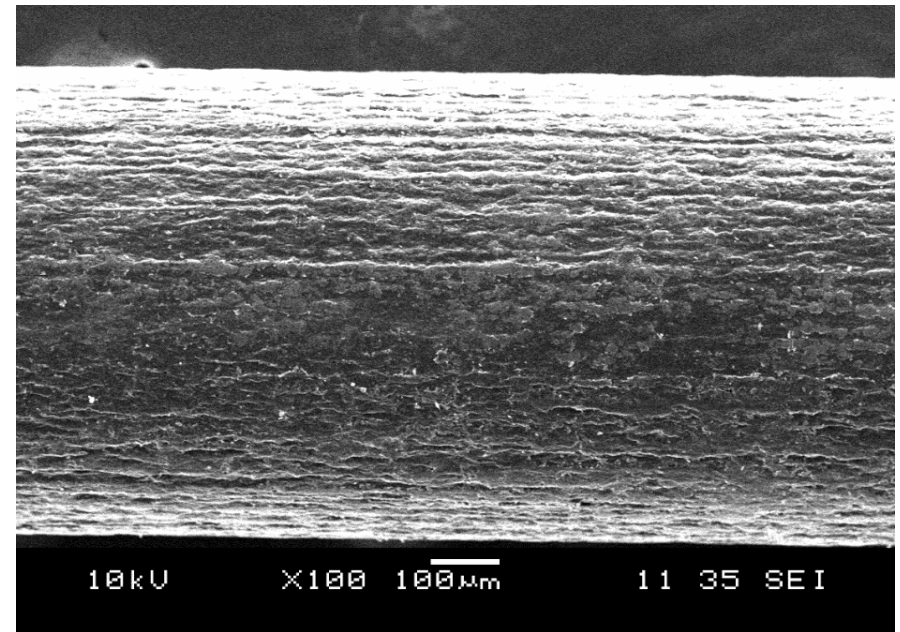


Looking at Surfaces

Optical Microscopy



Scanning Electron Microscopy

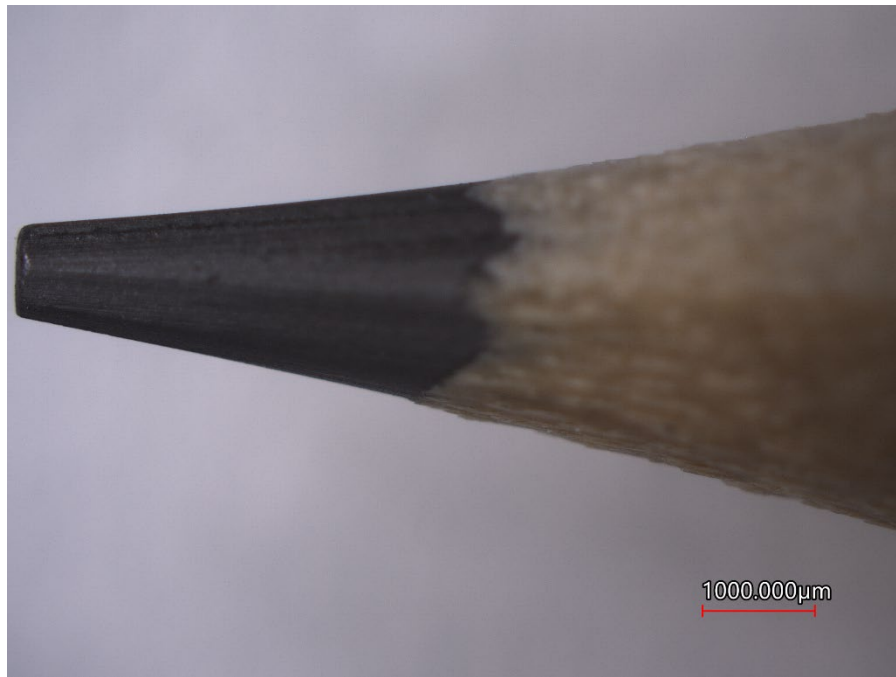


mechanical pencil “lead”

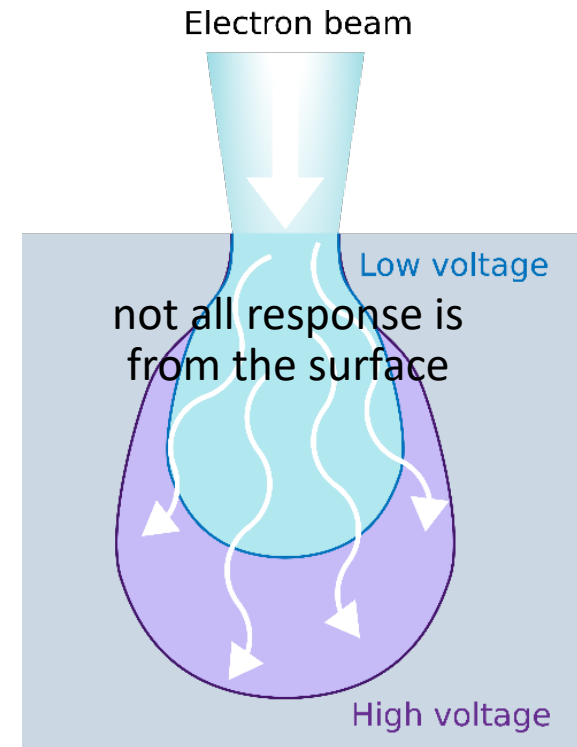


Looking at Surfaces

Optical Microscopy



Scanning Electron Microscopy

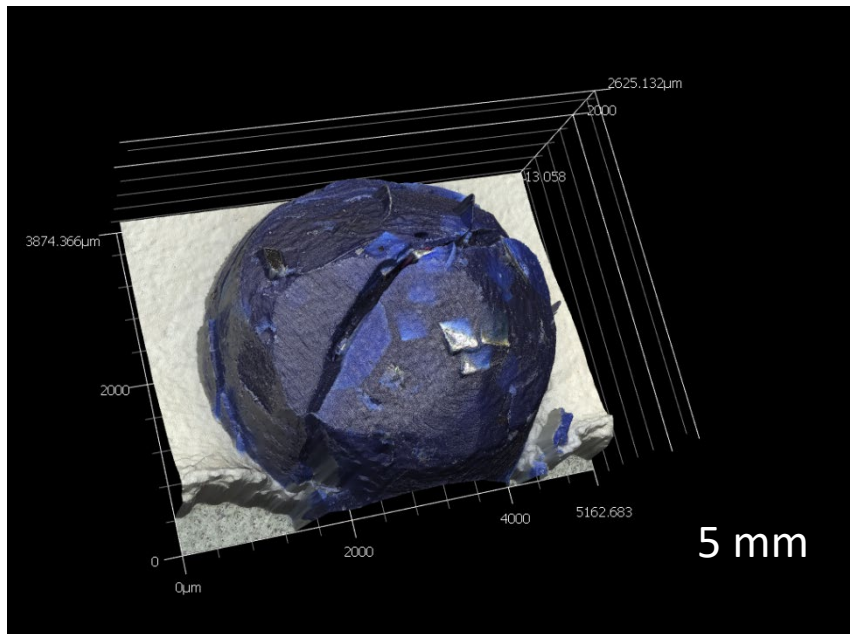


Adapted from
https://myscope.training/#/SEMlevel_2_13
(CC BY-SA 4.0)



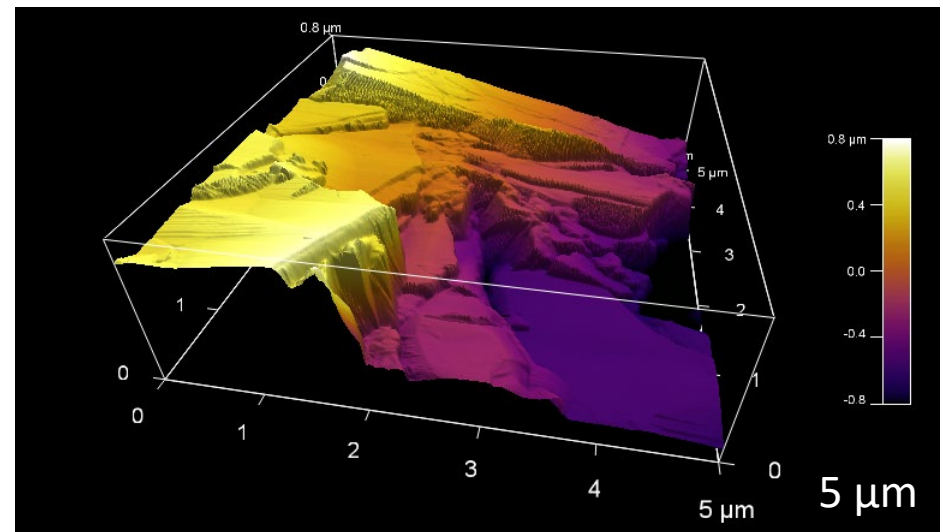
Surface XYZ Coordinates Needed

3D Optical Profilometry



blue glitter crayon tip

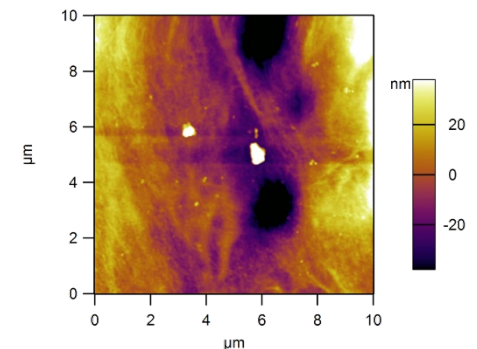
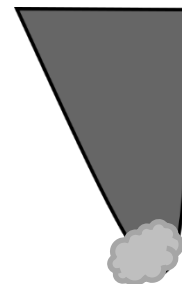
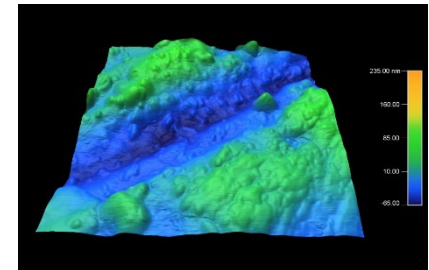
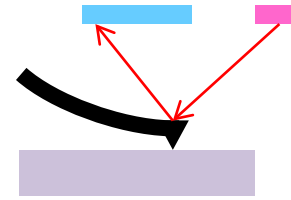
Atomic Force Microscopy



pencil "lead"

Topics for Today

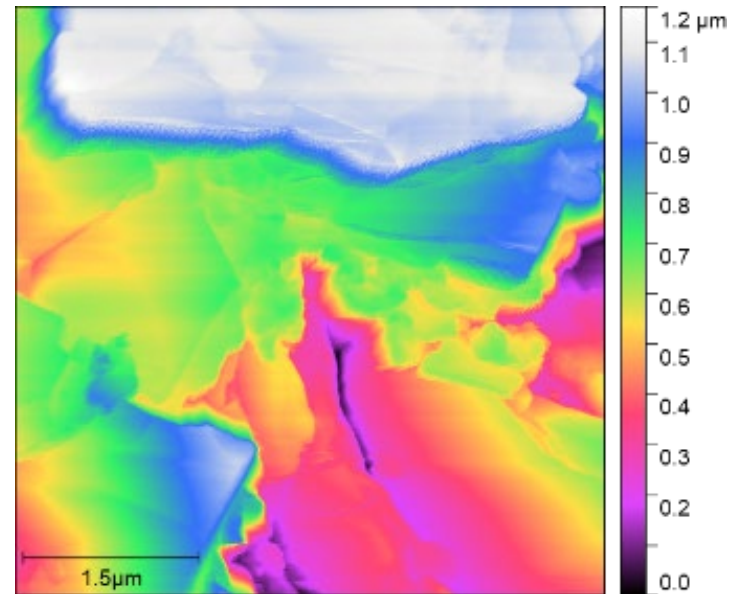
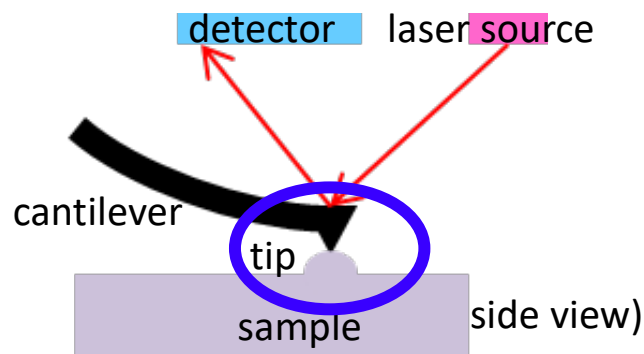
- How AFM works
- Featured applications
 - Topography
 - Profiles, step height
 - Roughness
 - Phase
 - Conductive AFM
- Issues and artifacts
- Image processing





What's an Atomic Force Microscope?

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

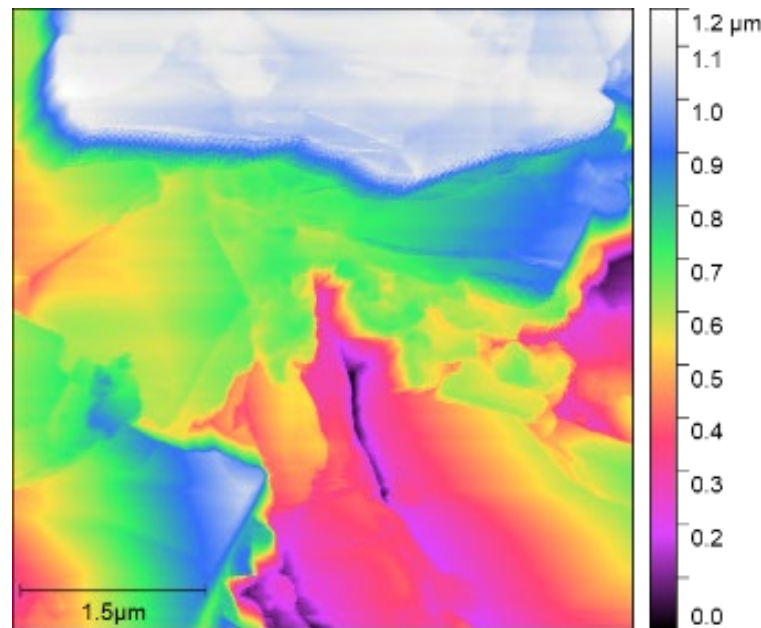
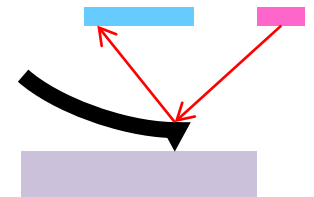


false-color surface topographs



What's an Atomic Force Microscope?

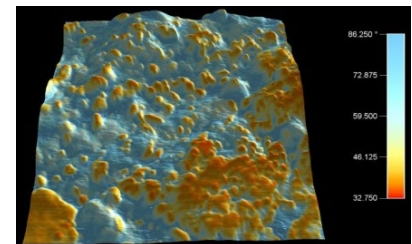
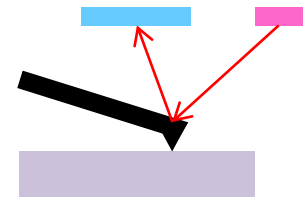
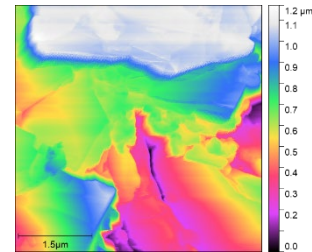
- “Atomic Force” — interactions between tip and sample
 - Not actual atomic resolution (usually)
 - Nanoscale lateral resolution (depends on tip)
 - Sub-angstrom vertical resolution
- “Microscope” — surface topograph (false color)





What's an Atomic Force Microscope?

- “Atomic Force” —interactions between tip and sample
 - Sub-angstrom vertical resolution
 - Not actual atomic resolution (usually)
 - Nanoscale lateral resolution (depends on tip)
- “Microscope” —surface topograph (false color)
- 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 choices

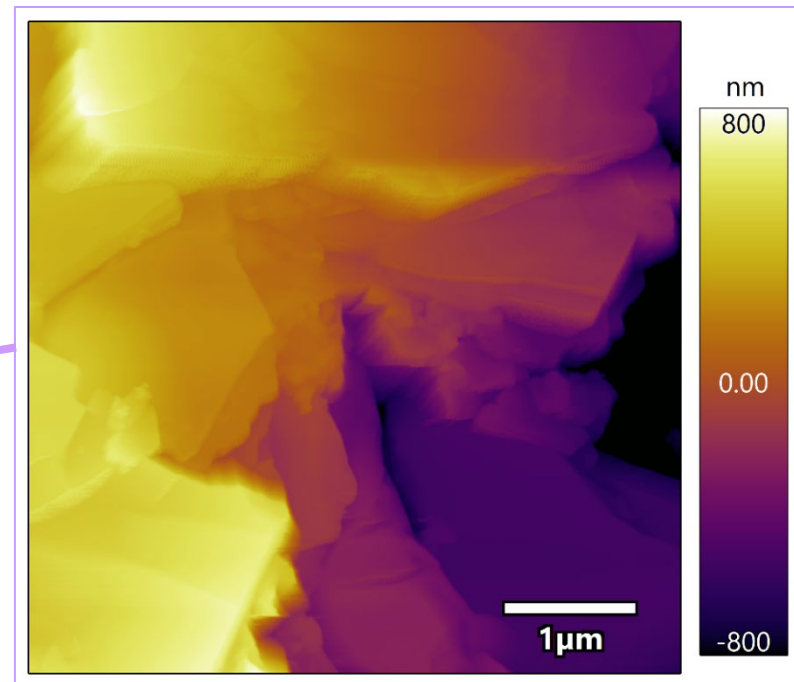
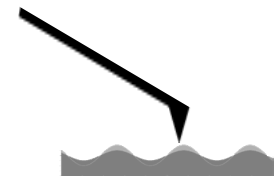


Turquoise, $1\mu\text{m} \times 1\mu\text{m}$
color overlay: phase

Typical AFM Scales

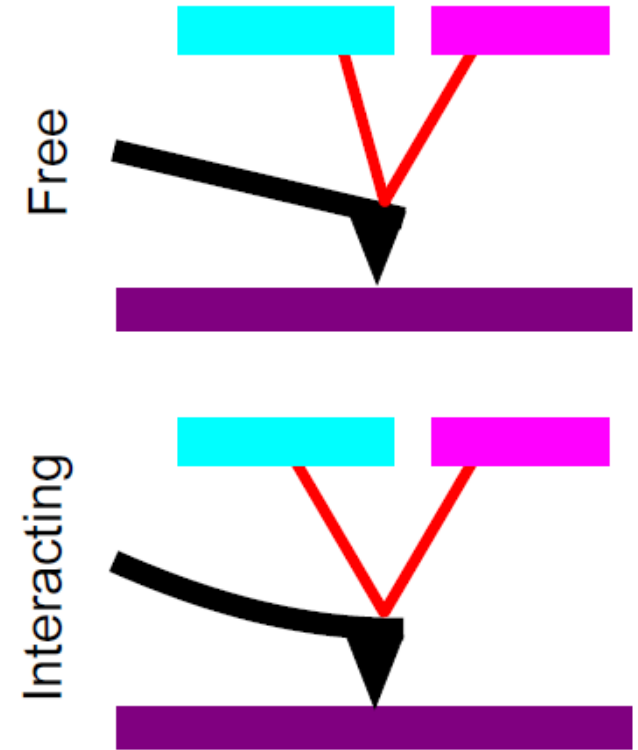
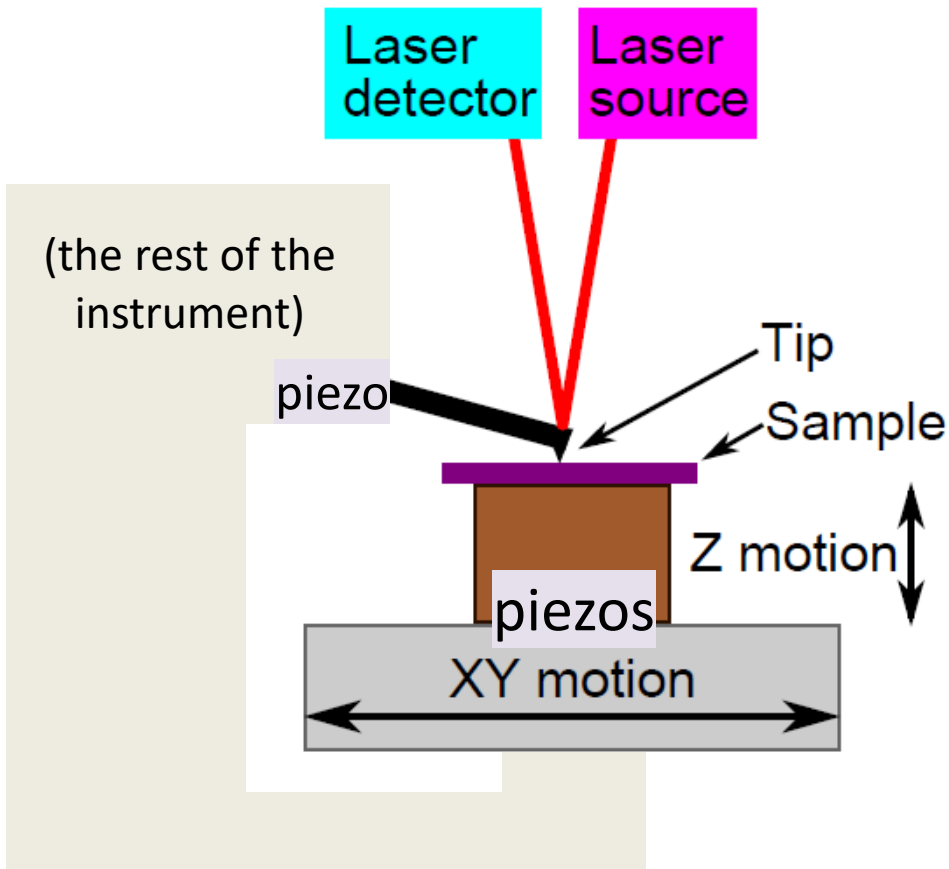
(only what's pretty common, not all of what's possible)

- Image sizes -- few to tens of μm^2
- Feature peak-to-valley -- \AA to μm
- Sample sizes -- mm to cm
- AFM measures surfaces





AFM Schematic

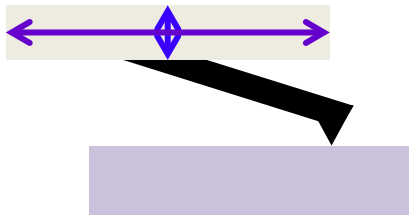




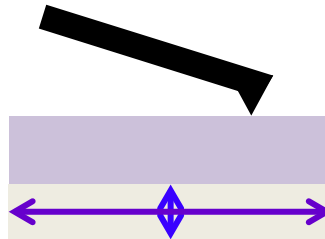
Scanners

scanning probe microscopy

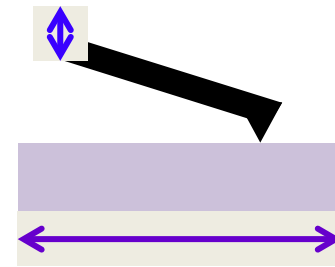
tip scanning



sample scanning



decoupled scanning

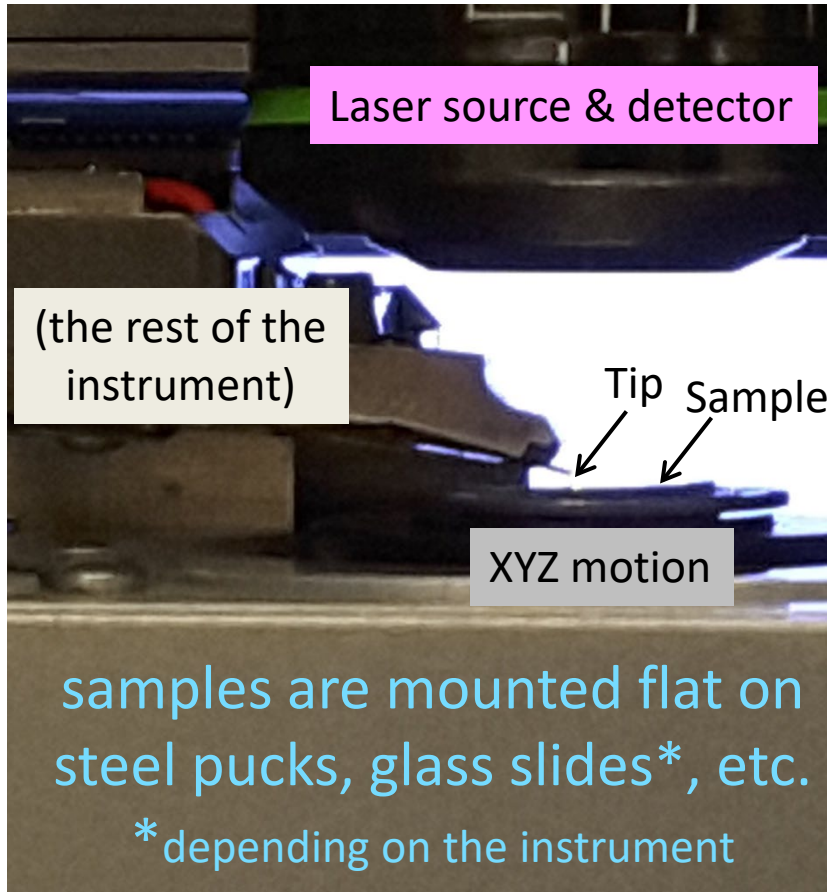


tapping is done close to or at the cantilever
(tapping mode will be discussed later)



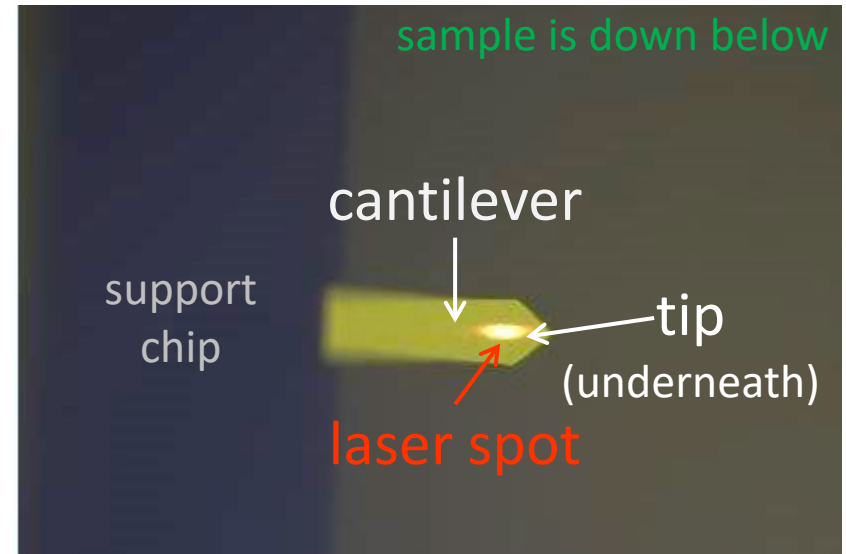


AFM Instrument



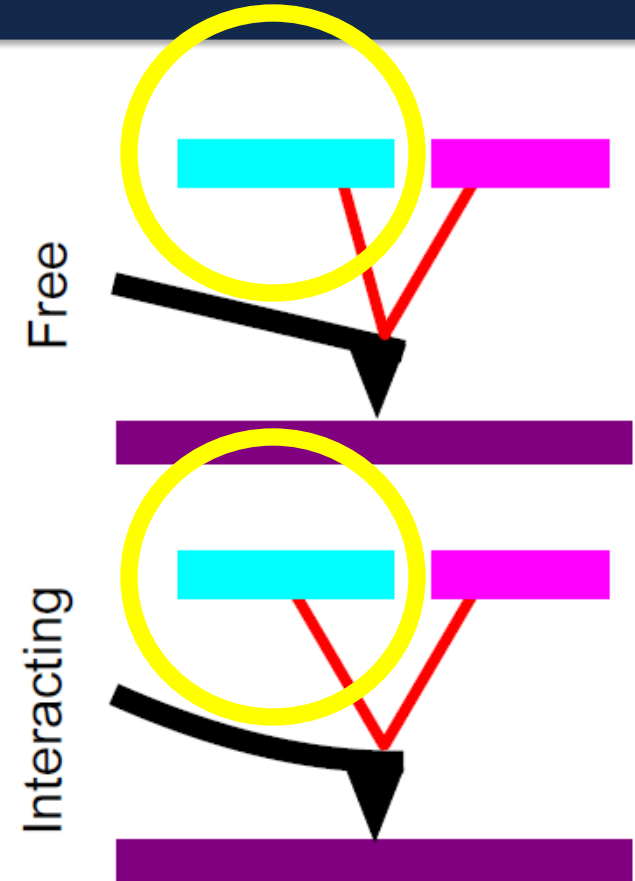
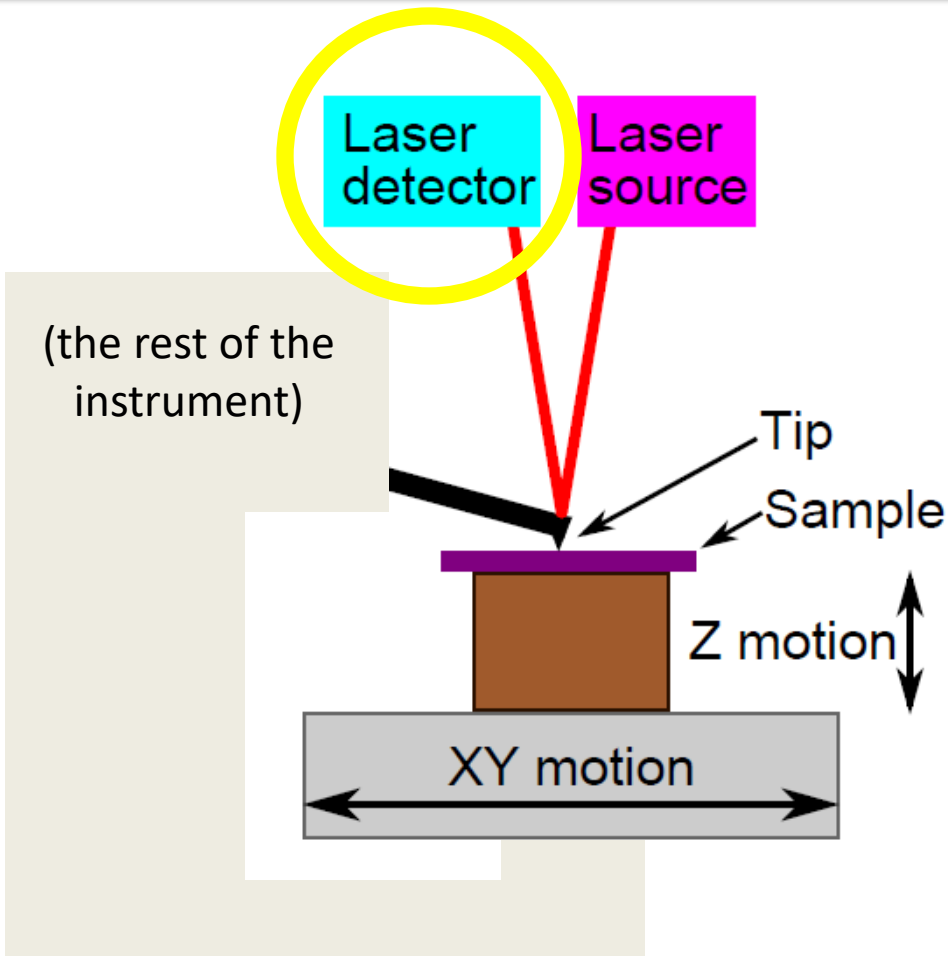
side view

top view





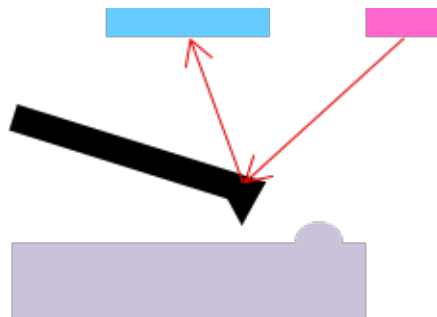
AFM Schematic





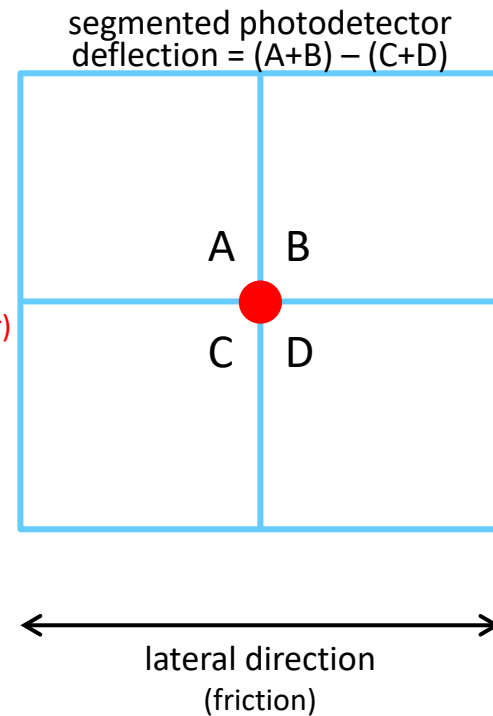
Laser Detection

non-interacting



(side view)

laser spot
(reflected from back of cantilever)

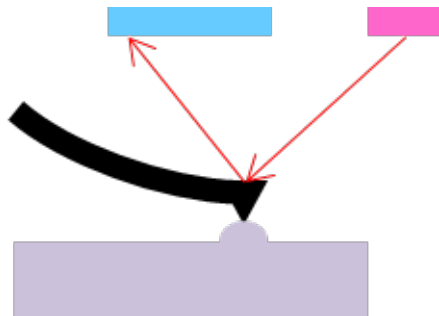


(exaggerated schematic)



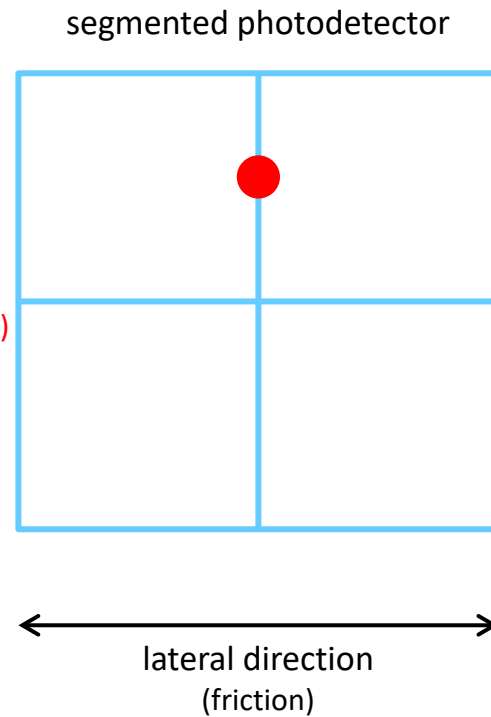
Laser Detection

sample pushing up



(side view)

laser spot
(reflected from back of cantilever)

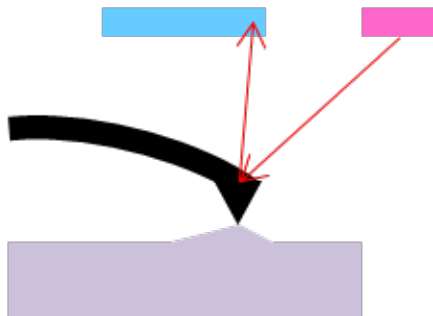


(exaggerated schematic)



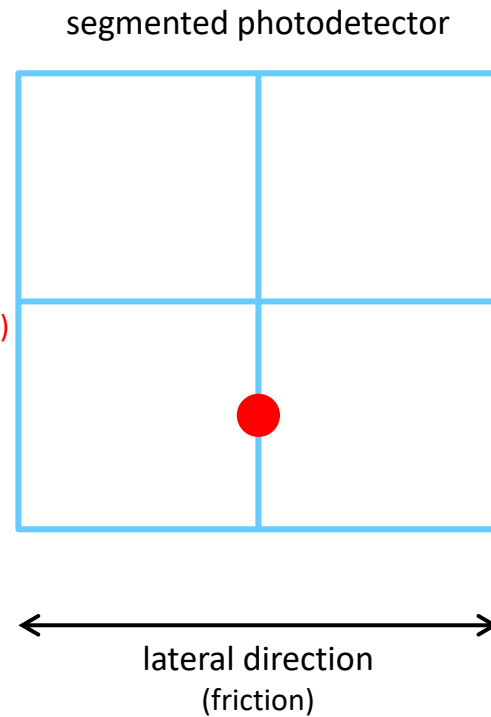
Laser Detection

sample pulling down



(side view)

laser spot
(reflected from back of cantilever)

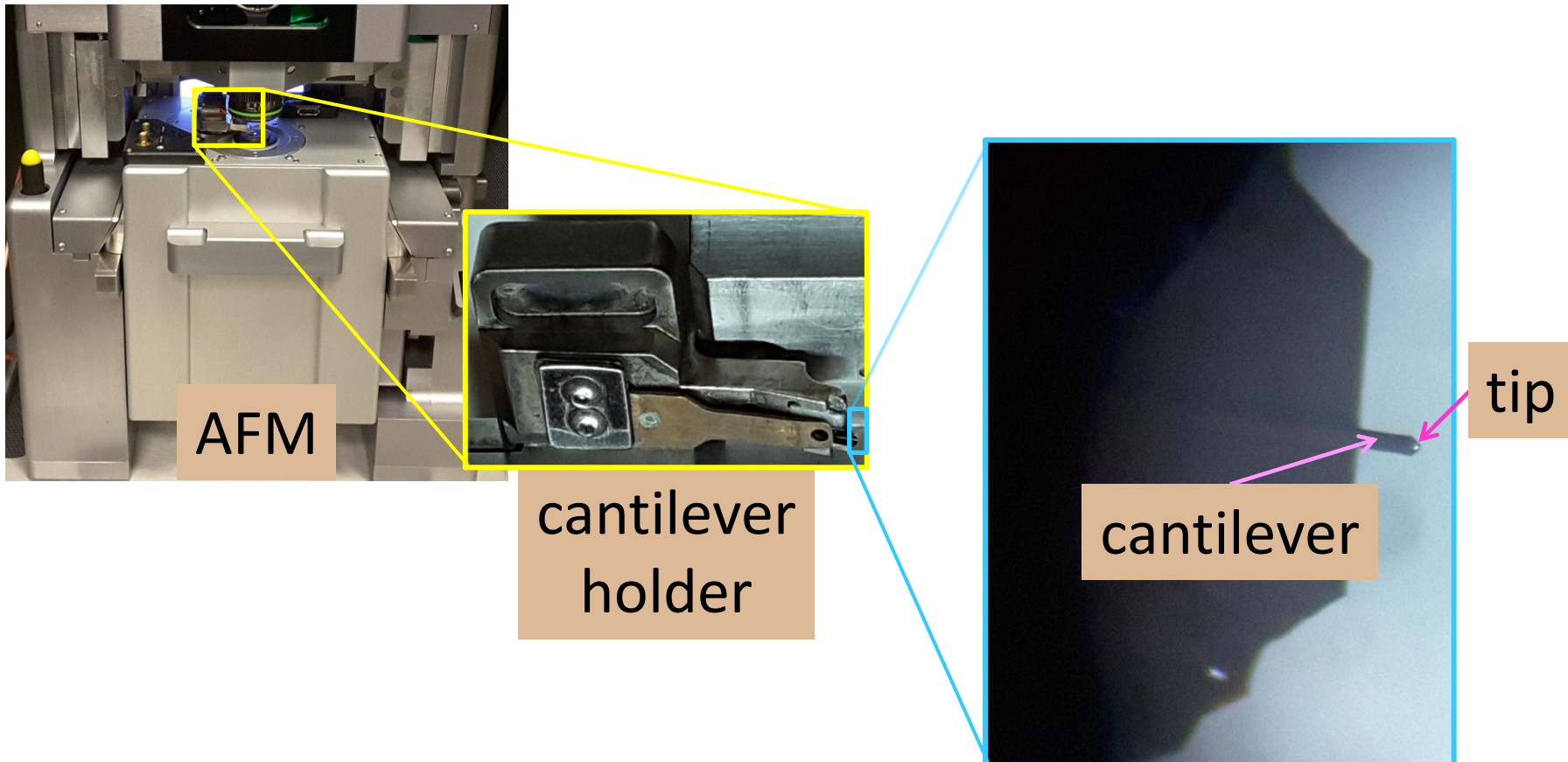


(exaggerated schematic)



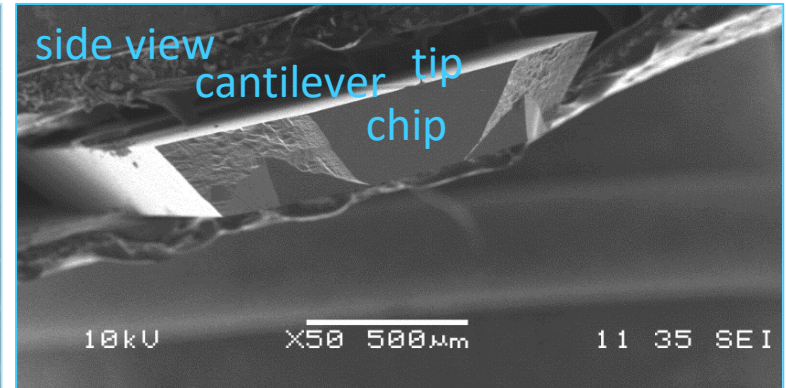
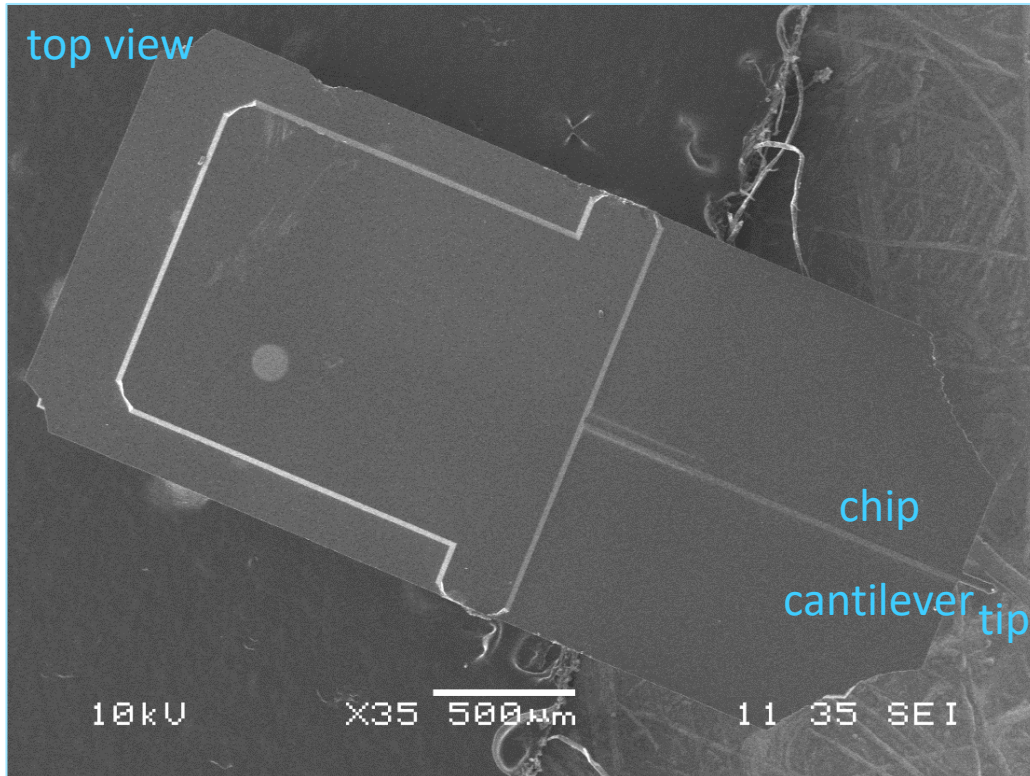
AFM Tips

scanning *probe* microscopy



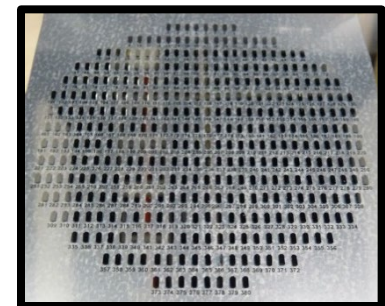


Tip Terminology



“probe”

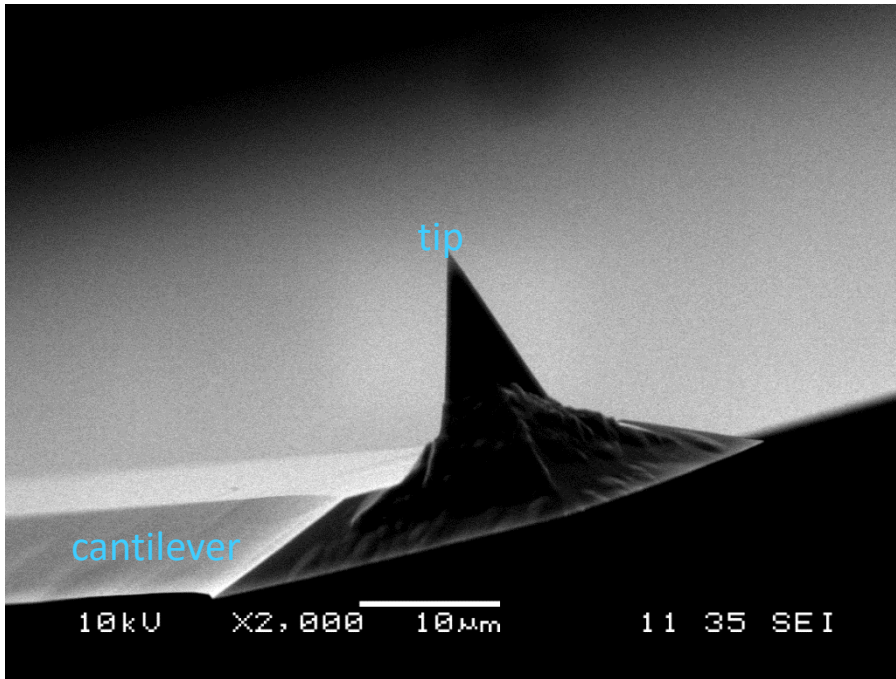
tips point upwards
in the box



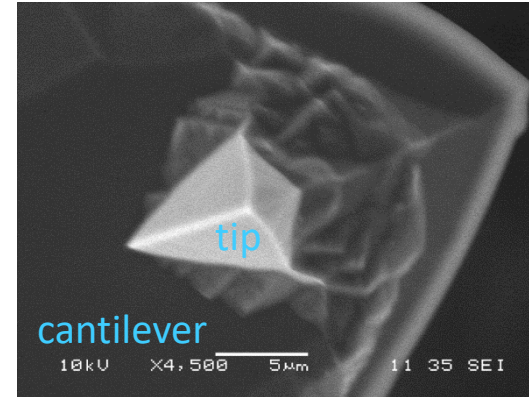
SEM images taken using MRL's JEOL 6060LV



Typical Tip

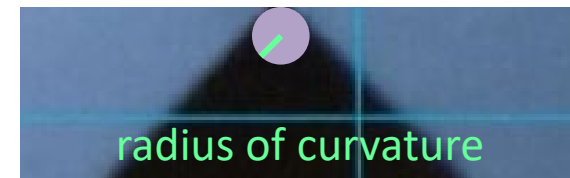


SEM images taken using MRL's JEOL 6060LV



common tip for imaging:

- tip radius of curvature < 10 nm
- silicon tip
- cantilever width $30 \mu\text{m}$
- cantilever length $125 \mu\text{m}$
- cantilever thickness $4 \mu\text{m}$





Tip Types


- Typical tapping tip cost ~\$21
- Specialized tips cost more
 - Coatings (electrical, magnetic) usually a couple more dollars per tip
 - Ultrasharp tips ~\$80-200
 - Coaxial microwave waveguide tips ~\$150
 - Colloidal probes, coated tips, made-to-order probes available



Non-Contact / Tapping Mode AFM Probes
General dynamic mode measurements
★ bestsellers >> new



Force Modulation (FM) AFM Probes
Multi-functional probes
★ bestsellers



Contact Mode AFM Probes
General static mode measurements
★ bestsellers



Life Science AFM Probes
Biological applications
★ bestsellers >> new



Ultra High Frequency AFM Probes
High speed measurements
★ bestsellers



Conductive AFM Probes
Electrical characterization (EFM, KPFM, SSRM, TUNA, etc.)
★ bestsellers >> new



Magnetic AFM Probes
Magnetic force microscopy (MFM)
★ bestsellers




Supersharp AFM Probes
Enhanced / atomic resolution measurements
★ bestsellers >> new



Diamond AFM Probes
The ultimate in hardness
★ bestsellers




Hardened / Enhanced Wear Resistance AFM Probes
Long scanning, hard samples
★ bestsellers



Nanoindentation and Lithography AFM Probes
Nanomechanics and Sample Modification
★ bestsellers >> new



High Aspect Ratio (HAR) AFM Probes
Deep trench measurements
★ bestsellers >> new



ScanAsyst*** AFM Probes
ScanAsyst* compatible probes
★ bestsellers



Silicon Nitride AFM Probes
Soft samples in air and liquid
★ bestsellers



Lateral Force Microscopy (LFM) AFM Probes
Frictional force measurements
★ bestsellers




Tipless AFM Cantilevers and Cantilever Arrays
For functionalization and gluing spheres
★ bestsellers



Colloidal AFM Probes
Various colloidal spheres for nanomechanics



Self-Sensing & Self-Actuating AFM Probes
The AFM technology of tomorrow
★ bestsellers



Sphere AFM Tips
Well defined sphere geometry for nanomechanics
>> new



Platinum Silicide AFM Probes
The ultimate probes for electrical characterization
★ bestsellers



Scanning Thermal Microscopy AFM Probes
Temperature and thermal conductivity measurements



Premounted AFM Probes
For Quesant / Ambios AFM systems
★ bestsellers

 **NANOANDMORE USA**
The Nanotech Facilitator

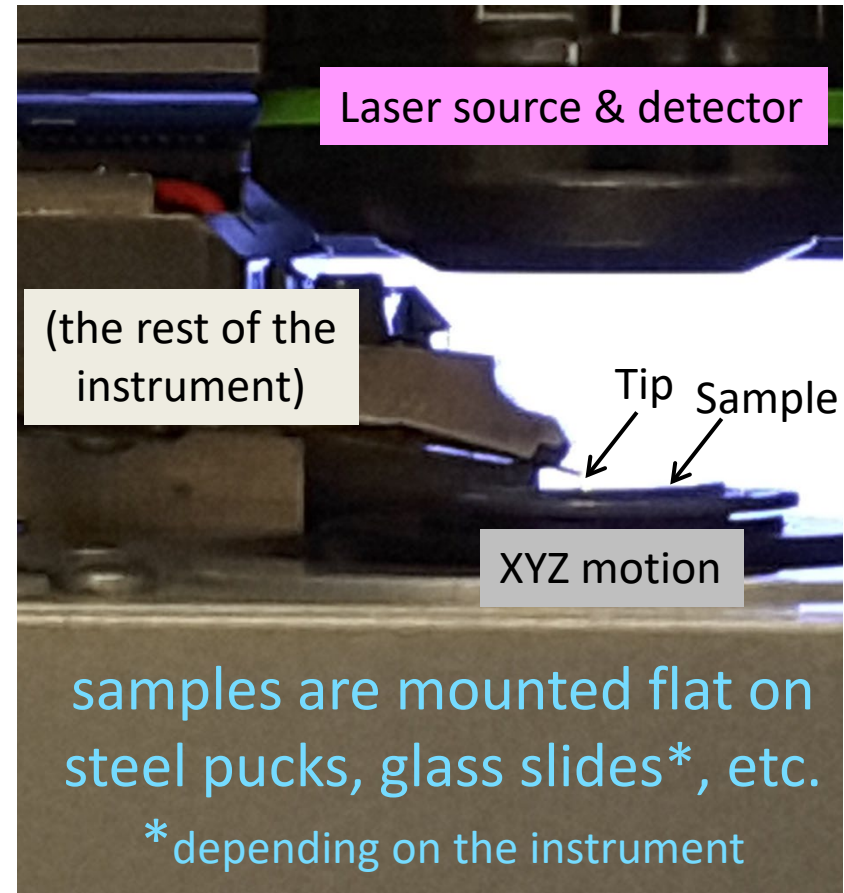
“How long does a tip last?”

- Tips are consumables
 - Contamination from samples
 - Wear from samples
 - Dropping them
- When your tip goes bad, just throw it out!
- Generally come in 10-packs
 - 50-packs if you scan a whole lot



The Process

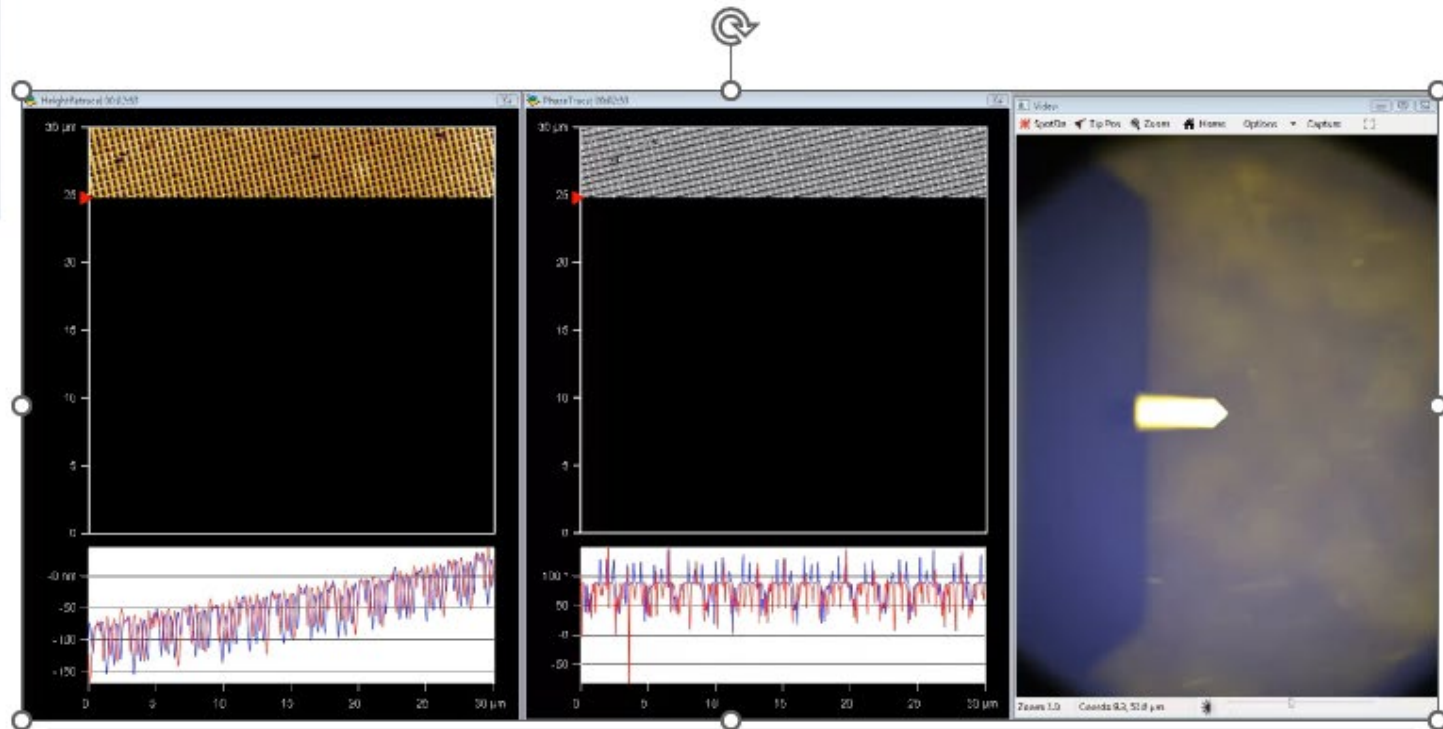
- Mount tip
- Mount sample
- Scan
- Process image
- Extract numbers
(application-dependent)





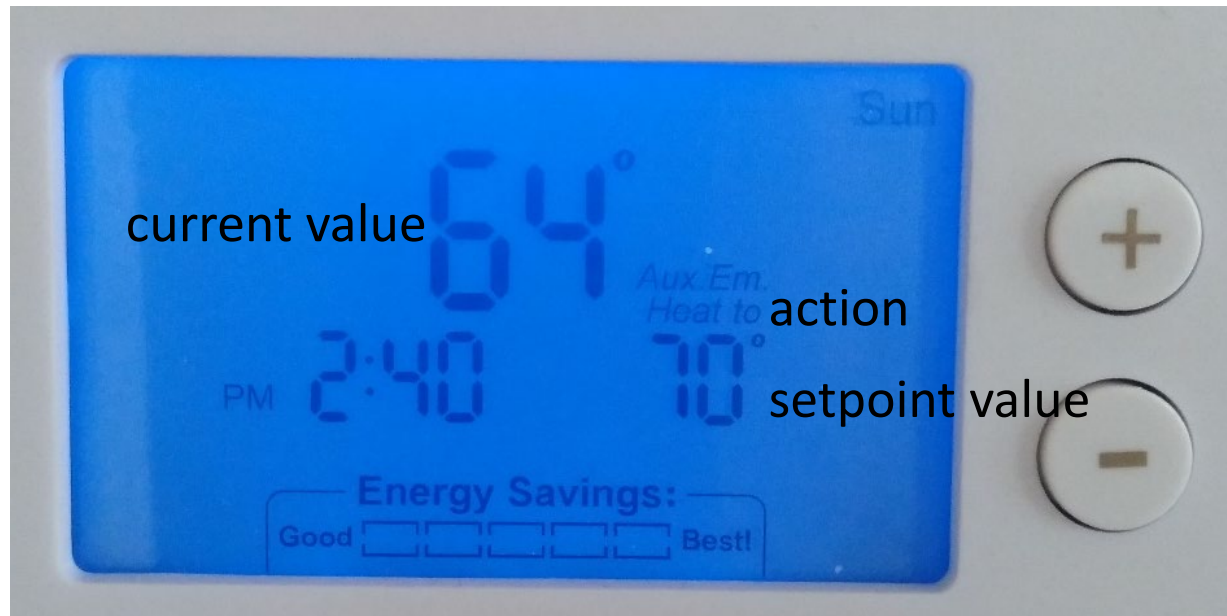
Raster Scanning

Move probe and sample with respect to one another to build up an image



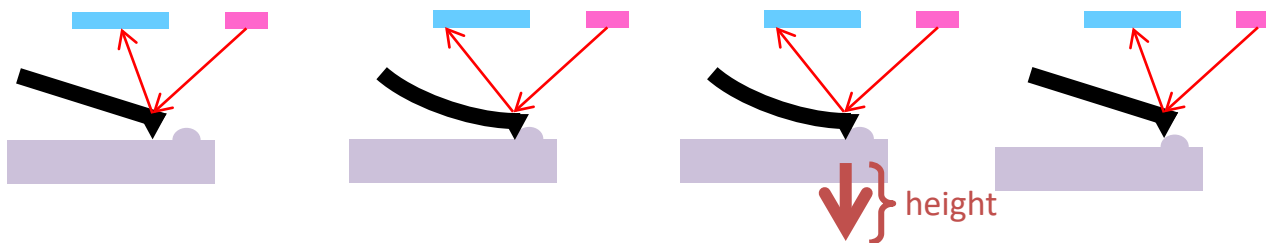


Feedback



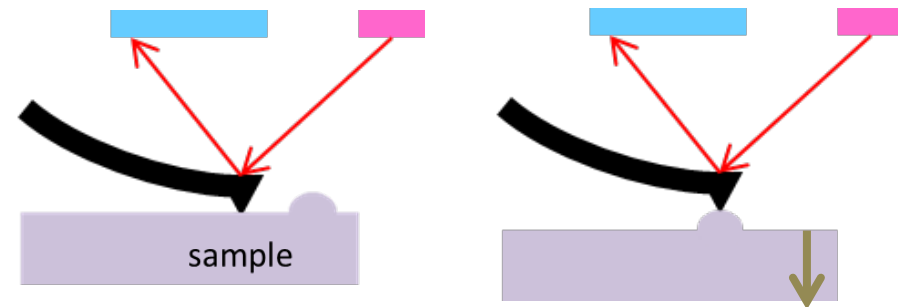
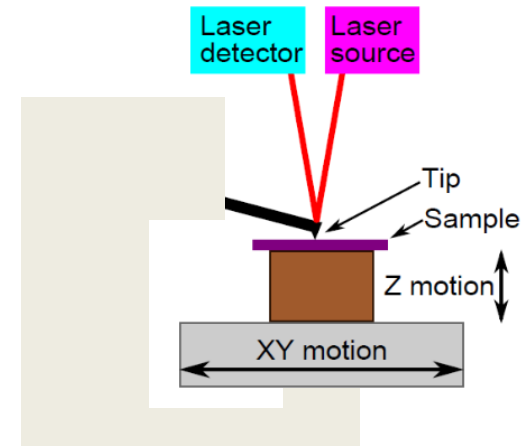
Feedback

- z piezo extension adjusted to keep **feedback signal** equal to setpoint
 - too much force—move away
 - too little force—move closer
 - deflection for **contact mode**, usually amplitude for **tapping mode**
- distance extended or retracted describes the height of the feature



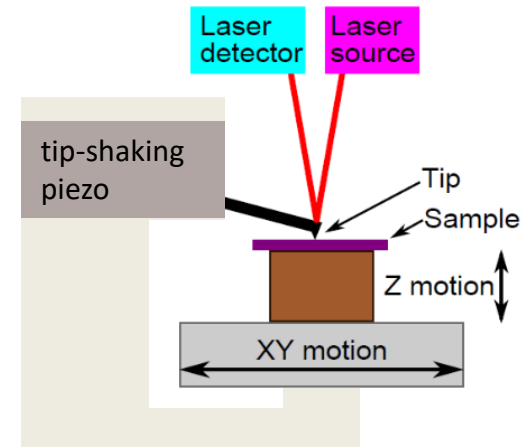
Contact Mode Imaging

- Drag tip along surface like a stylus profilometer (or like a record player)
- Adjust tip—sample separation to keep cantilever deflection constant
 - Traces sample topography
 - Some AFMs move tip; some move sample

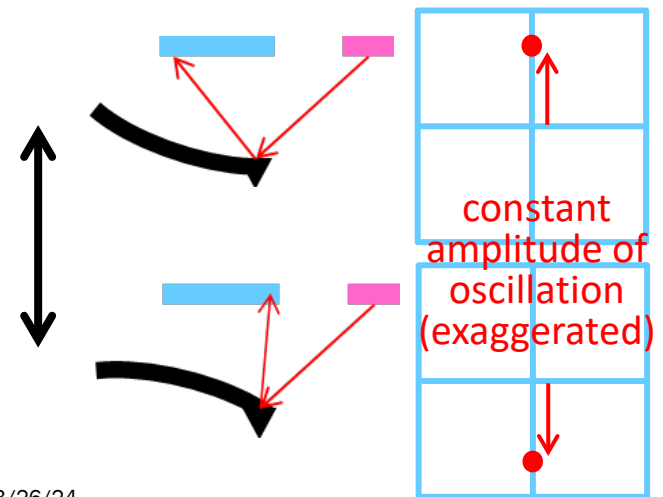


Tapping Mode Imaging

- Standard mode for AFM topography
- Intermittent contact, tapping, AC, amplitude modulation mode
- Not constantly in contact with the surface
- Driven, oscillating cantilever
- Tip—sample interactions affect oscillation

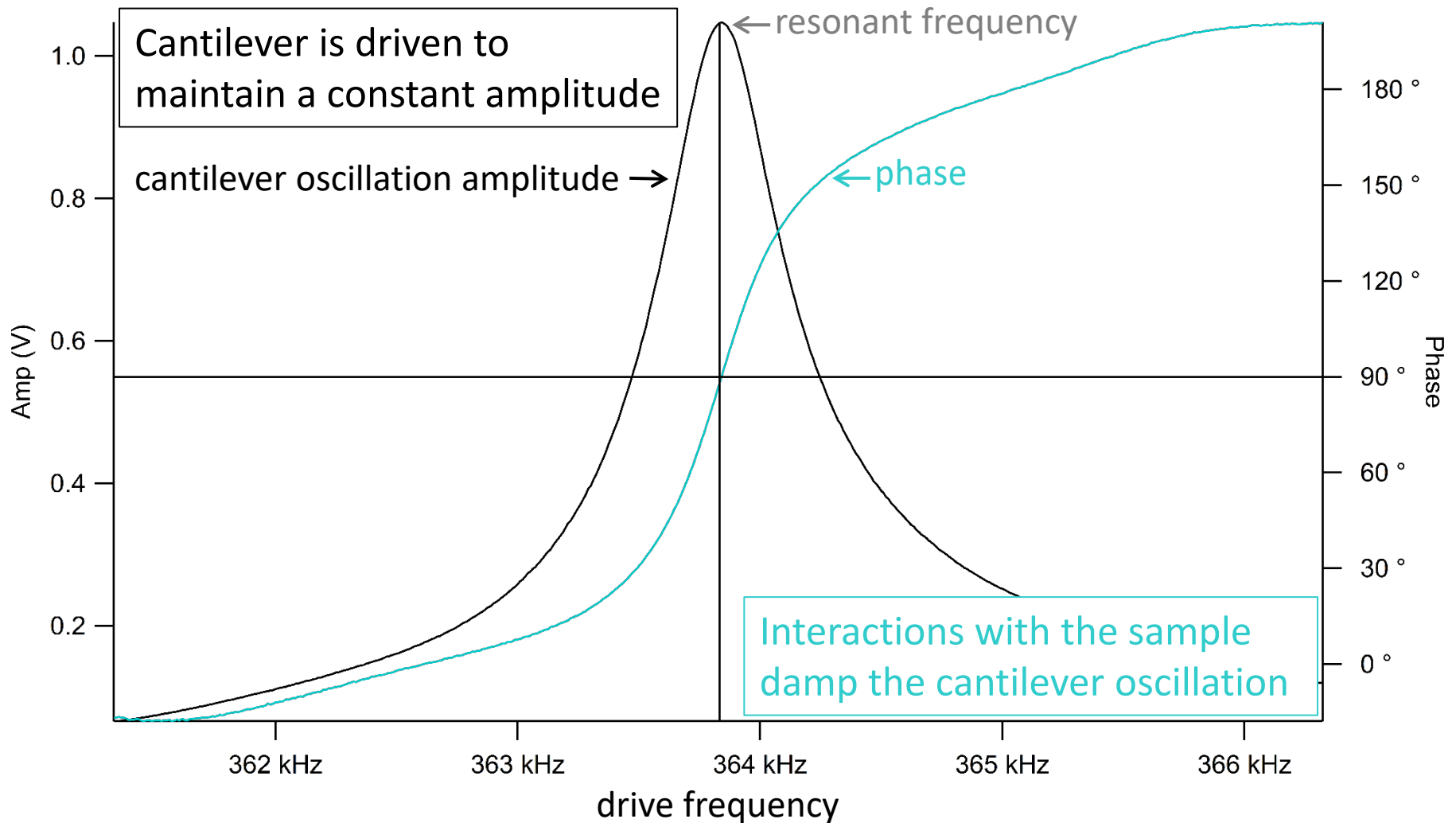


tip oscillates at tens of kHz to MHz





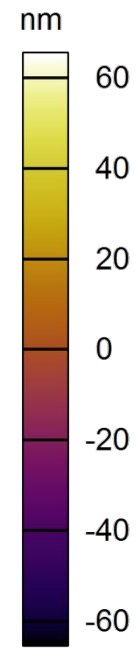
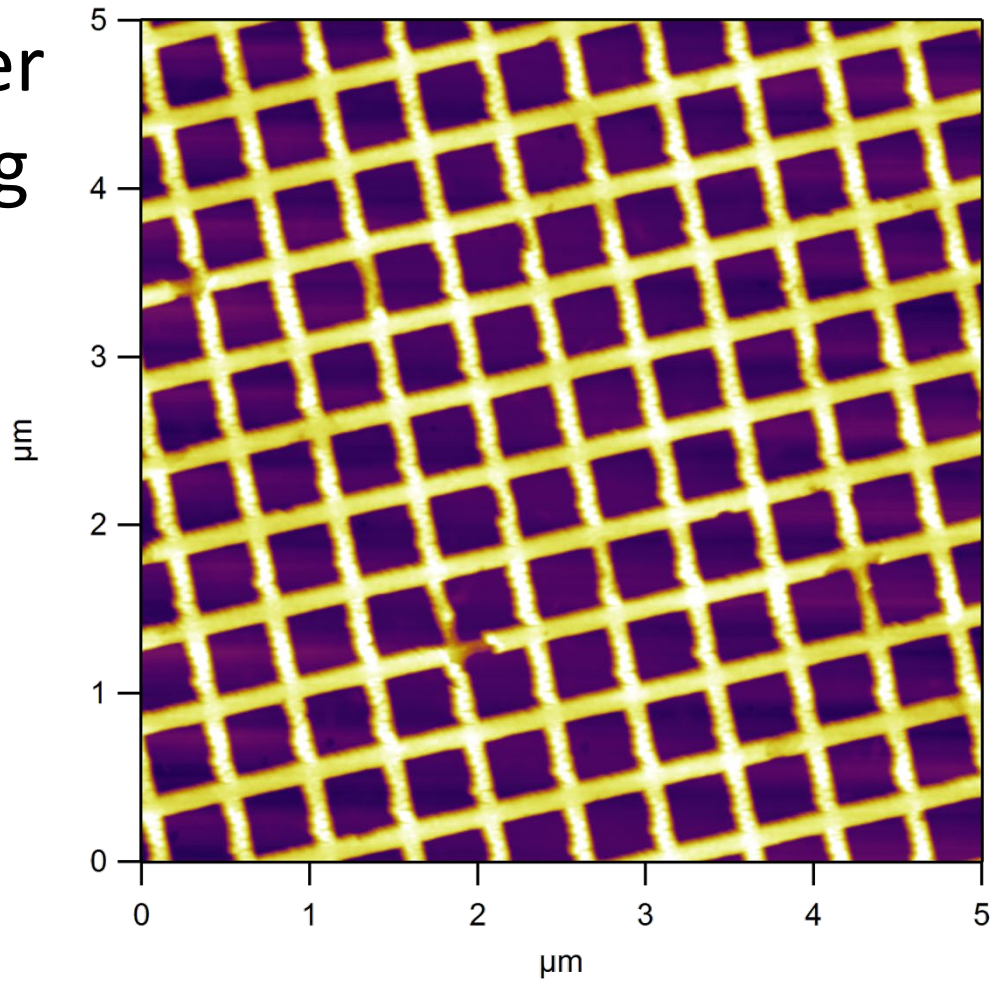
Tuning the Cantilever





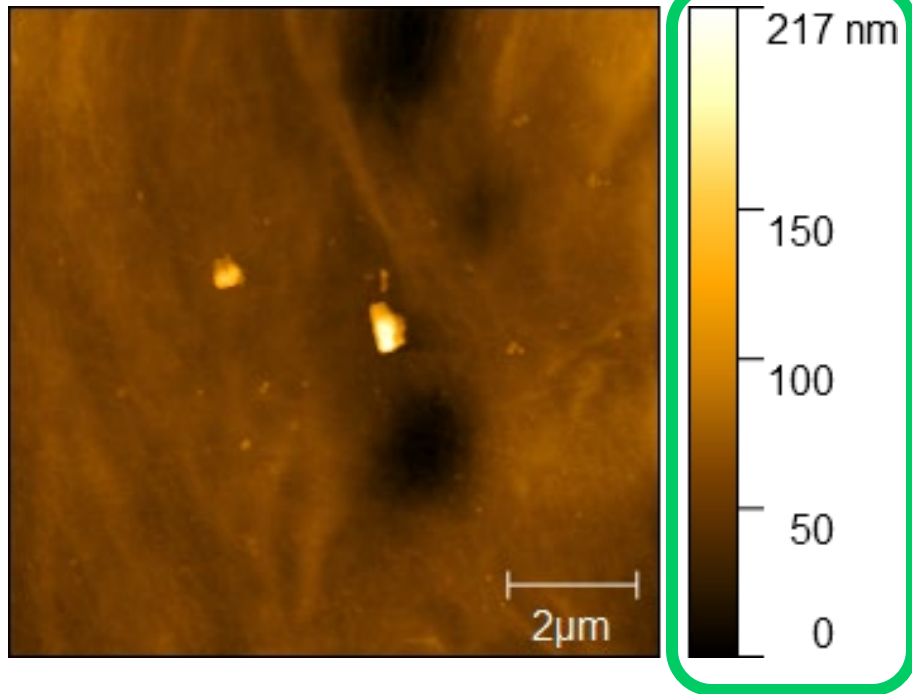
Application: Imaging

Polymer
Grating

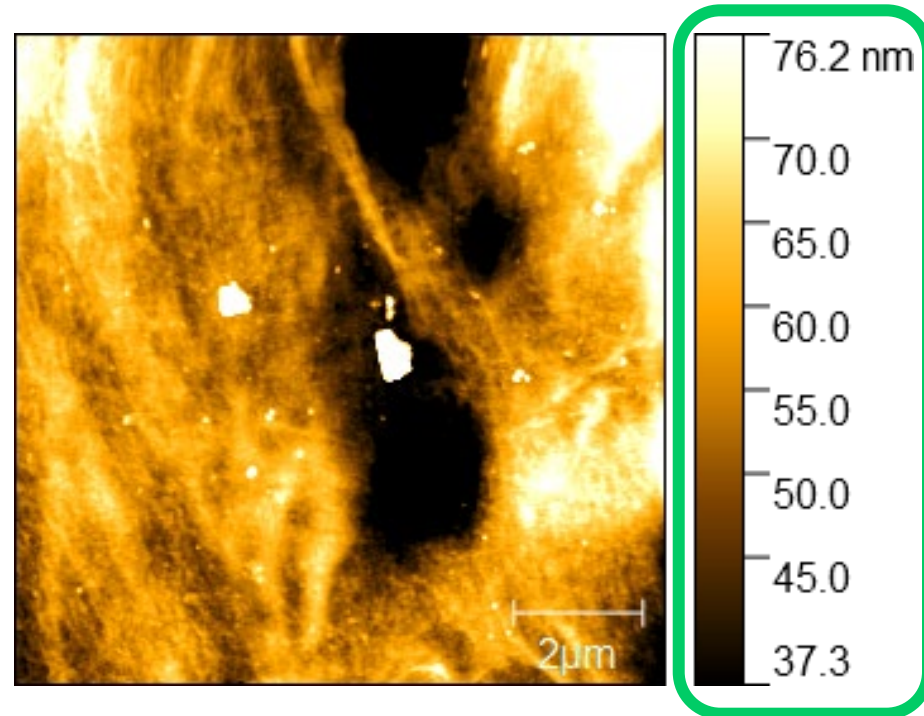


range of colors,
not heights in
the image

Reading the Colorscale

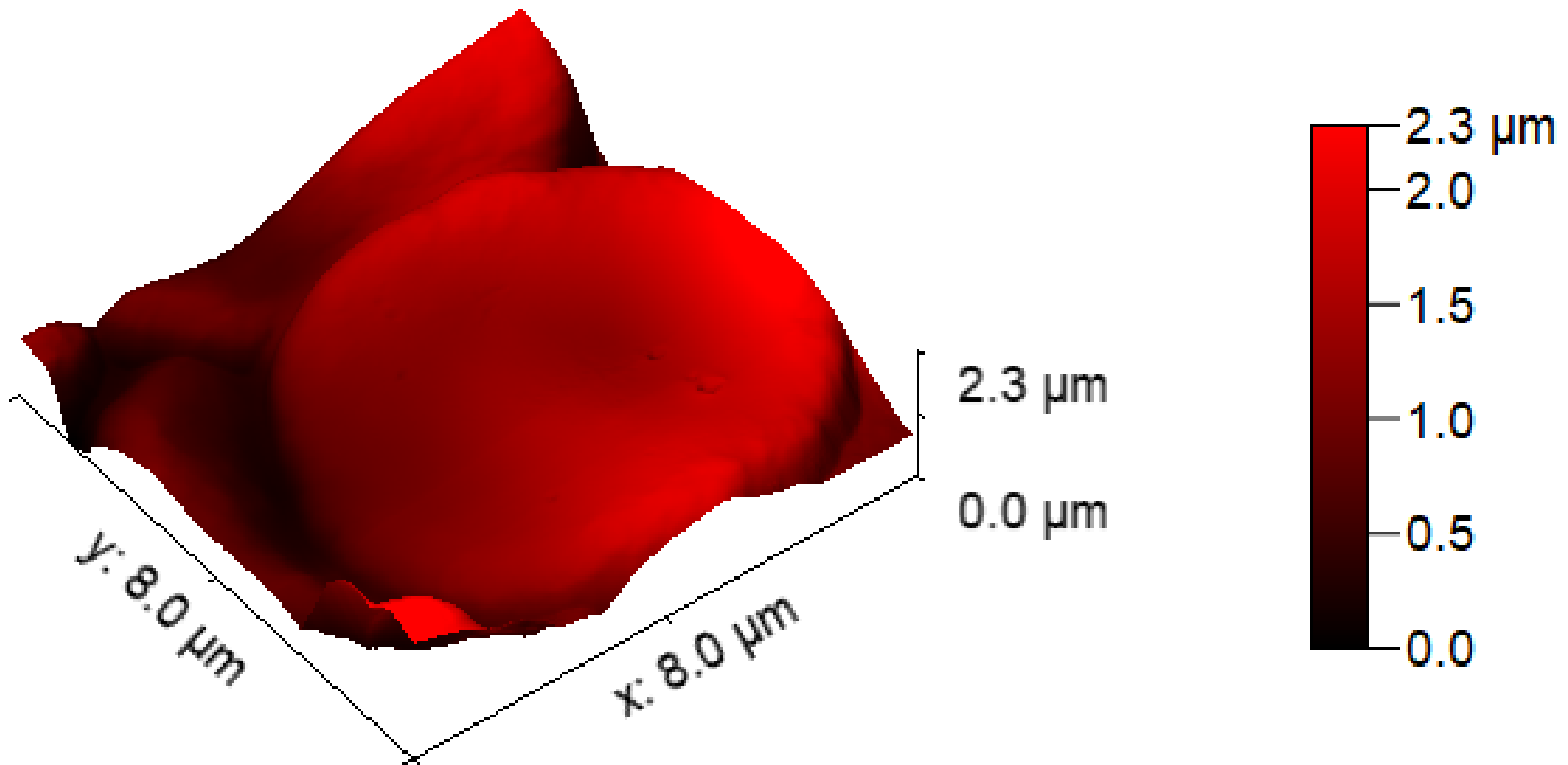


same image, different color ranges
color range of the displayed image,
not necessarily all heights on the surface



BOPP/PE polymer blend (toothbrush packaging), 10 μ m x 10 μ m AFM topograph

Interpreting 3D Images



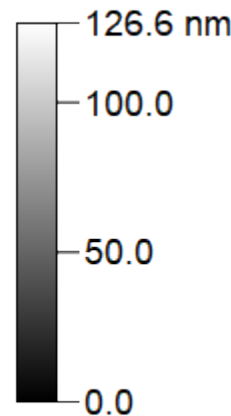
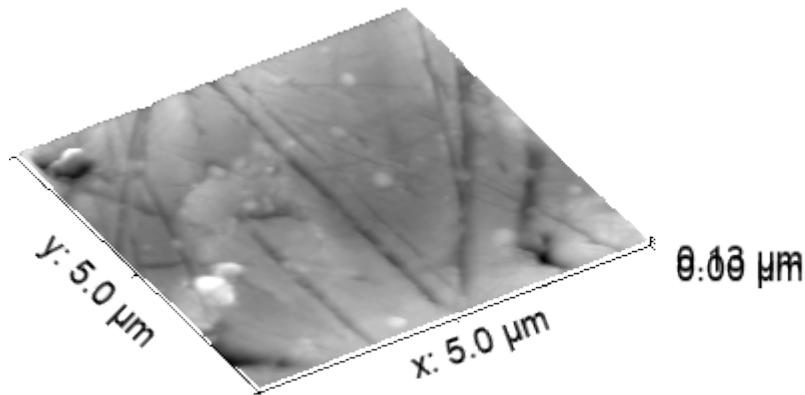
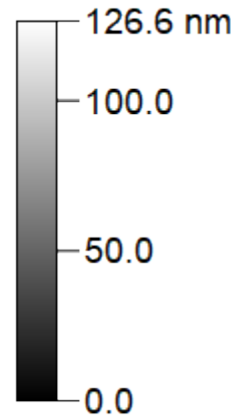
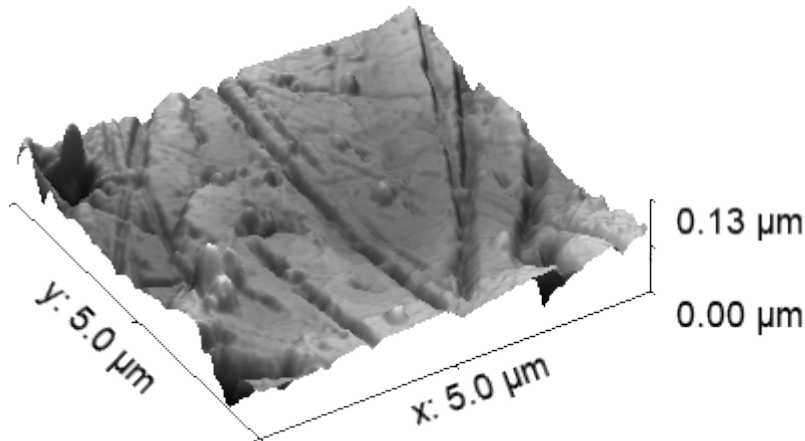
blood cells, 8μm x 8μm AFM topograph



Interpreting 3D Images

not necessarily 1:1:1 z:x:y

z often exaggerated compared to xy to convey texture information



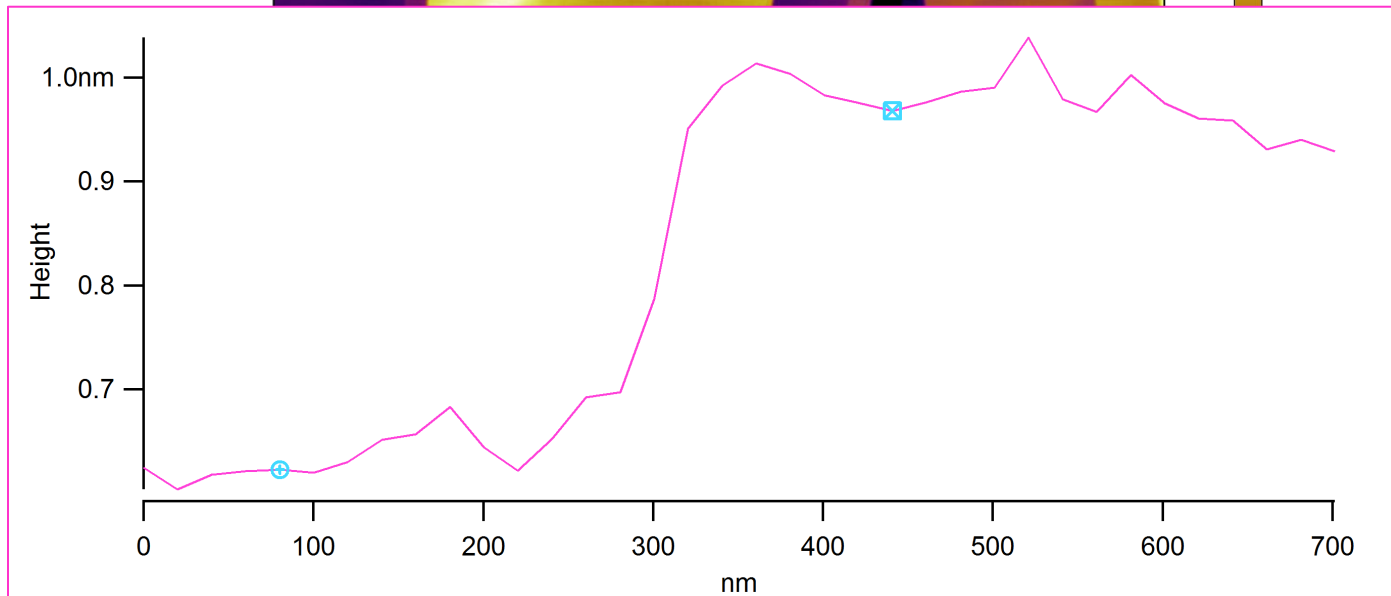
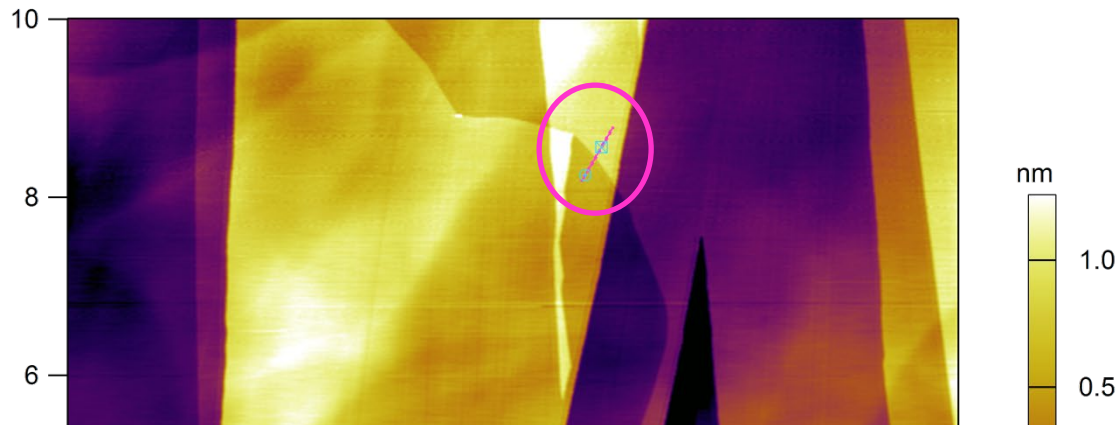
steel sample disk
5 μm AFM topograph

(blood and steel samples
courtesy Physics 403 Lab 2022)



Application: Step Heights

HOPG

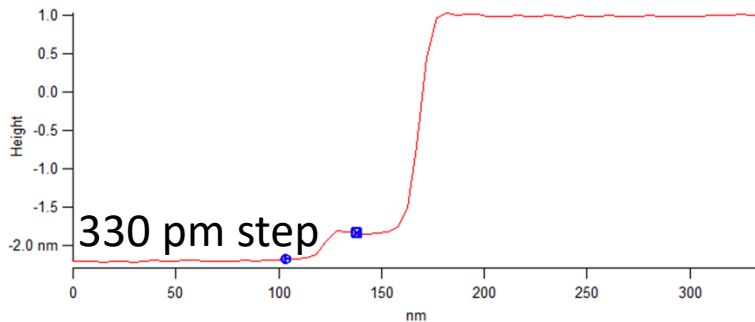
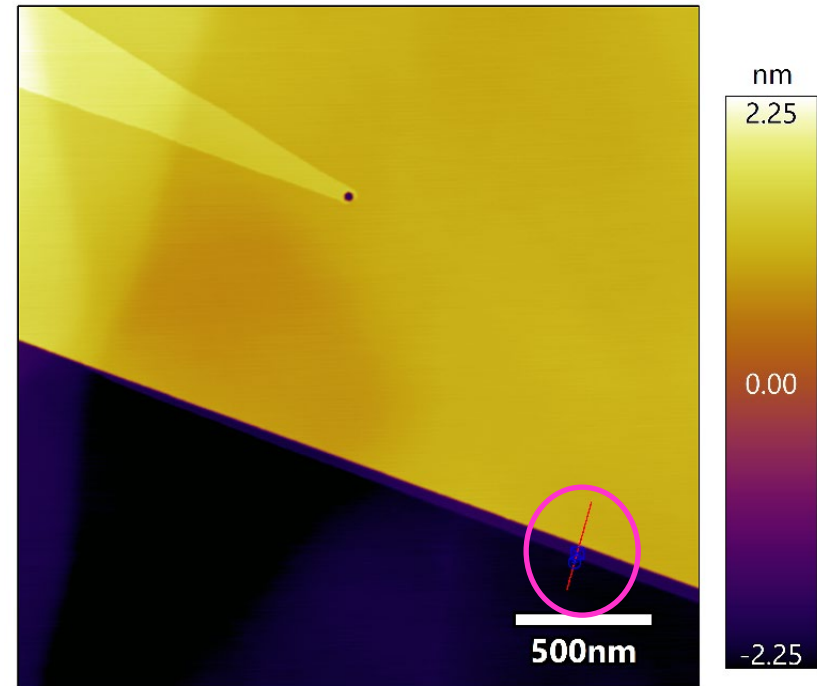




Application: Step Heights

HOPG

(highly oriented pyrolytic graphite)



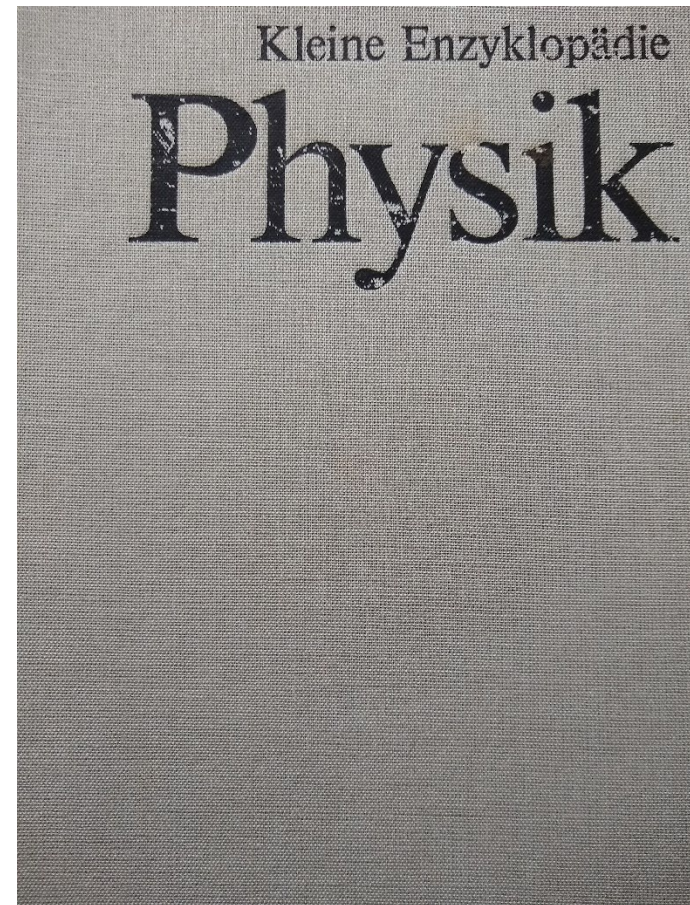
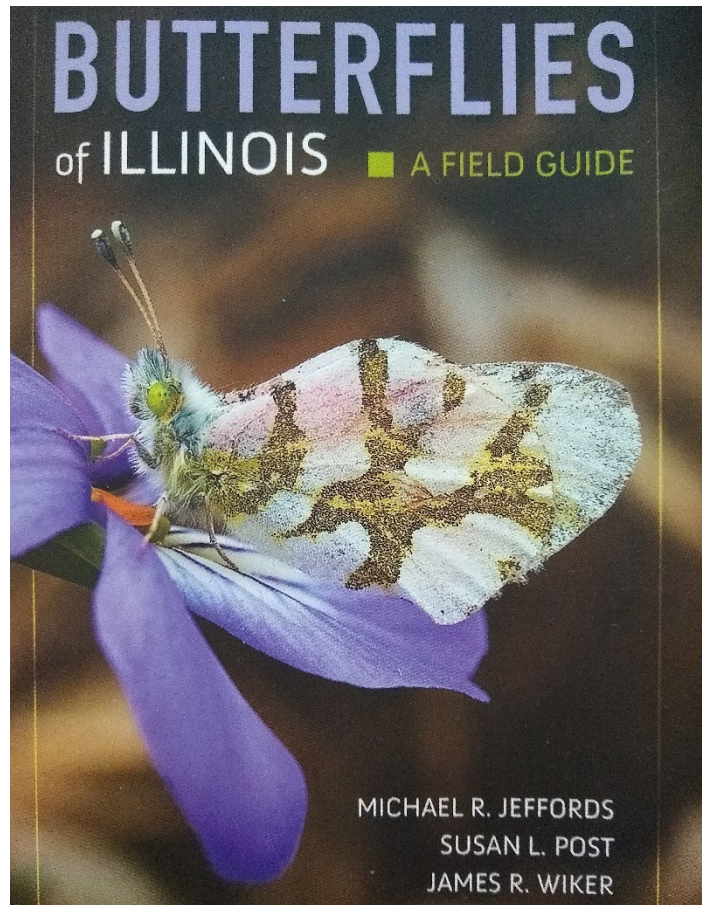
Xa	2.04 μm
Ya	458.97 nm
Za	-2.18 nm
Xb	2.05 μm
Yb	492.15 nm
Zb	-1.85 nm
dx	9.31 nm
dy	33.18 nm
dz	332.26 pm

HOPG_0006HeightTraceMod0



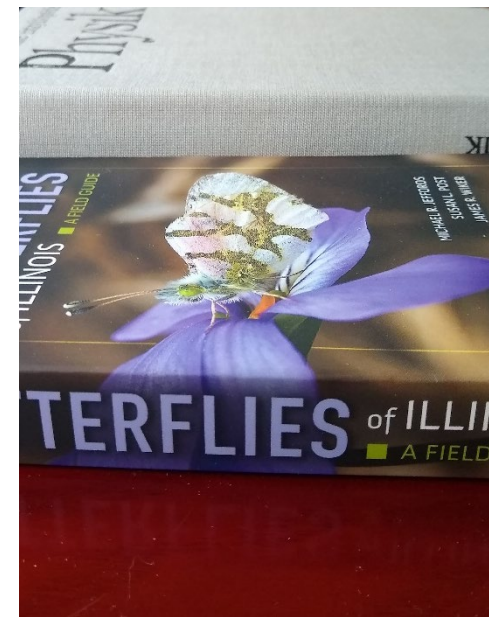
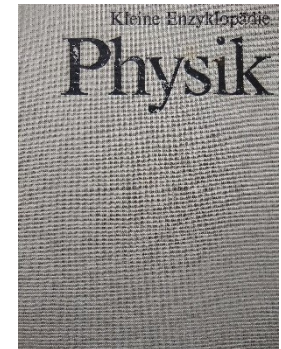
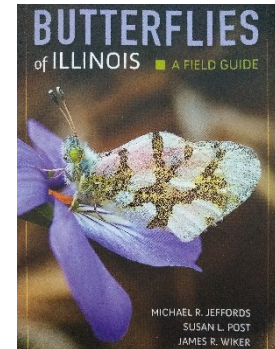
Step Heights and Thicknesses

Which book is thicker?



Step Height: Relative Height

- Film thickness is measured by step height
- Measure a height difference
 - Leave some bare substrate (patches are OK)
 - Scratch down to the substrate
 - Multilayer material—exposed underlayer





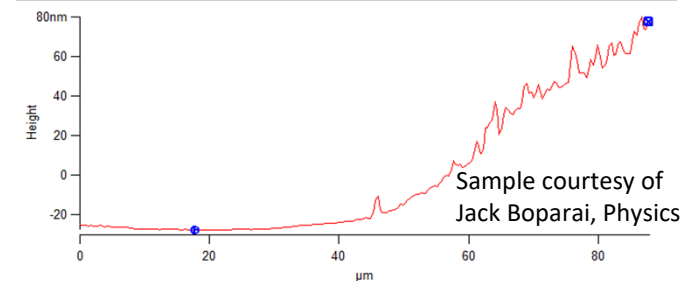
Step Height/Film Thickness: Complementary Techniques

If your step's too broad for the AFM
(edge width $> \sim 80\mu\text{m}$), try...

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

} Need a height difference (step) like AFM

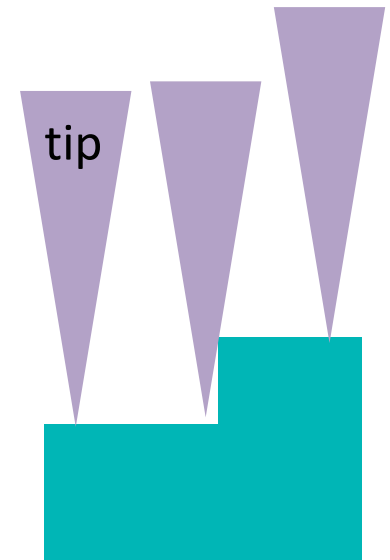
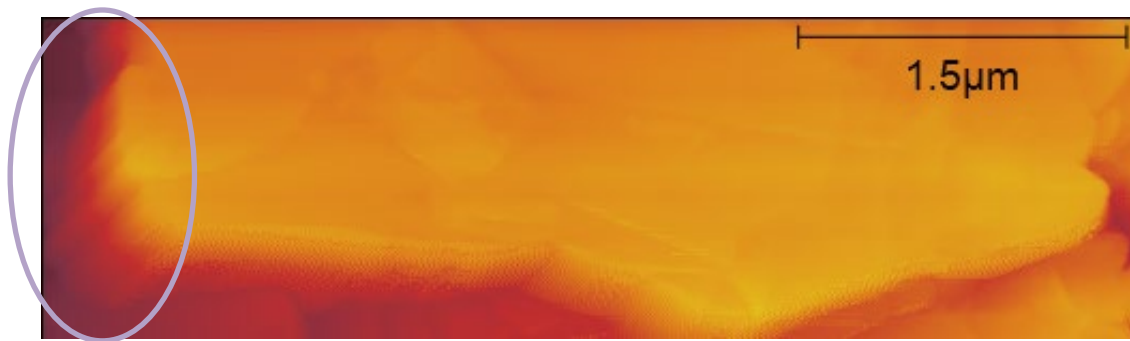
} Continuous film (no steps)
May need to know density



Width Measurements

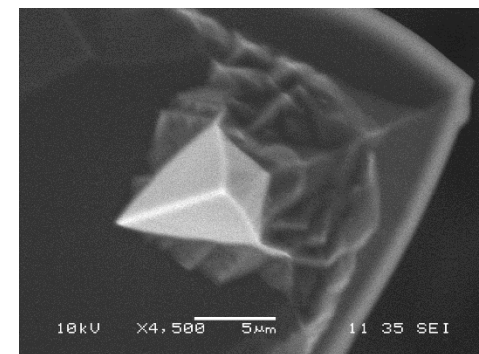
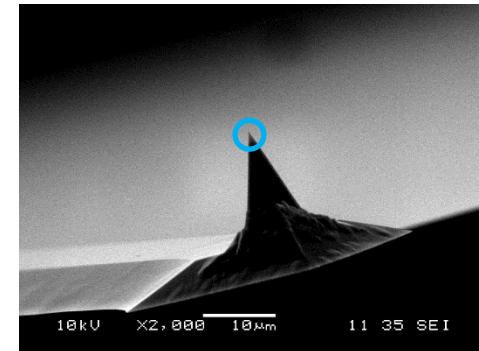
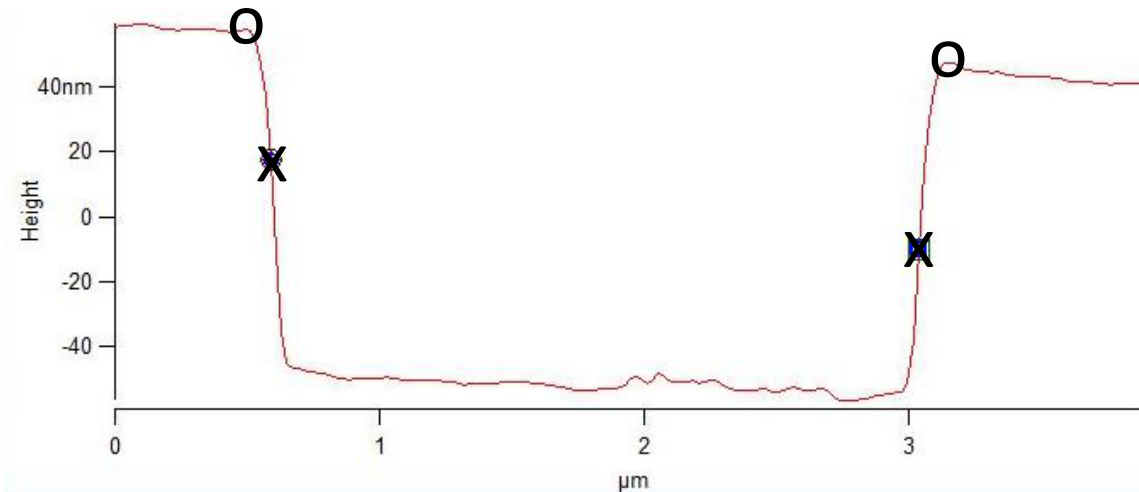
Beware of tip shape convolution

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



Width Measurements

- 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 (nanoscale)
 - Stylus profilometry
 - 3D optical profilometry

What is the roughness of this landscape?



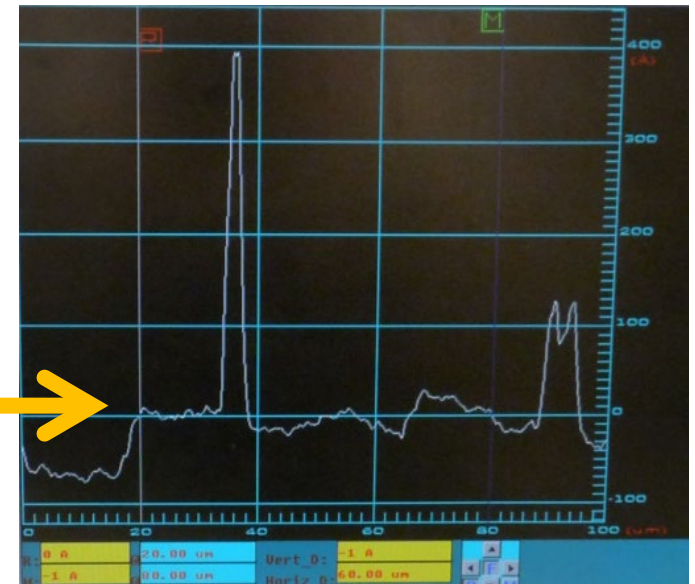
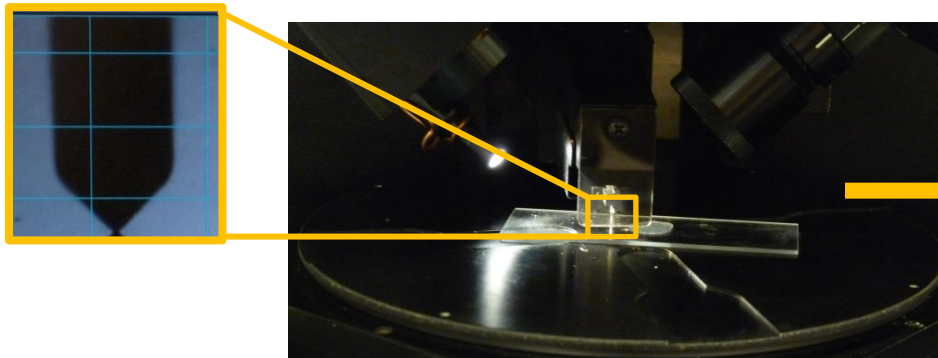
Michael Jeffords and Susan Post, University of Illinois Prairie Research Institute
<https://photojournalingm-s.smugmug.com/Colorado-and-Kansas/i-3tJ3DZk/A>



Complementary: Stylus Profilometry



2D stylus profilometry
(line profiles)
(diamond tip)

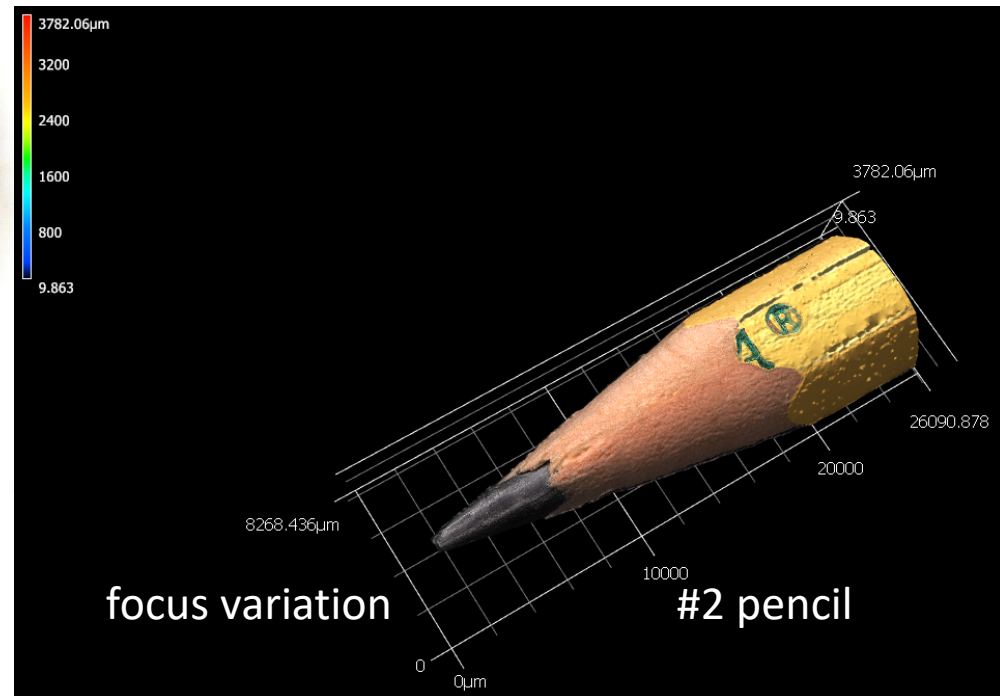




Complementary: Optical Profilometry

go.illinois.edu/MRL3DOpticalProfilometry

ladybug imaged during Cena y Ciencias using the Keyence VK-X1000
image by Kathy Walsh, MRL Facilities
sample courtesy of Julio Soares, MRL Facilities





Qualitative Comparison

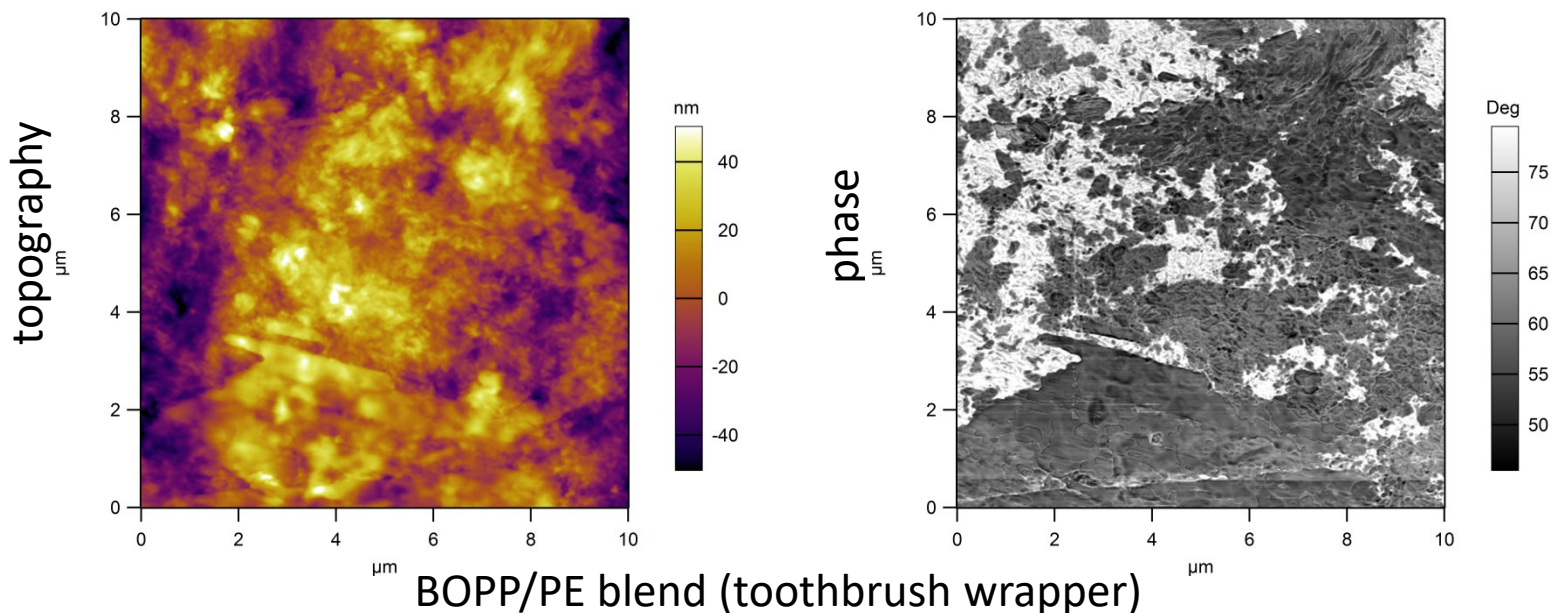
	AFM	2D Stylus Profilometry	3D Optical Profilometry
Vertical resolution	outstanding	OK	OK
Field of view	small	large	large
Data type	image	line	image
Max sample size	depends on instrument (~cm to large)	large	large
Max feature height	few μm	mm	mm
Force on sample	light	moderate	none
Speed	moderate	really fast	fast



Mechanical Characterization

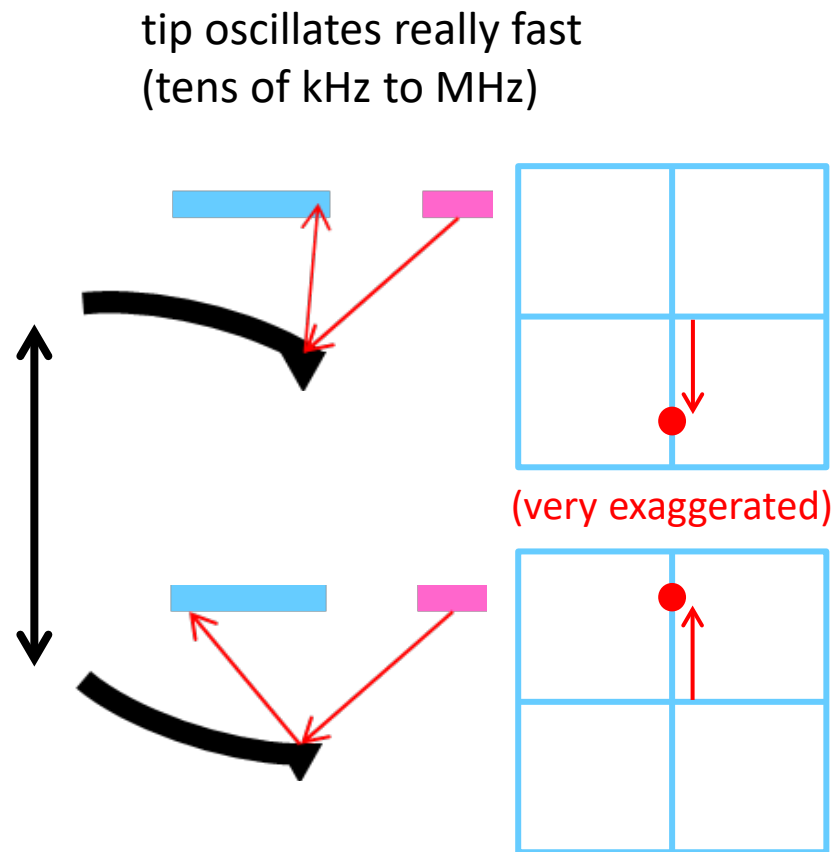
Visual impact of mechanical differences

- Phase (tapping mode)
- Force modulation, AM-FM, contact resonance, etc.
- Maps of quantitative measurement results (force mapping)



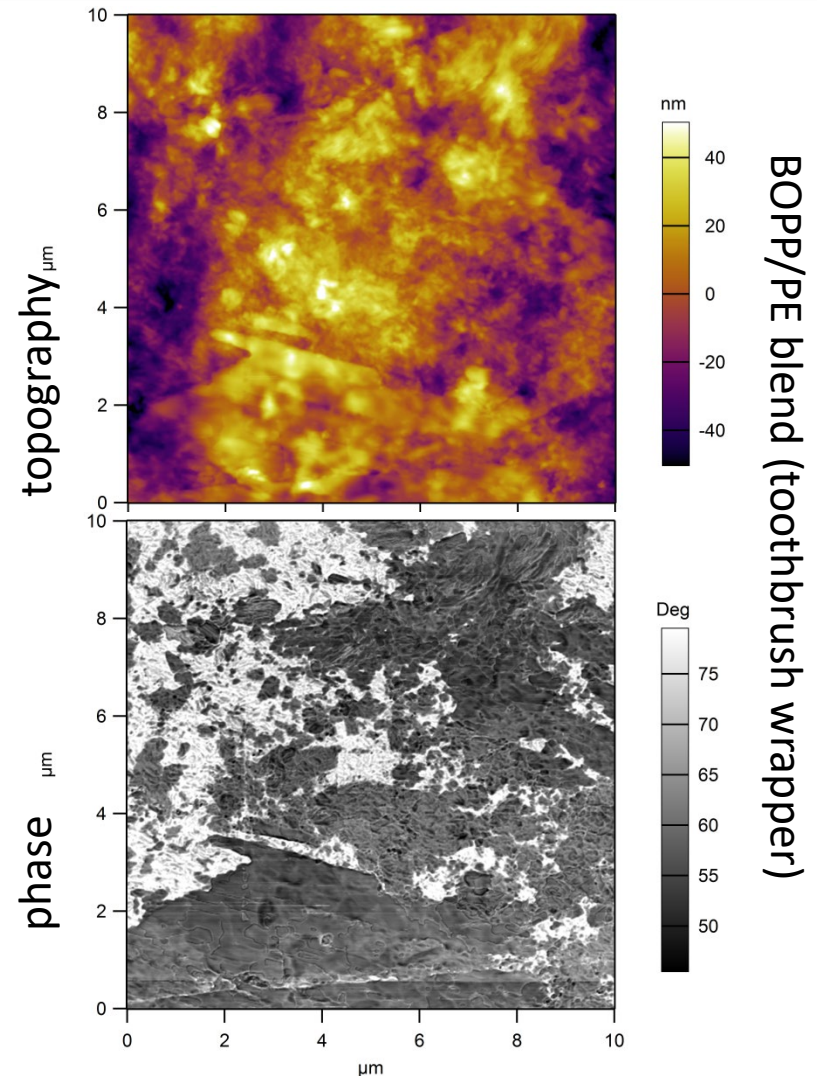
Tapping Mode Imaging: Phase

- Oscillating cantilever
- Tip—surface interactions affect oscillation
 - Cantilever driven sinusoidally to keep a constant amplitude
 - Dissipative interactions cause a phase lag (delay)
 - Viscous areas
 - Sticky areas



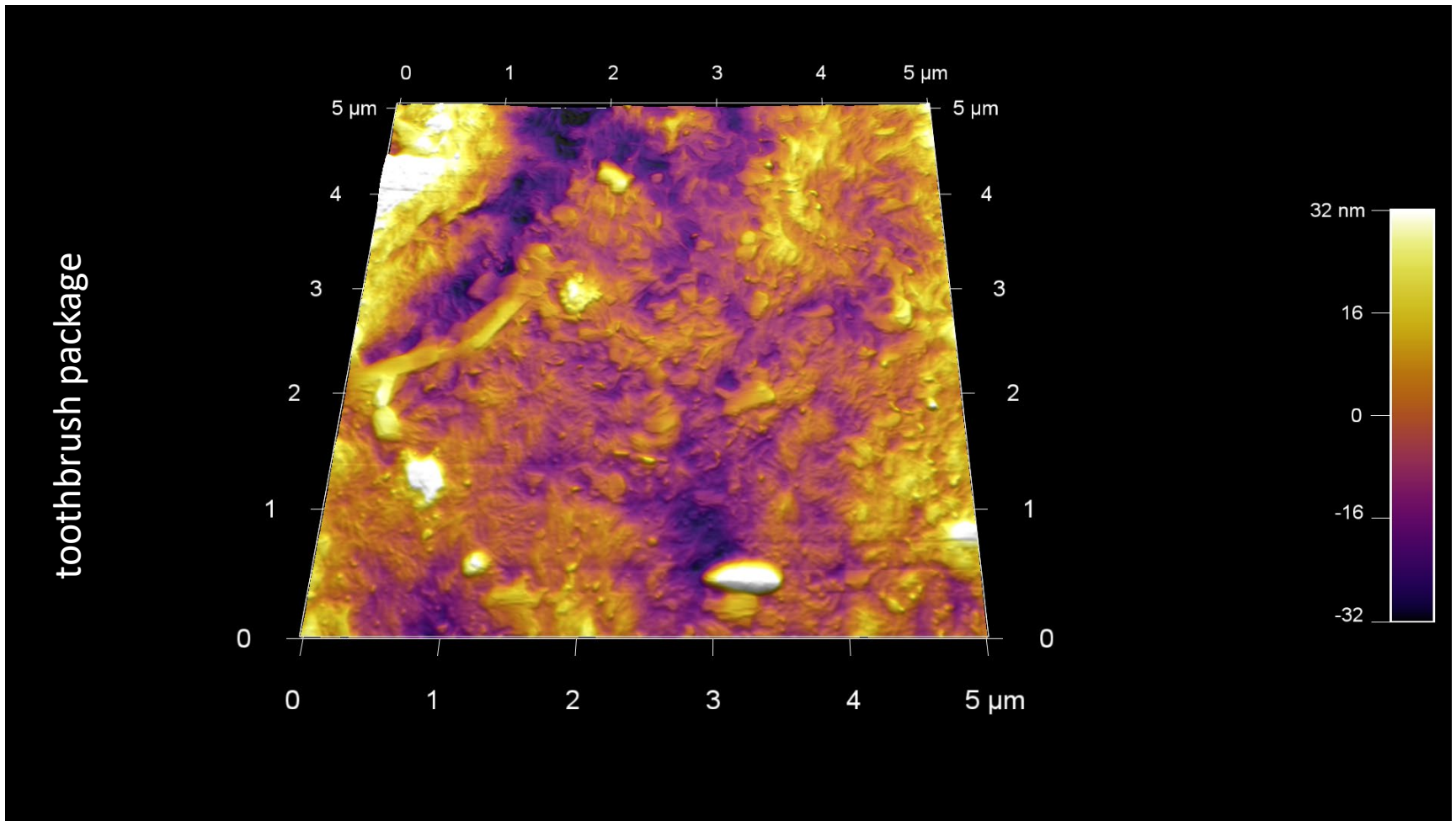
Phase (Qualitative)

- Tapping mode imaging
- Contrast in phase image shows differences in mechanical properties
 - Qualitative, not quantitative
 - Great for mixtures
 - Great for soft materials deposited on hard surfaces



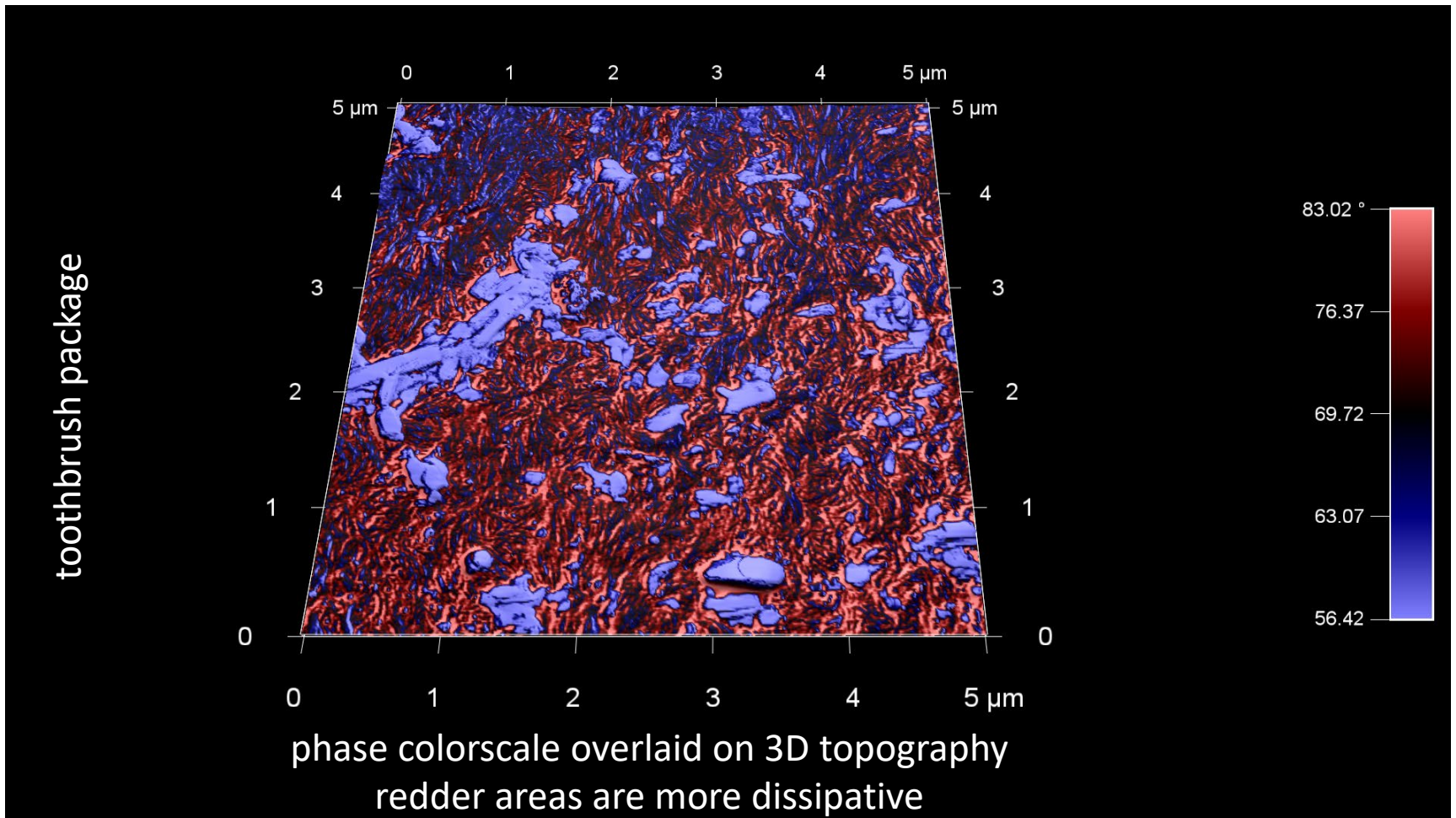


Topography



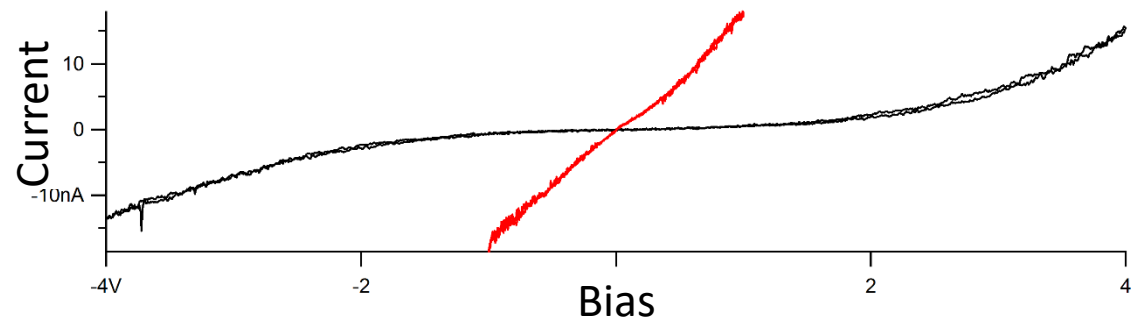
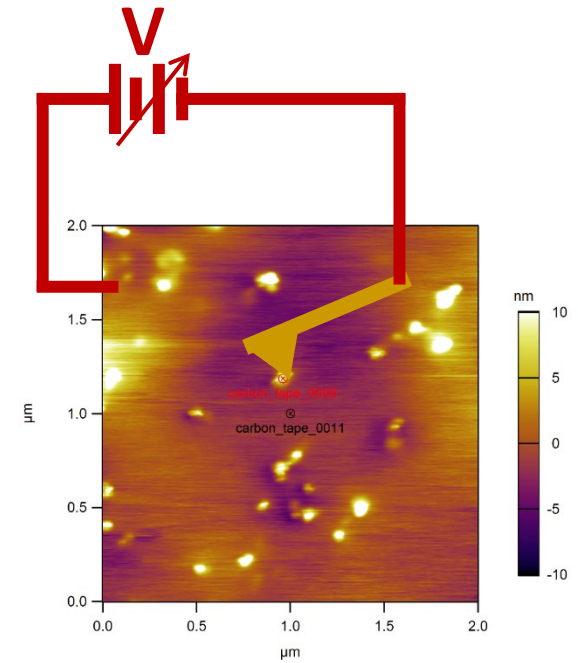
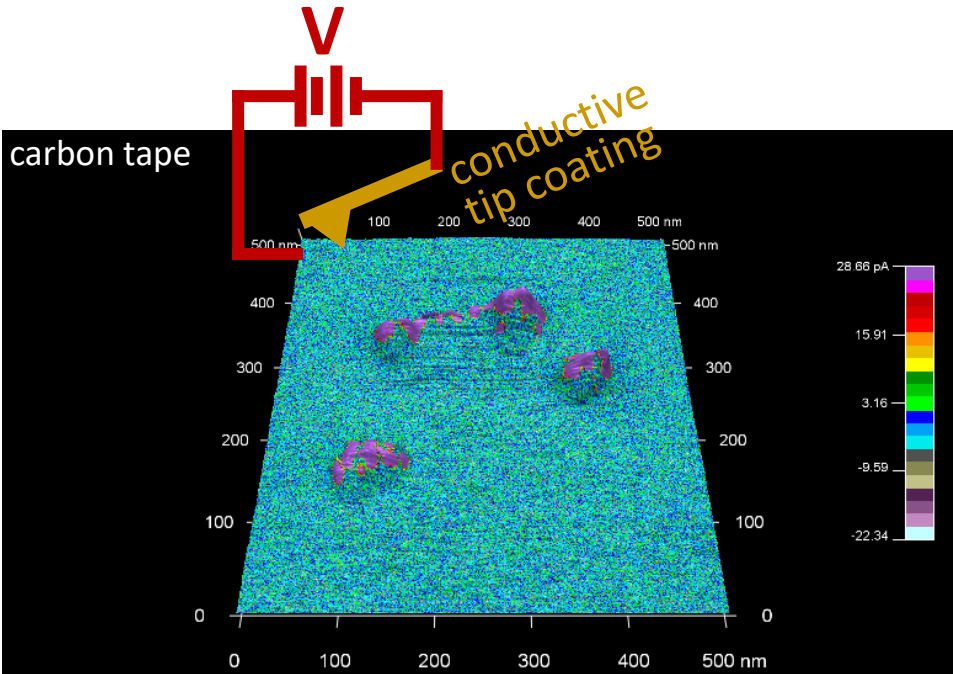


Topography with Colors from Phase





Common Application: Conductive AFM





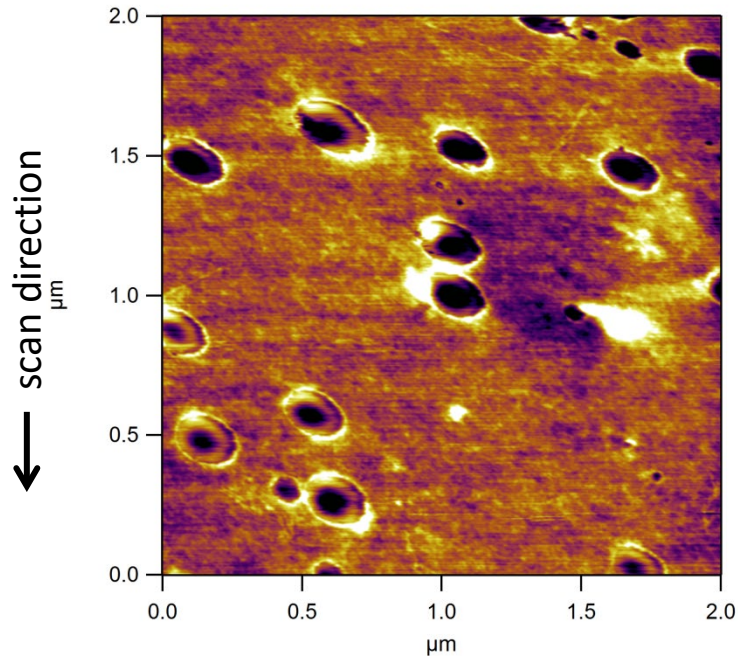
Artifacts and Image Processing

- Recognize what is (or is not) informative about the sample
- Images may not always exactly represent the sample: sample drift, tip condition/geometry
- Display data in an informative way
 - Correct for sample tilt, etc.

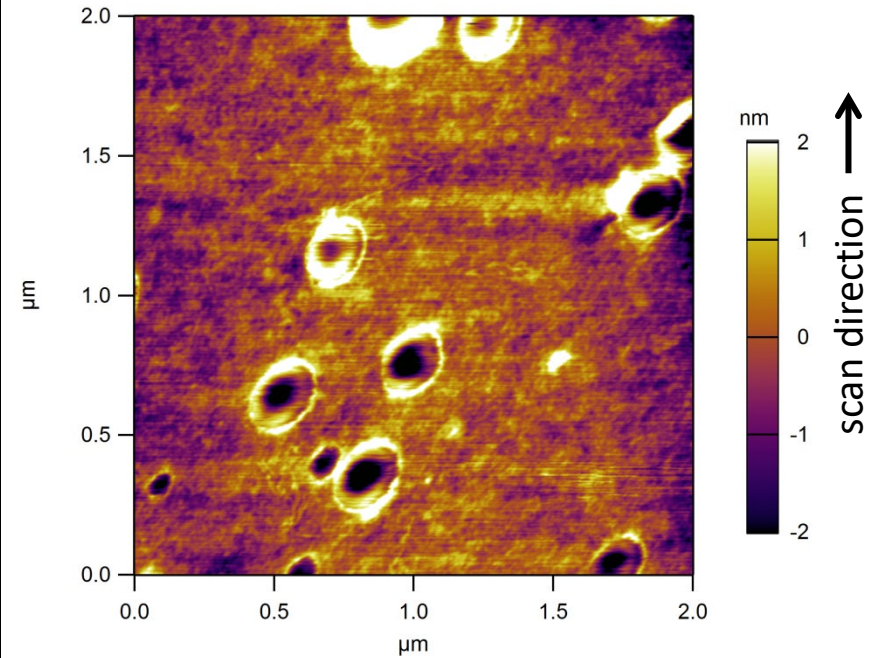


Sample Drift

Scanning downwards...



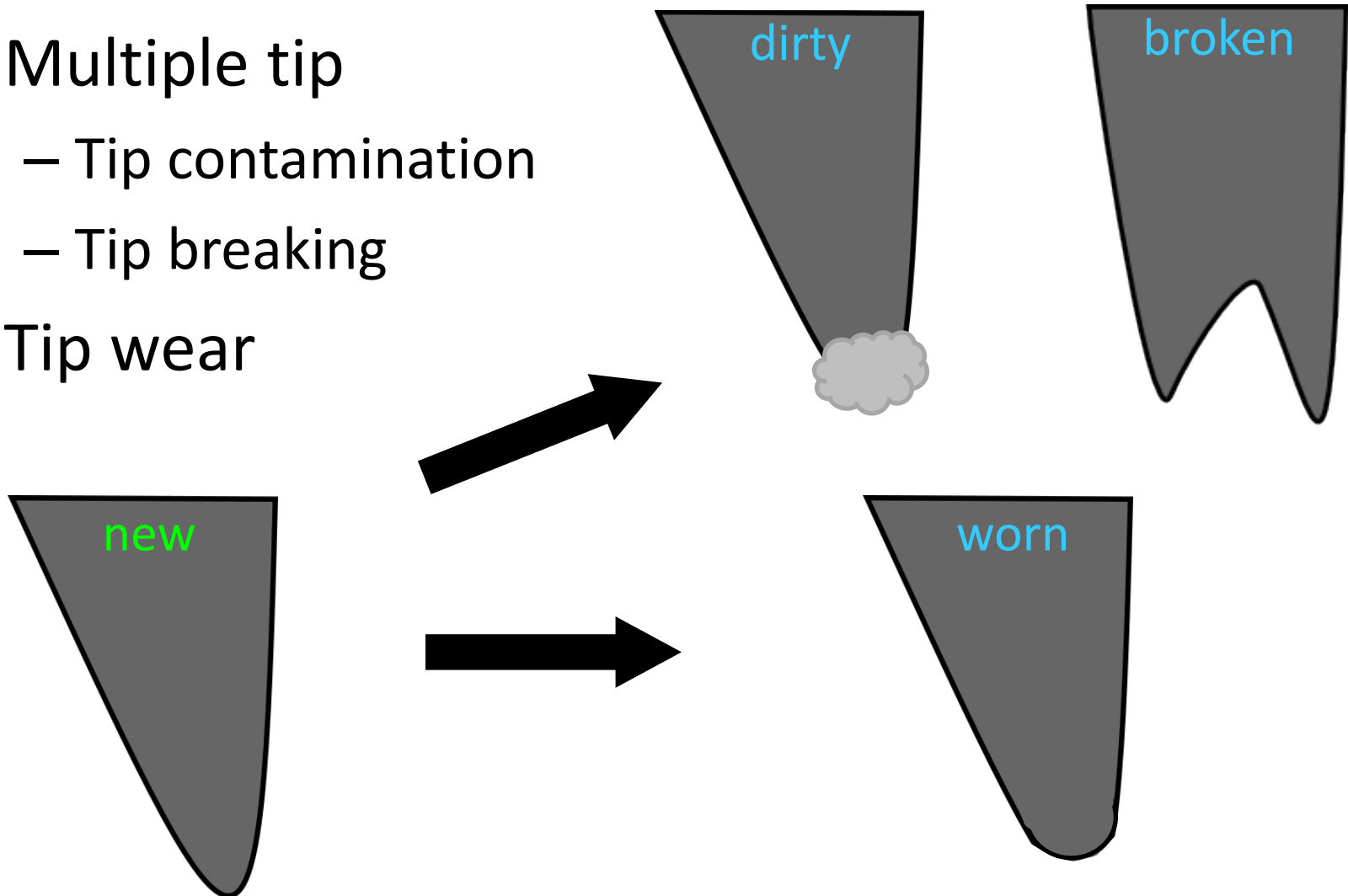
... then scanning upwards



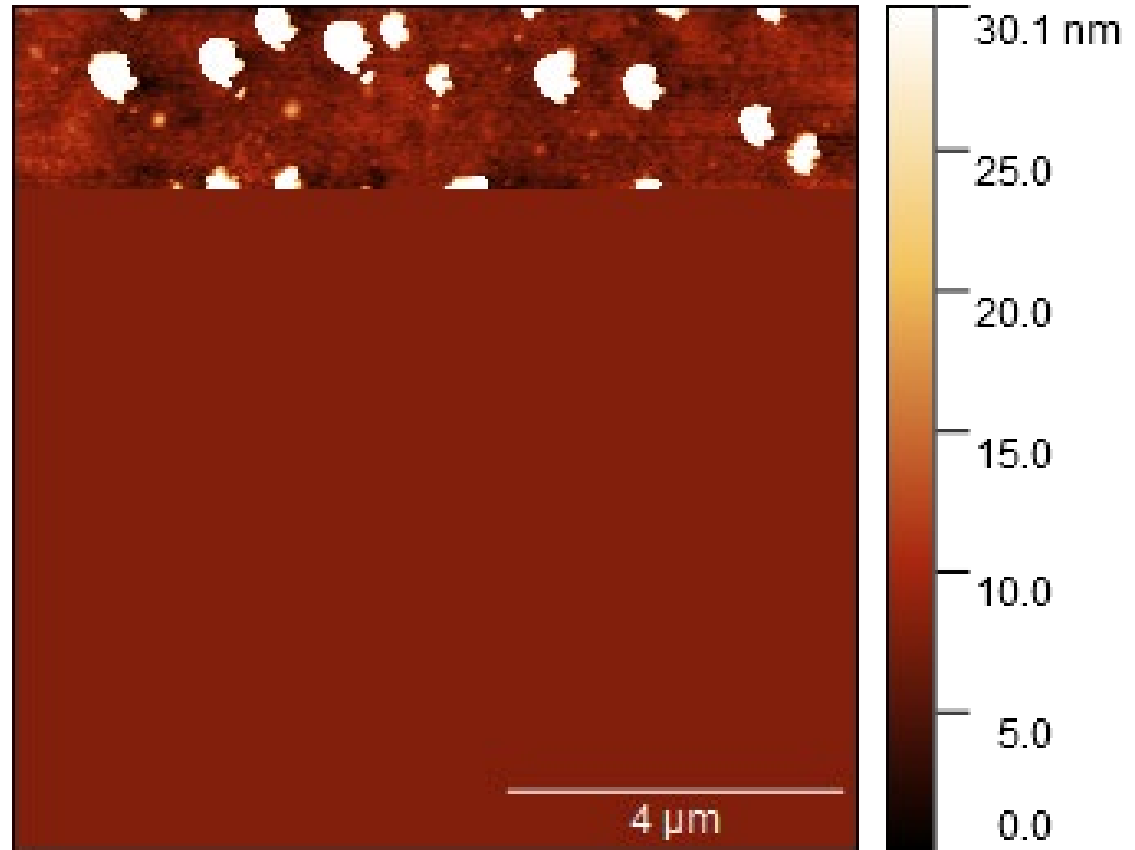
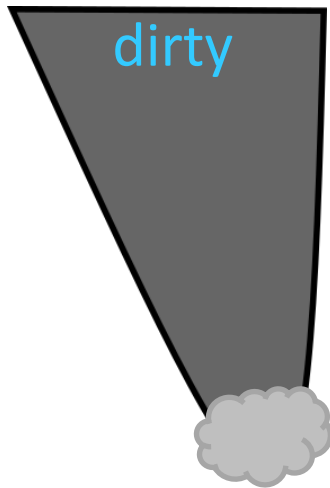
chewing gum

Tip Artifacts

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



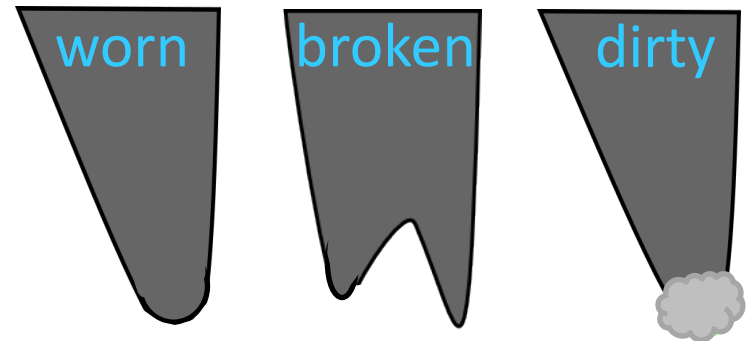
Contaminated Tip



10μm partial scan

Tip Artifacts

- Tip shape change
 - Multiple tip
 - Tip contamination
 - Tip breaking
 - Tip wear
- Tip height change
 - Tip contamination
 - Tip wear



Line-by-Line Subtraction

- Difference from line to line
- Tip condition changes, curvature
- Polynomial subtraction

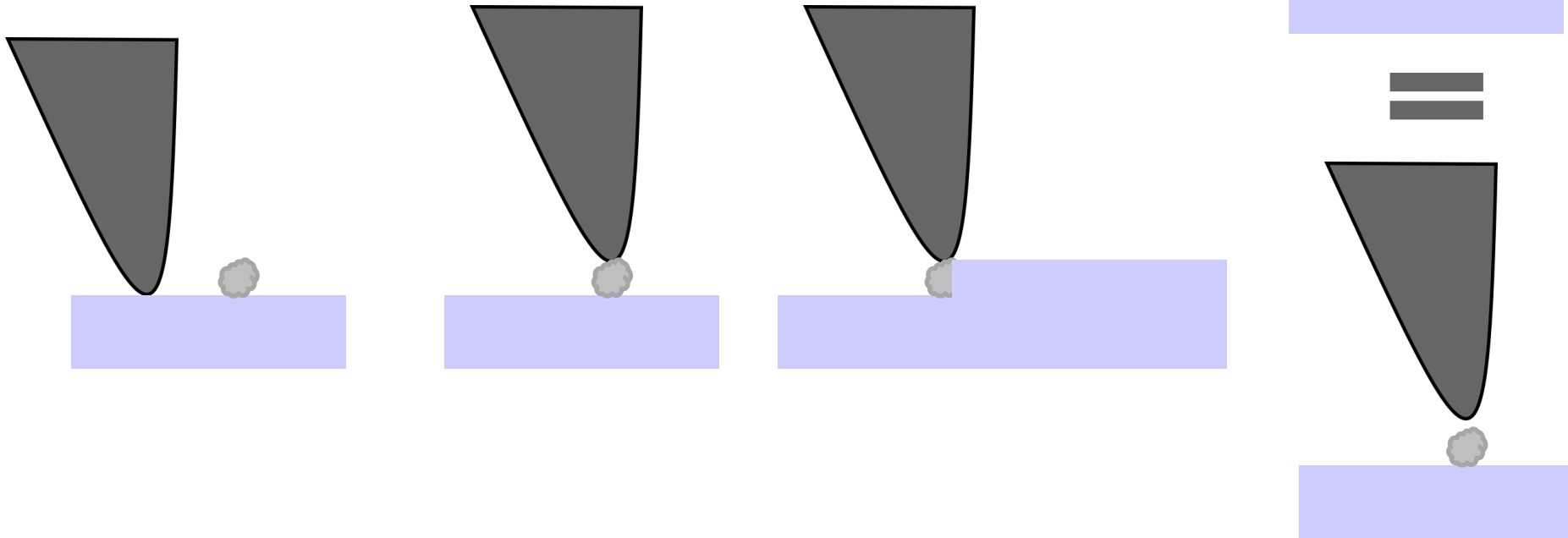




Image Processing

raw image

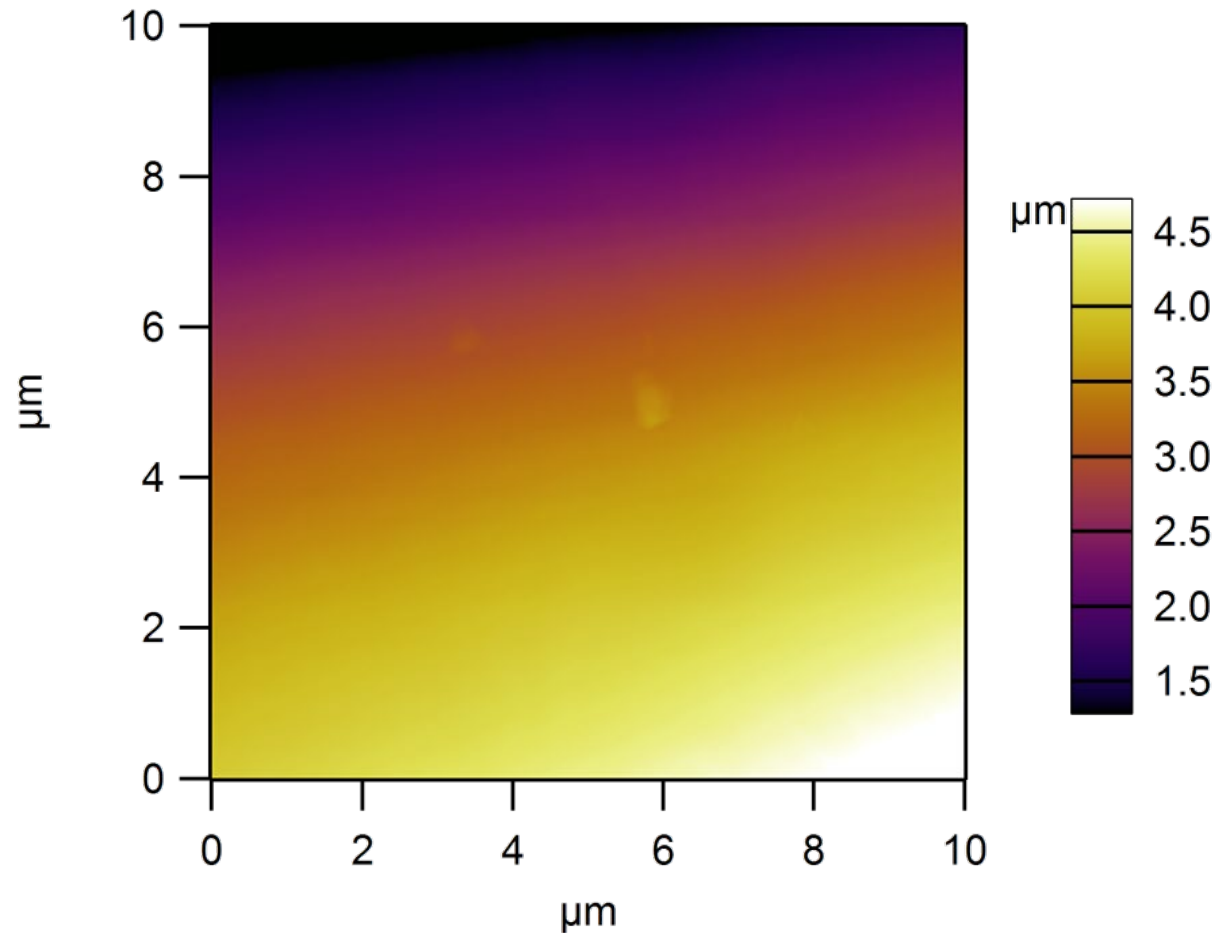




Image Processing

line subtraction

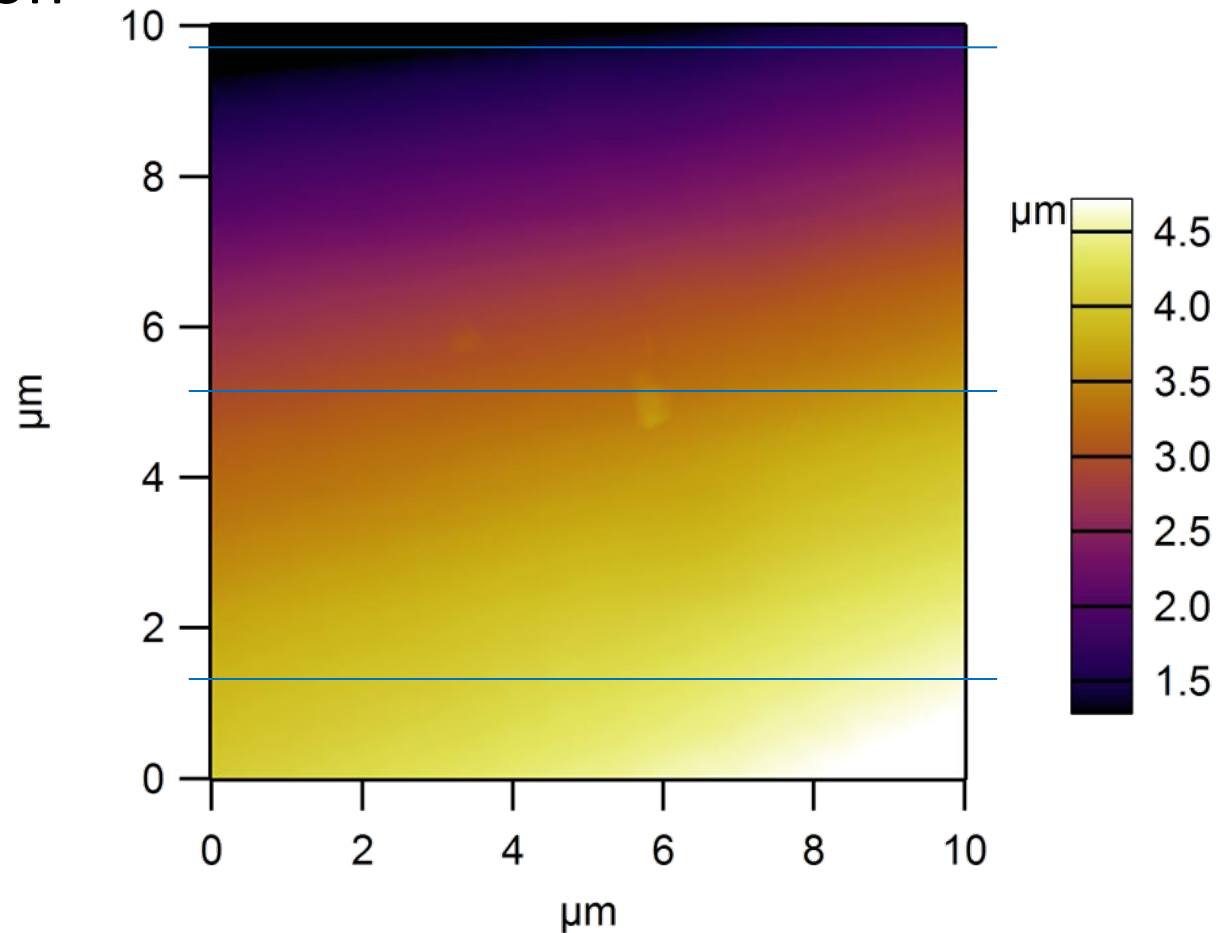




Image Processing

line subtraction

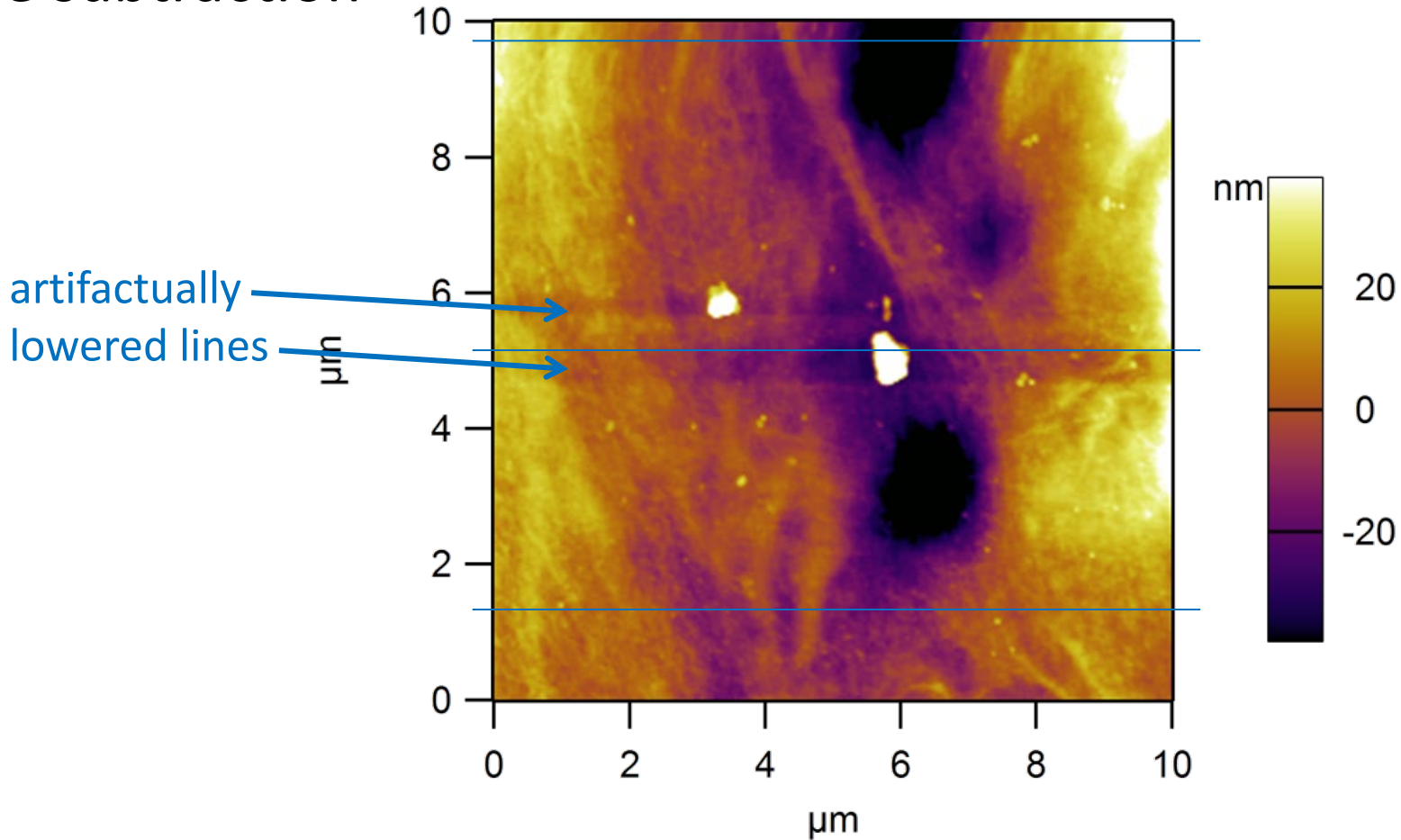




Image Processing

line subtraction:
mask outlier areas

areas to ignore
when processing

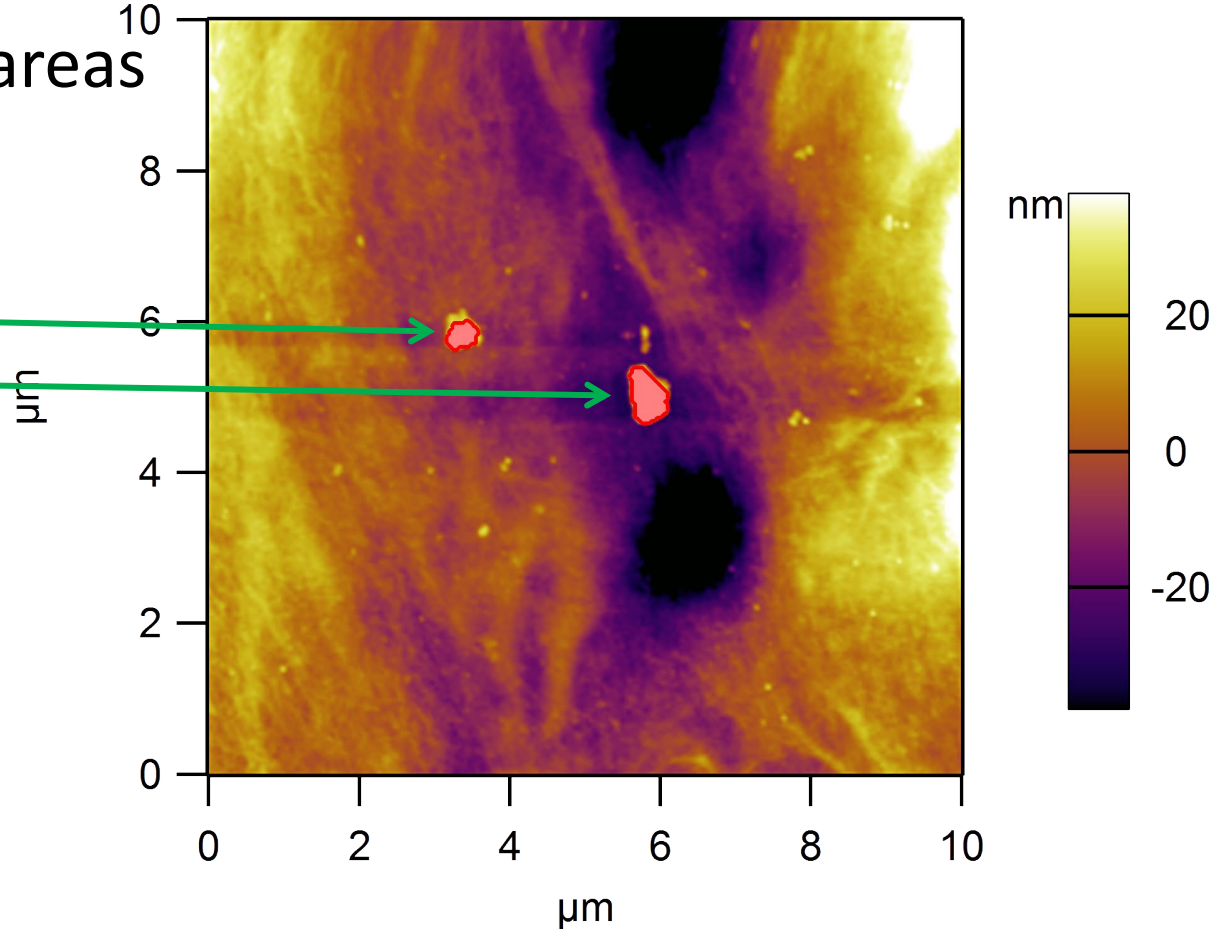


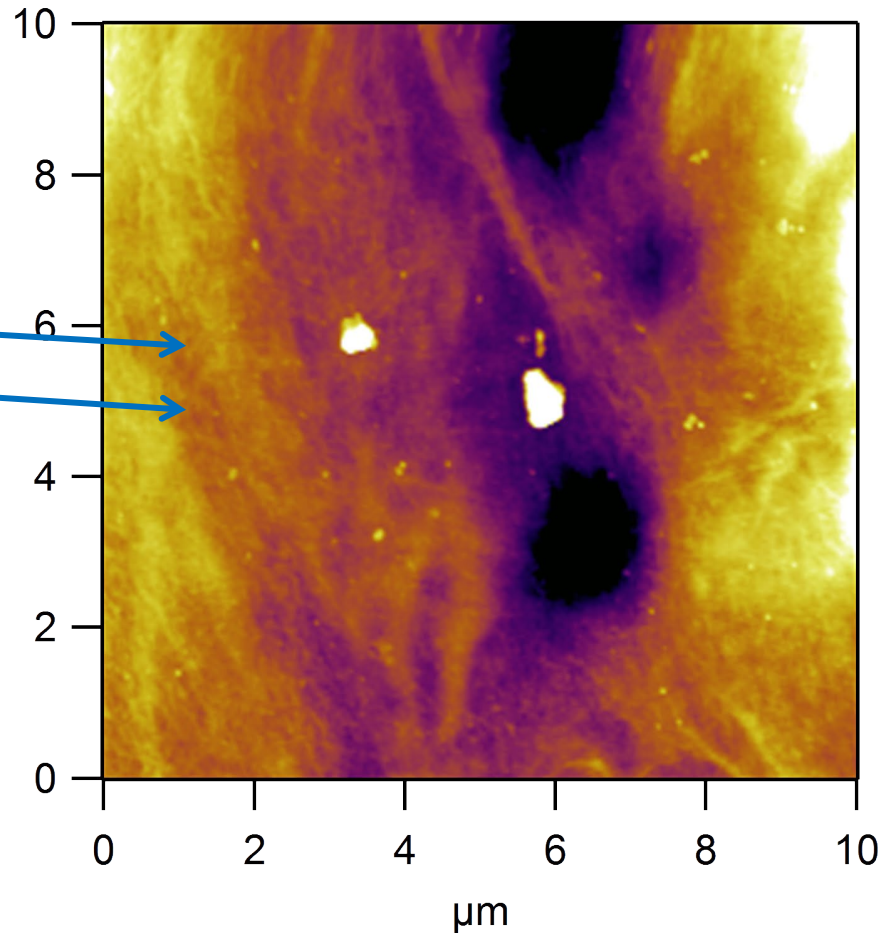


Image Processing

line subtraction:
masked flatten

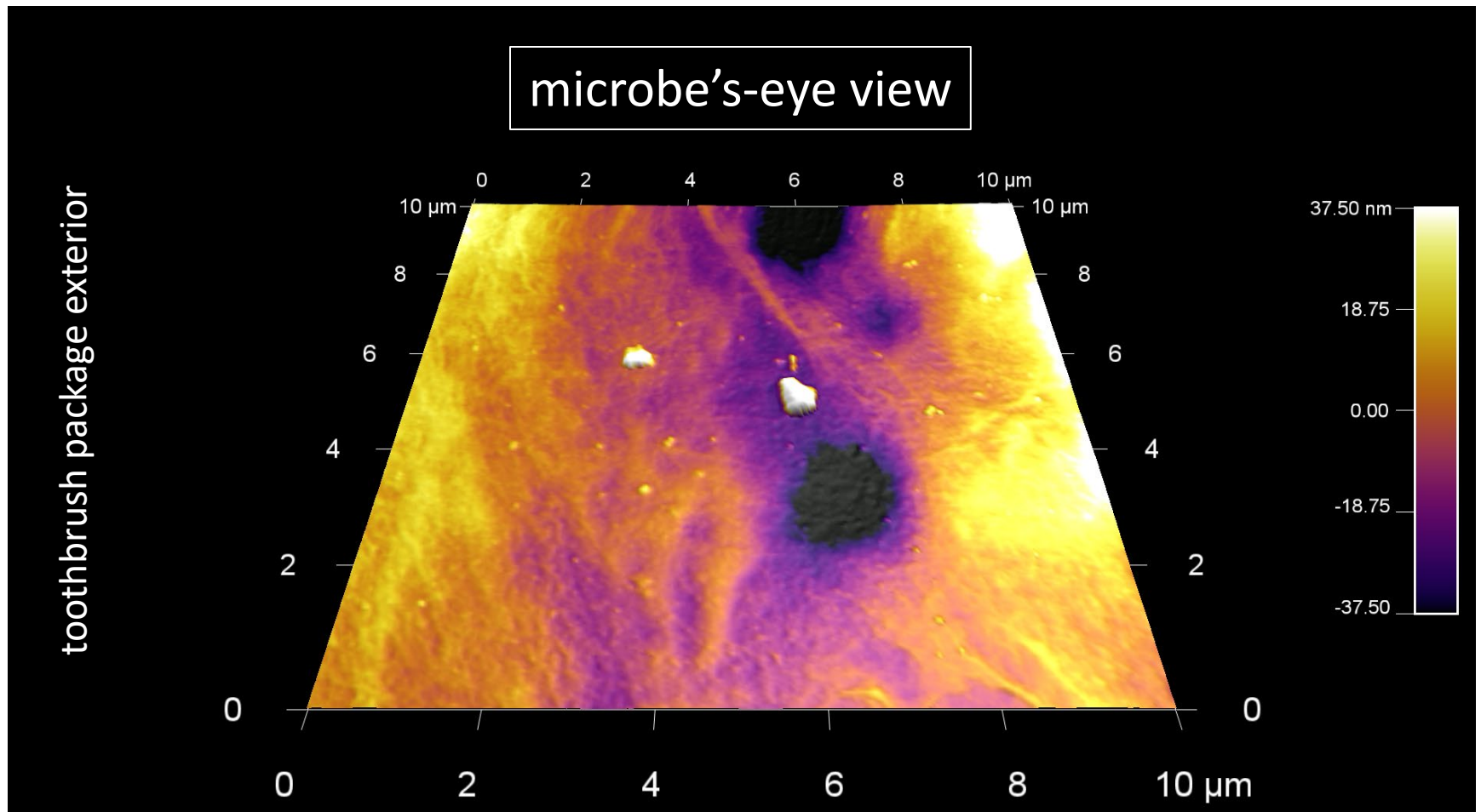
no more
streaks

μm





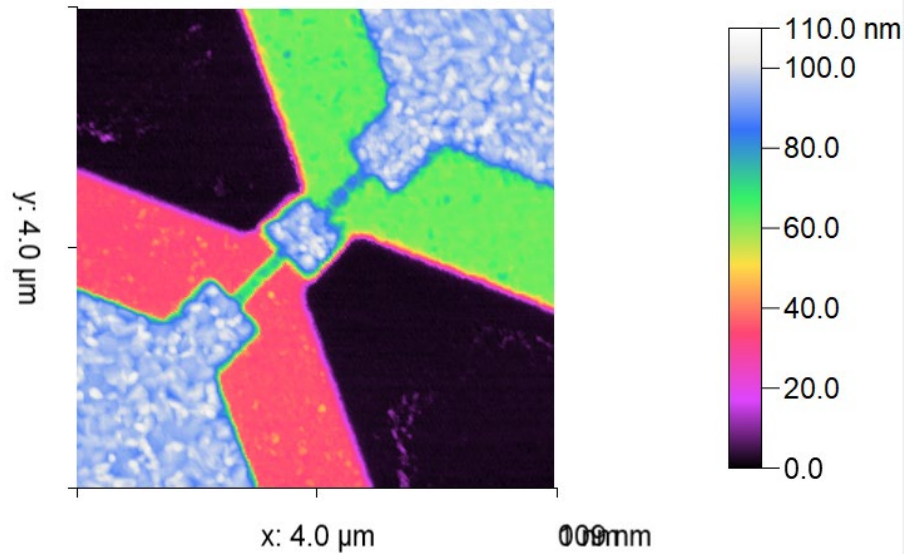
3D Display



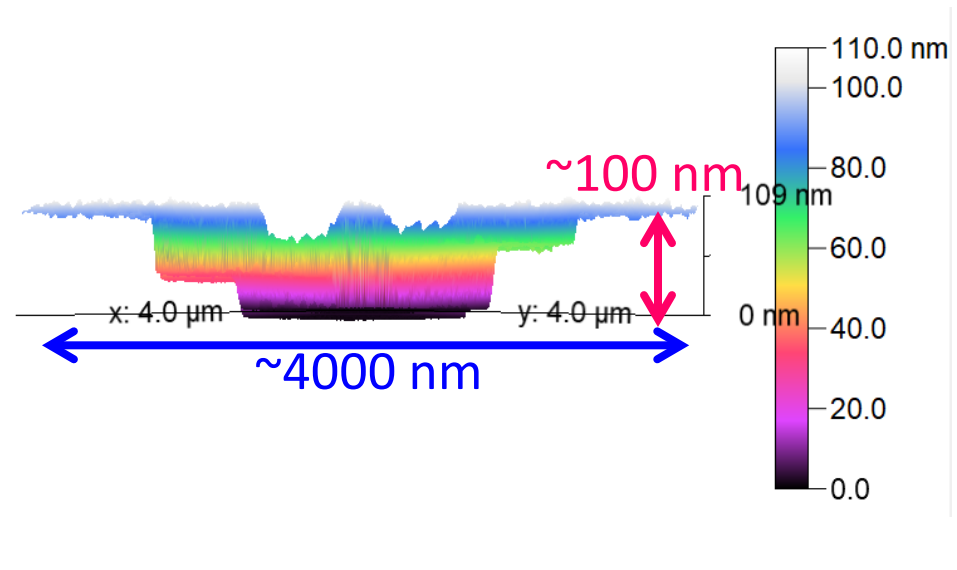


3D Display

face on



side view

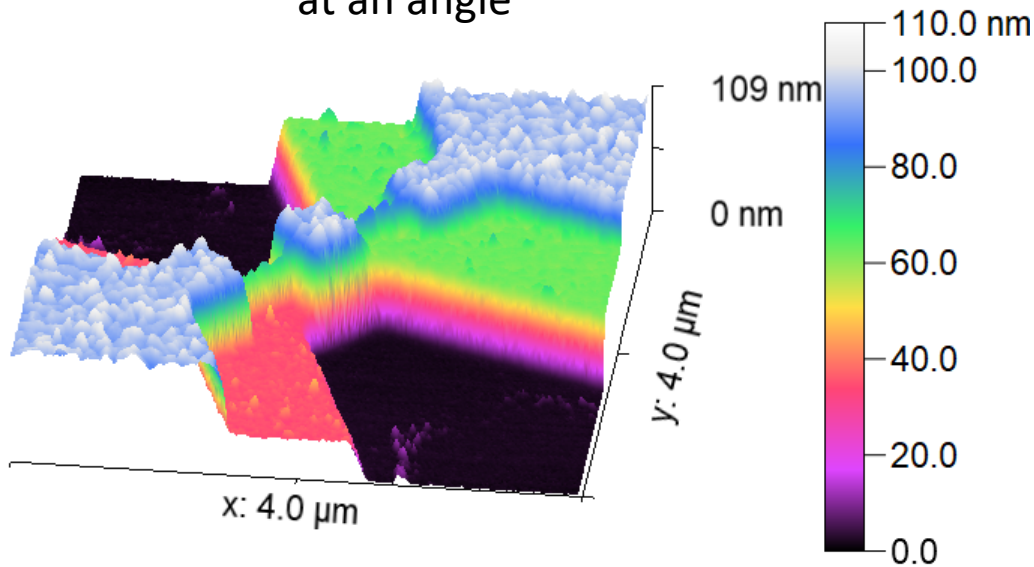


raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM

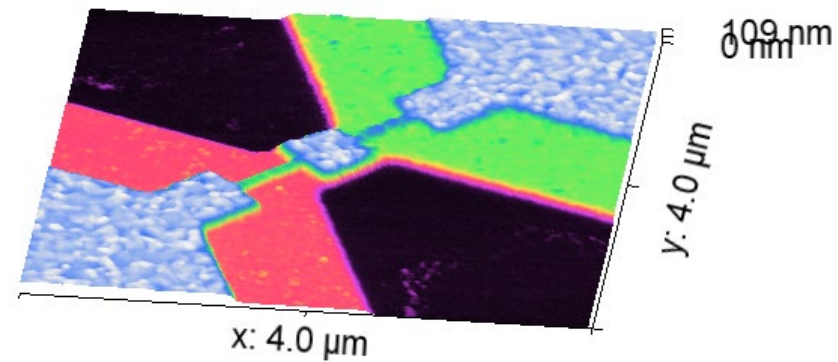


3D Display—z:xy

at an angle



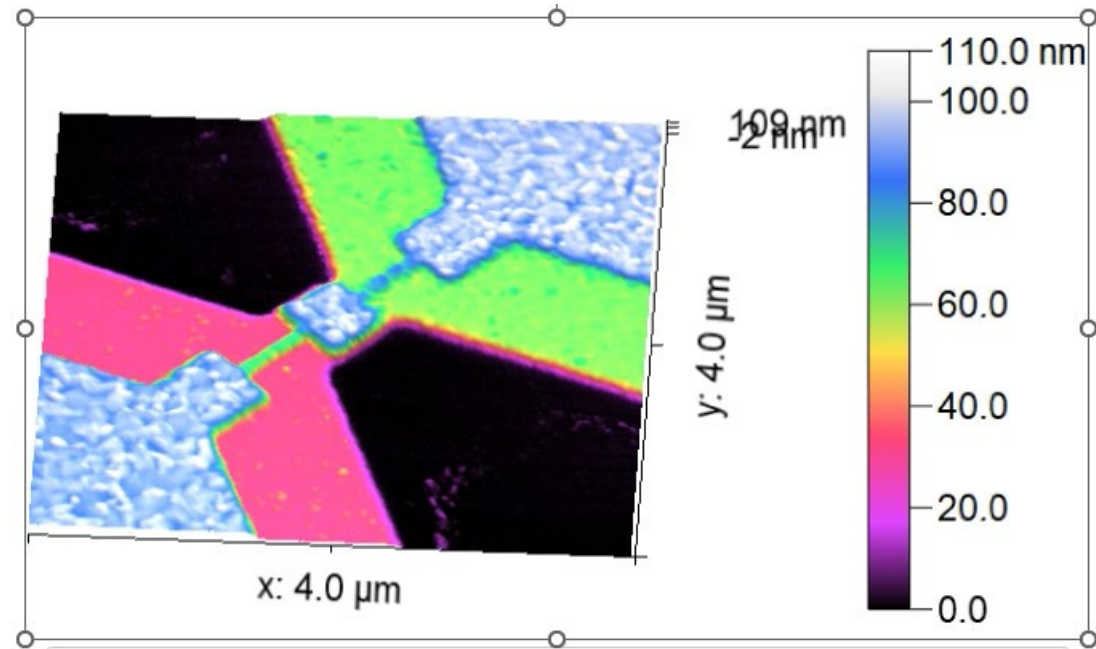
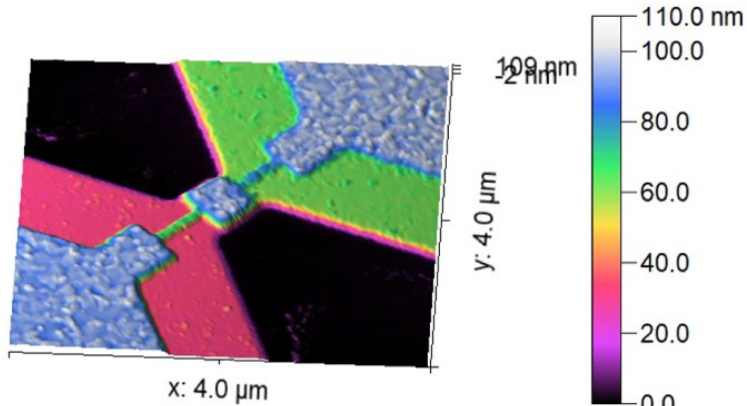
true z:xy



raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM



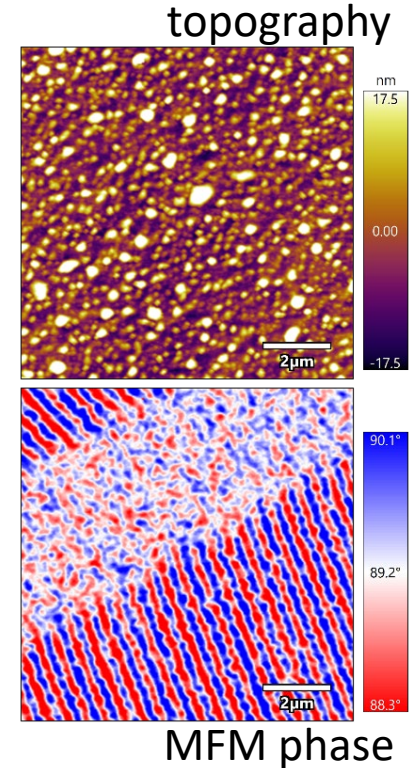
3D Display—Lighting Angle



raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM








Many Other Applications










- Nanolithography/nanomanipulation
- LFM (friction, lateral force microscopy)
- EFM (electrostatic force microscopy)
- KPFM (SKPM, Kelvin probe)
- MFM (magnetic force microscopy)
- PFM (piezoresponse force microscopy)
- ... and these generally don't need extra gear (except different tips)

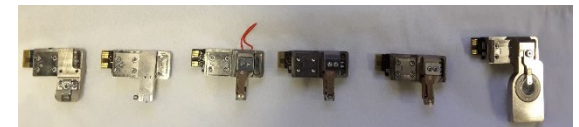
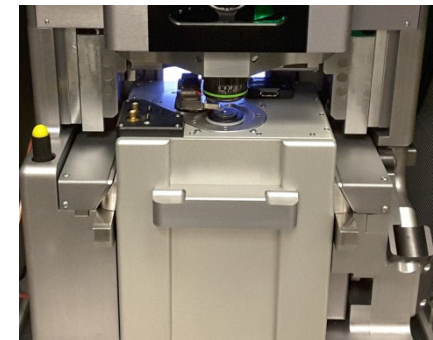




Attachments on the MRL AFMs

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

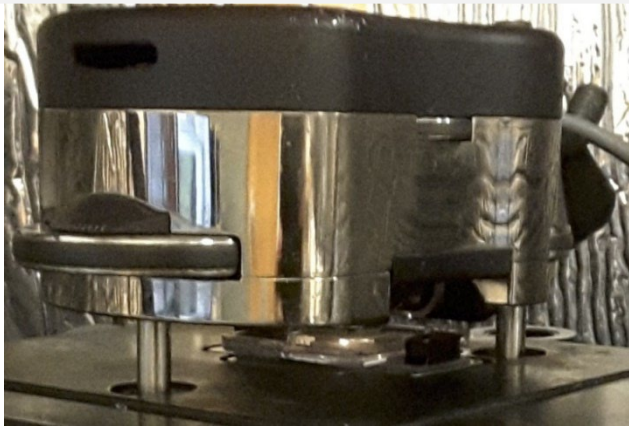
-  blueDrive Photothermal Excitation
-  Fast Force Mapping
-  Dual-Gain ORCA Conductive AFM
-  Piezoresponse Force Microscopy (HV-PFM)
-  Contact Resonance Viscoelastic Mapping Mode
-  AM-FM Viscoelastic Mapping Mode
-  Scanning Tunneling Microscopy (STM)
-  Air Temperature Controller (ATC)
-  Droplet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



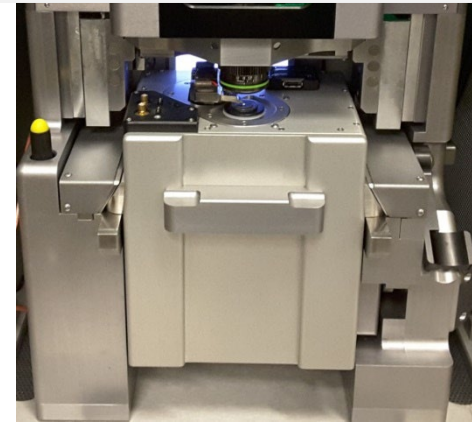


MRL AFMs—B12 MRL

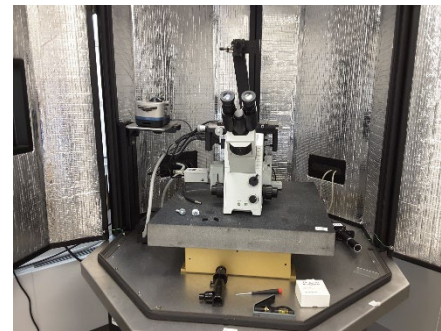
Asylum Research MFP-3D-SA (2 of these)
15 μm z range, 90 μm x 90 μm scan size



Asylum Research Cypher
5 μm z range, 30 μm x 30 μm scan size



0026 Supercon: Asylum Research MFP-3D-Bio
on an inverted optical fluorescence microscope

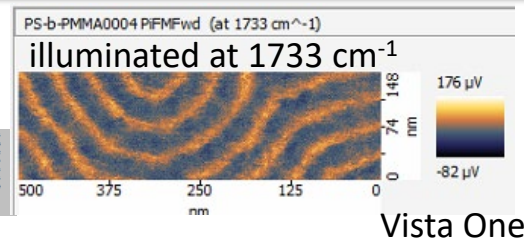


Related Instruments at MRL

Highly localized chemical information

- Molecular Vista PiFM

- Photo-induced force microscopy and spectroscopy



- Horiba TERS/TEPL

- Tip-enhanced Raman spectroscopy

- Neaspec Nano-IR

- AFM + infrared

Profilometry

- Dektak stylus profilometer

- Keyence 3D optical profiler



Keep Learning

- MRL Webinar Series
 - go.illinois.edu/MRLYouTubeChannel
 - Basics of Atomic Force Microscopy (Kathy Walsh)
 - The Versatility of Nanomechanics with AFM (Jessica Spear)
 - 3D Optical Profilometry (Julio Soares and Kathy Walsh)
- Kathy Walsh, kawalsh@illinois.edu

