



**Grainger College  
of Engineering**

UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

# Atomic Force Microscopy

**Kathy Walsh**

Senior Research Scientist

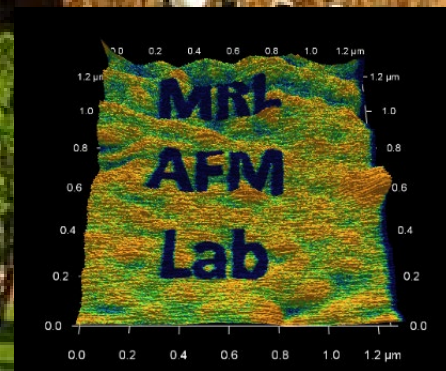
**Scanning Probe Microscopy**

Materials Research Laboratory

Central Research Facilities

Physics 403

7/21/21





# Illinois Materials Research Lab Central Research Facilities

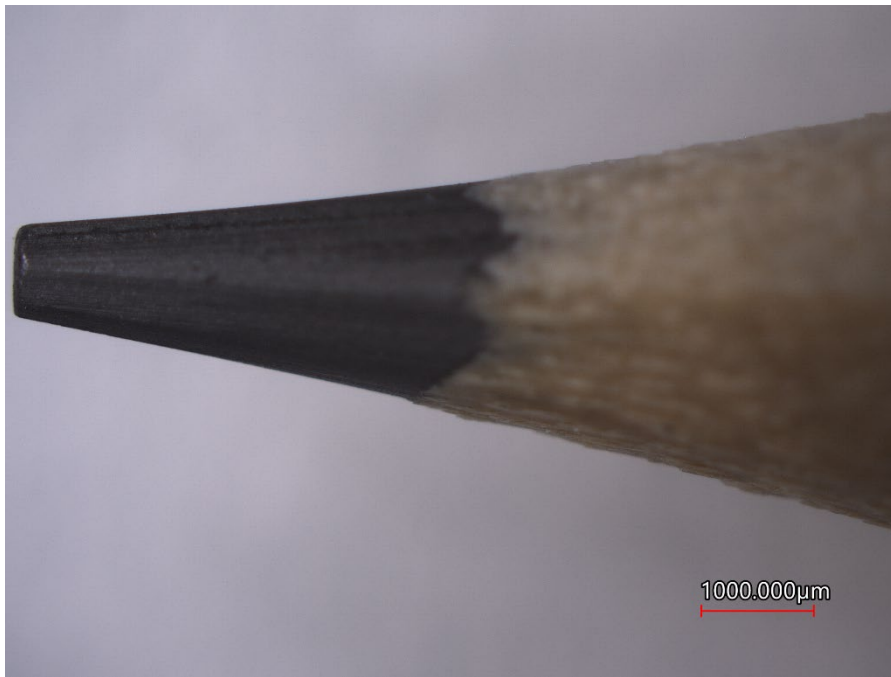
- User facility—anyone can be trained
  - UIUC and non-UIUC researchers welcome
  - Undergraduate researchers welcome
  - Staff collaboration or analysis available
- [mrl.illinois.edu/facilities](http://mrl.illinois.edu/facilities)
- [mrl-facilities@illinois.edu](mailto:mrl-facilities@illinois.edu)



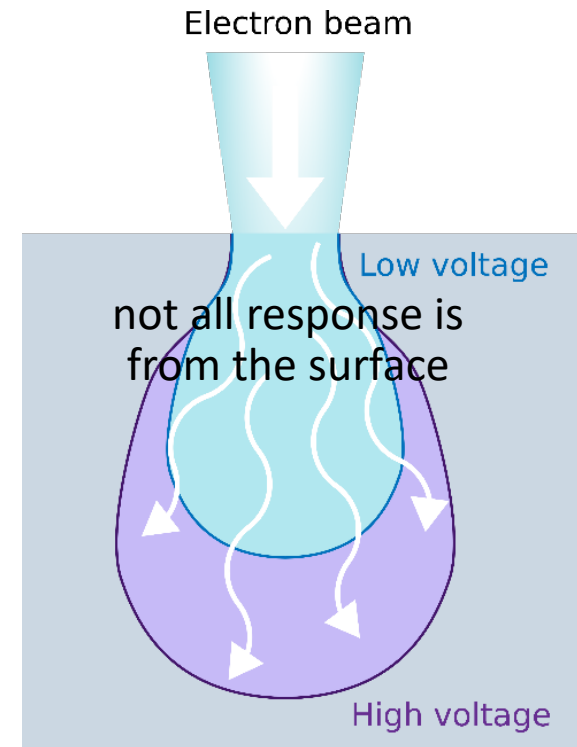


# Looking at Surfaces

## Optical Microscopy



## Scanning Electron Microscopy



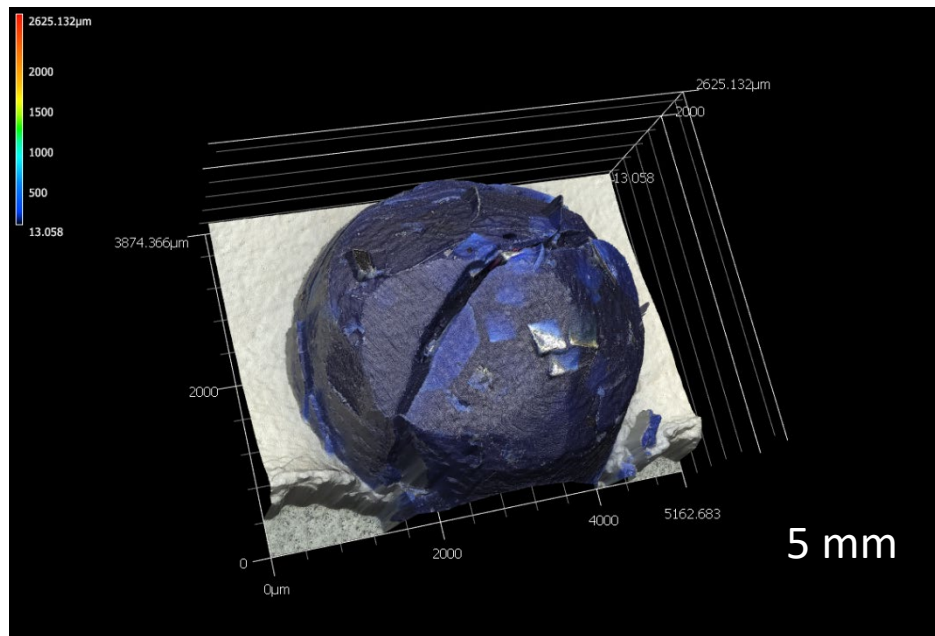
Adapted (cropped) from  
[https://myscope.training/#/SEMlevel\\_2\\_13](https://myscope.training/#/SEMlevel_2_13)  
(CC BY-SA 4.0)



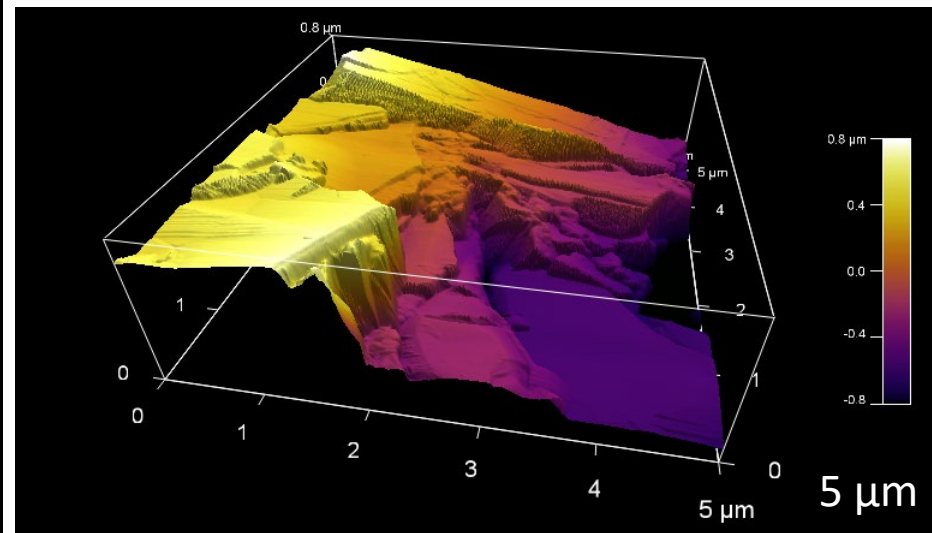
# Surface XYZ Coordinates Needed

## 3D Optical Profilometry

## Atomic Force Microscopy



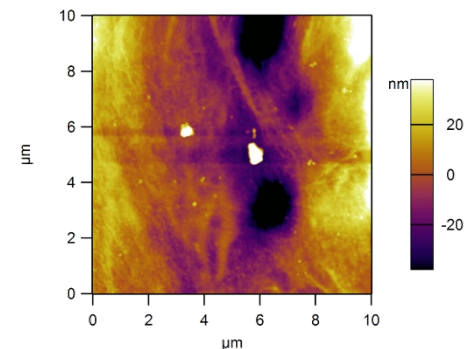
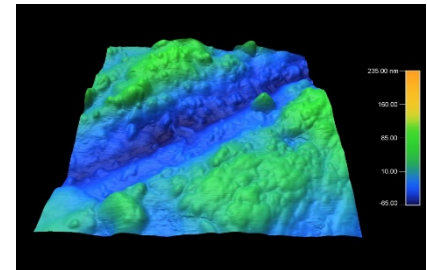
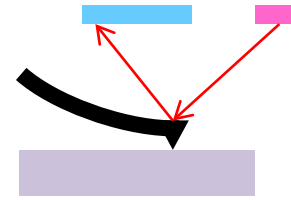
blue glitter crayon tip



pencil "lead"

# Topics for Today

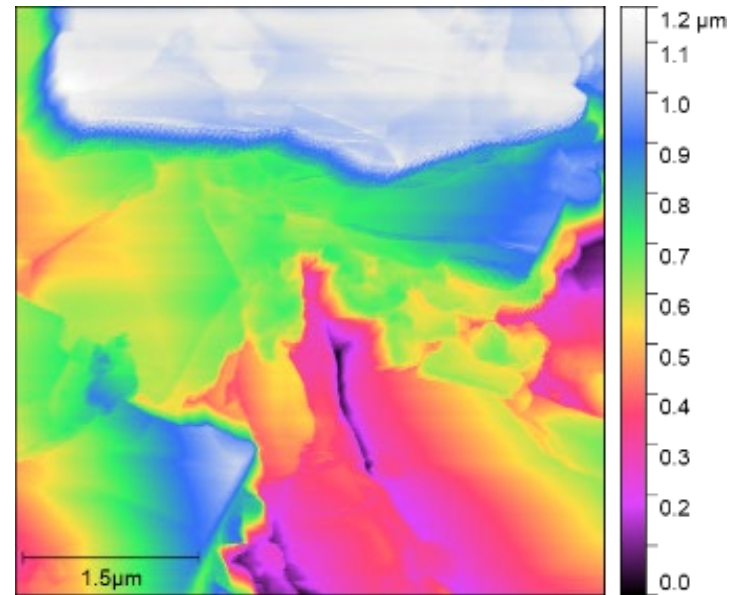
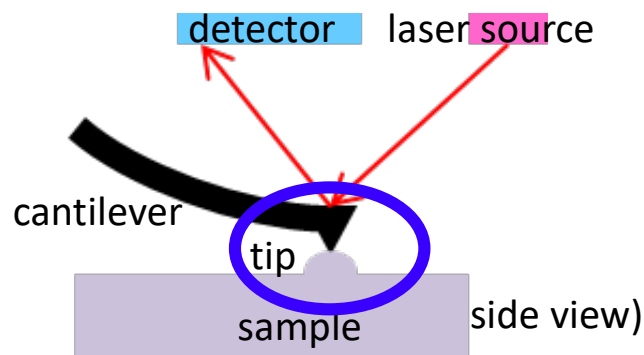
- How AFM works
- Featured applications
  - Topography
    - Profiles, step height
    - Roughness
  - Phase
  - Conductive AFM
  - Working in fluid
- Issues and artifacts
- Image processing





# What's an Atomic Force Microscope?

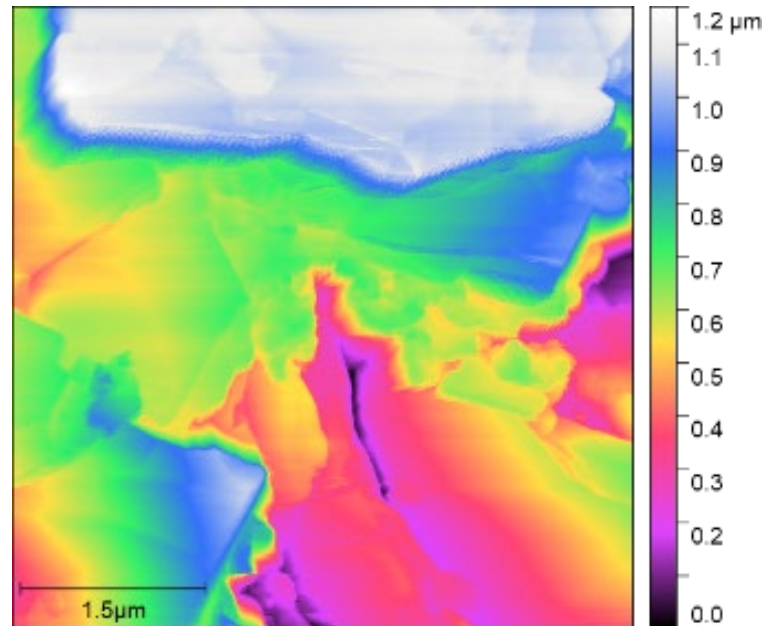
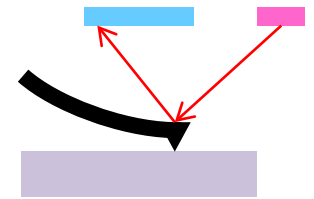
“Atomic Force” Microscopy—forces between atoms in the tip and atoms in the sample



false-color surface topographs

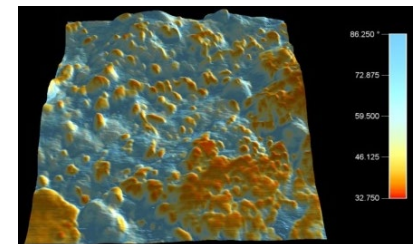
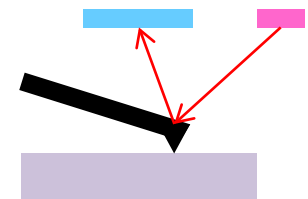
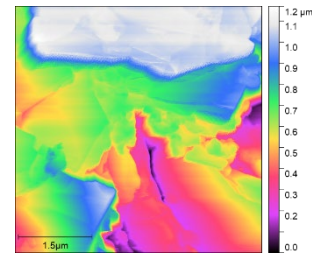
# What's an Atomic Force Microscope?

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



# What's an Atomic Force Microscope?

- “Atomic Force” — interactions between tip and sample
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  - 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



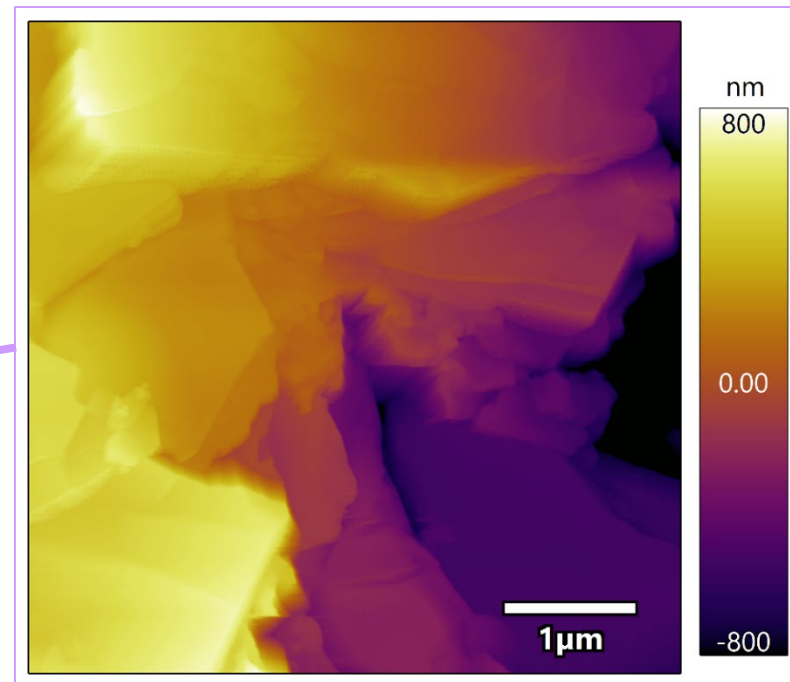
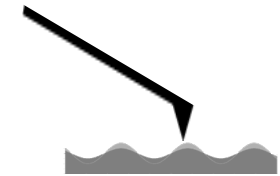
Turquoise,  $1\mu\text{m} \times 1\mu\text{m}$   
color overlay: phase



# Typical AFM Scales

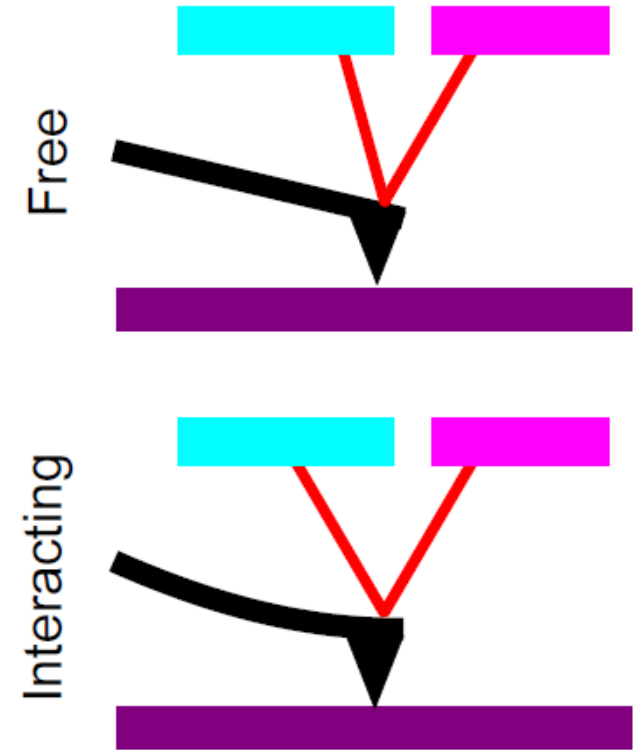
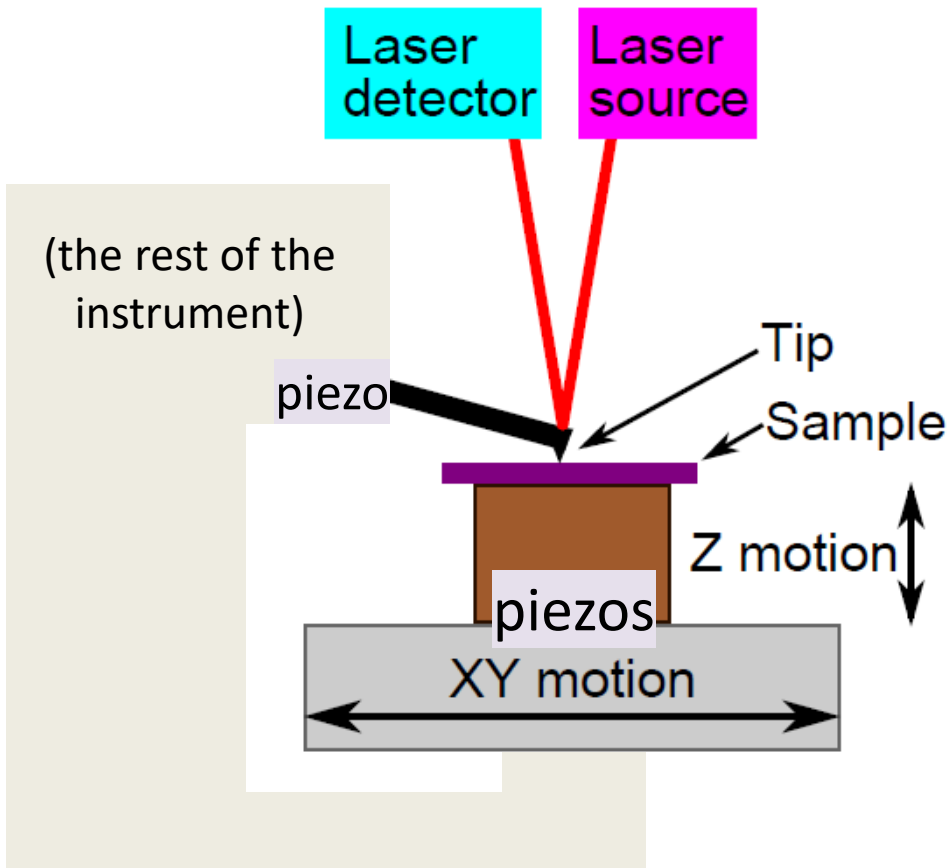
(only what's pretty common, not all of what's possible)

- Image sizes -- few to tens of  $\mu\text{m}^2$
- Feature peak-to-valley --  $\text{\AA}$  to  $\mu\text{m}$
- Sample sizes -- mm to cm
- AFM measures surfaces





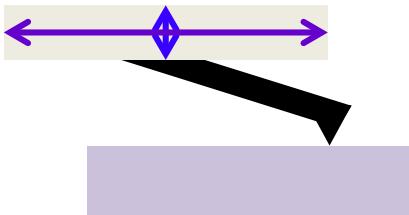
# AFM Schematic



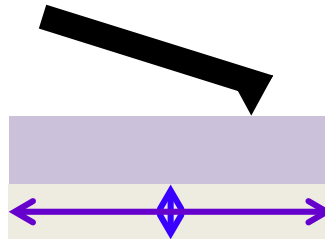
# Scanners

## *scanning* probe microscopy

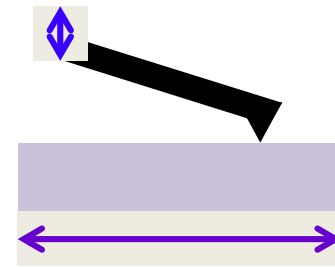
tip scanning



sample scanning



decoupled scanning

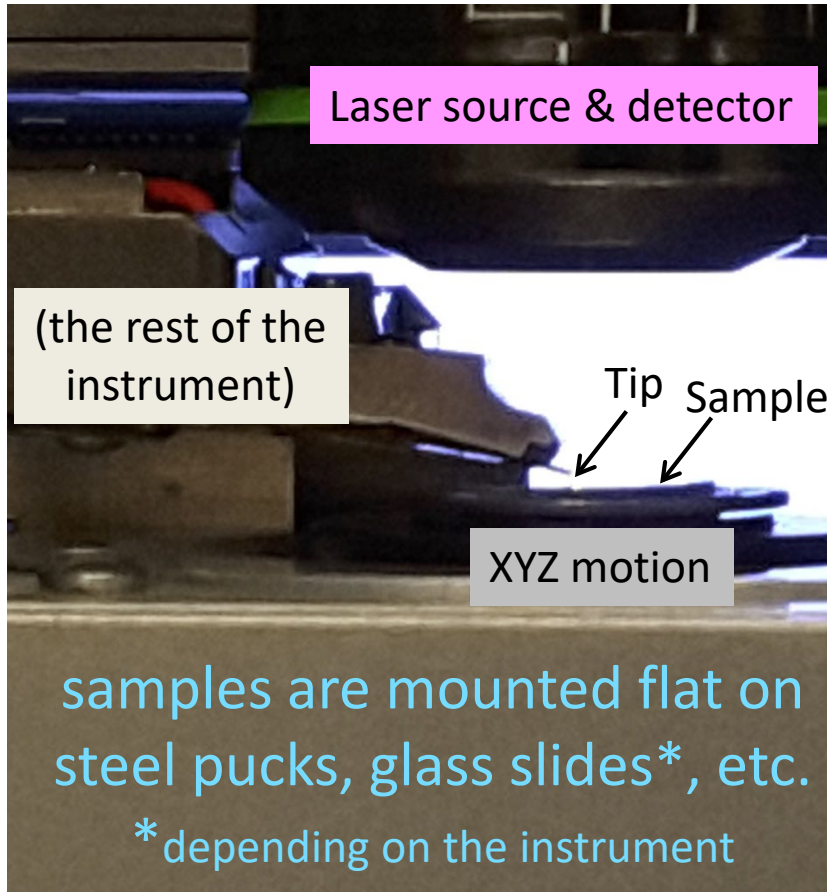


tapping is always done as close to the tip as possible  
(tapping mode will be discussed later)



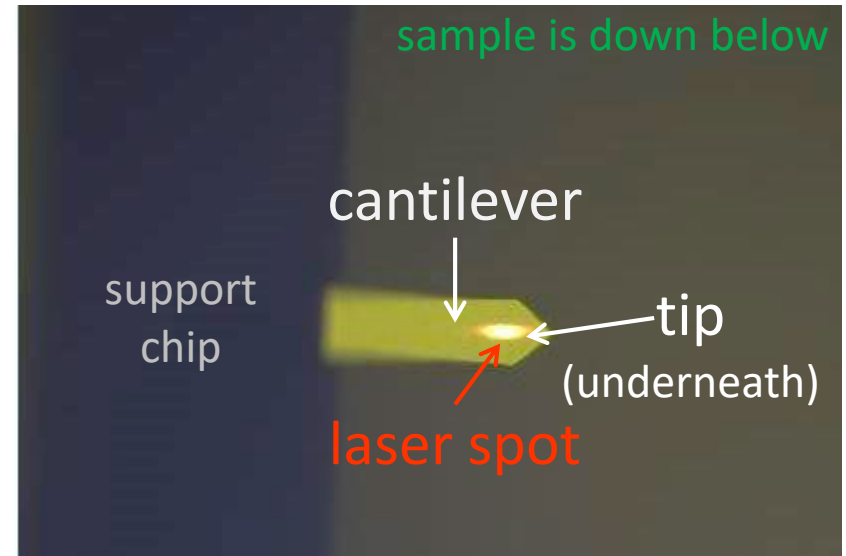


# AFM Instrument

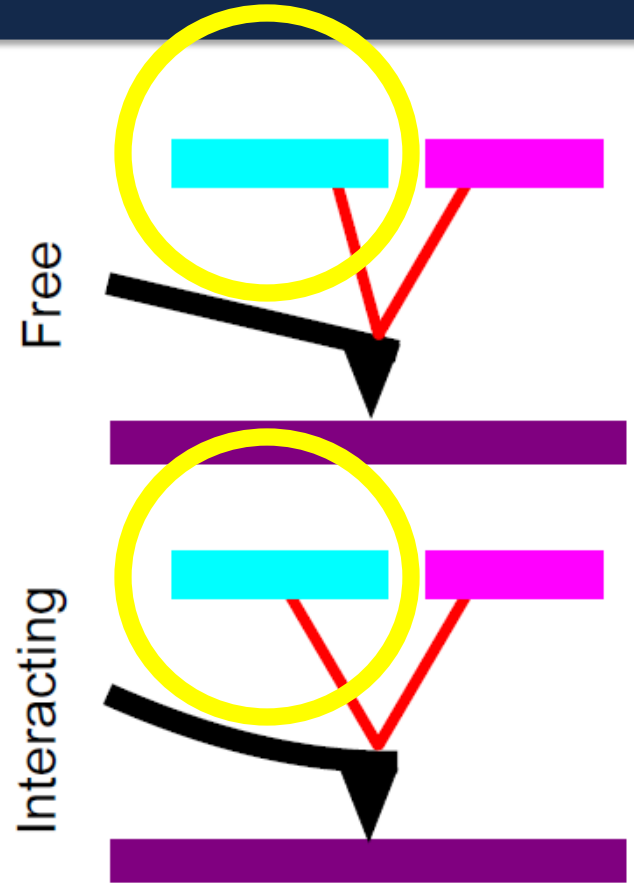
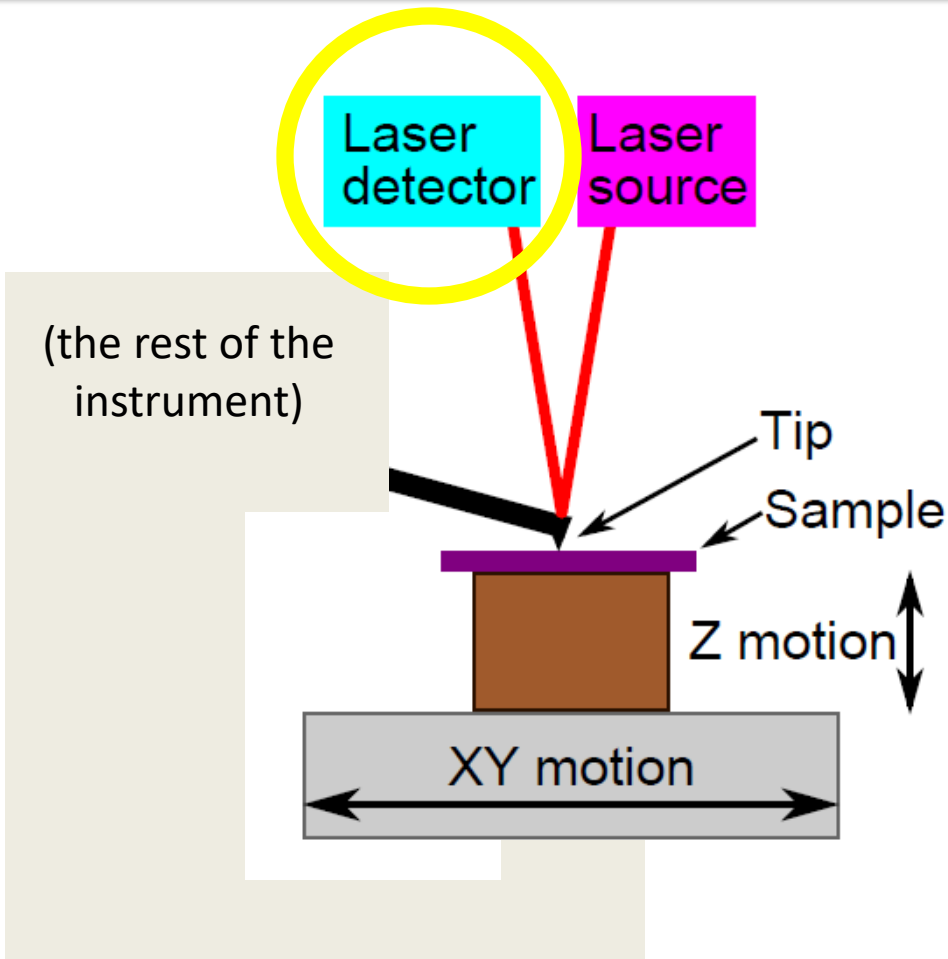


side view

top view



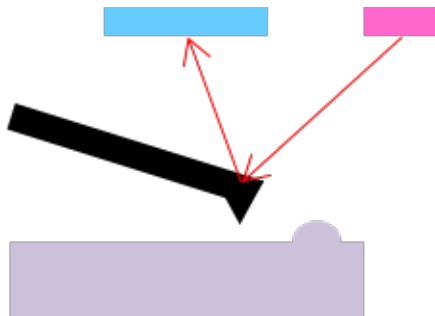
# AFM Schematic





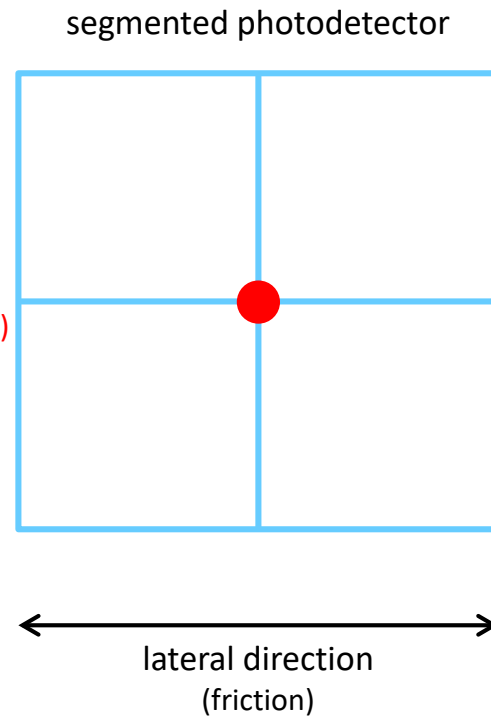
# Laser Detection

non-interacting



(side view)

laser spot  
(reflected from back of cantilever)

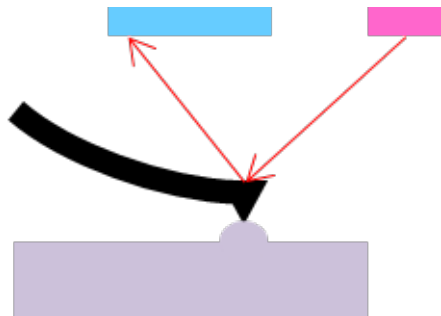


(exaggerated schematic)



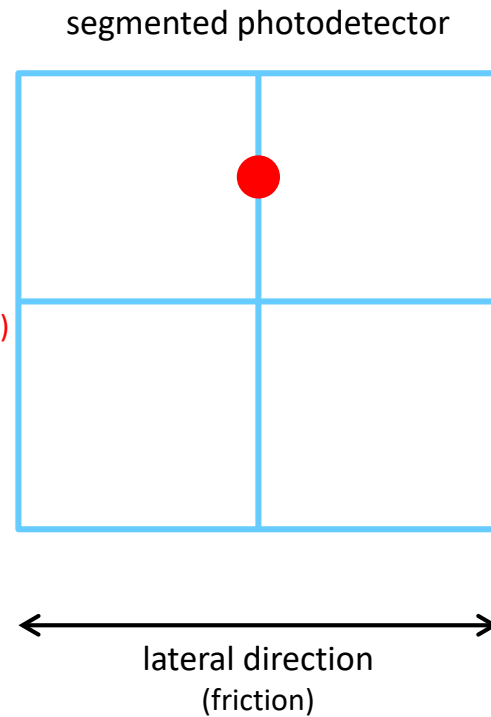
# Laser Detection

sample pushing up



(side view)

laser spot  
(reflected from back of cantilever)

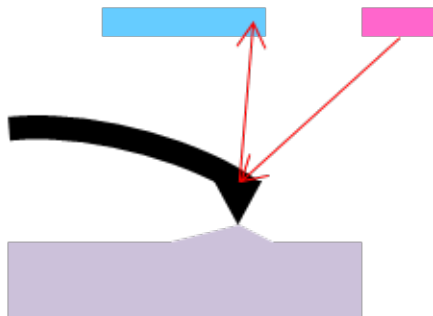


(exaggerated schematic)



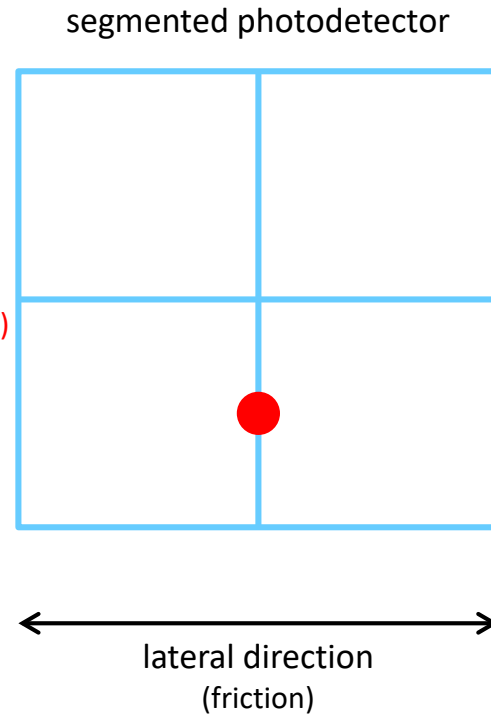
# Laser Detection

sample pulling down



(side view)

laser spot  
(reflected from back of cantilever)



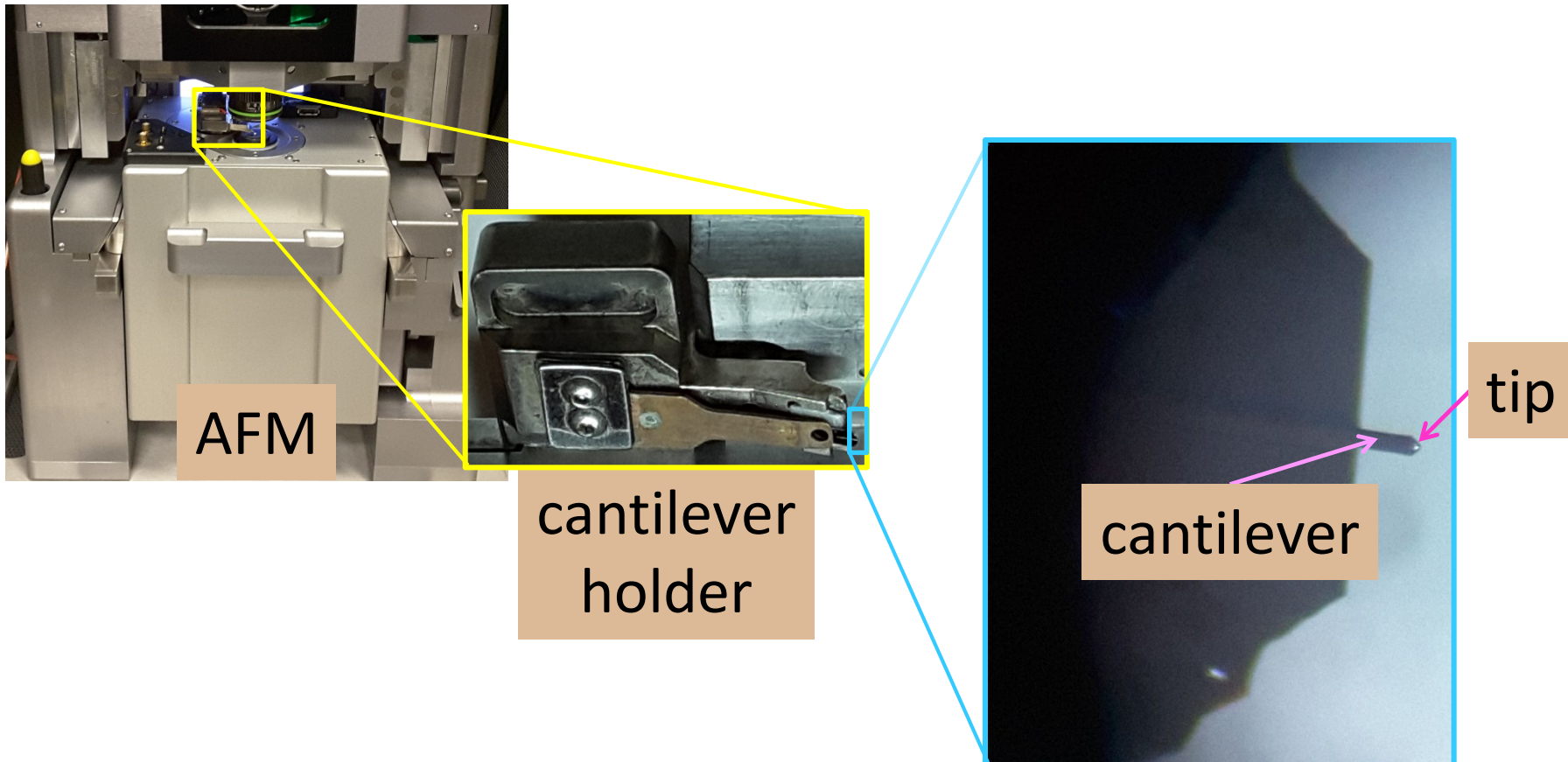
(exaggerated schematic)





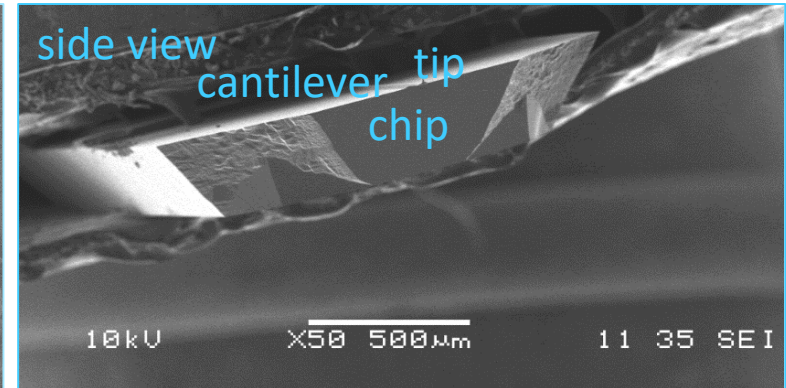
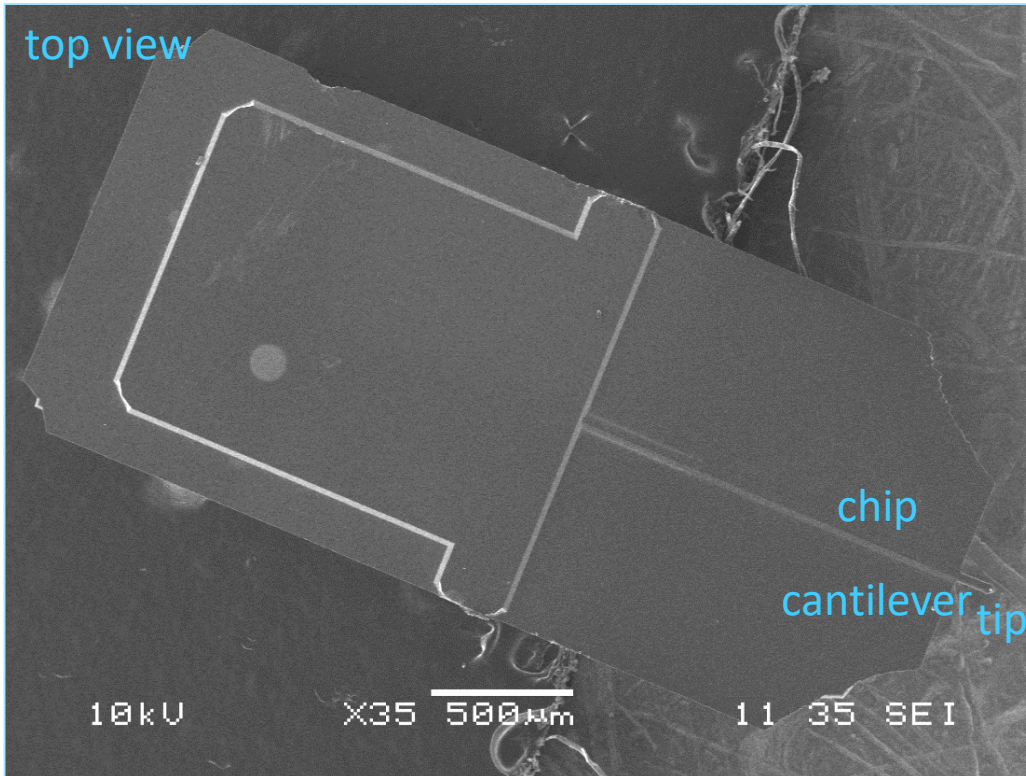
# AFM Tips

scanning *probe* microscopy



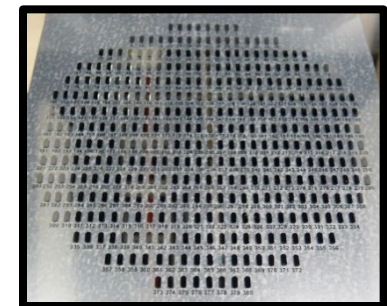


# Tip Terminology



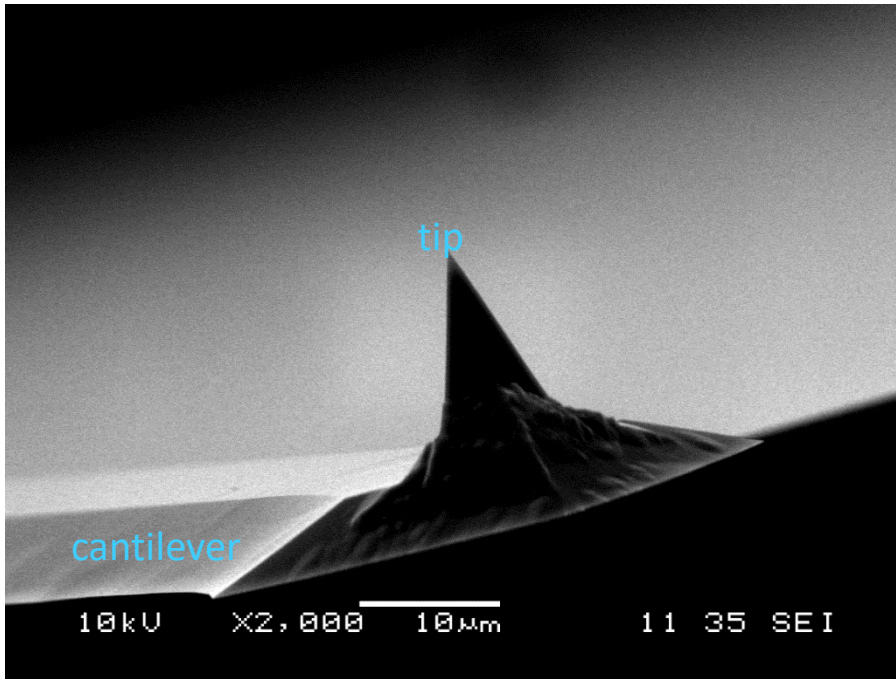
“probe”

tips point upwards  
in the box

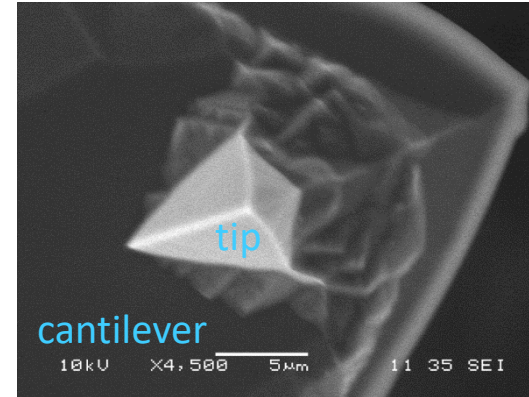


SEM images taken using MRL's JEOL 6060LV

# Typical Tip

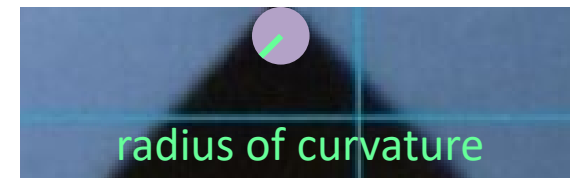


SEM images taken using MRL's JEOL 6060LV



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  $4\ \mu\text{m}$





# Tips for Good Results


- Typical tapping tip cost ~\$21
- Specialized tips cost more
  - Coatings (electrical, magnetic) usually a couple more dollars per tip
  - High aspect ratio or 2 nm radius tips ~\$70-80
  - 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

  
The Nanotech Facilitator

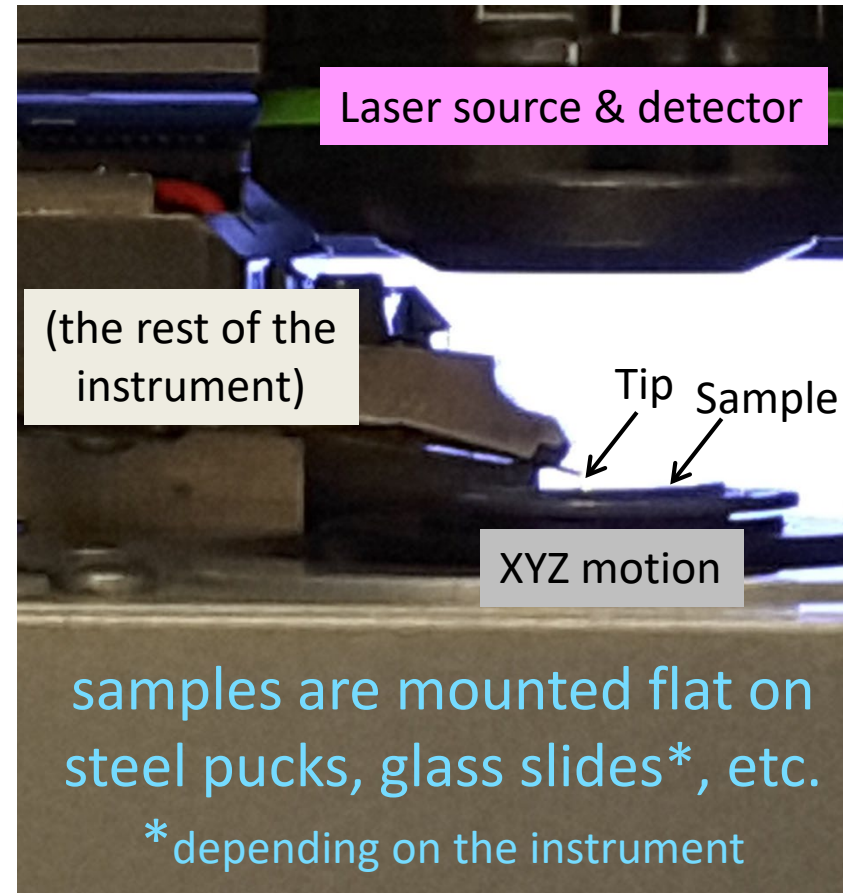
# “How long does a tip last?”

- 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 for frequent AFM'ers



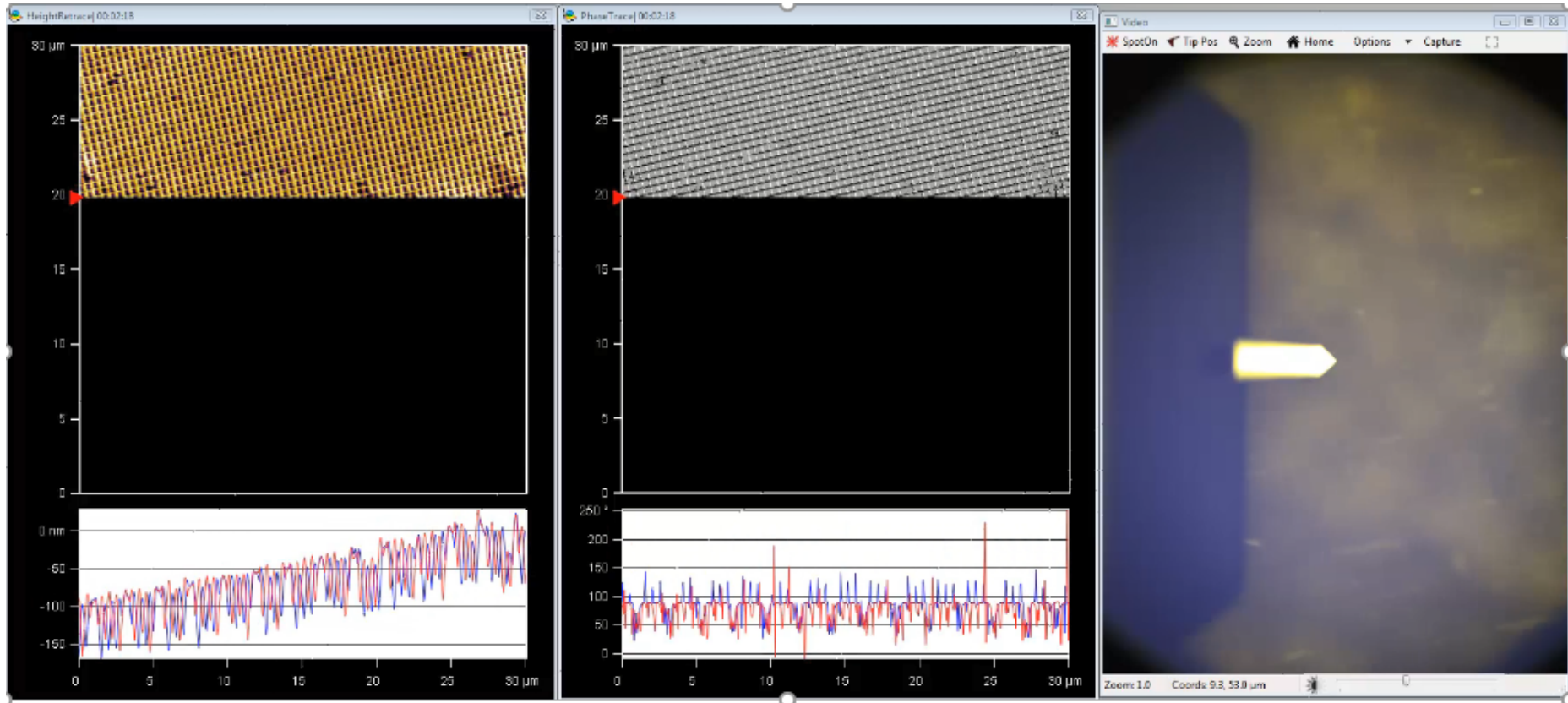
# The Process

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



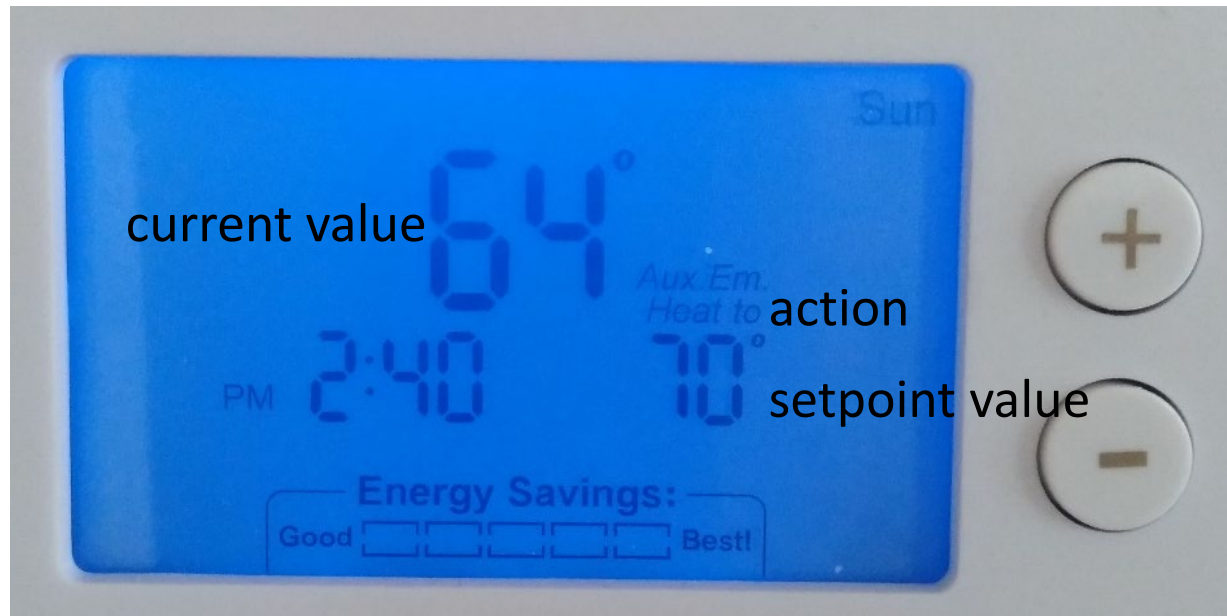


# Raster Scanning on the AFM





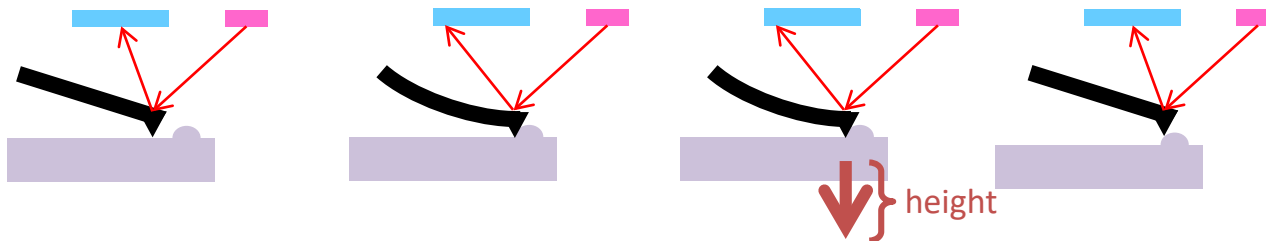
# Feedback





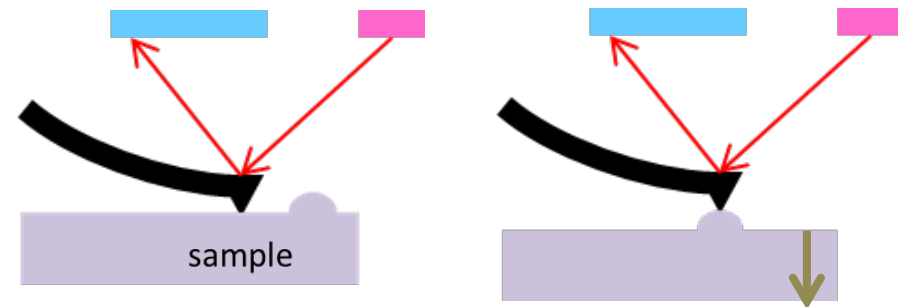
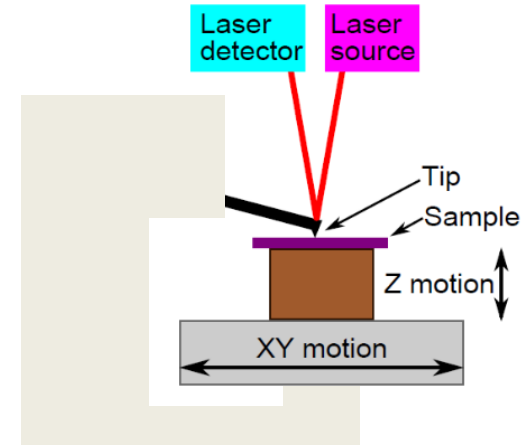
# Feedback

- 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



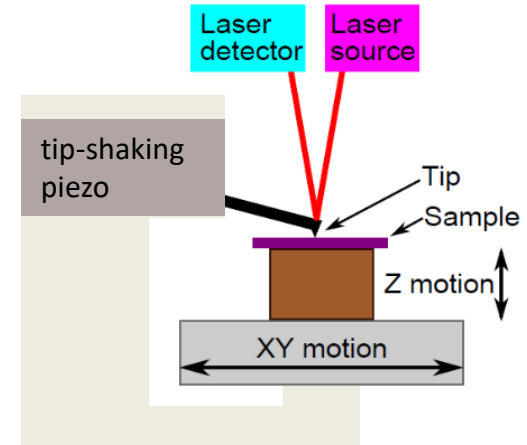
# Contact Mode Imaging

- 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

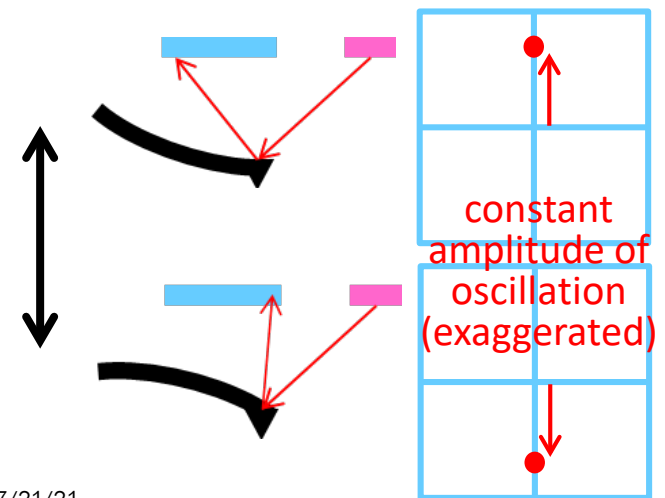


# Tapping Mode Imaging

- 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

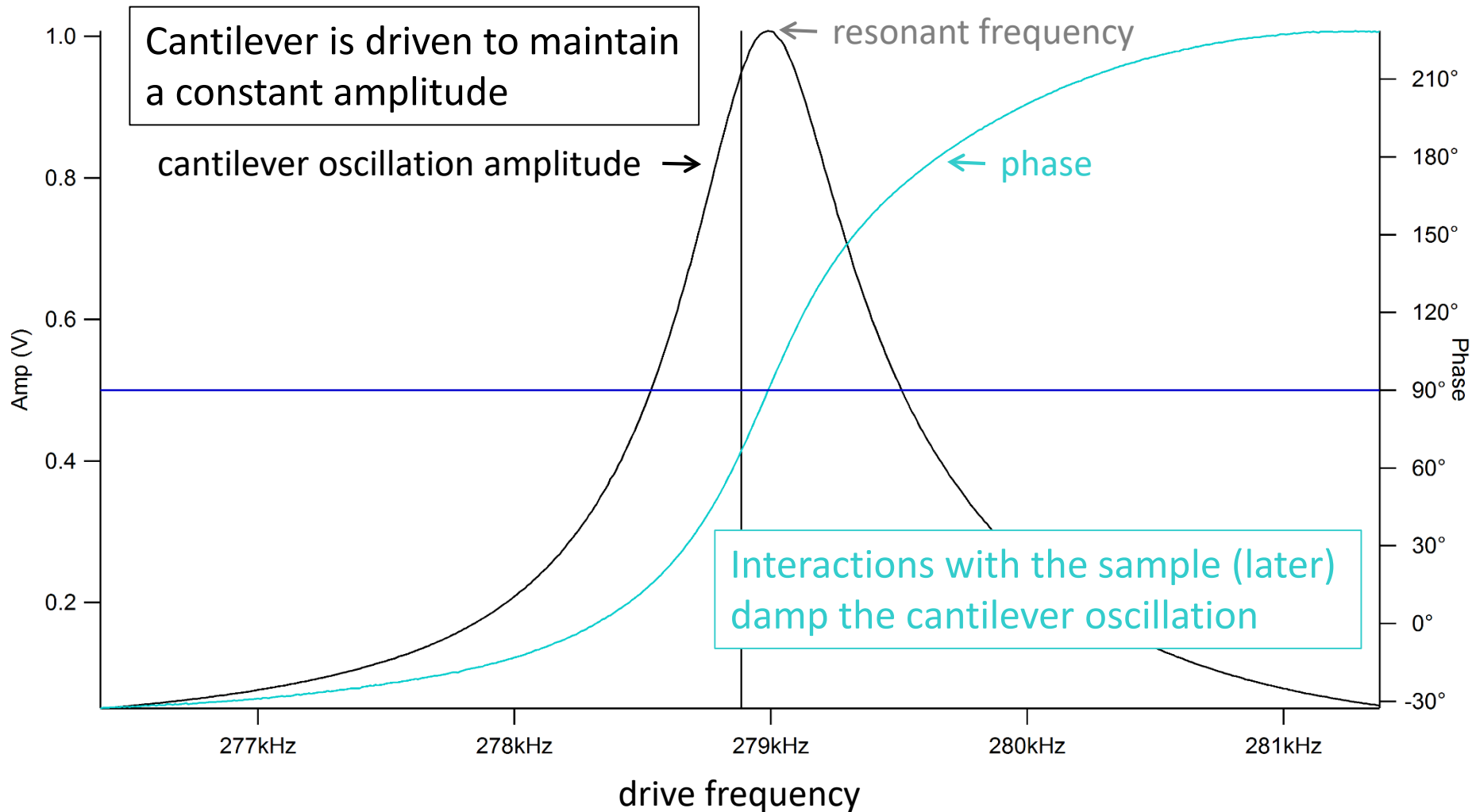


tip oscillates at tens of kHz to MHz





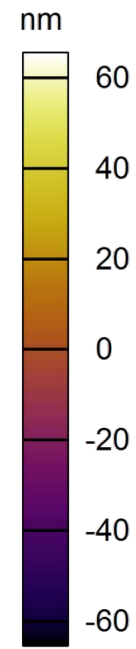
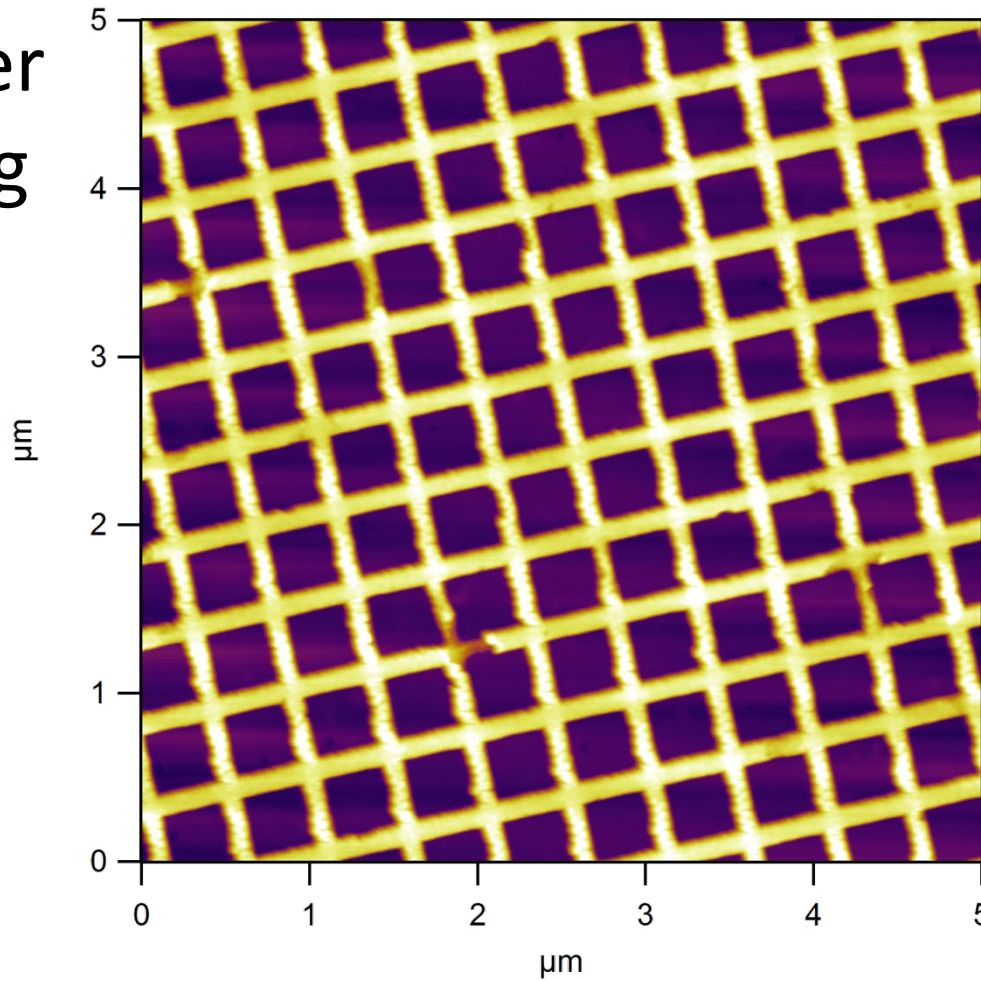
# Tuning the Cantilever





# Application: Imaging

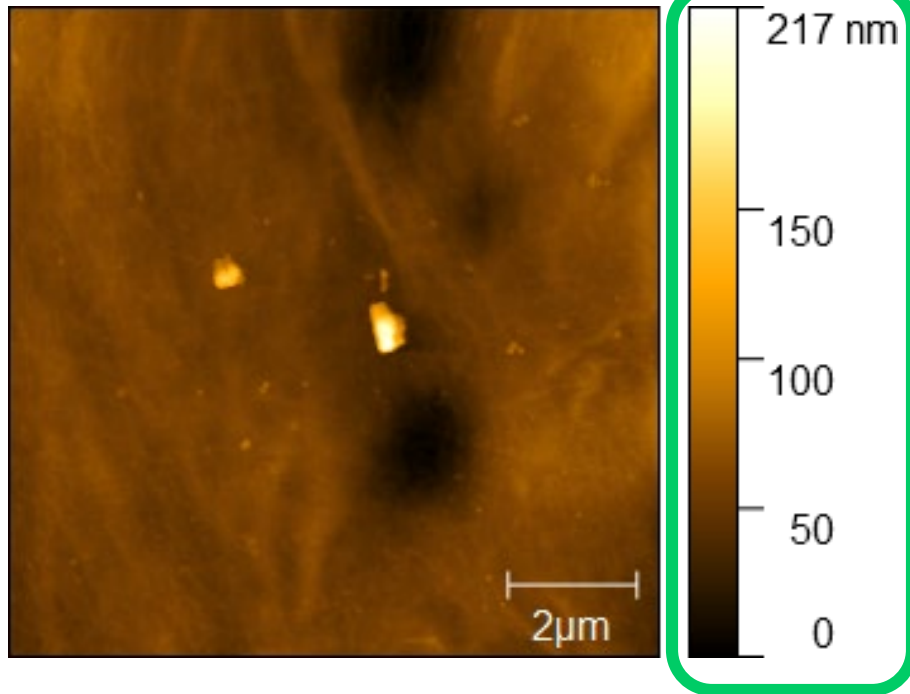
Polymer  
Grating



range of colors,  
not heights in  
the image

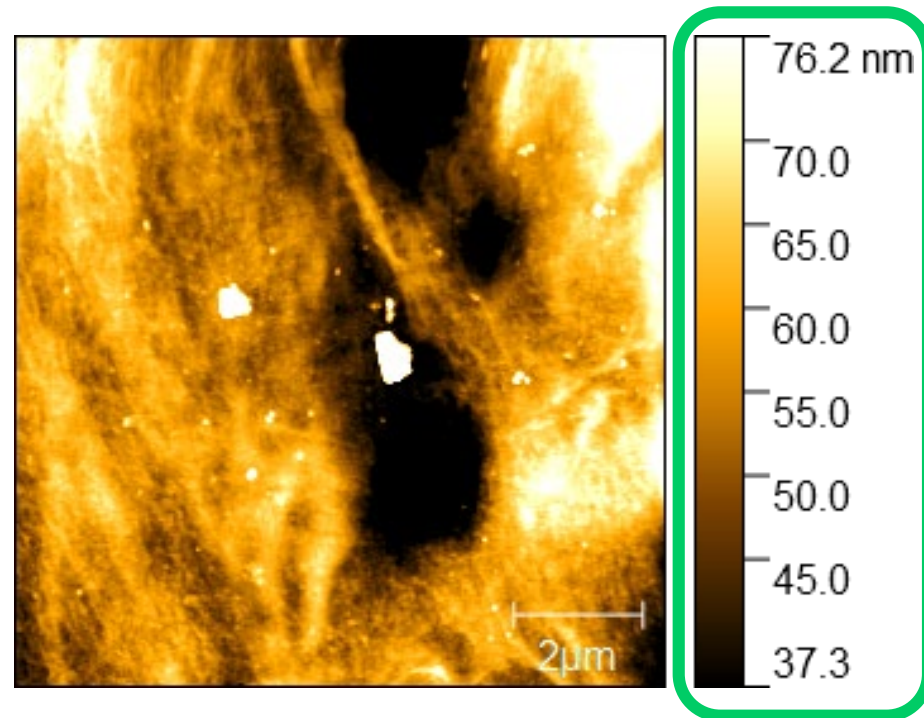


# Reading the Colorscale



same image, different color ranges

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

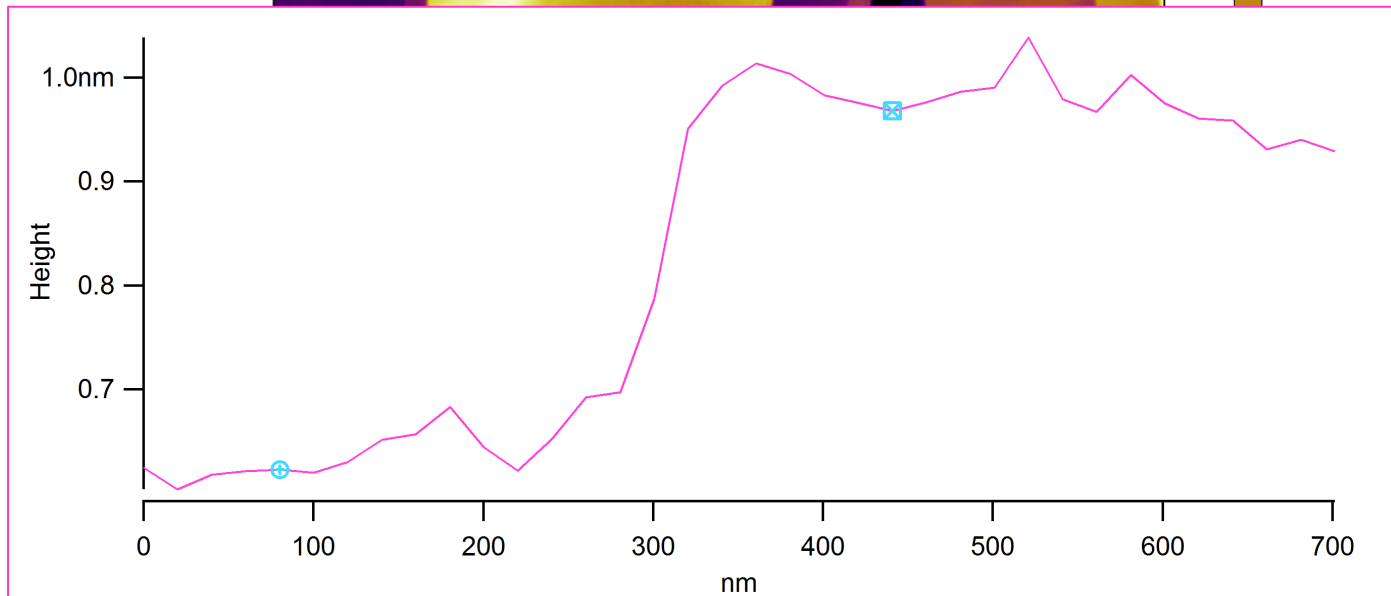
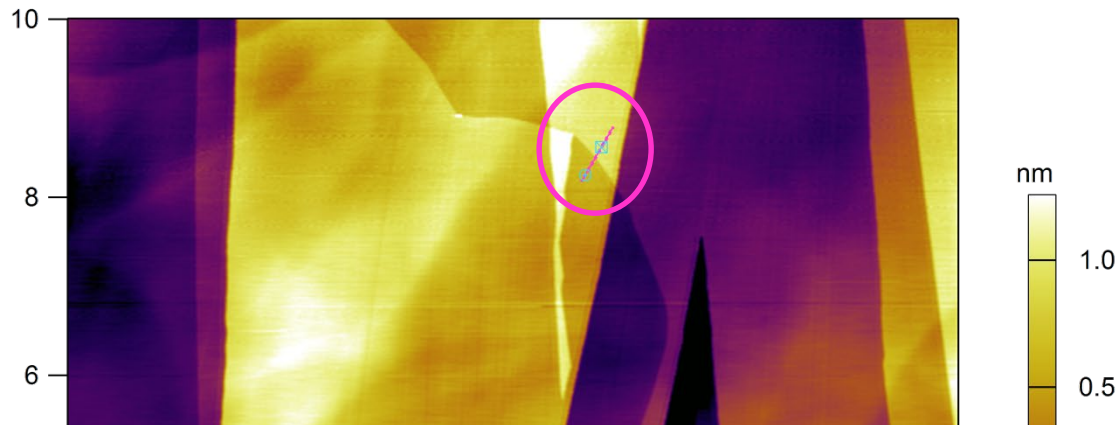


BOPP/PE polymer blend (toothbrush packaging), 10 μm x 10 μm AFM topograph



# Application: Step Heights

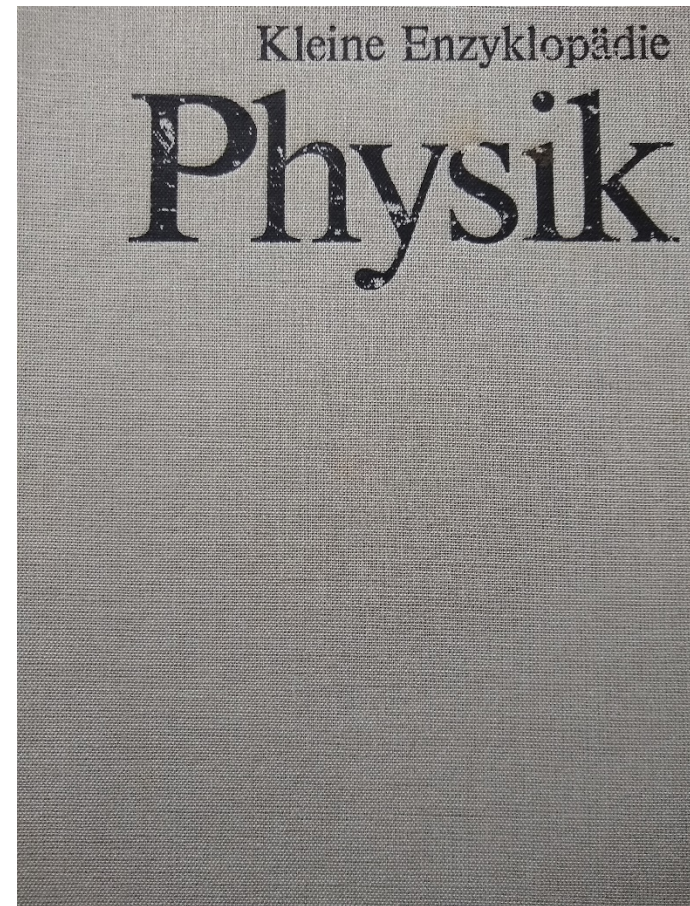
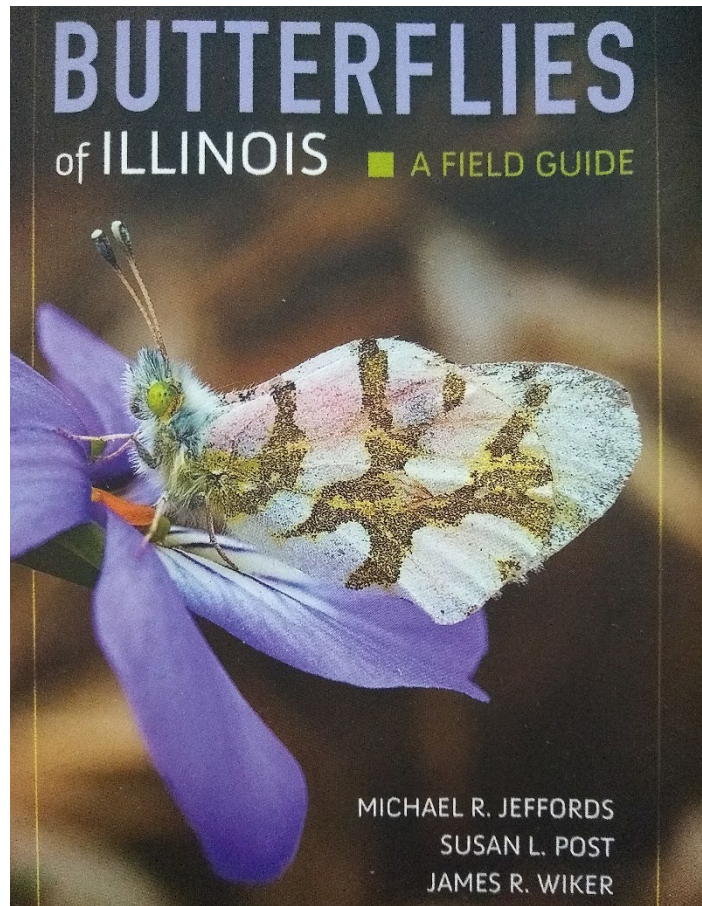
HOPG





# Step Heights and Thicknesses

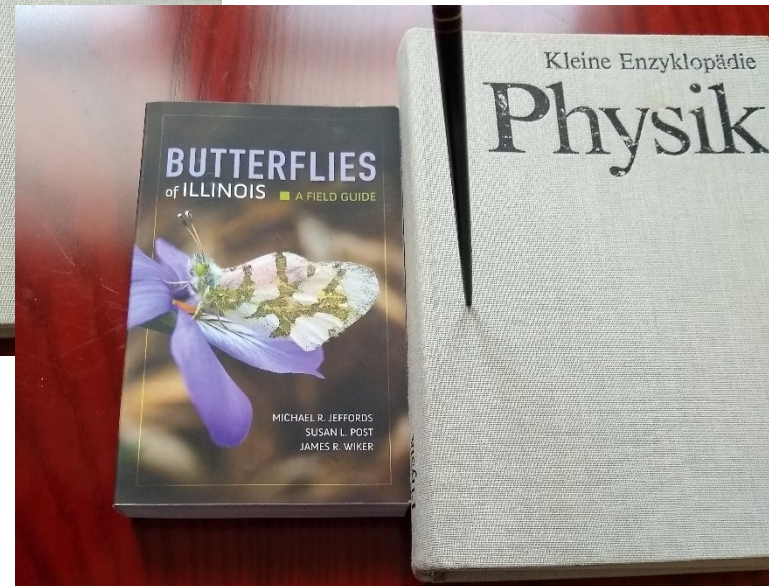
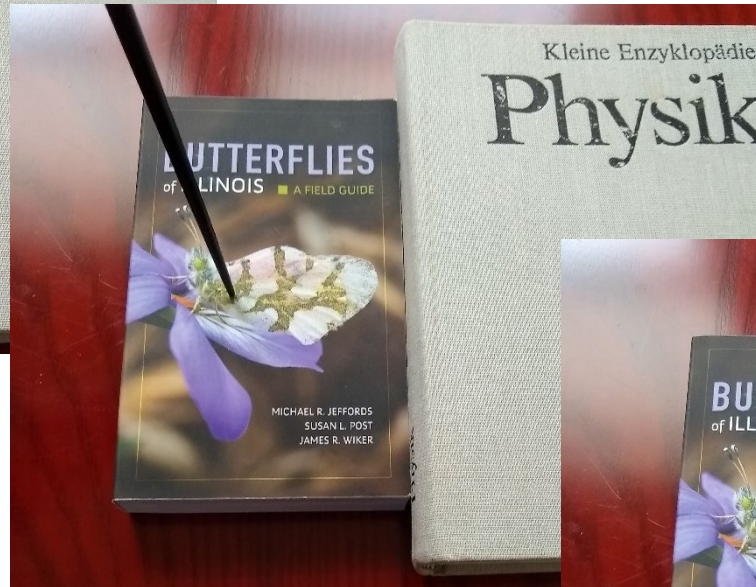
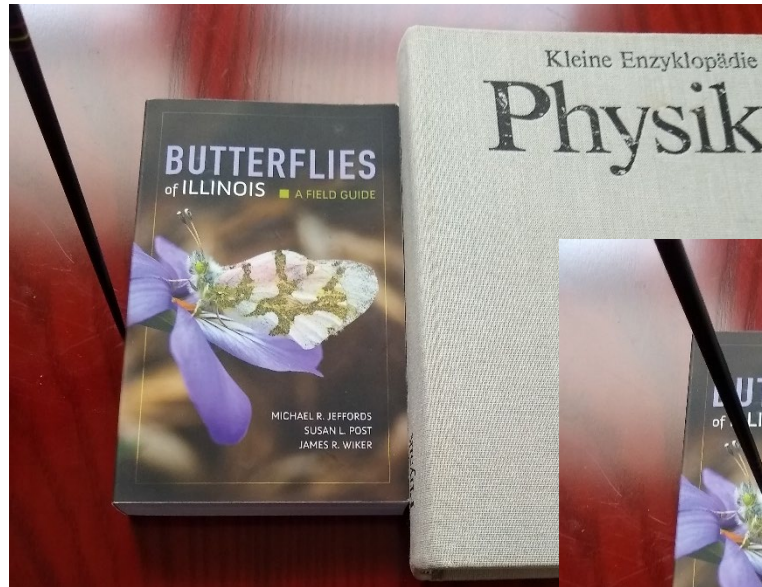
Which book is thicker?





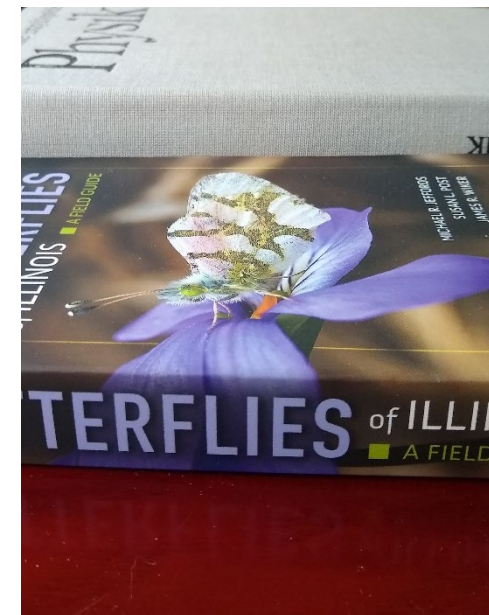
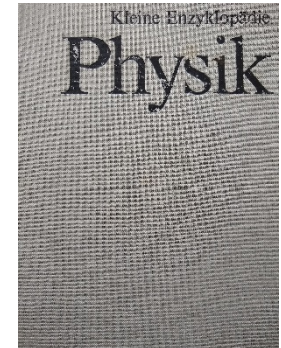
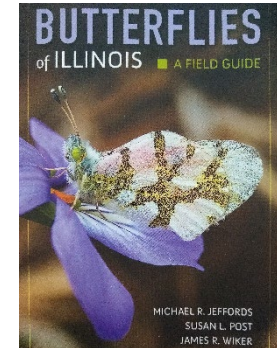


# Step Heights and Thicknesses



# 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





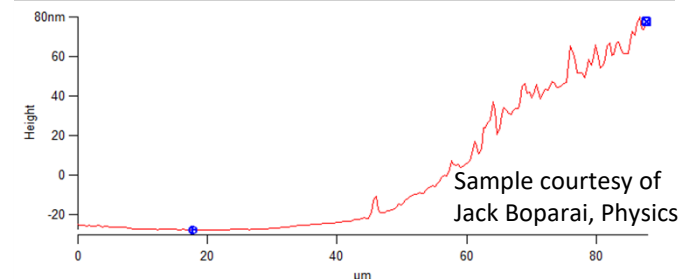
# Step Height/Film Thickness: Complementary Techniques

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)
- X-ray Fluorescence (XRF)
- Rutherford Backscattering Spectrometry (RBS)

Need a height difference (step) like AFM

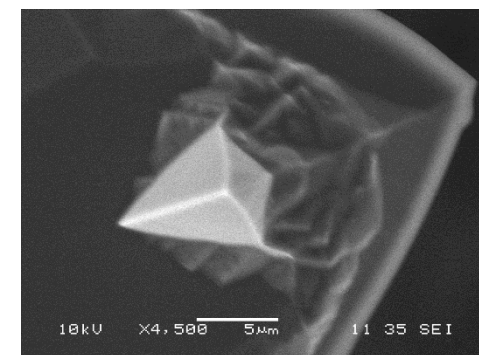
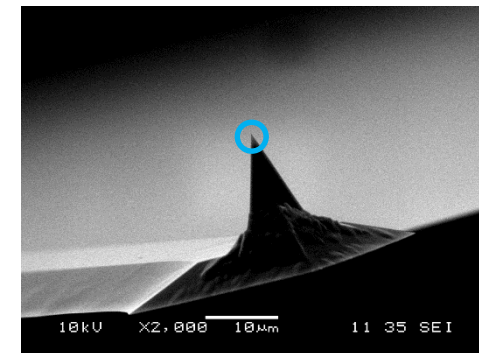
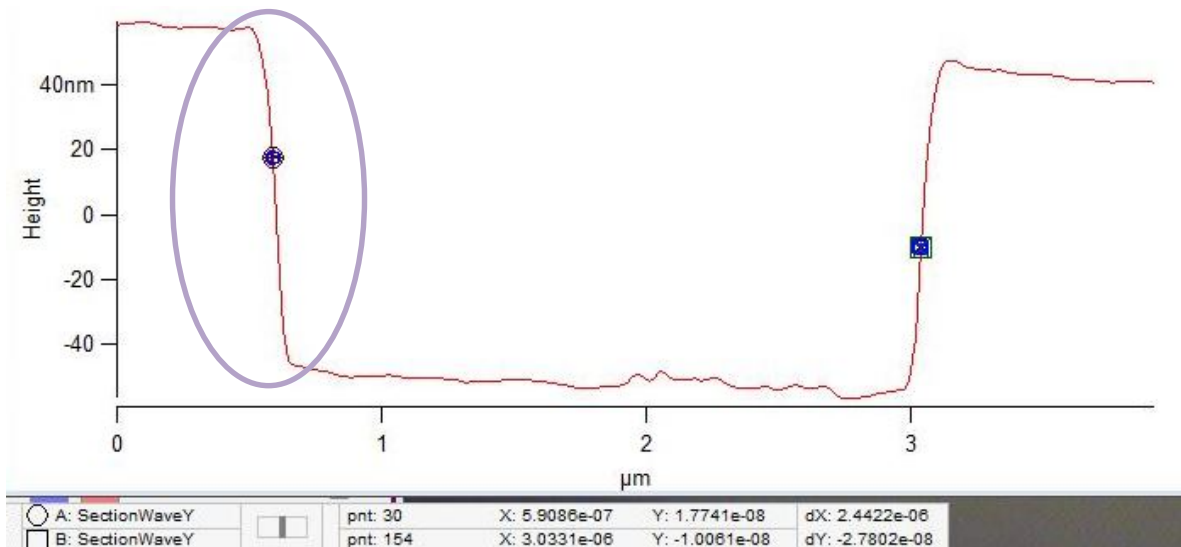
Continuous film (no steps)  
May need to know density



# AFM and Widths

## Beware of tip shape convolution

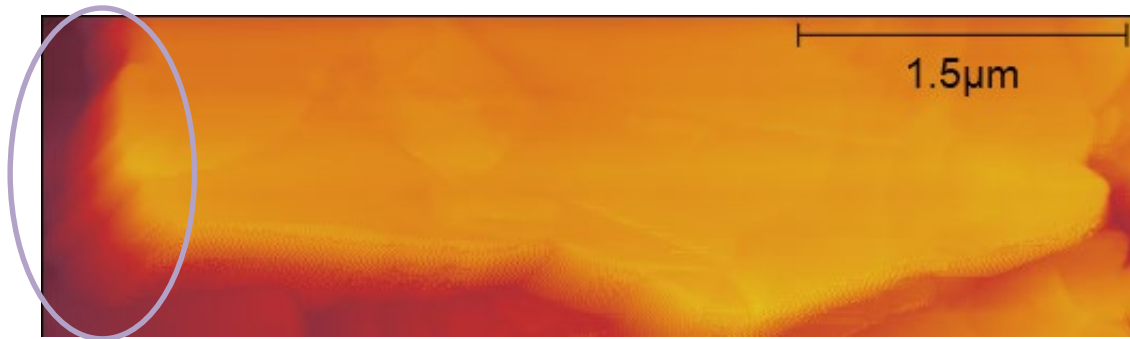
- As depth increases, pyramidal tips get broader
- Steep drop-offs look less sharp
- High aspect ratio tips are available



# AFM and Widths

## Beware of tip shape convolution

- As depth increases, pyramidal 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?



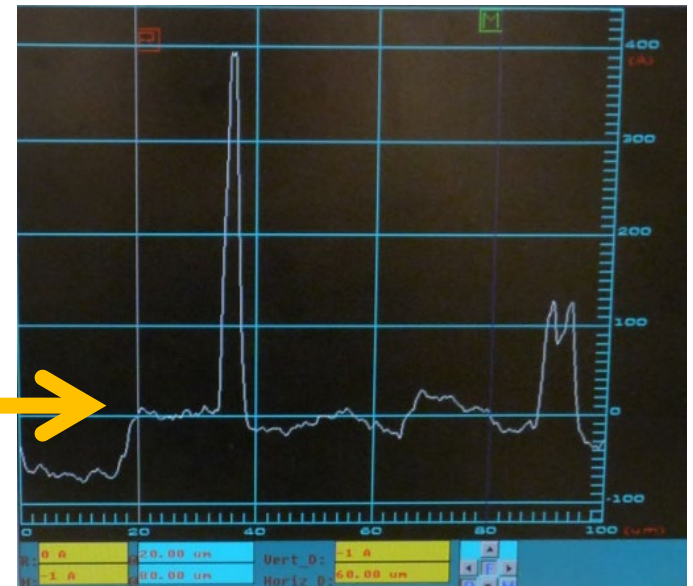
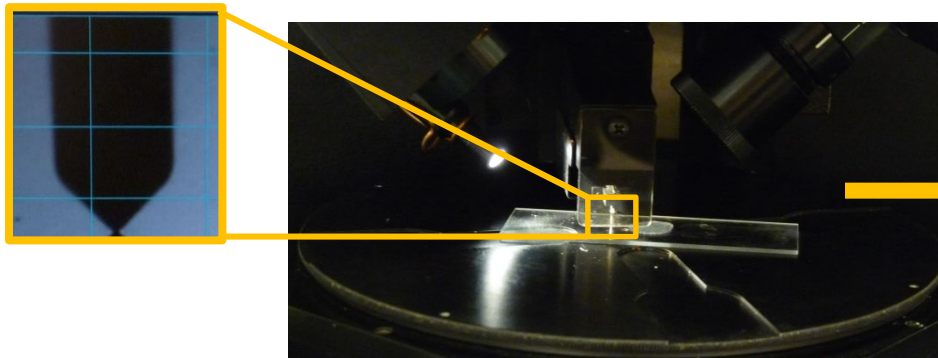
Michael Jeffords and Susan Post, University of Illinois Prairie Research Institute  
<https://photojournalingm-s.smugmug.com/Colorado-and-Kansas/i-3tJ3DZk/A>



# Complementary: Stylus Profilometry



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

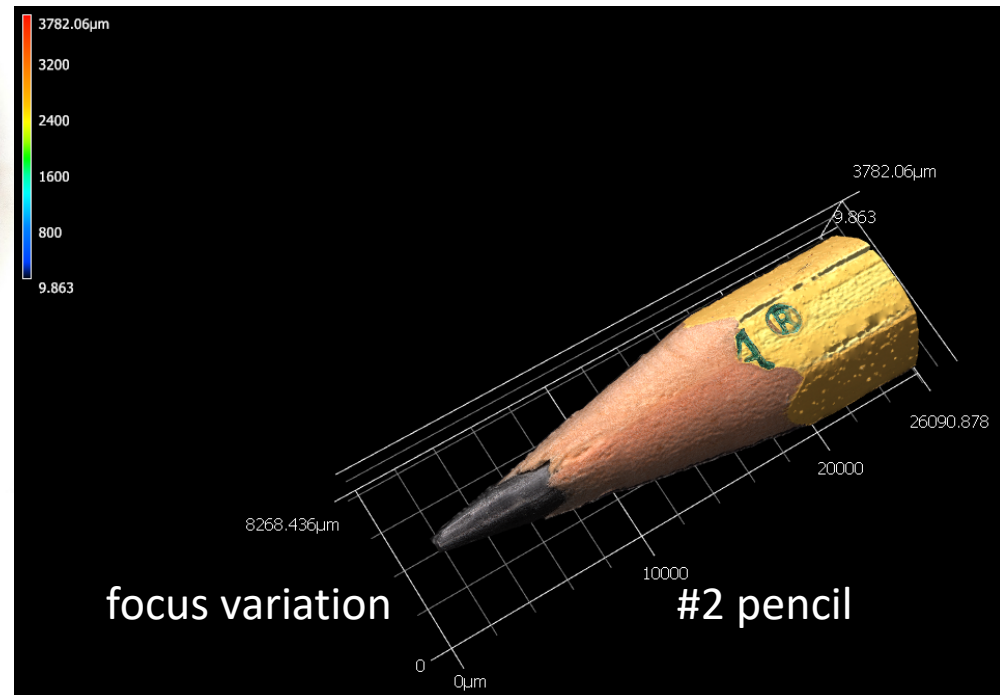




# Complementary: Optical Profilometry

[go.illinois.edu/MRL3DOpticalProfilometry](http://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

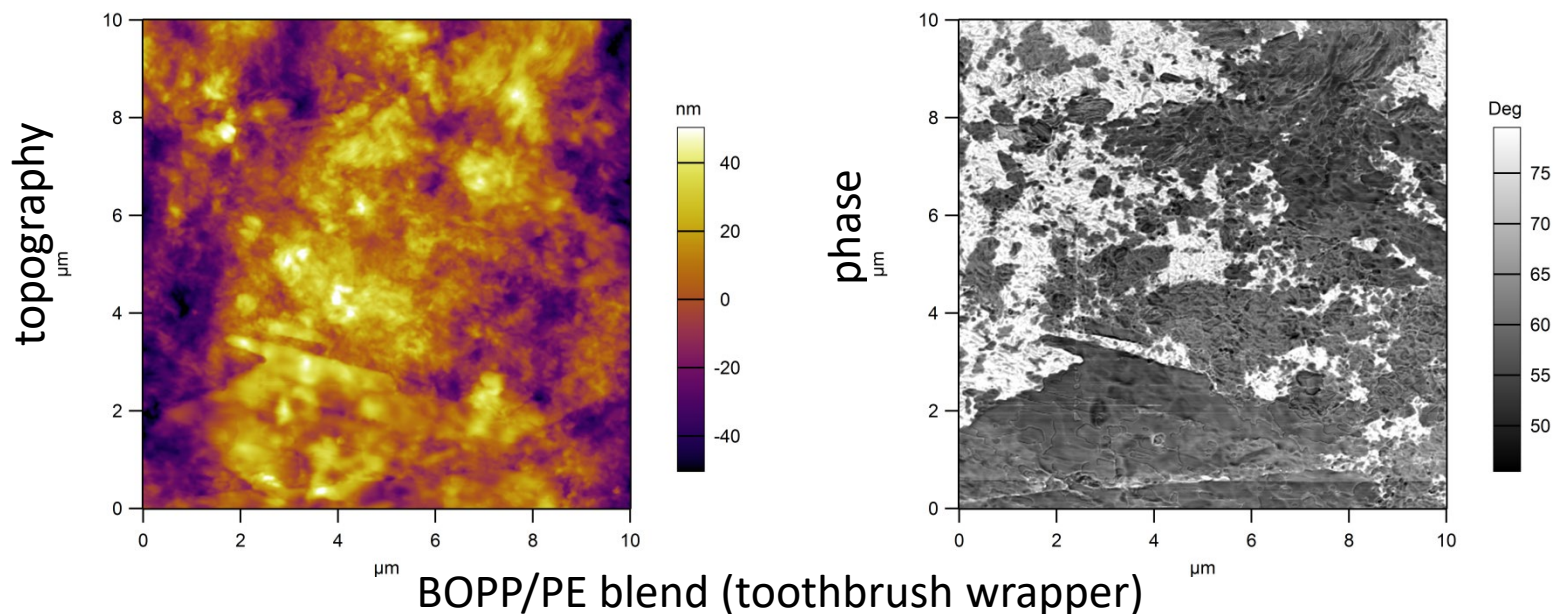
	AFM	2D Stylus Profilometry	3D Optical Profilometry
Vertical resolution	outstanding	OK	OK
Field of view	small	large	large
Data type	image	line	image
Max sample size	depends on instrument (~cm to large)	large	large
Max feature height	few $\mu\text{m}$	mm	mm
Force on sample	light	moderate	none
Speed	moderate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



# Mechanical Characterization

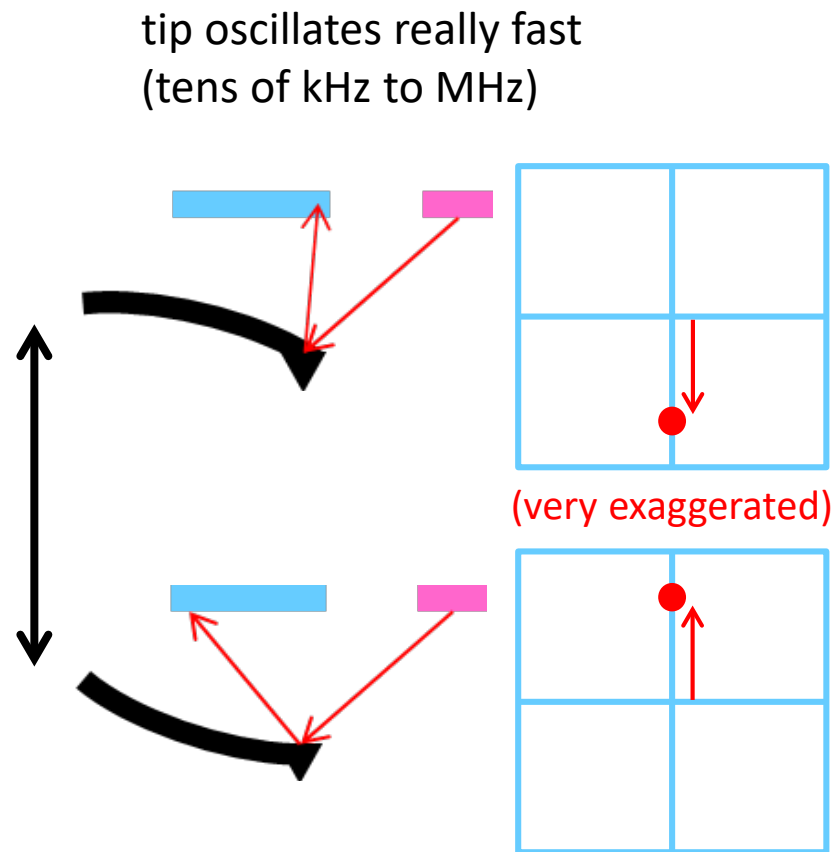
## Visual impact of mechanical differences

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



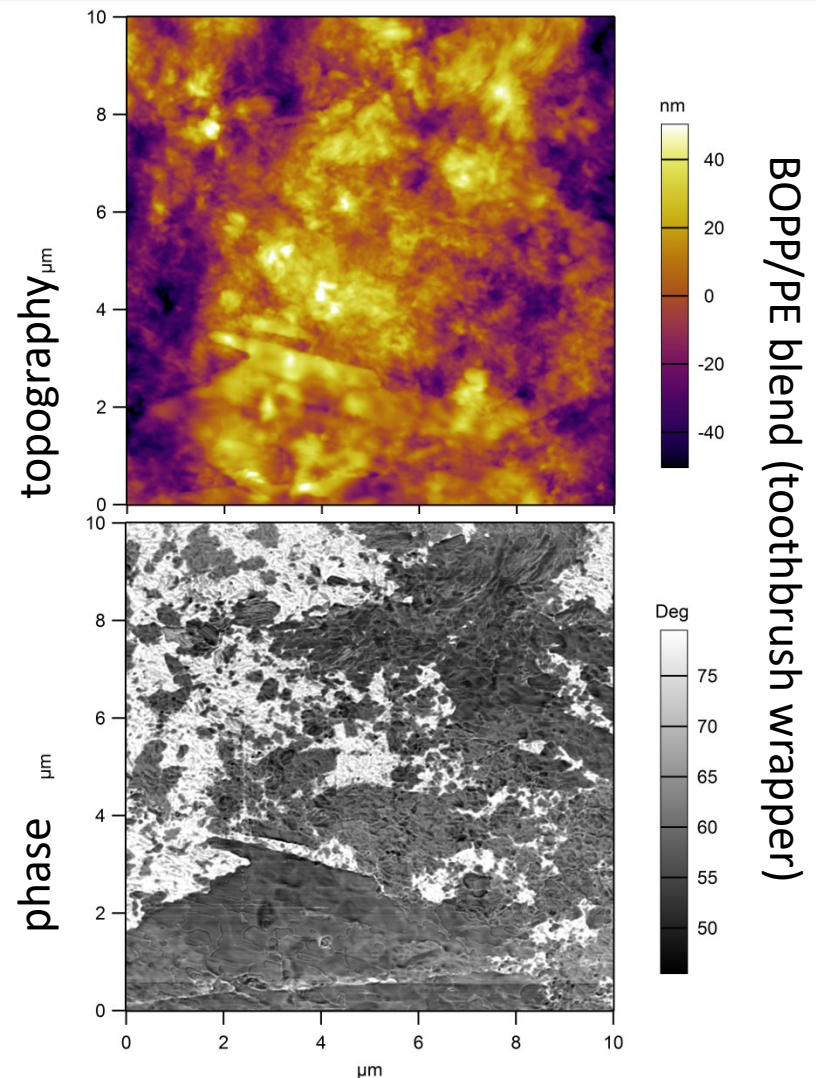
# Tapping Mode Imaging: Phase

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



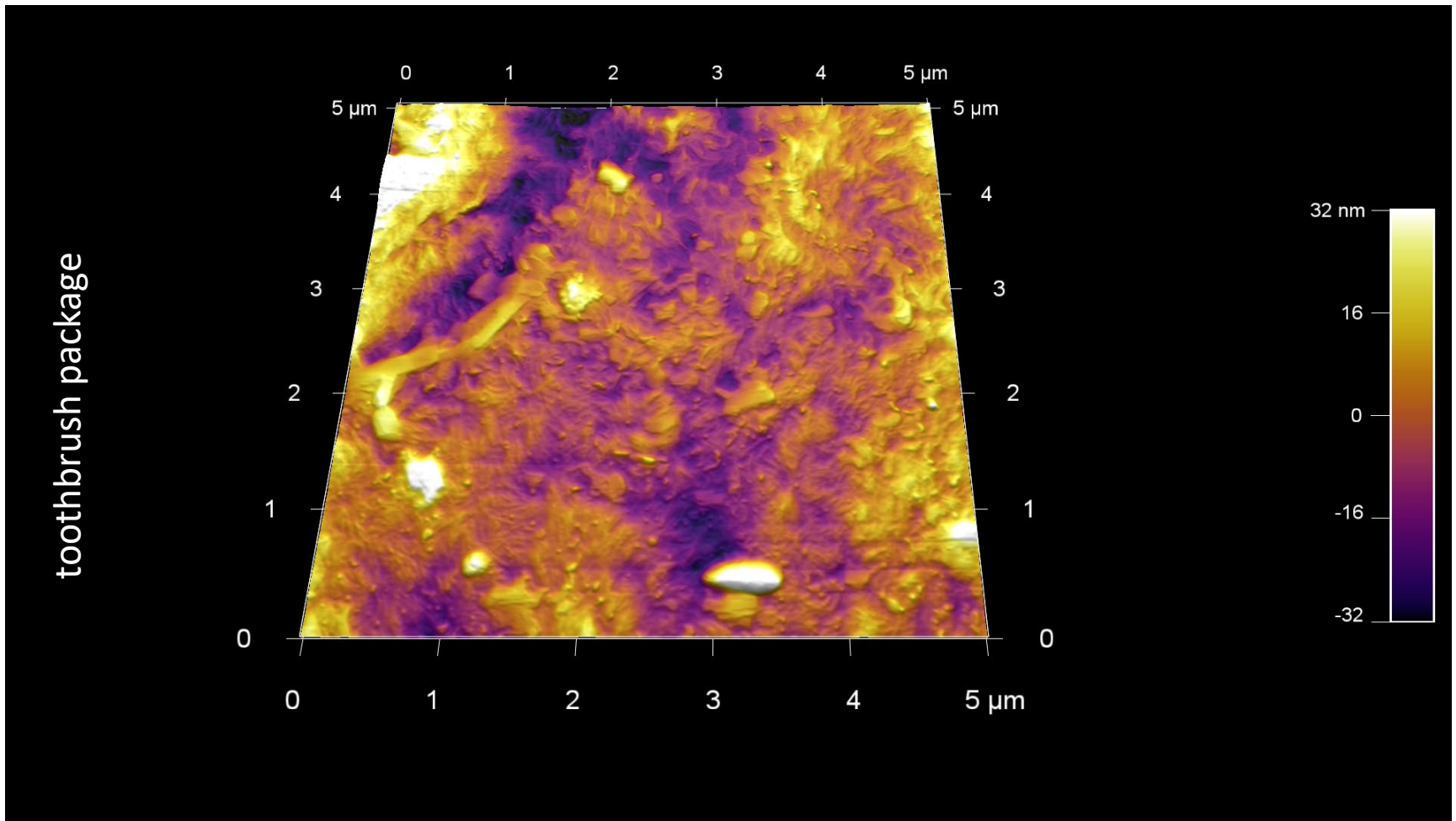
# Phase (Qualitative)

- 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



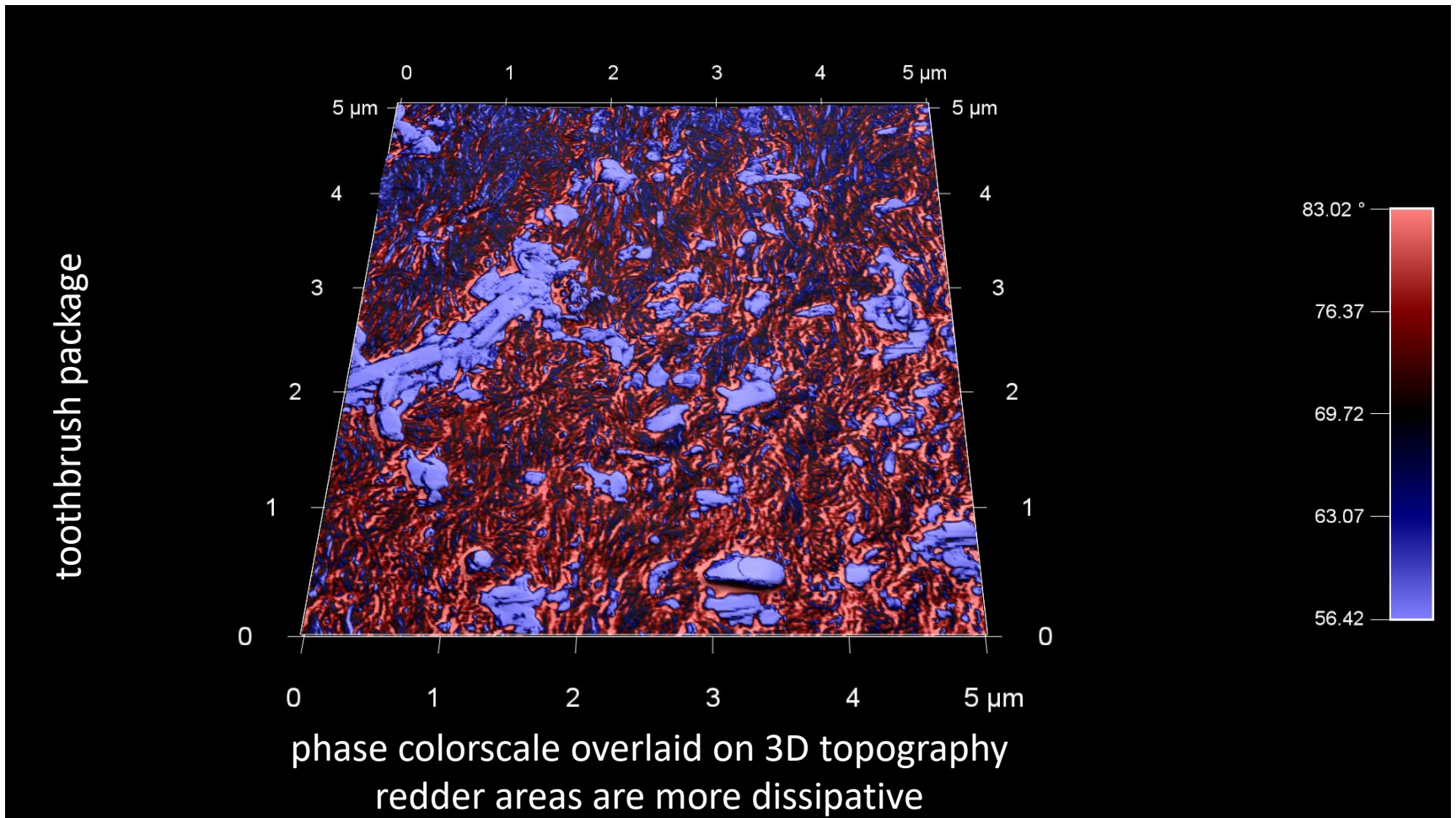


# Topography



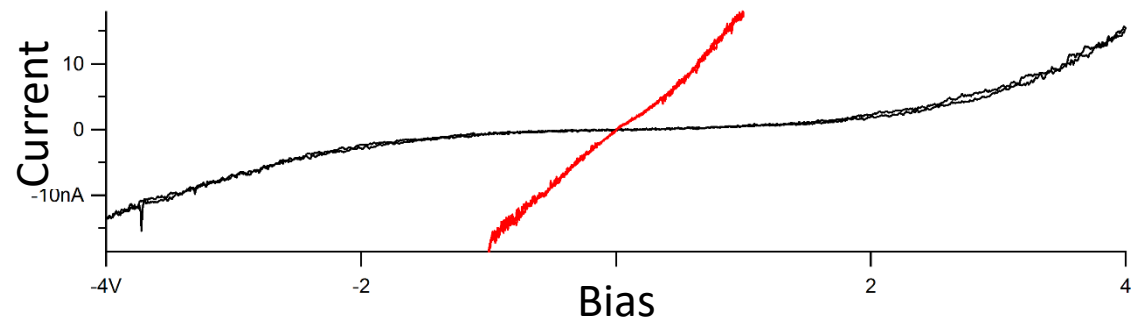
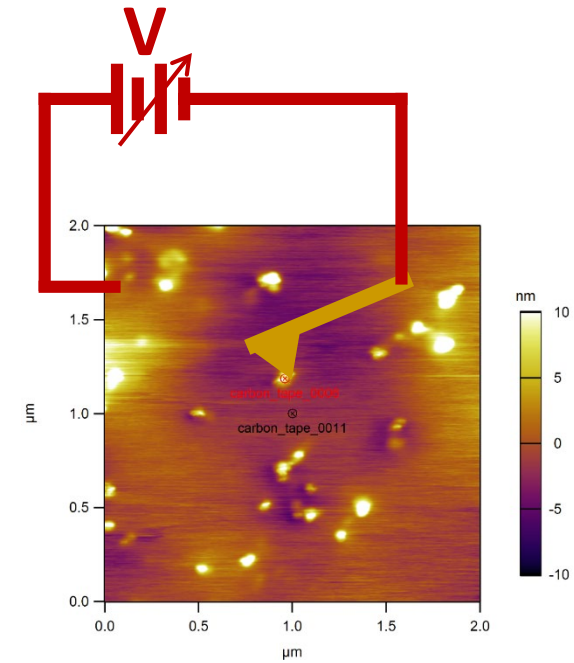
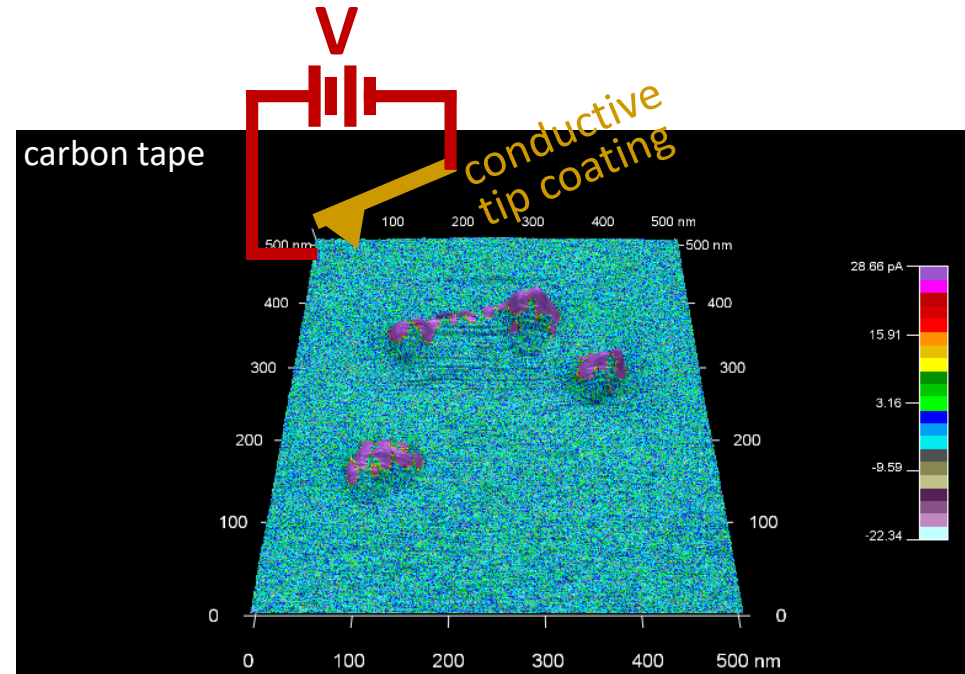


# Topography with Colors from Phase



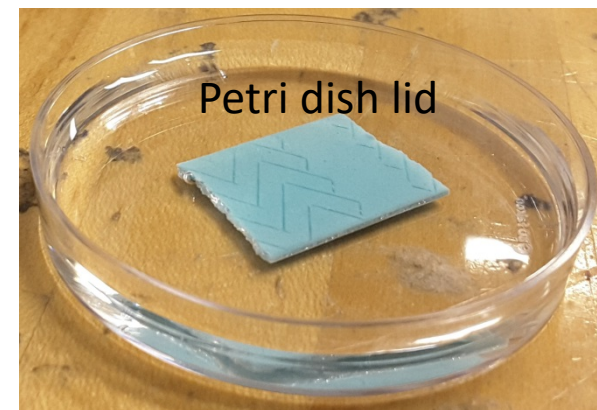
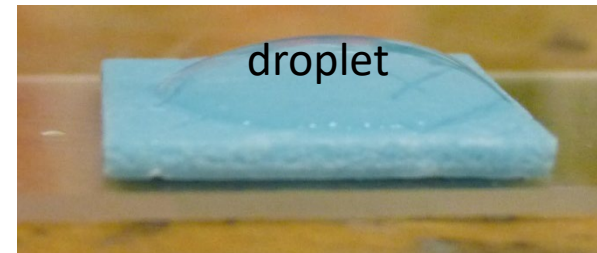


# Application: Conductive AFM



# Application: Fluid

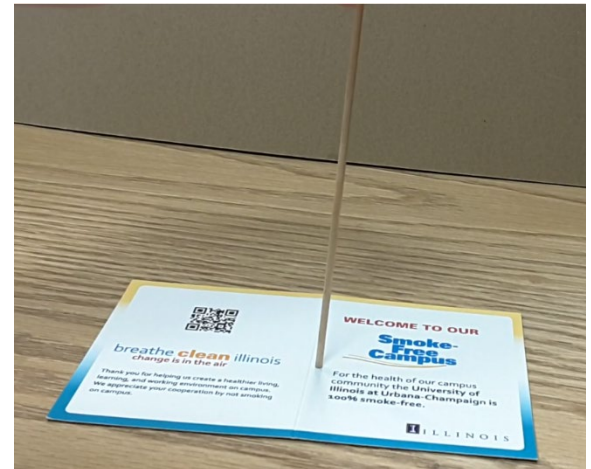
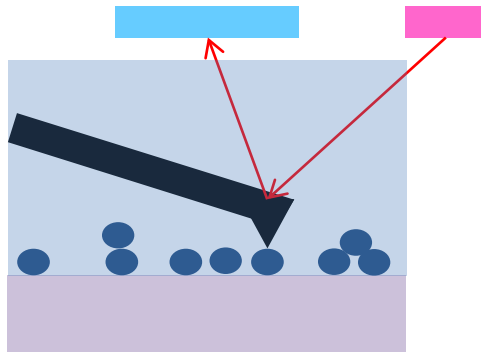
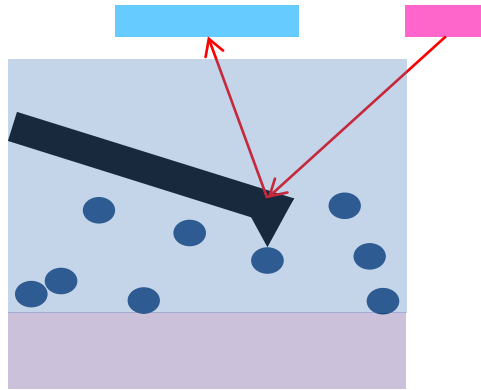
- Can image and do some mechanical measurements in fluid
- Different setups
  - Droplet of fluid on sample
  - Submerged sample in open dish
  - Closed fluid cell
- Fluid is trickier
  - Setup (need to be more careful)
  - Hydrodynamics (partial solution: photothermal cantilever excitation)







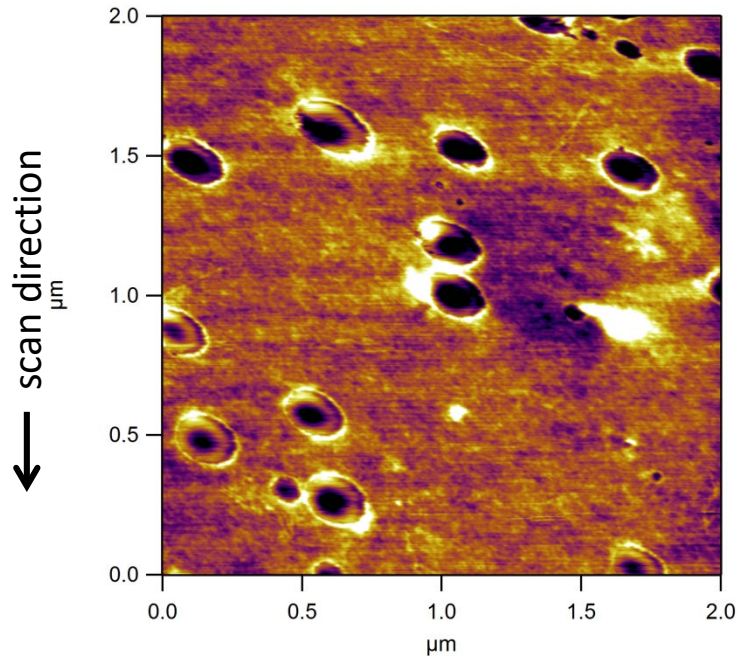
# Samples Shouldn't Float or Flex



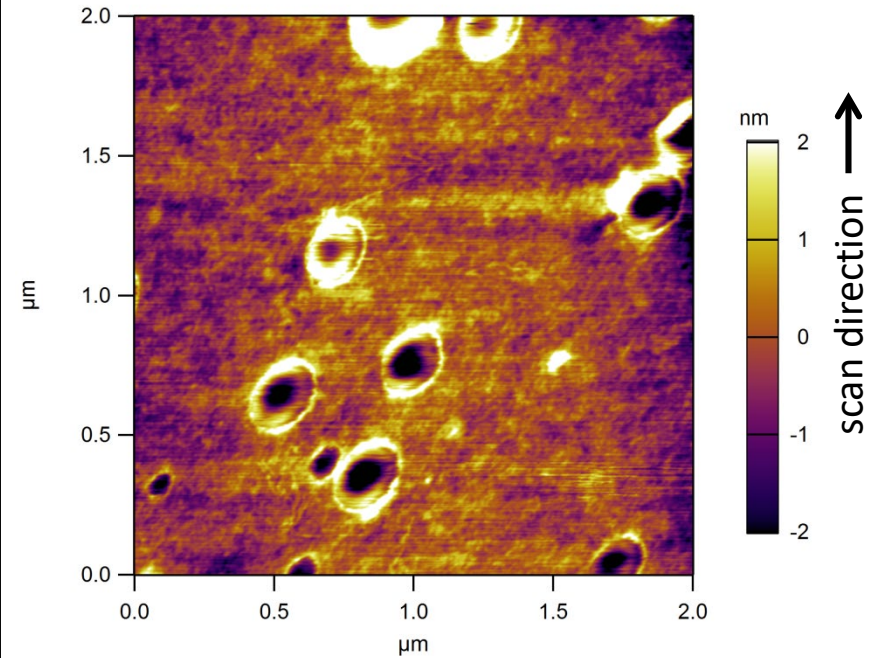


# Sample Drift

Scanning downwards...



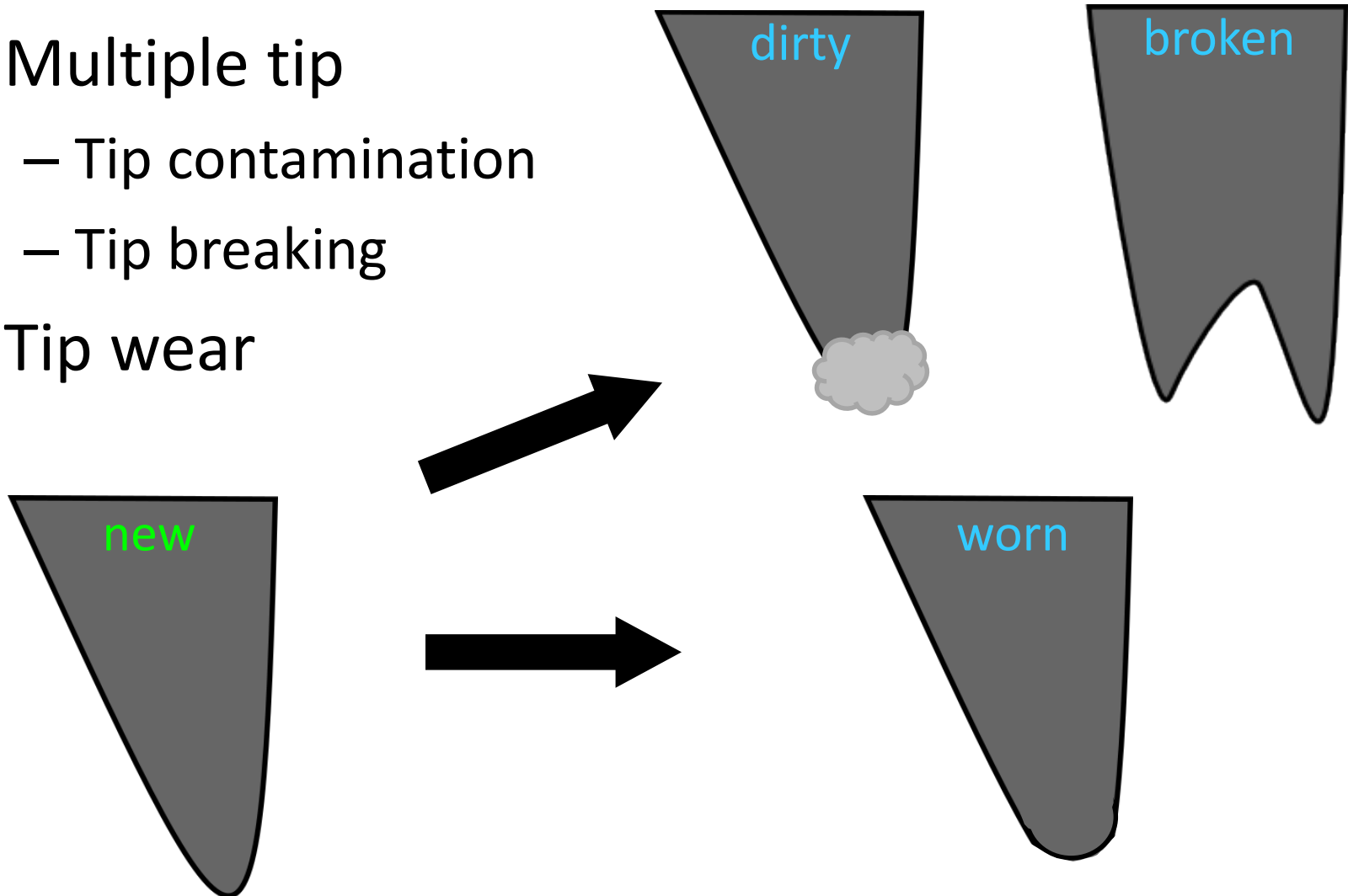
... then scanning upwards



chewing gum

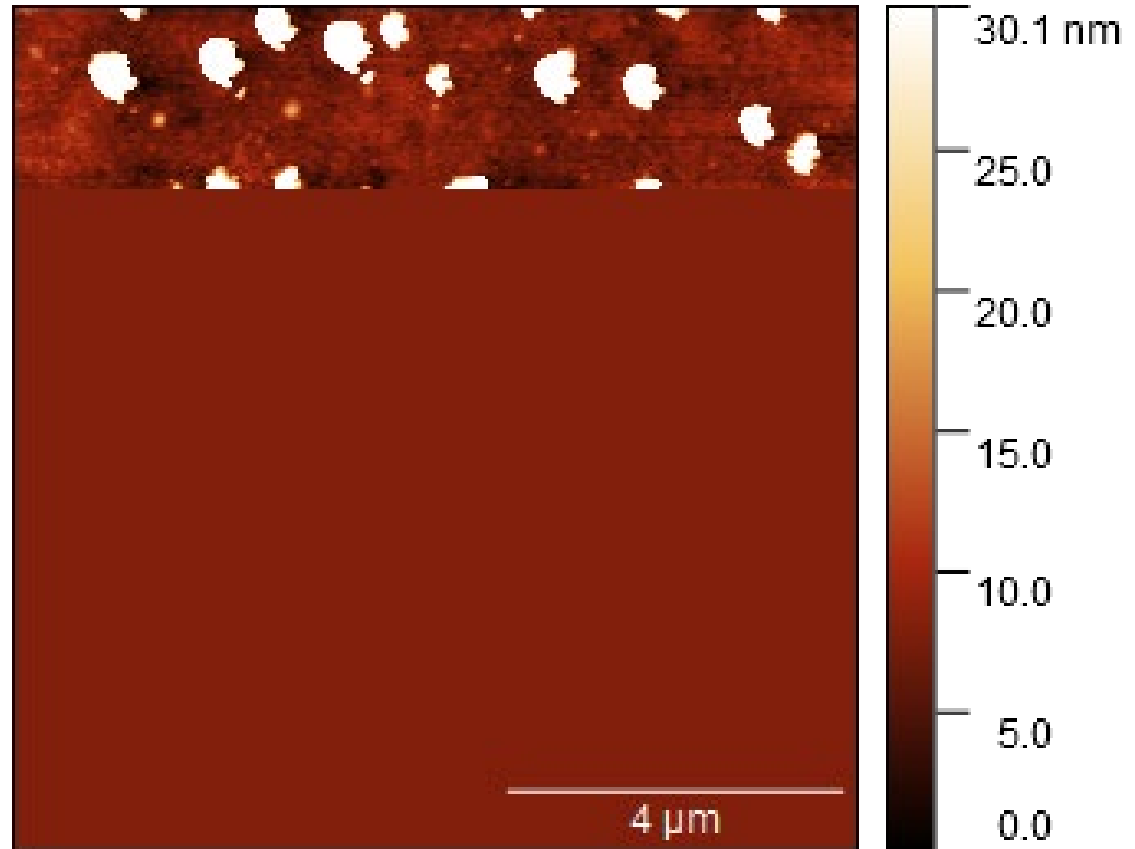
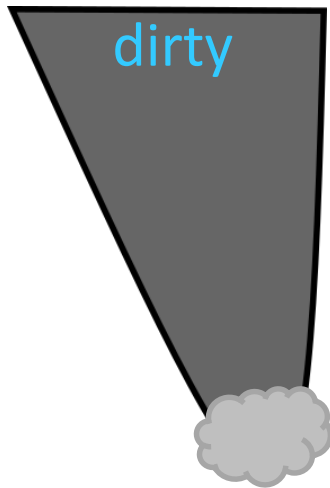
# Tip Artifacts

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





# Contaminated Tip



10 μm partial scan



# Image Processing

Do background subtraction first!



rockhopper penguin colony

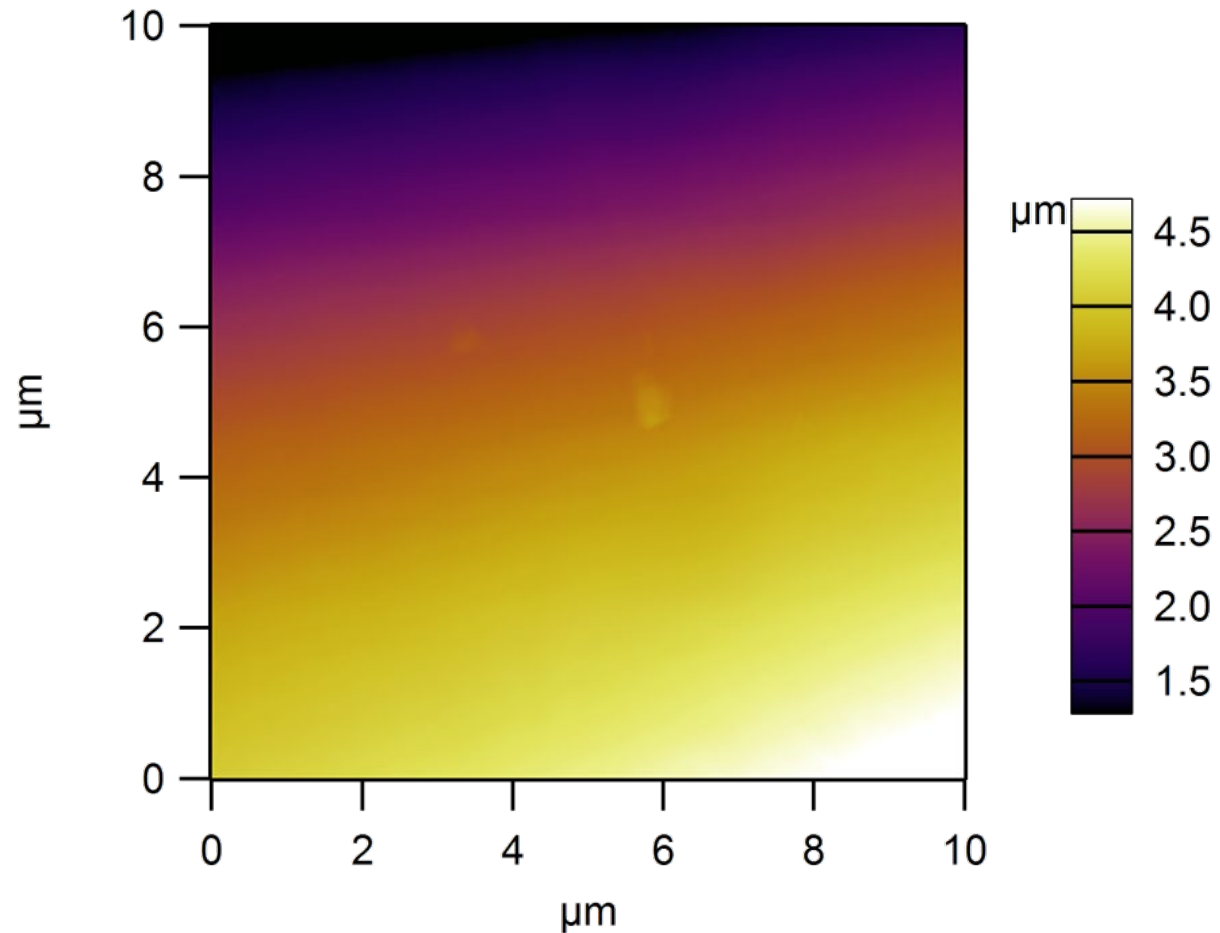


photography by Michael Jeffords and Susan Post, Prairie Research Institute



# Image Processing

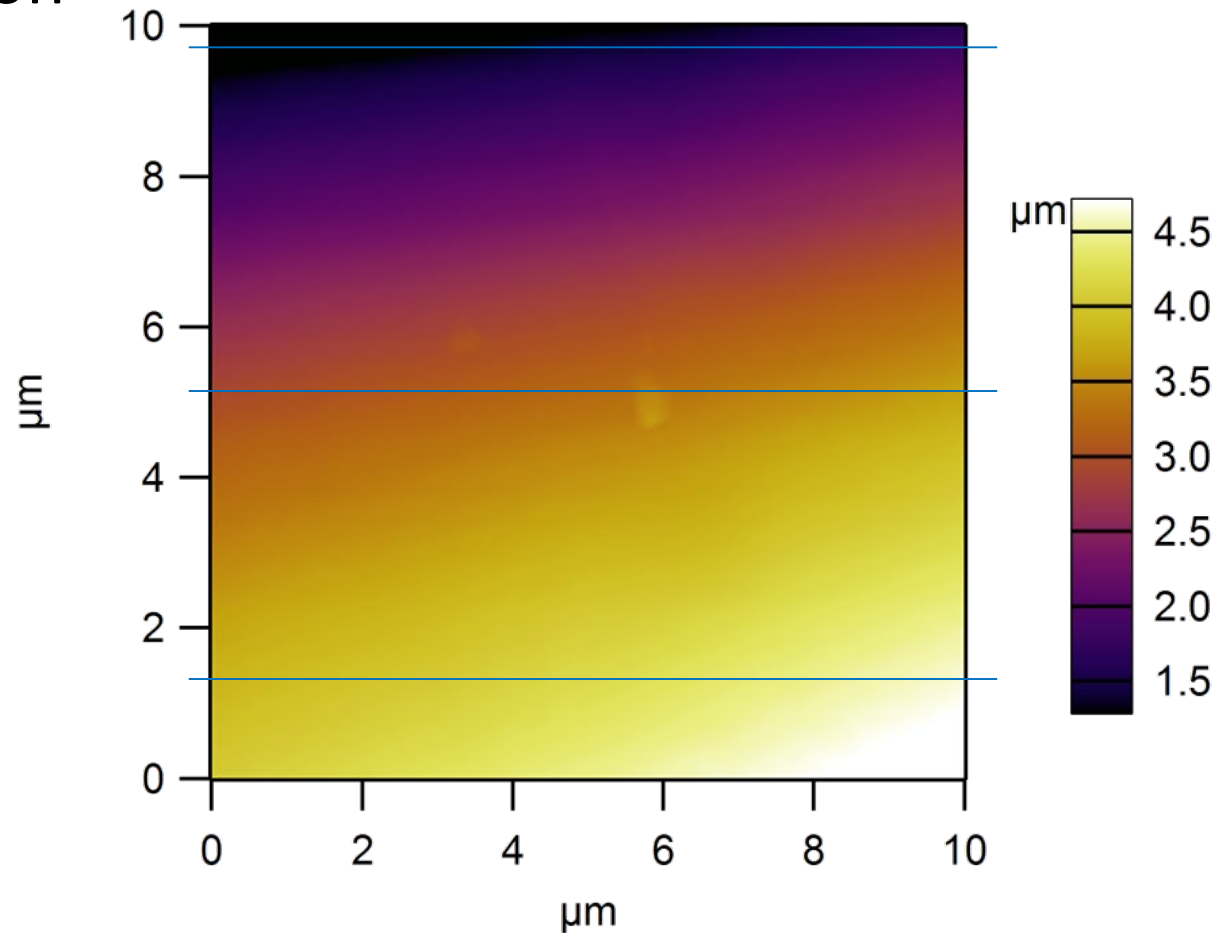
raw image





# Image Processing

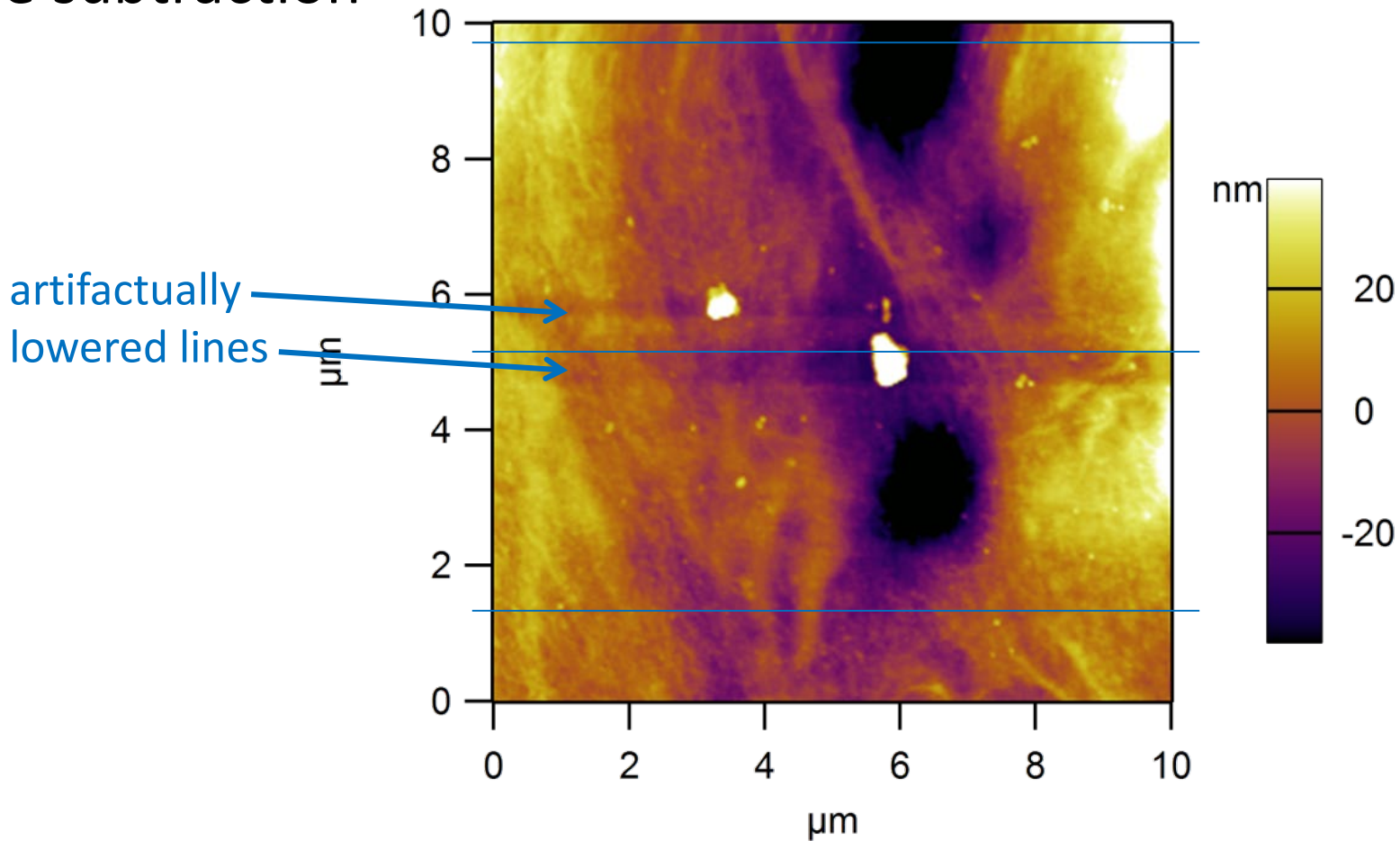
line subtraction





# Image Processing

line subtraction



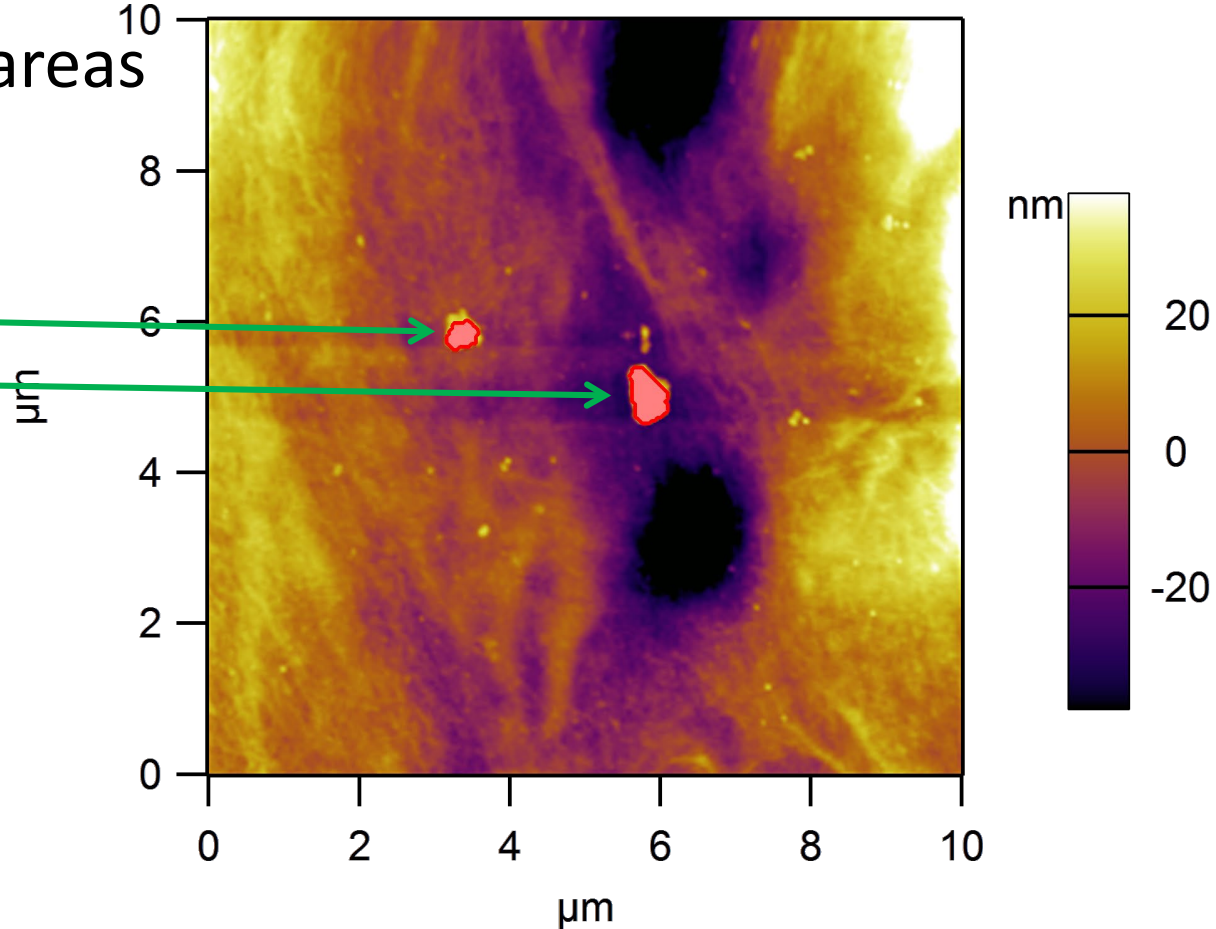




# Image Processing

line subtraction:  
mask outlier areas

areas to ignore  
when processing

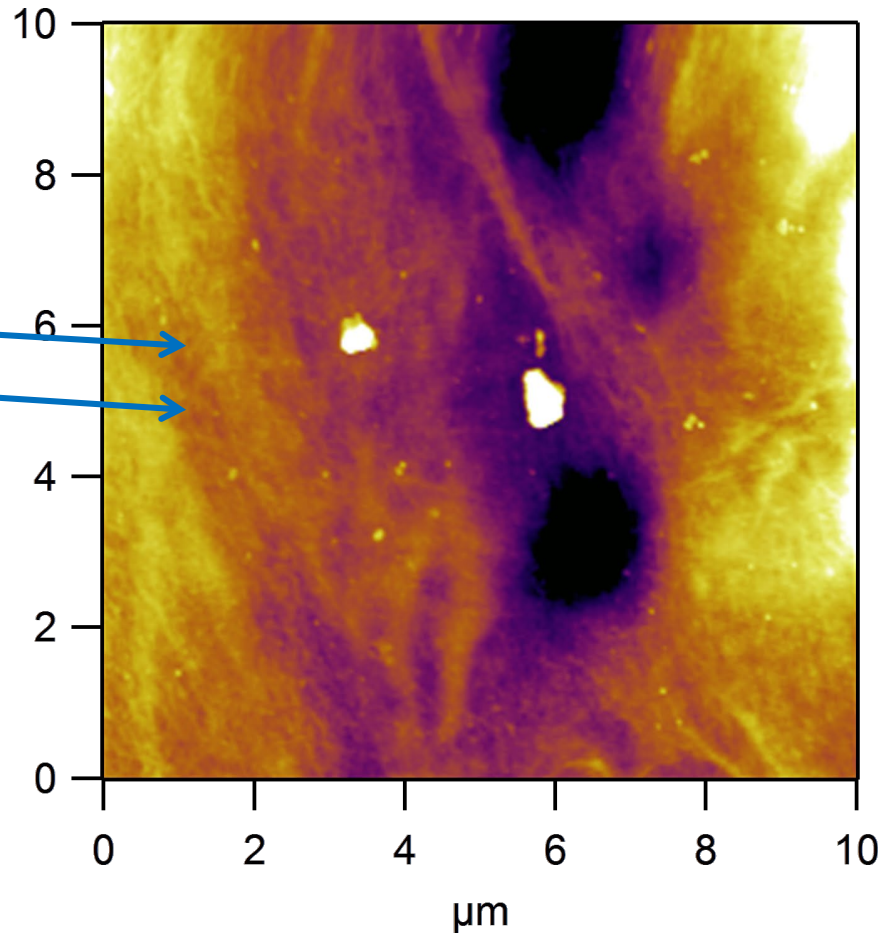




# Image Processing

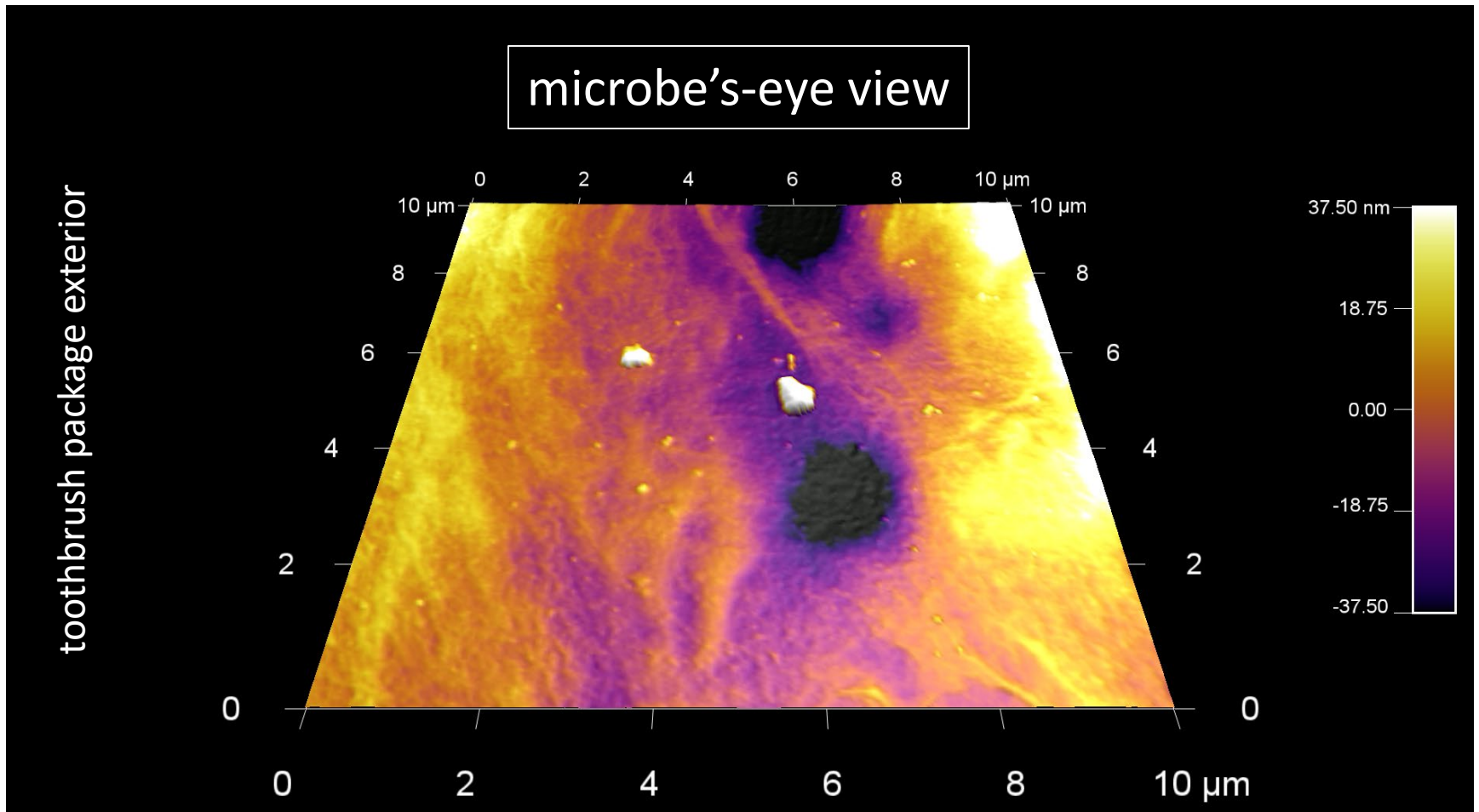
line subtraction:  
masked flatten

no more  
streaks





# Image Display

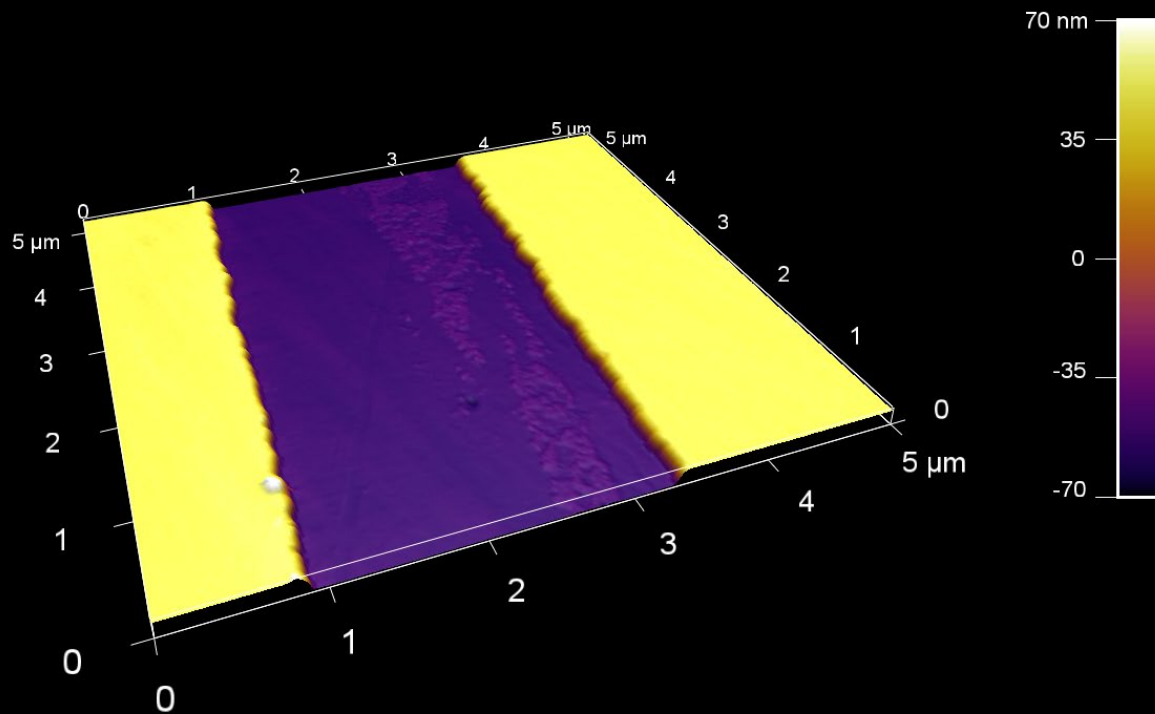




# Image Display

photomask

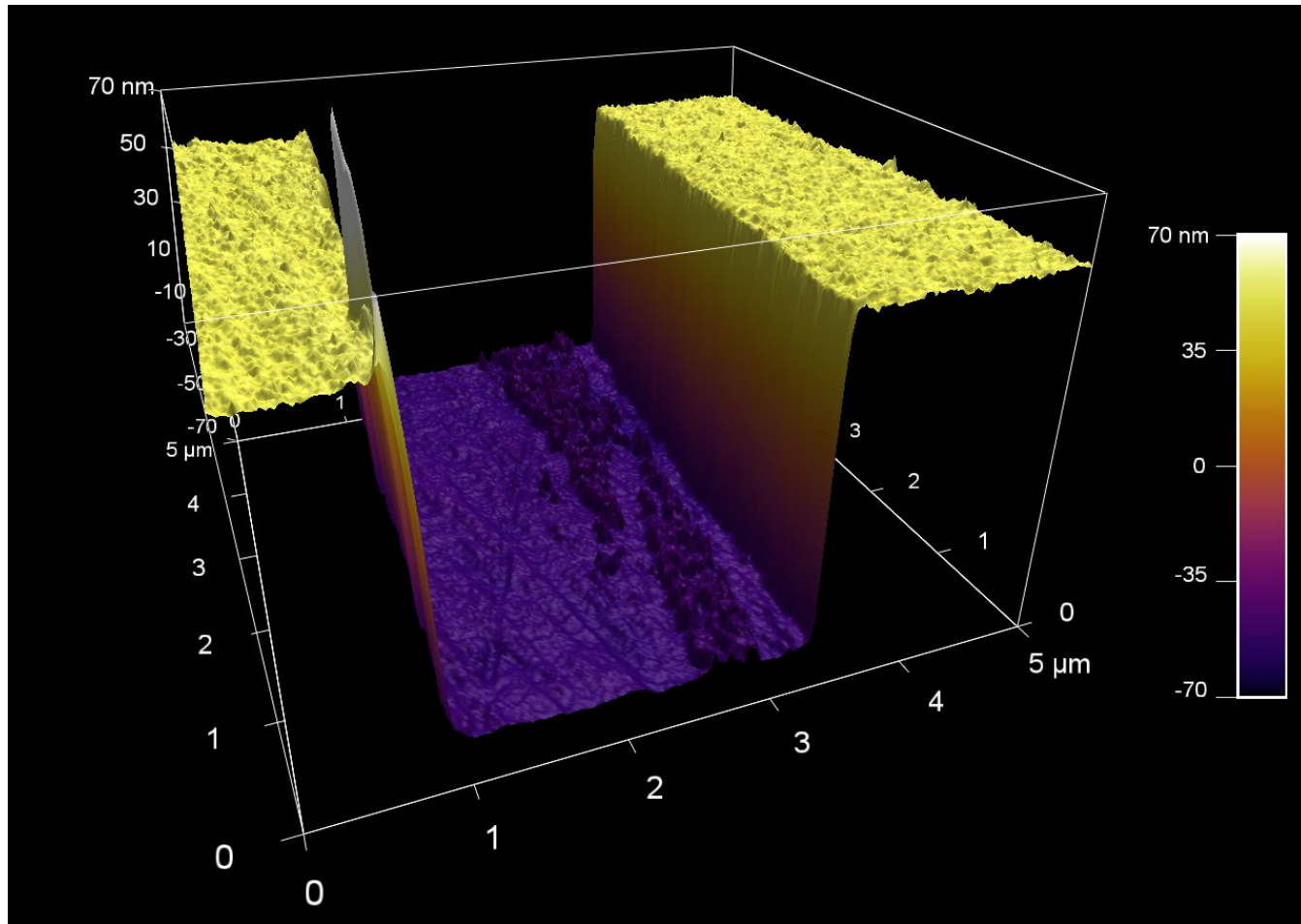
microbe's-eye view





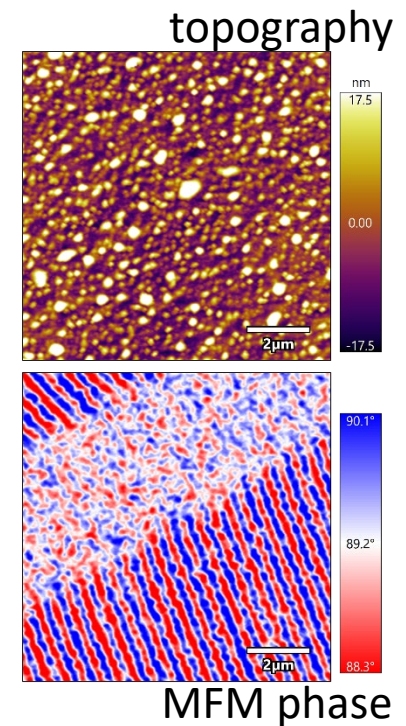
# Image Display

photomask


















# Many Other Applications

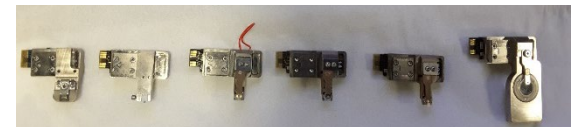
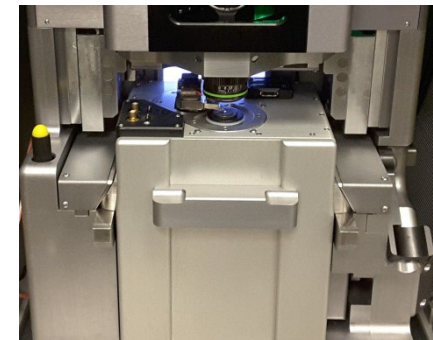
- Nanolithography/nanomanipulation
- LFM (friction, lateral force microscopy)
- EFM (electrostatic force microscopy)
- KPFM (SKPM, Kelvin probe)
- MFM (magnetic force microscopy)
- PFM (piezoresponse force microscopy)
- ... and these generally don't need extra gear (except different tips)





# Attachments on the MRL AFMs

-  ORCA Conductive AFM
-  Scanning Microwave Impedance Microscopy (sMIM)
-  Environmental Controller
-  BioHeater
-  PolyHeater (up to 300°C)
-  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

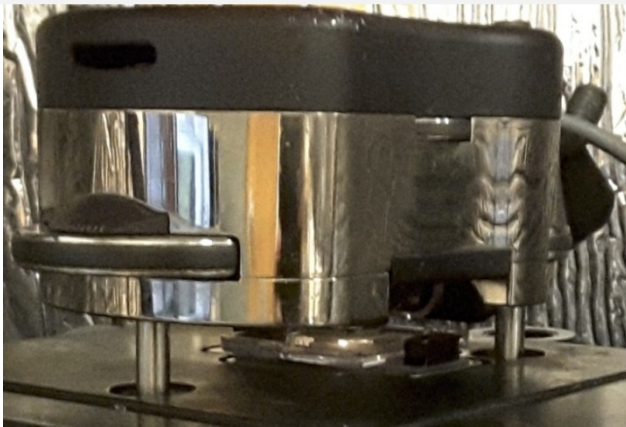




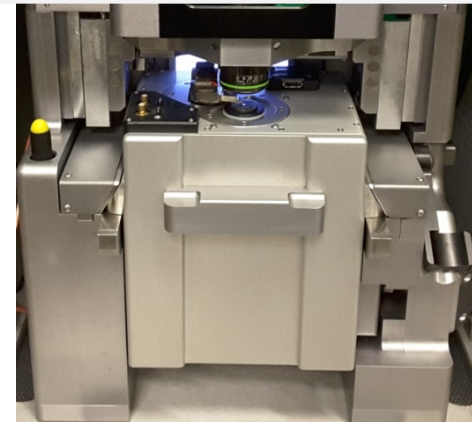
# MRL AFMs—B12 MRL

Lab tour today, 7/21, 4pm

Asylum MFP-3D-SA (2 of these)  
15 $\mu\text{m}$  z range, 90 $\mu\text{m}$  x 90 $\mu\text{m}$  scan size



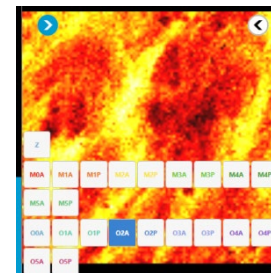
Asylum Cypher  
5 $\mu\text{m}$  z range, 30 $\mu\text{m}$  x 30 $\mu\text{m}$  scan size





# Related Instruments at MRL

- Neaspec Nano-IR
  - AFM + infrared
  - Highly localized chemical information
- Coming soon: Horiba TERS
  - Tip-enhanced Raman spectroscopy



Julio Soares, MRL

- Dektak stylus profilometer
- Keyence 3D optical profiler



# Keep Learning

- MRL Webinar Series
  - [go.illinois.edu/MRLYouTubeChannel](http://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](mailto:kawalsh@illinois.edu)

