Grainger College of Engineering
UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

Atomic Force Microscopy

Kathy Walsh

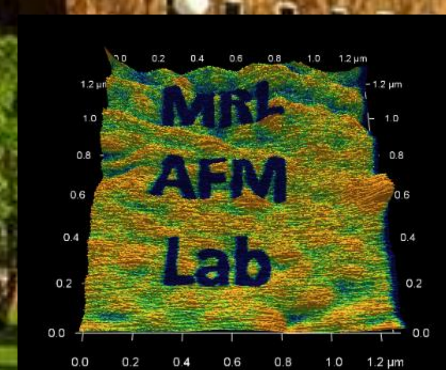
Senior Research Scientist

Scanning Probe Microscopy

Materials Research Laboratory

Central Research Facilities

Physics 403
7/20/22

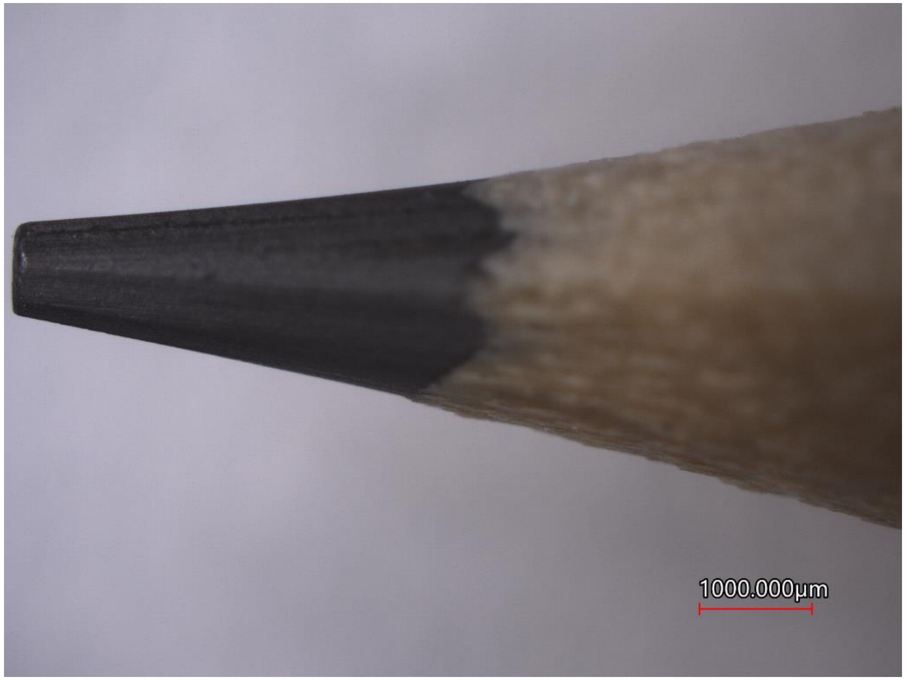


Illinois Materials Research Lab Central Research Facilities

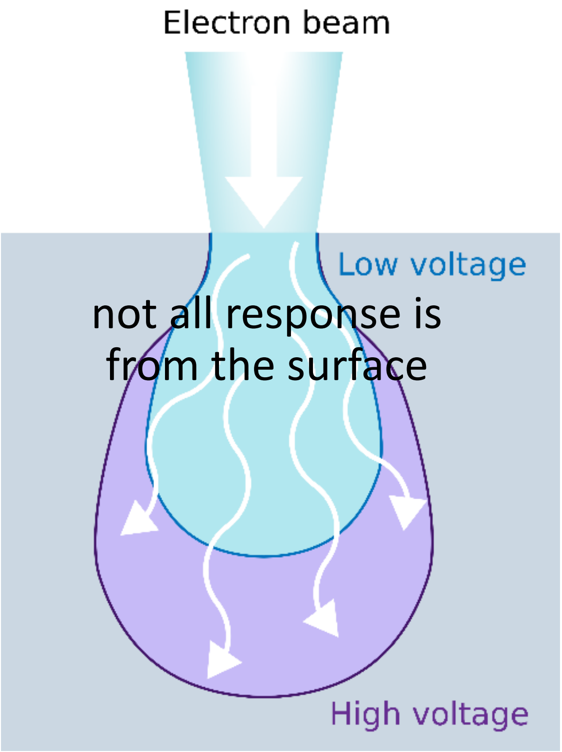
- User facility—anyone can be trained
  - UIUC and non-UIUC researchers welcome
  - Undergraduate researchers welcome
  - Staff collaboration or analysis available
- mrl.illinois.edu/facilities
- mrl-facilities@illinois.edu

Looking at Surfaces

Optical Microscopy



Scanning Electron Microscopy

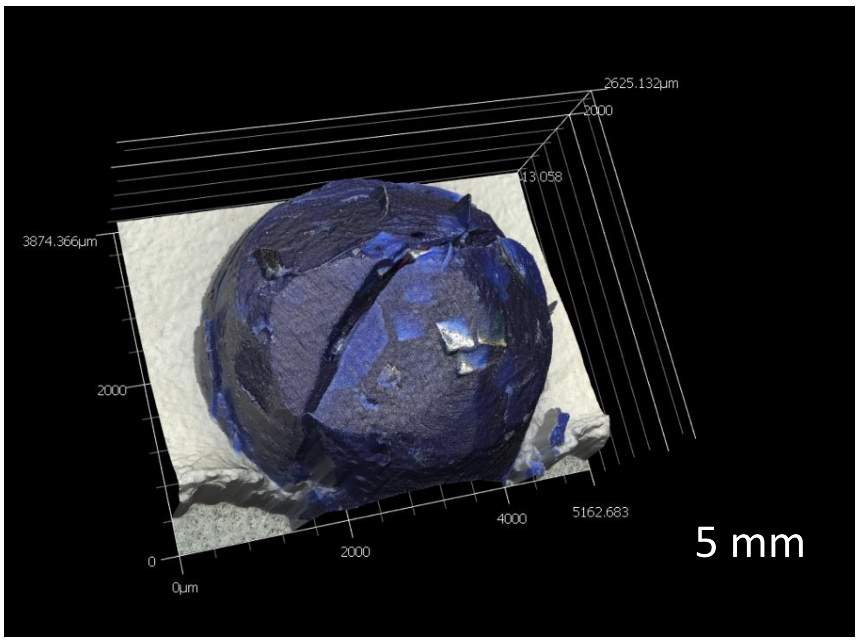


Adapted from
https://myscope.training/#/SEMlevel_2_13
(CC BY-SA 4.0)

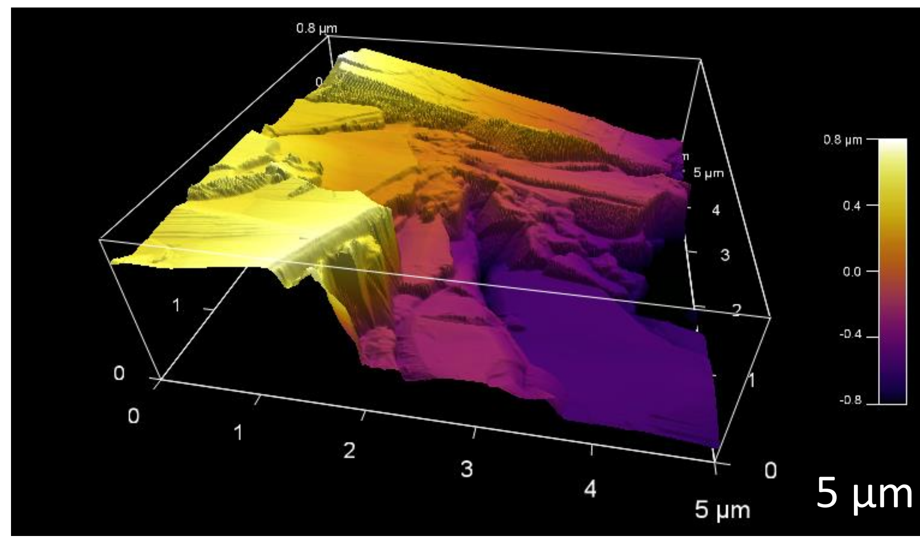
Surface XYZ Coordinates Needed

3D Optical Profilometry

Atomic Force Microscopy



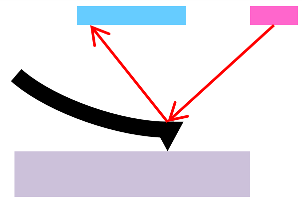
blue glitter crayon tip

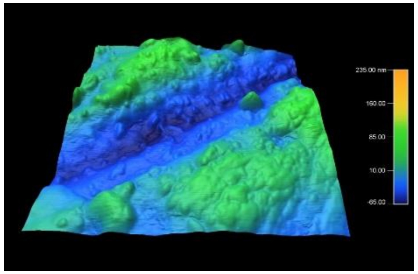


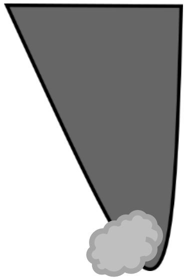
pencil “lead”

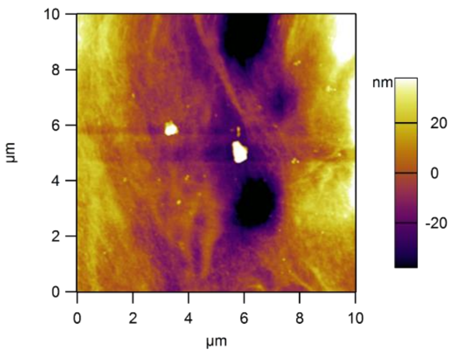
Topics for Today

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



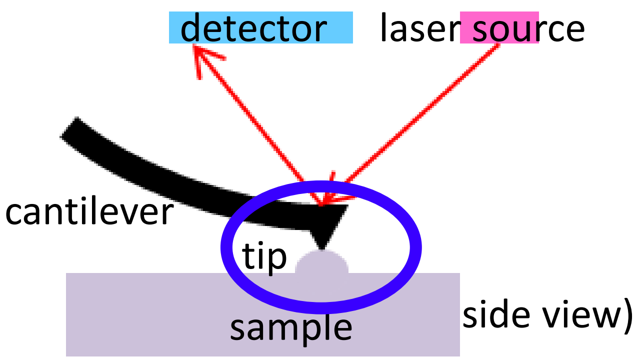


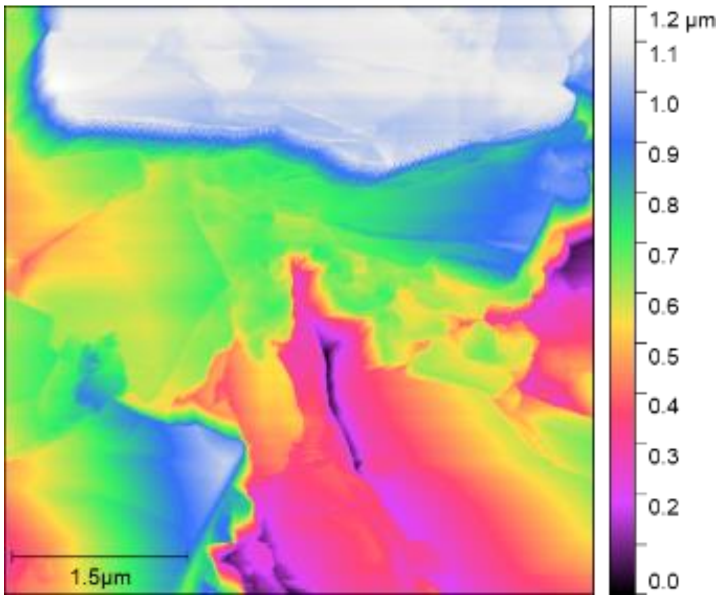




What's an Atomic Force Microscope?

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

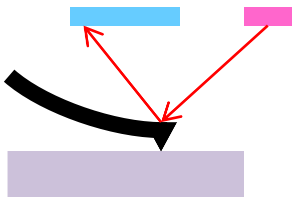




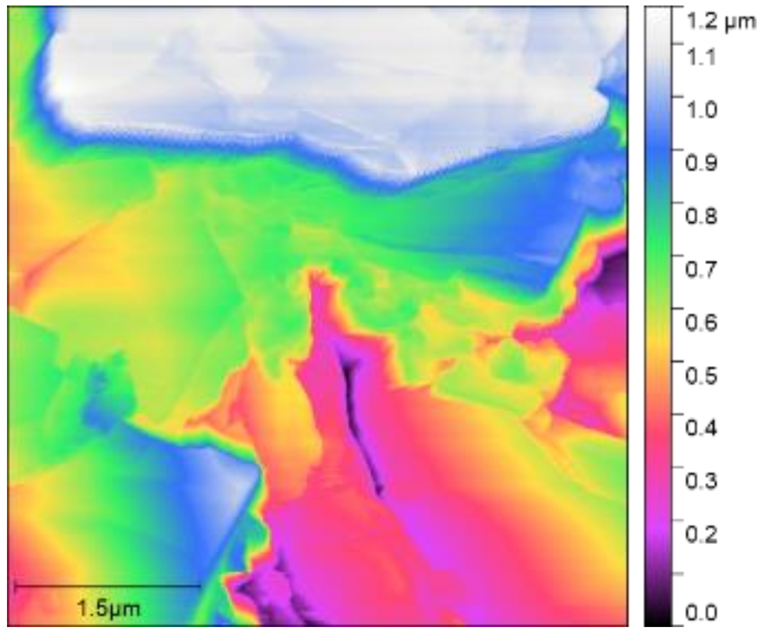
false-color surface topographs

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
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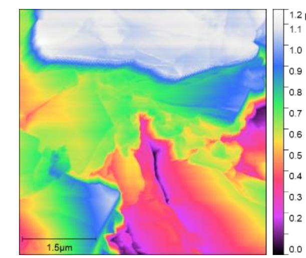
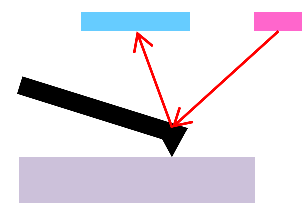
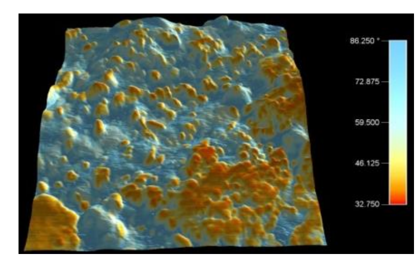
- “Microscope”—surface topograph (false color)



- 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

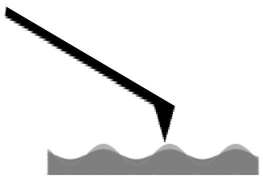


Turquoise, 1µm x 1µm
color overlay: phase

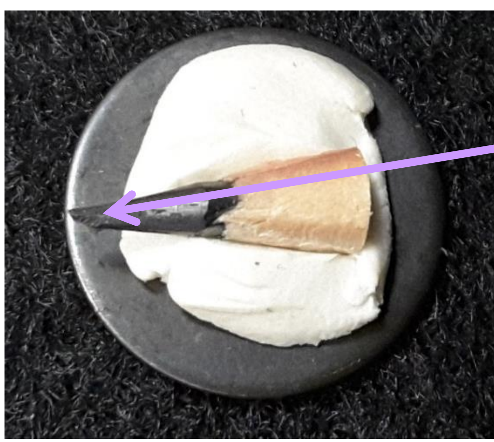
Typical AFM Scales

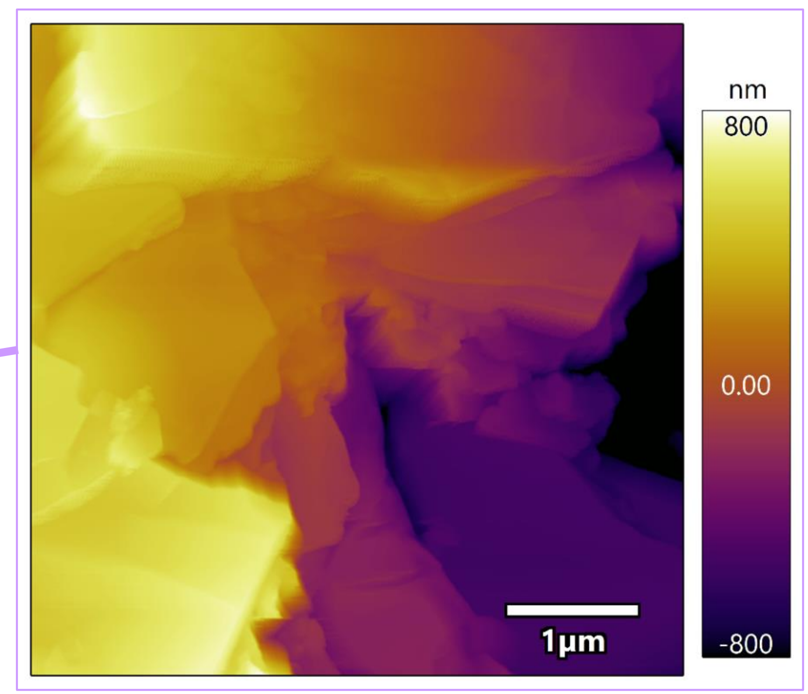
(only what's pretty common, not all of what's possible)

- Image sizes -- few to tens of μm^2
- Feature peak-to-valley -- Å to μm

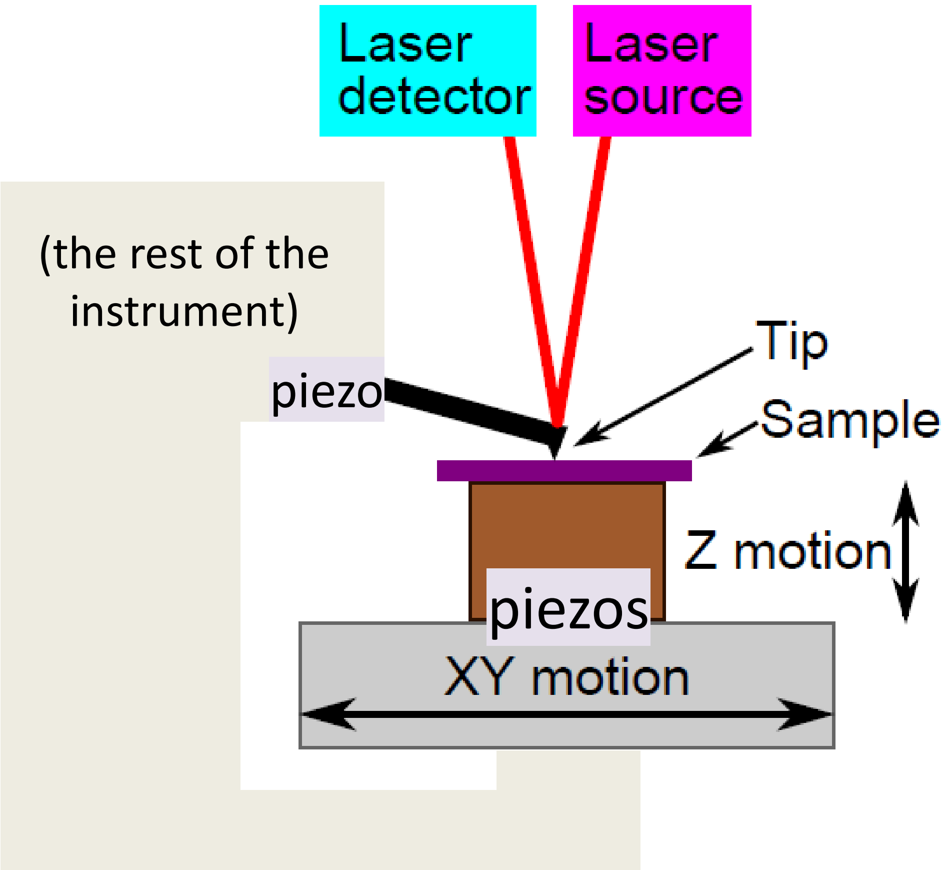


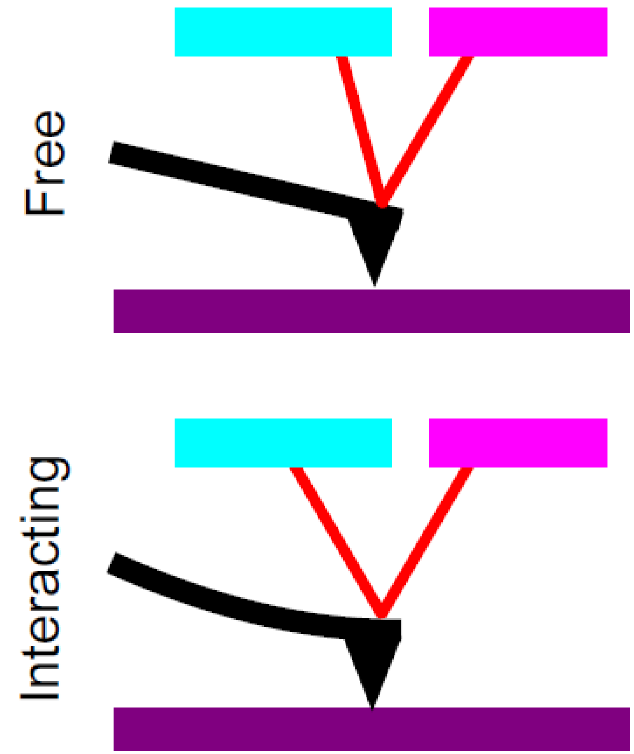
- Sample sizes -- mm to cm
- AFM measures surfaces





AFM Schematic

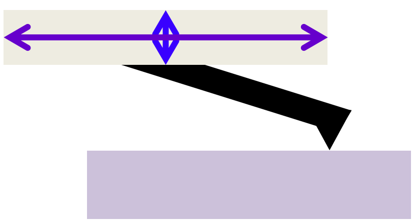




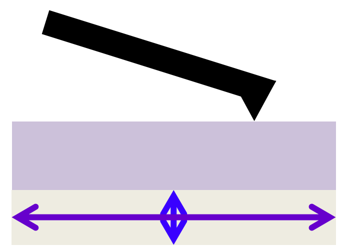
Scanners

scanning probe microscopy

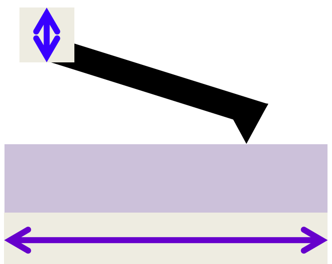
tip scanning



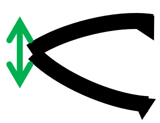
sample scanning



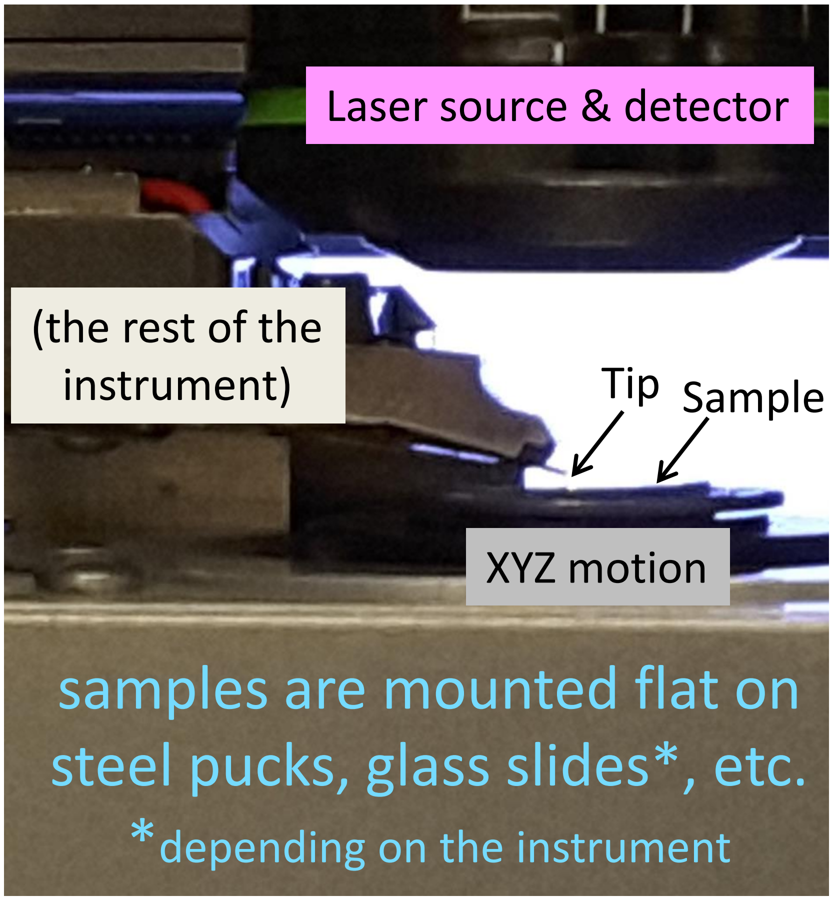
decoupled scanning



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

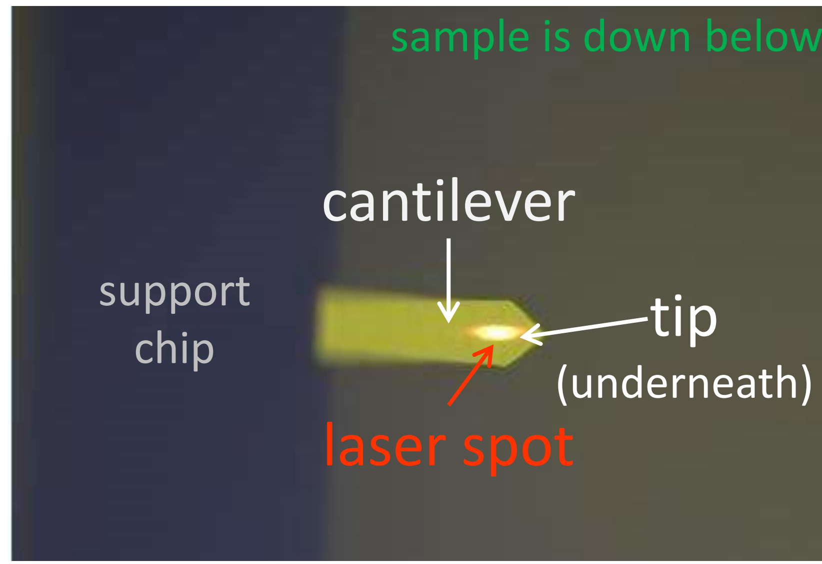


AFM Instrument

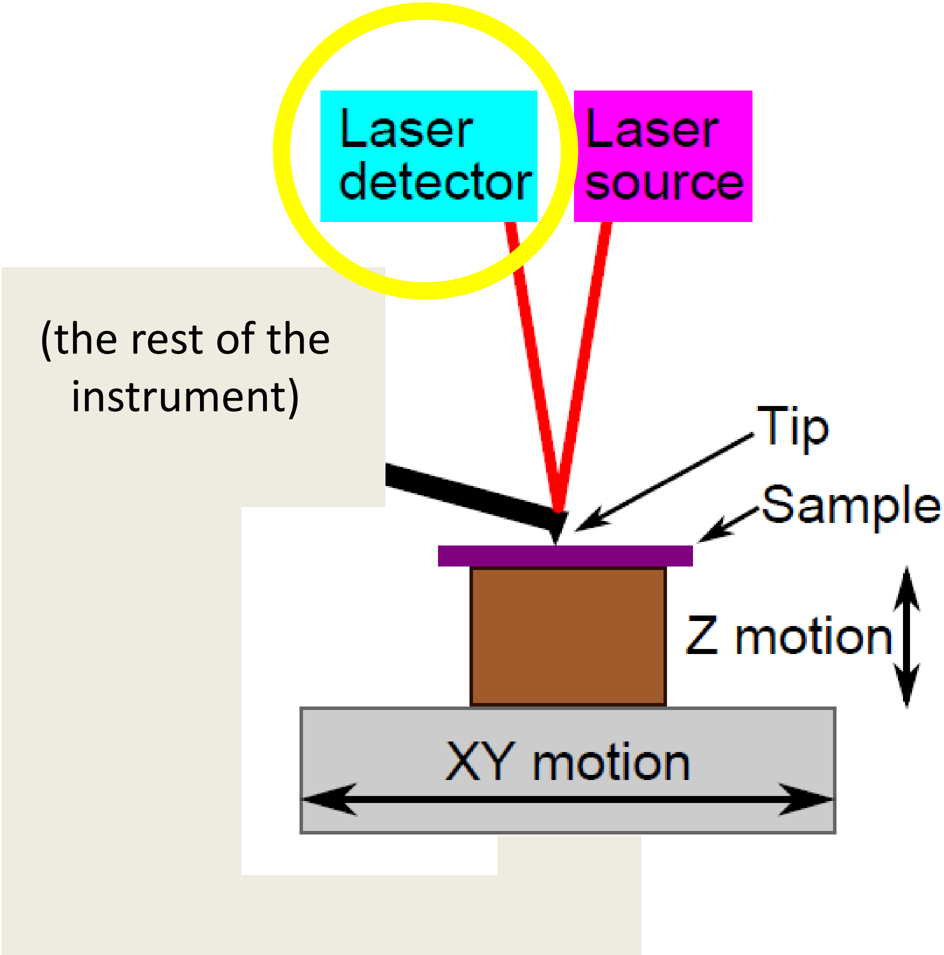


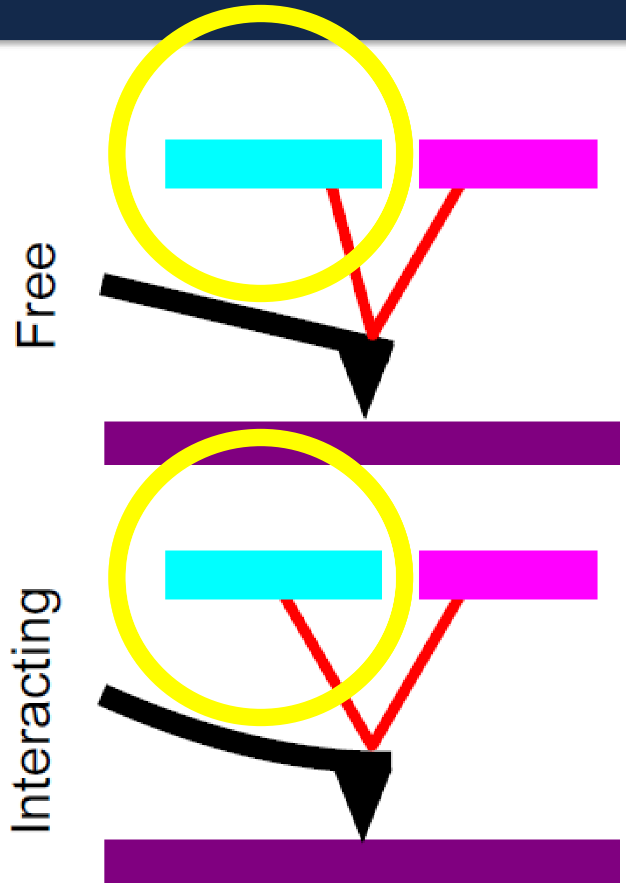
side view

top view



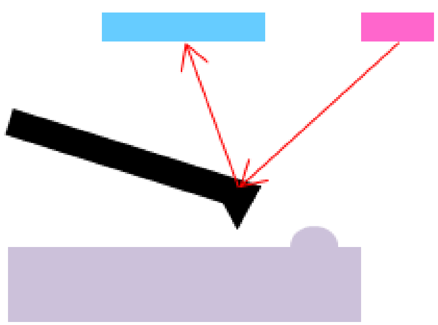
AFM Schematic



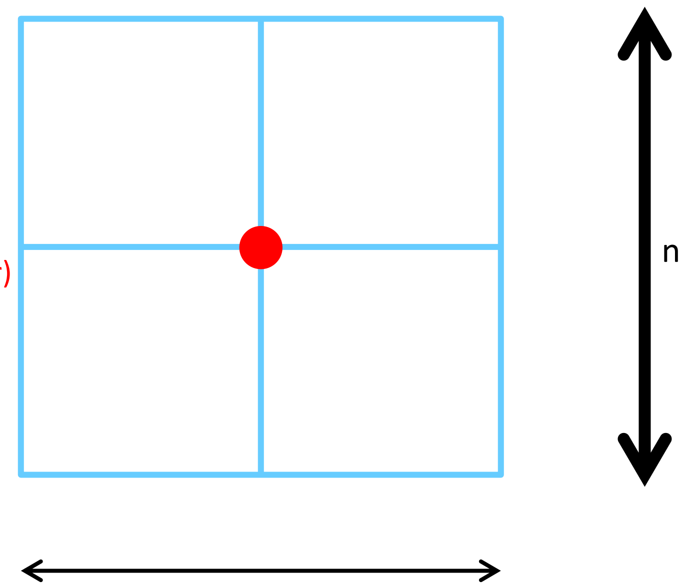


Laser Detection

non-interacting



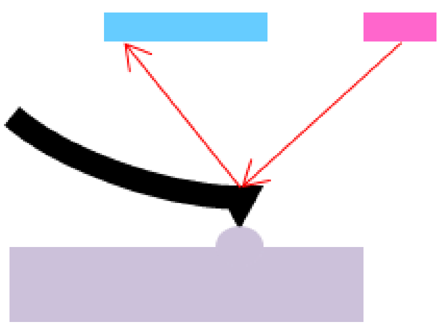
(side view)



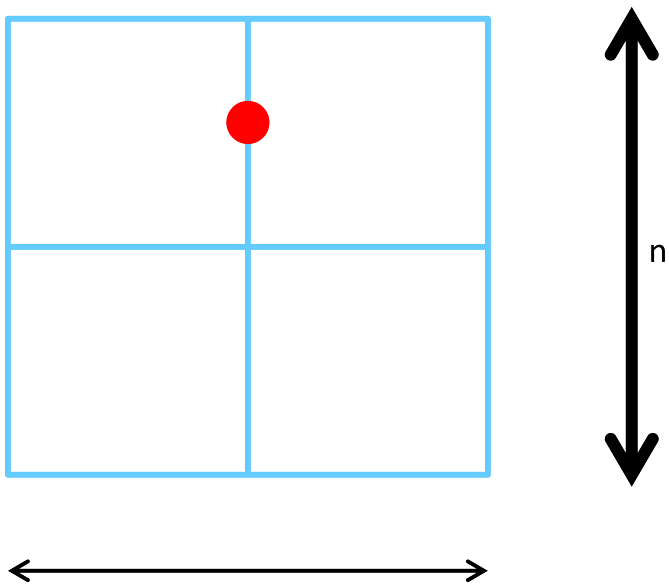
(exaggerated schematic)

Laser Detection

sample pushing up



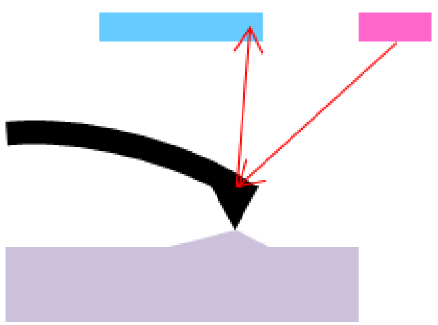
(side view)



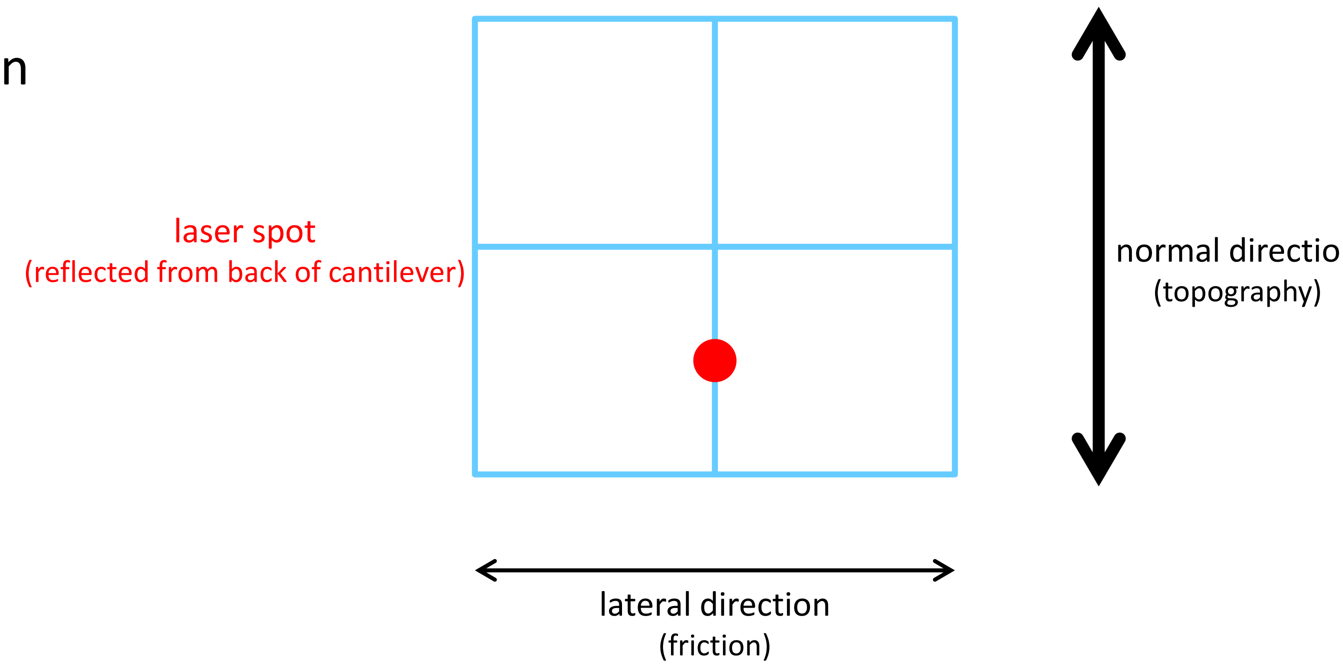
(exaggerated schematic)

Laser Detection

sample pulling down



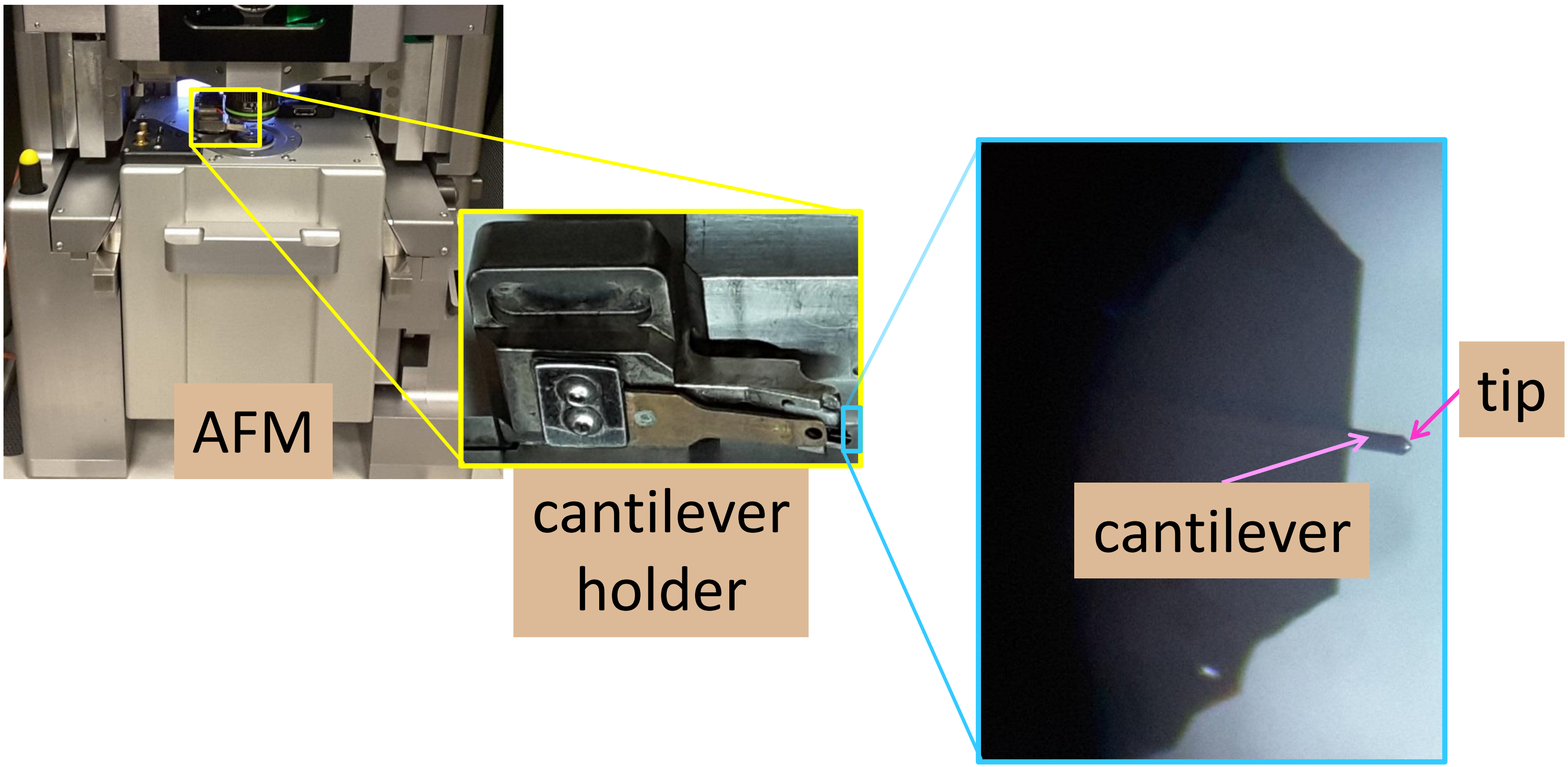
(side view)



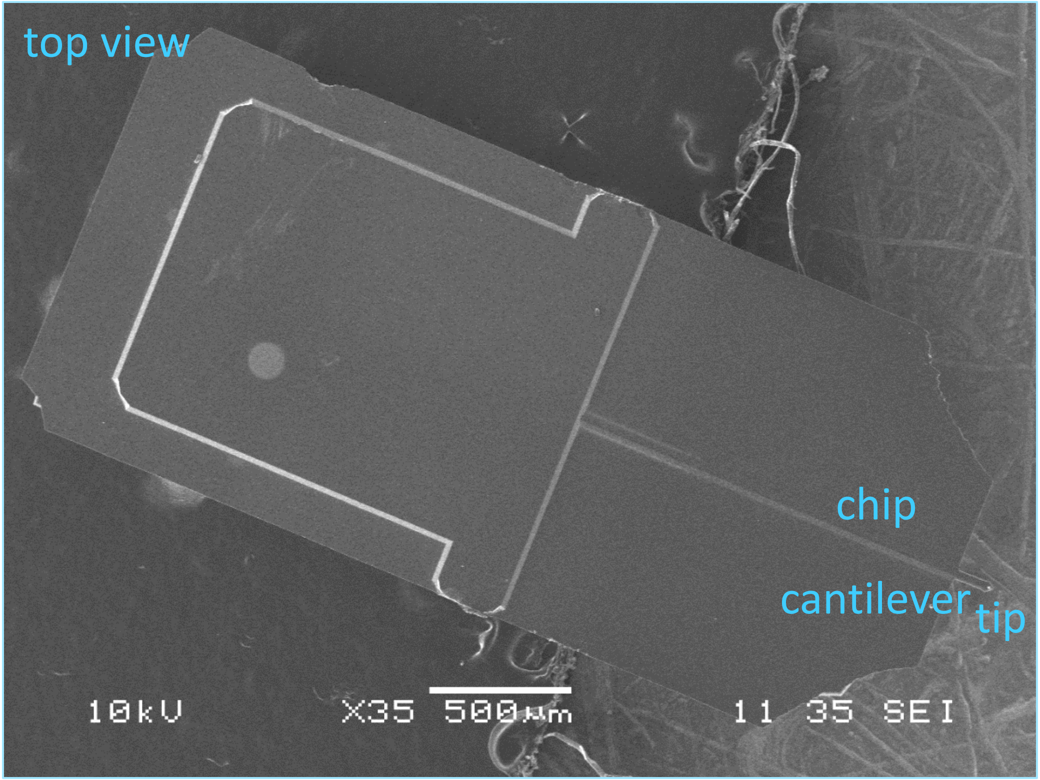
(exaggerated schematic)

AFM Tips

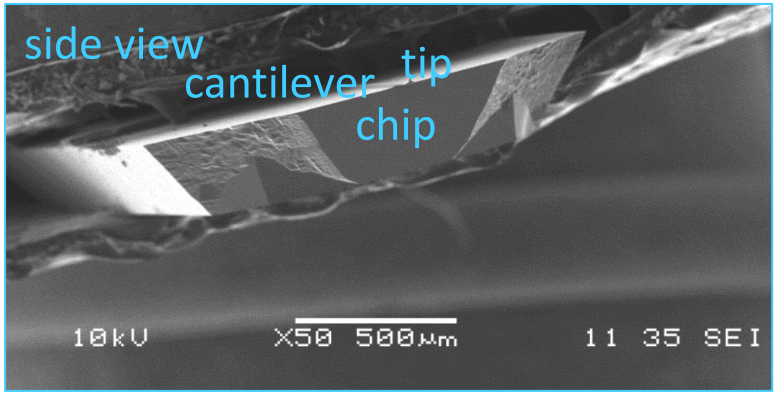
scanning *probe* microscopy



Tip Terminology

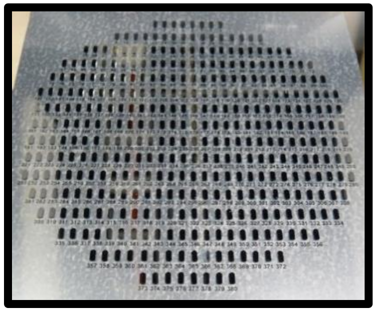


SEM images taken using MRL's JEOL 6060LV

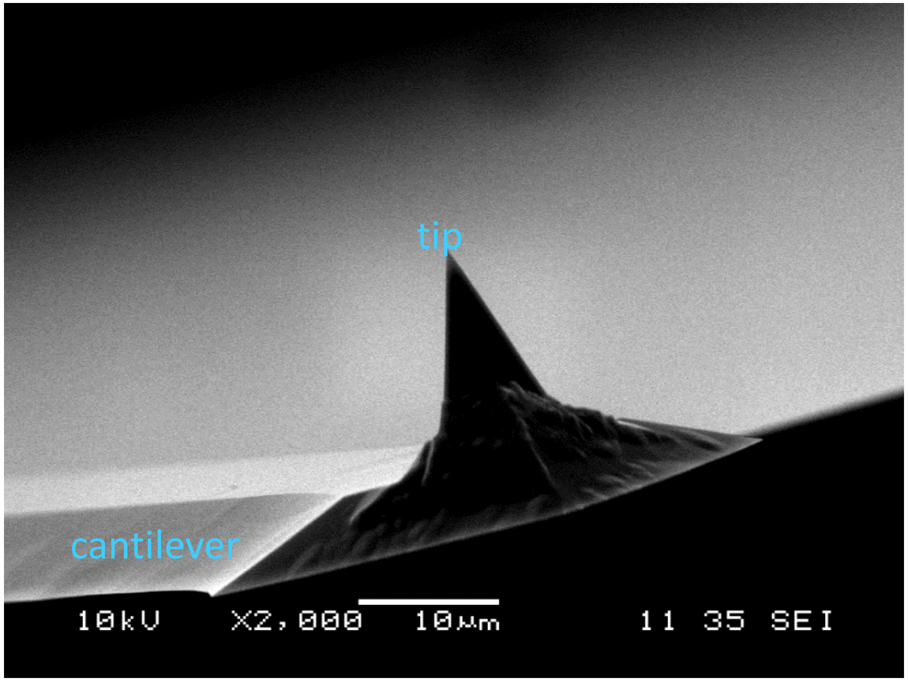


"probe"

tips point upwards in the box



Typical Tip



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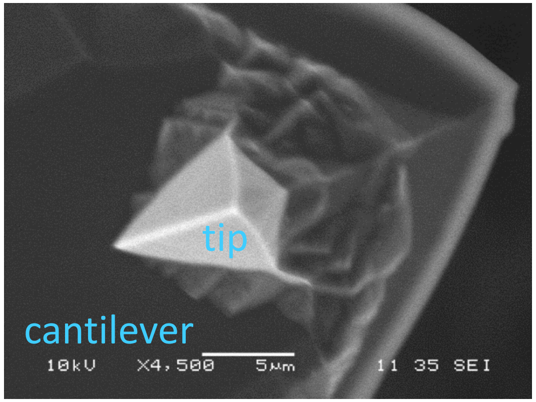
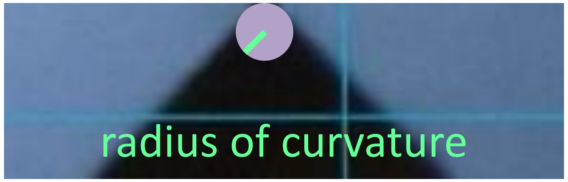


common tip for imaging:

- tip radius of curvature < 10 nm
- silicon tip
- cantilever width 30 μm
- cantilever length 125 μm
- cantilever thickness 4 μm



Tip Types

- 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

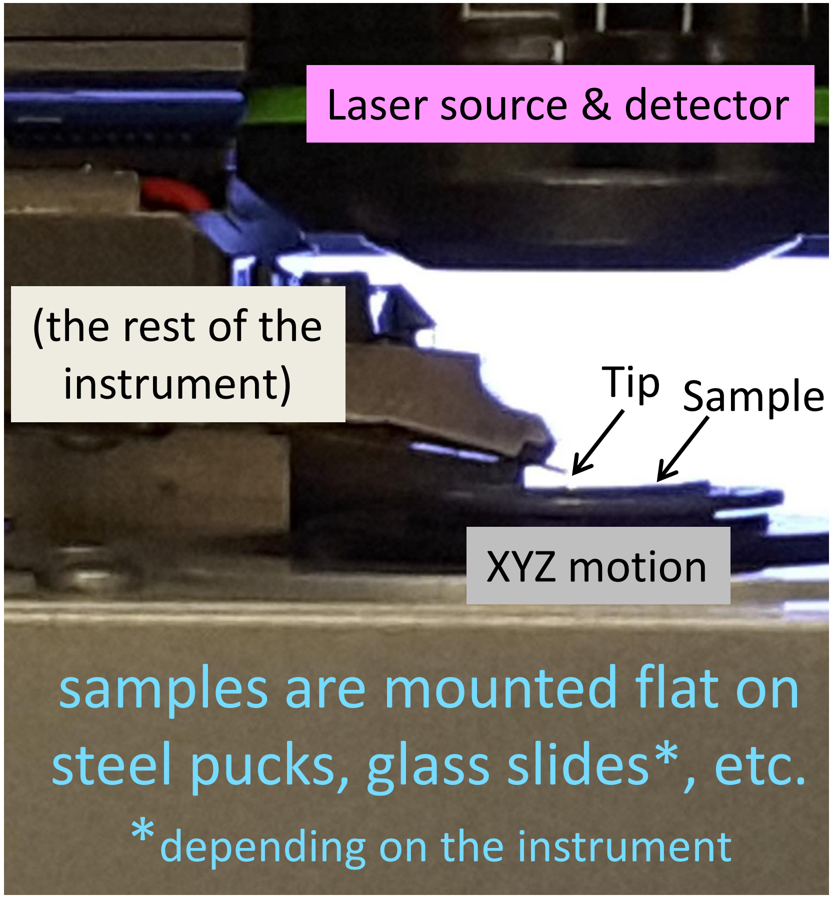
“How long does a tip last?”

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

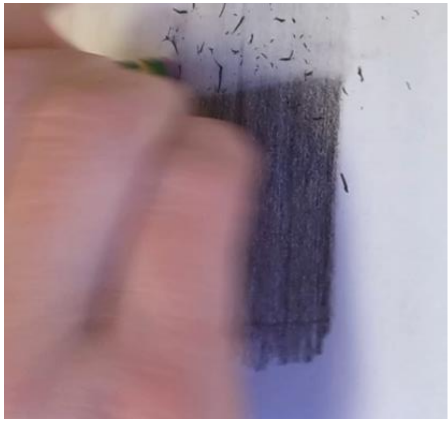
- When your tip goes bad, just throw it out!
- Generally come in 10-packs
  - 50-packs for frequent AFM’ers

The Process

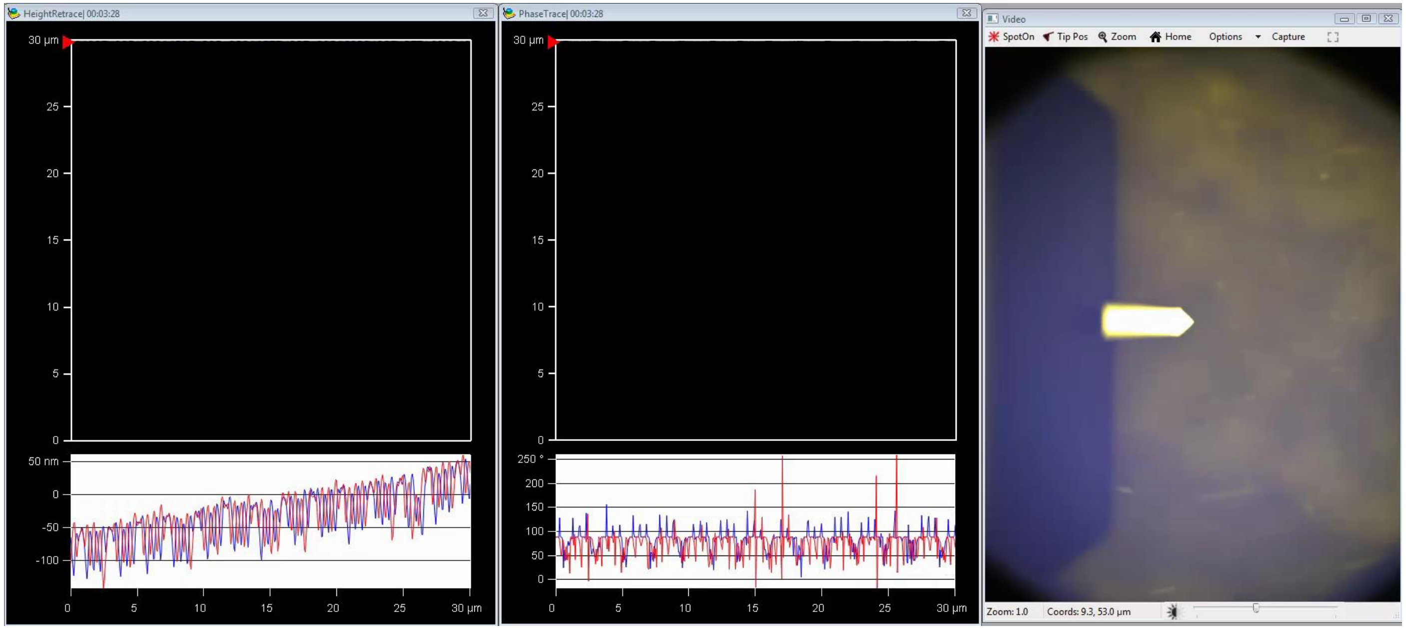
- Mount tip
- Mount sample
- Scan
- Process image
- Extract numbers (application-dependent)



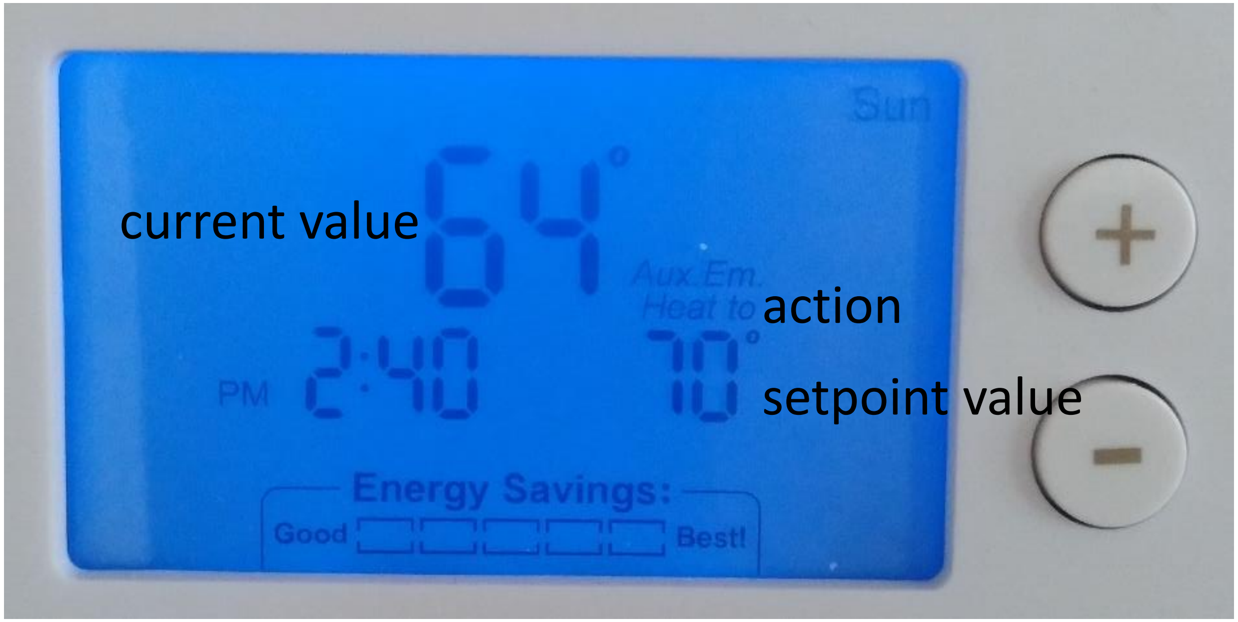
Raster Scanning



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

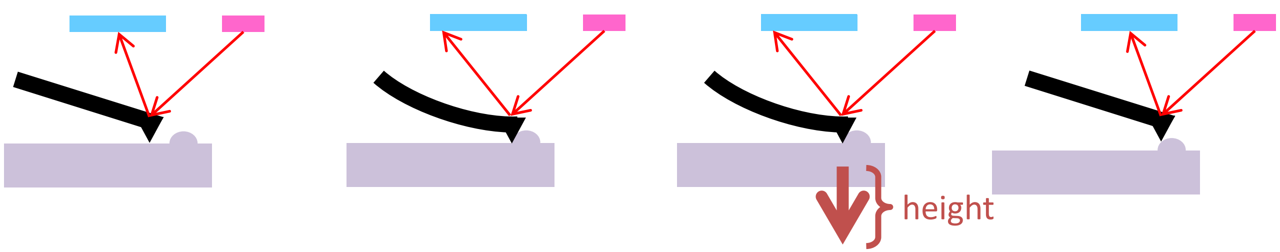


Feedback


Sun
current value
64°
Aux.Em.
Heat to
action
PM 2:40
70°
setpoint value
Energy Savings:
Good
Best!

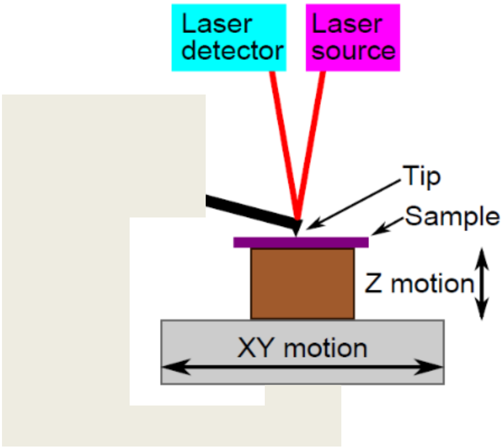
Feedback

- 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

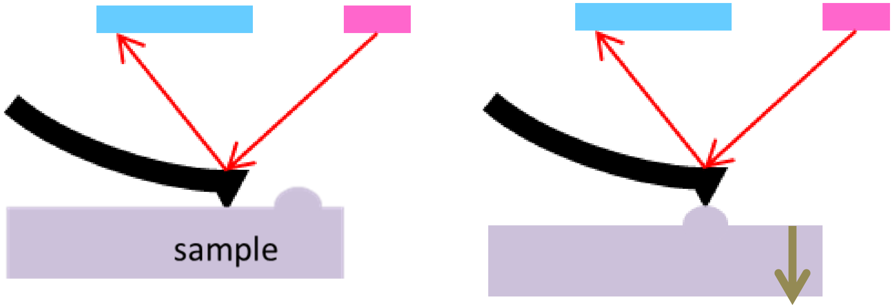


Contact Mode Imaging

- Drag tip along surface like a stylus profilometer (or like a record player)

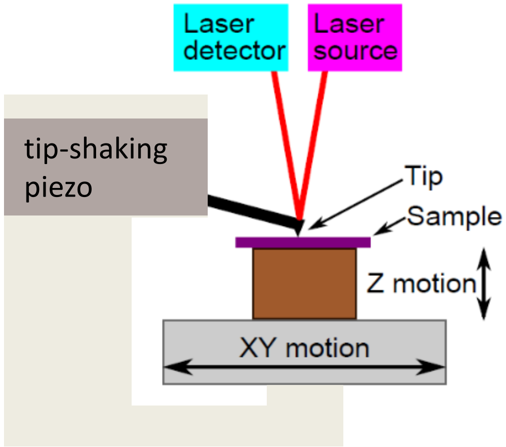


- Adjust tip—sample separation to keep cantilever deflection constant
  - Traces sample topography
  - Some AFMs move tip; some move sample

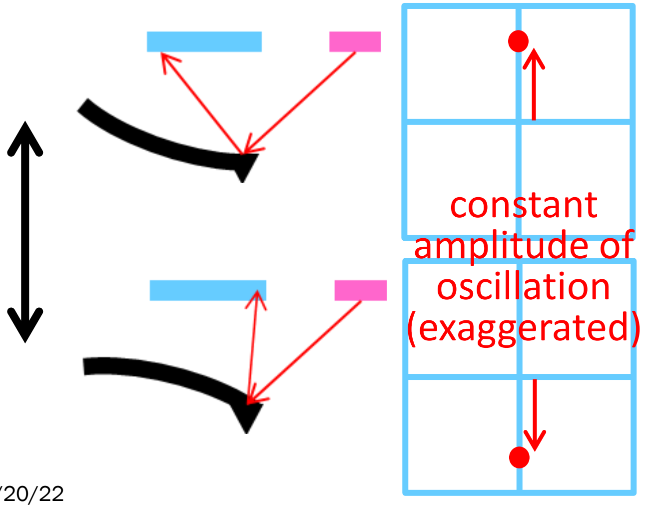


Tapping Mode Imaging

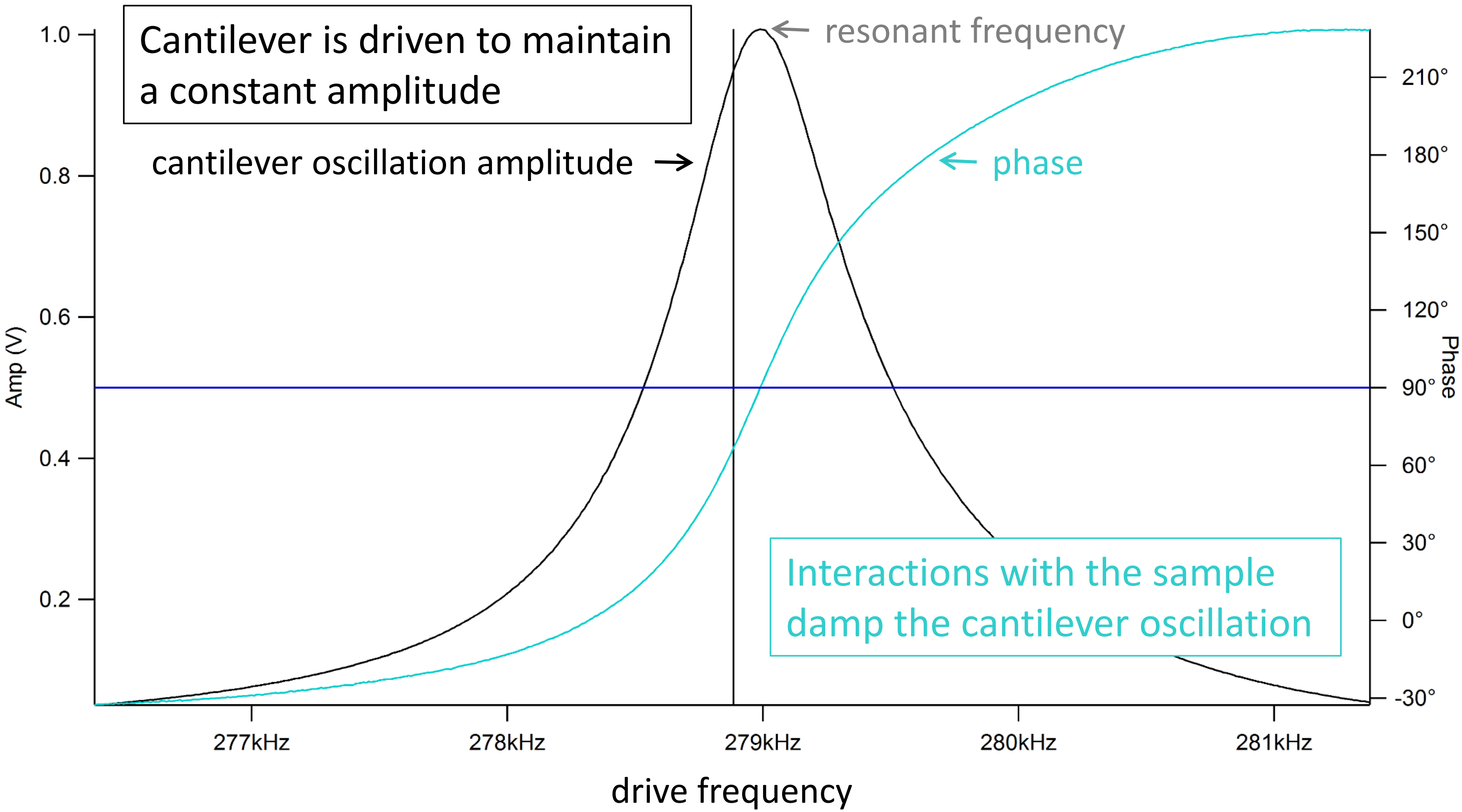
- Standard mode for AFM topography
- Intermittent contact, tapping, AC, amplitude modulation mode
- Not constantly in contact with the surface
- Driven, oscillating cantilever
- Tip—sample interactions affect oscillation



tip oscillates at tens of kHz to MHz

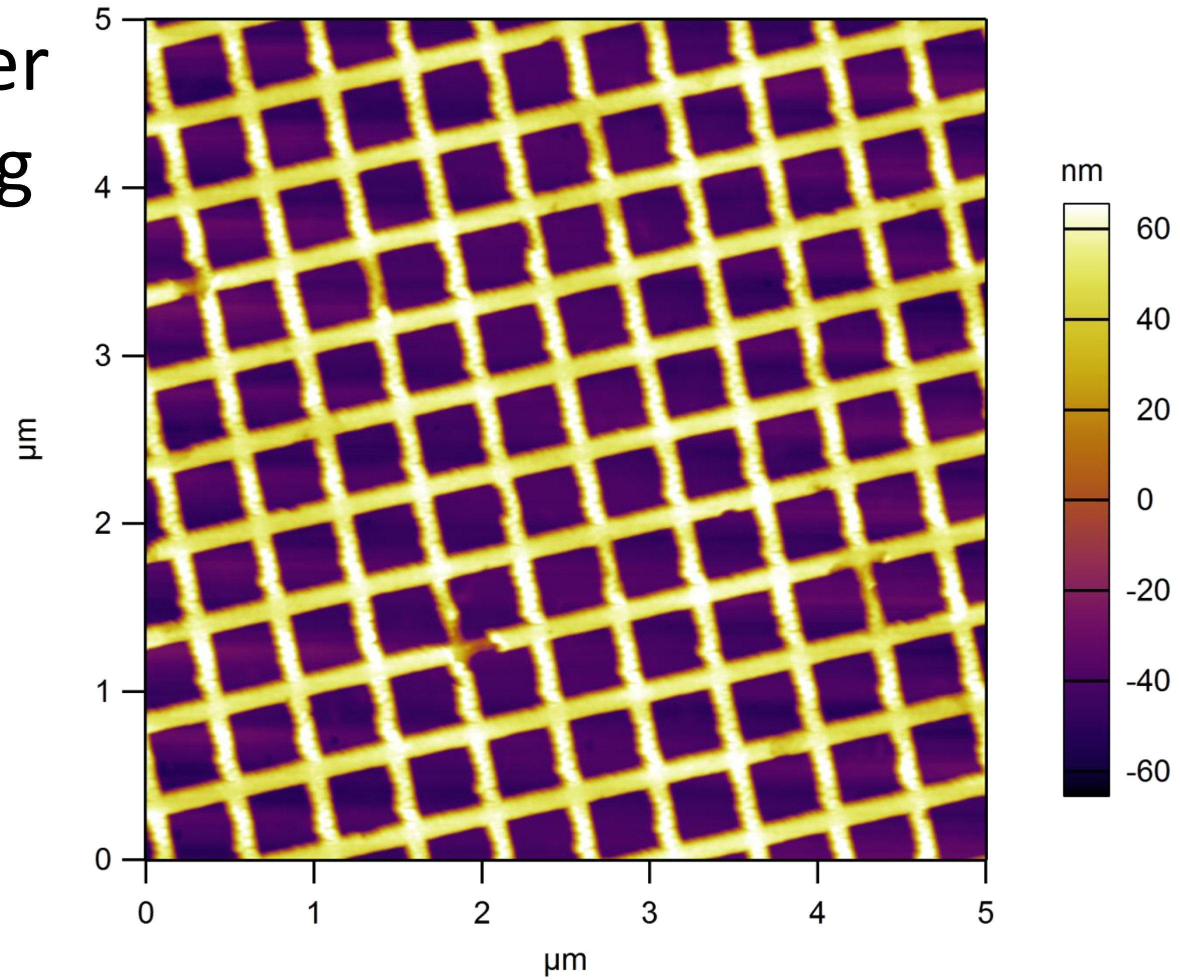


Tuning the Cantilever



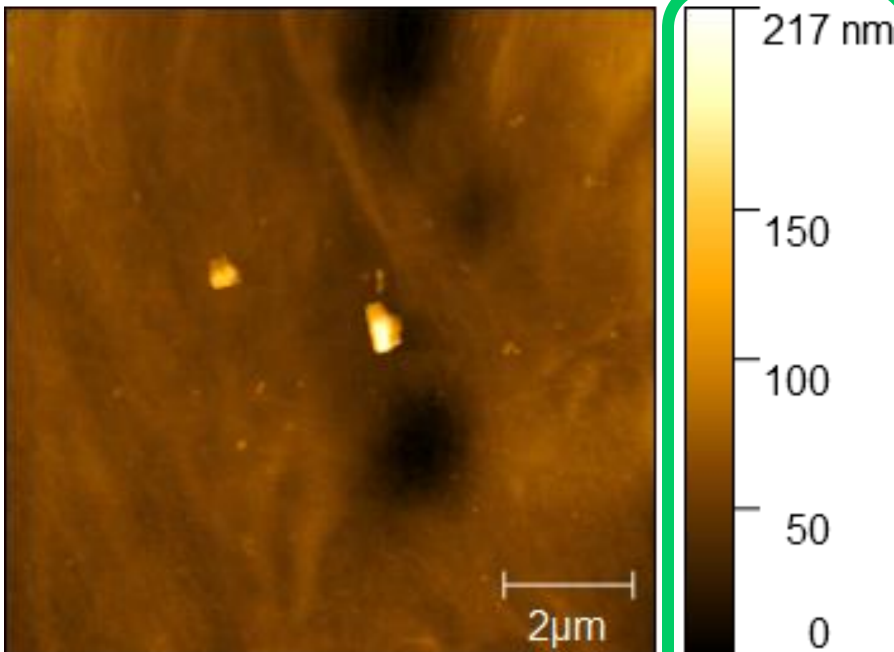
Application: Imaging

Polymer Grating



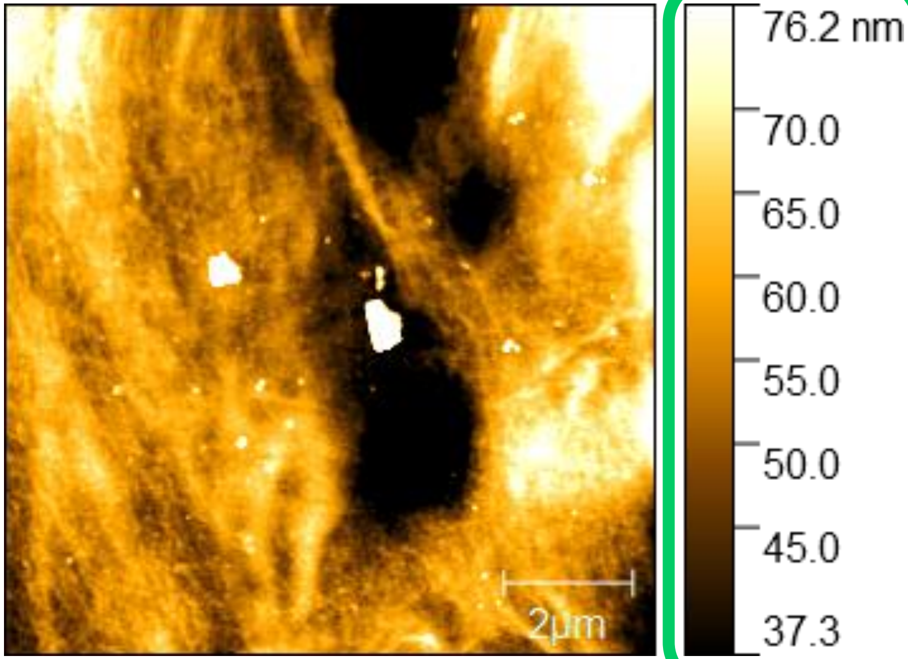
range of **colors, not heights** in the image

Reading the Colorscale



same image, different color ranges

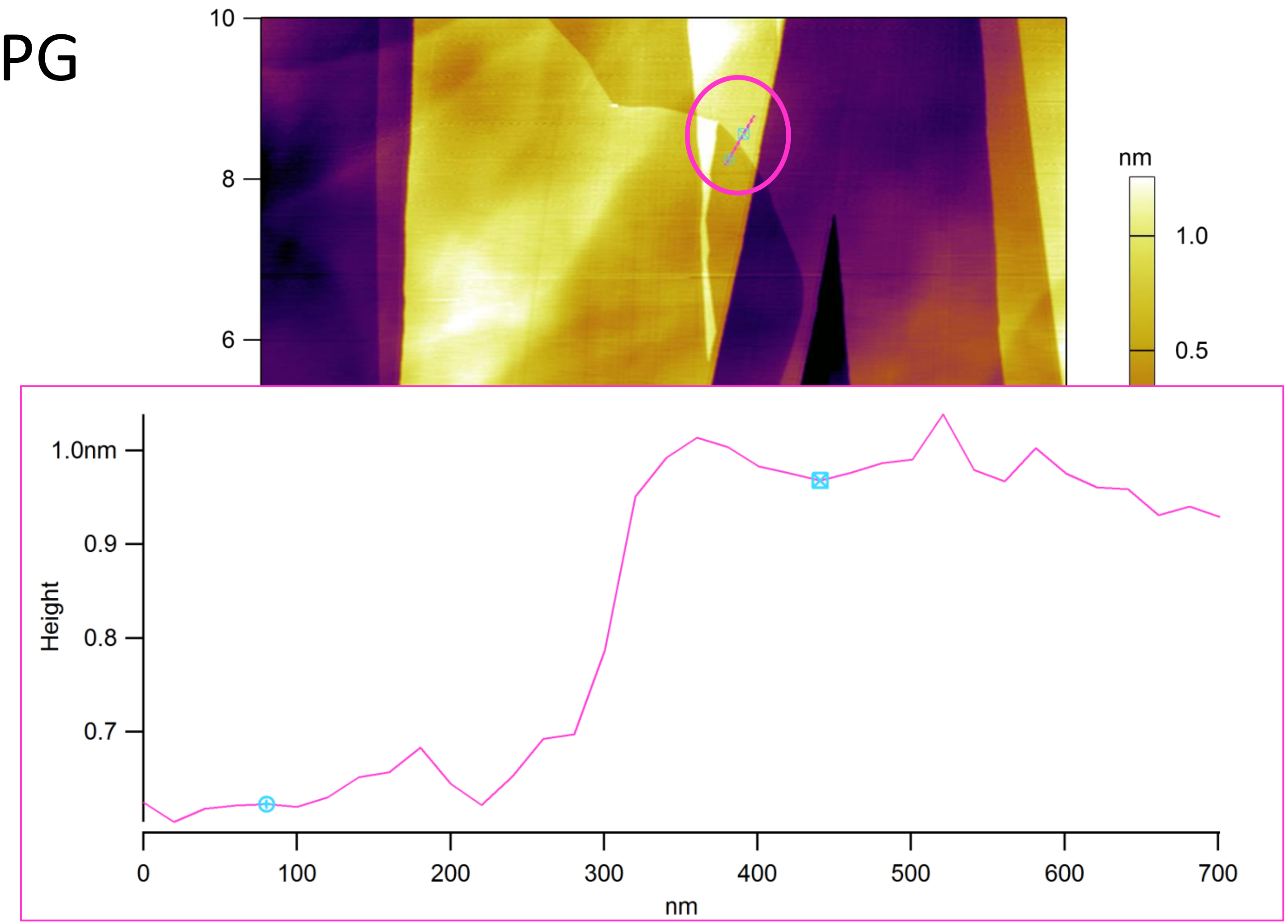
color range of the displayed image,
not necessarily all heights on the surface



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

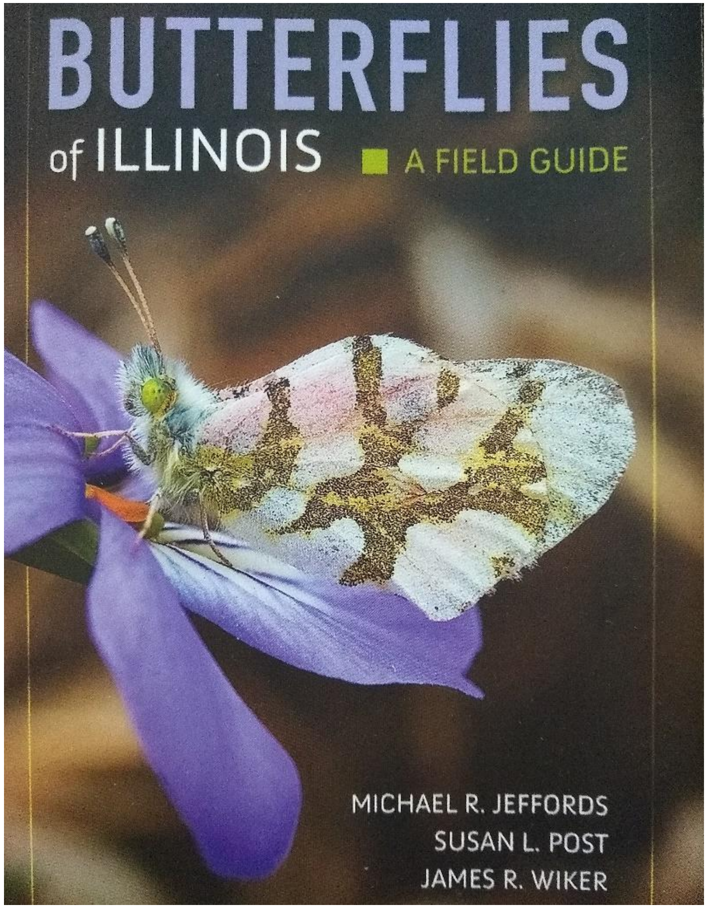
Application: Step Heights

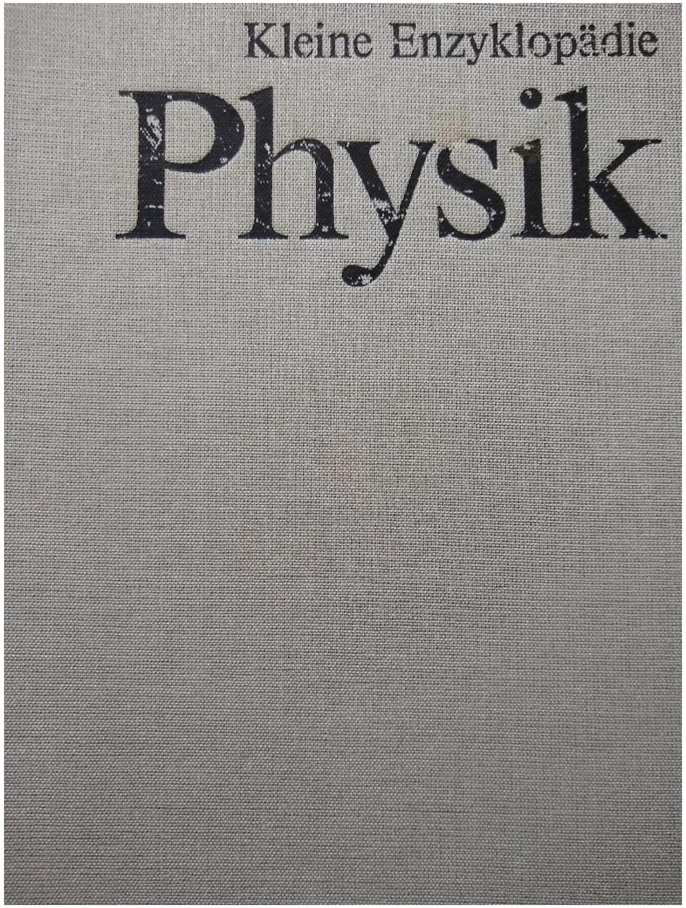
HOPG



Step Heights and Thicknesses

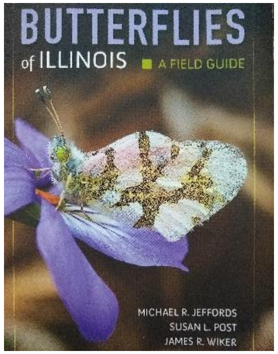
Which book is thicker?

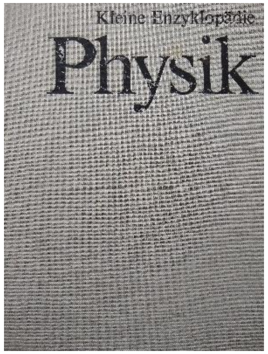




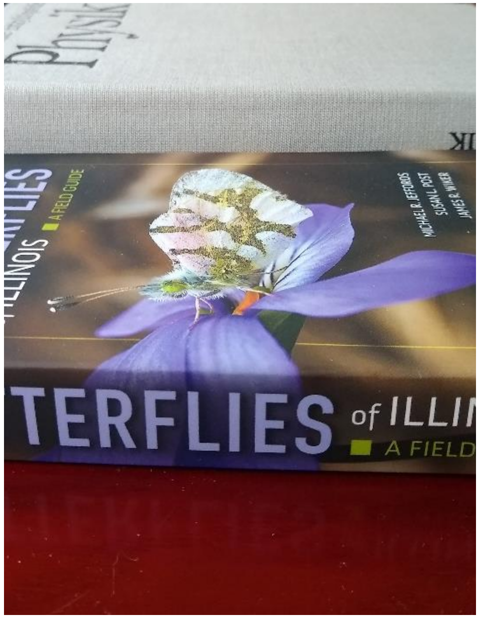
Step Height: Relative Height

- Film thickness is measured by step height



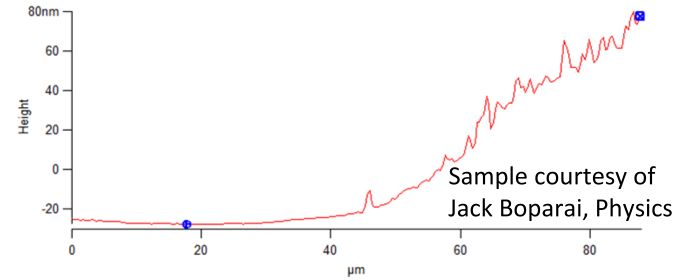


- Measure a height difference
  - Leave some bare substrate (patches are OK)
  - Scratch down to the substrate
  - Multilayer material—exposed underlayer



Step Height/Film Thickness: Complementary Techniques

If your step's too broad for the AFM (edge width >~80um), try…



- Stylus profilometry
- 3D optical profilometry

Need a height difference (step) like AFM

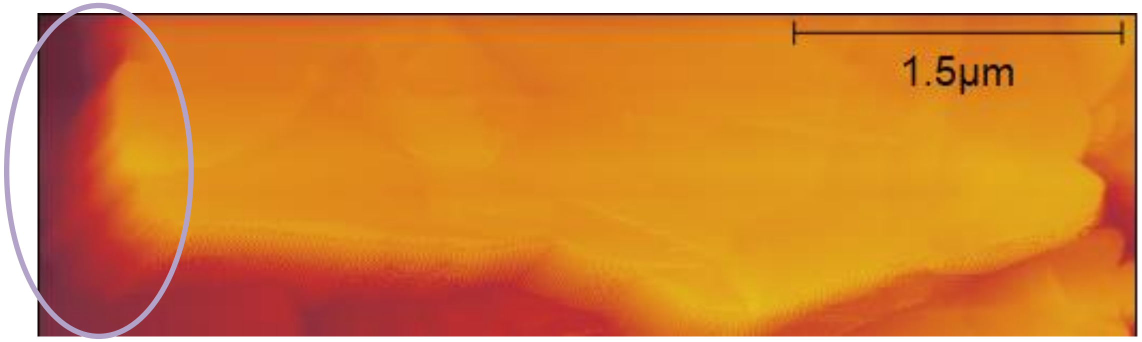
- X-ray Reflectivity (XRR)
- X-ray Fluorescence (XRF)
- Rutherford Backscattering Spectrometry (RBS)

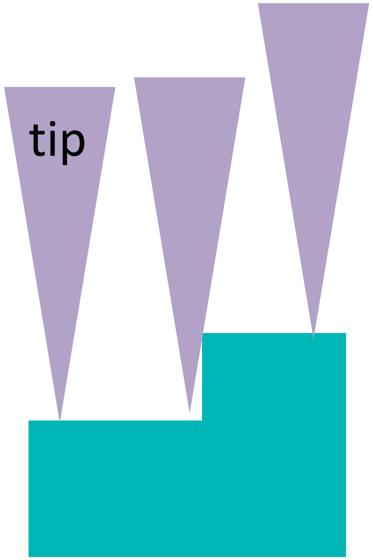
Continuous film (no steps)
May need to know density

Width Measurements

Beware of tip shape convolution

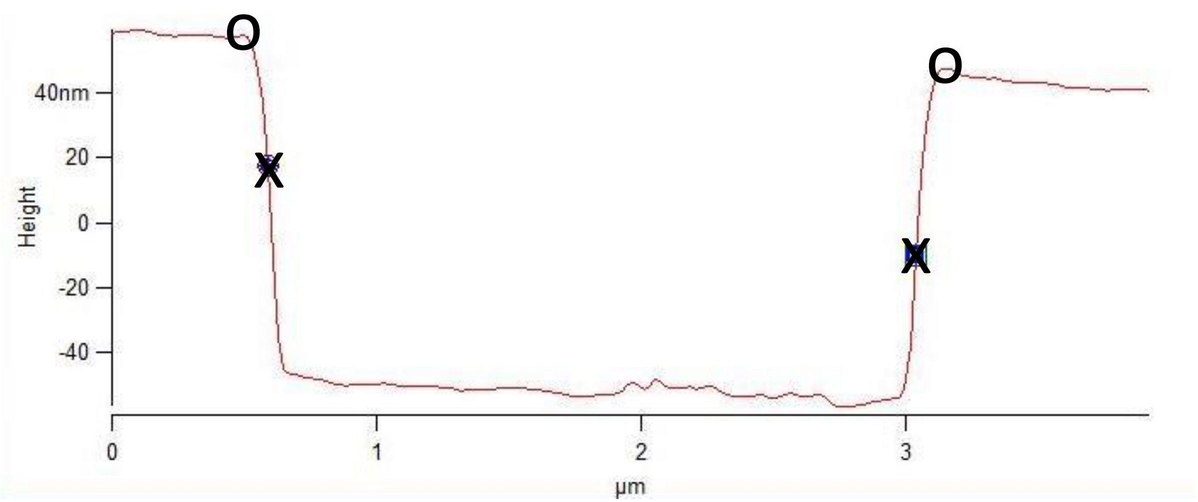
- As depth increases, tips get broader
- Steep drop-offs look less sharp
- High aspect ratio tips are available

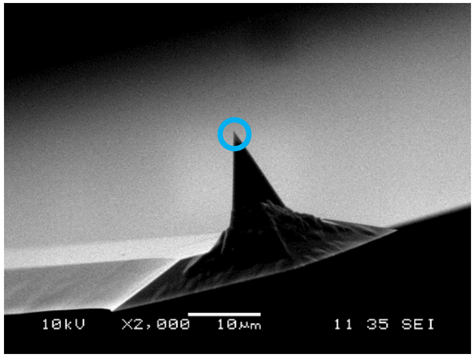


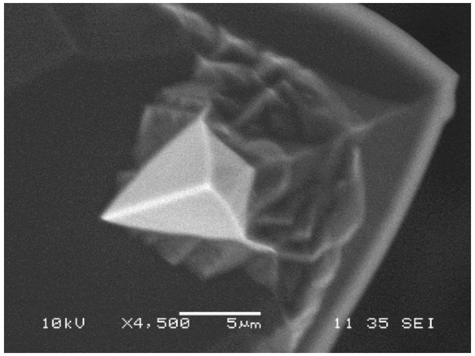


Width Measurements

- 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?

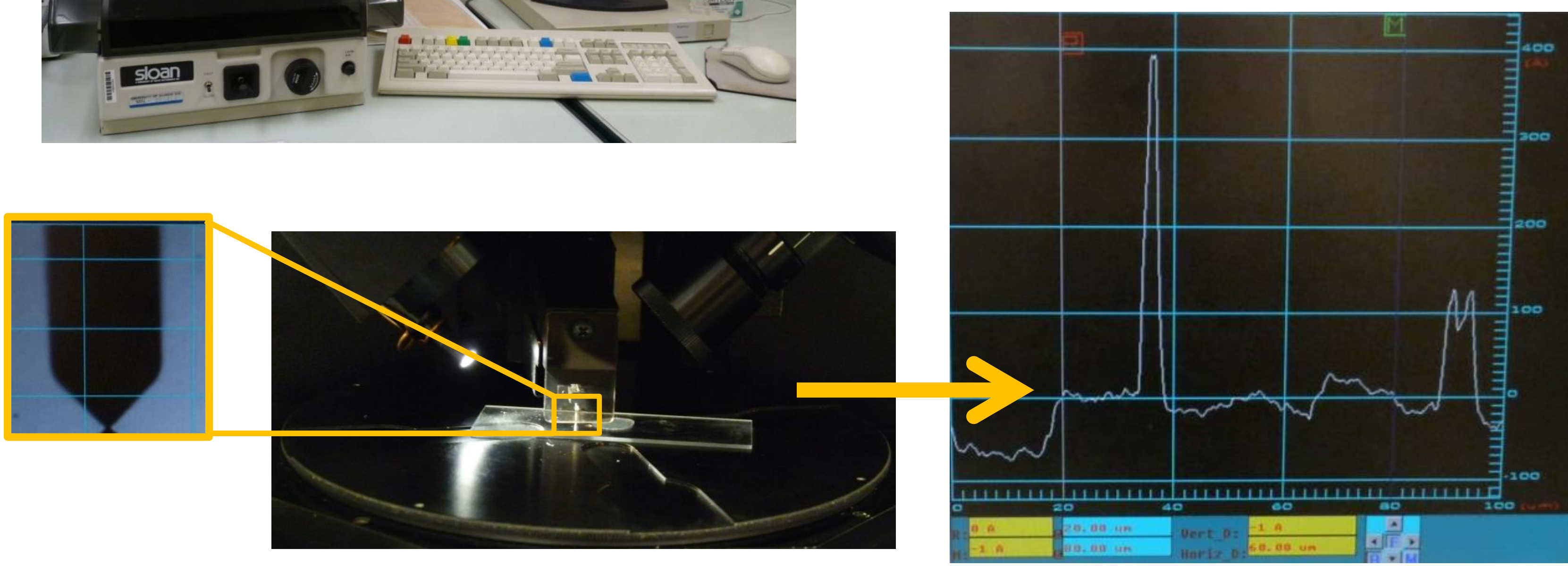
Michael Jeffords and Susan Post, University of Illinois Prairie Research Institute
https://photojournalingm-s.smugmug.com/Colorado-and-Kansas/i-3tJ3DZk/A

Complementary: Stylus Profilometry

2D stylus profilometry

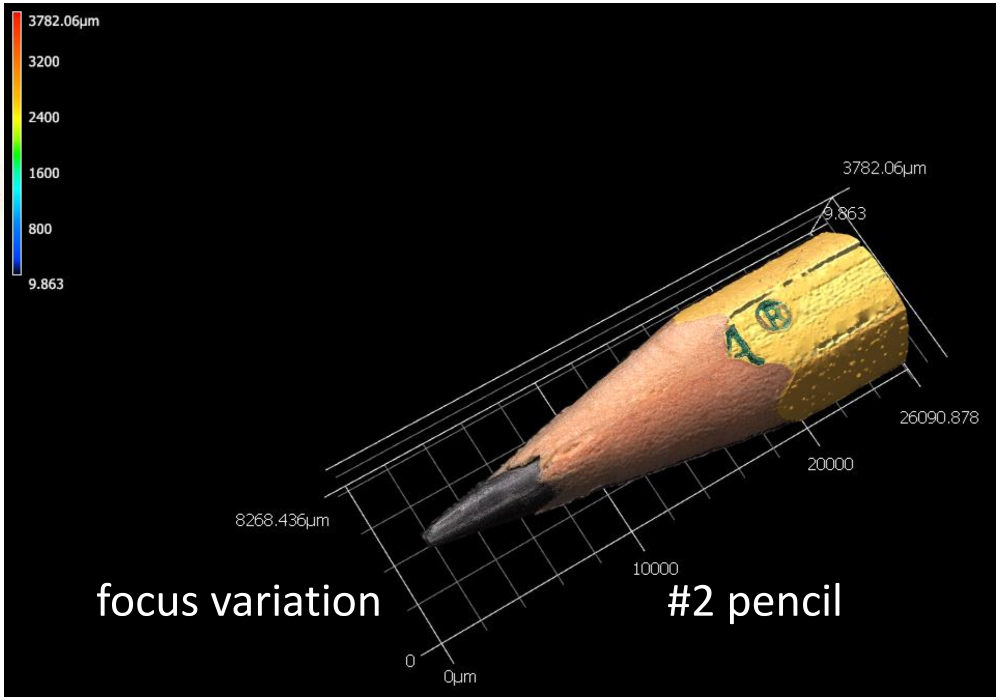
(line profiles)

(diamond tip)



Complementary: Optical Profilometry

go.illinois.edu/MRL3DOpticalProfilometry



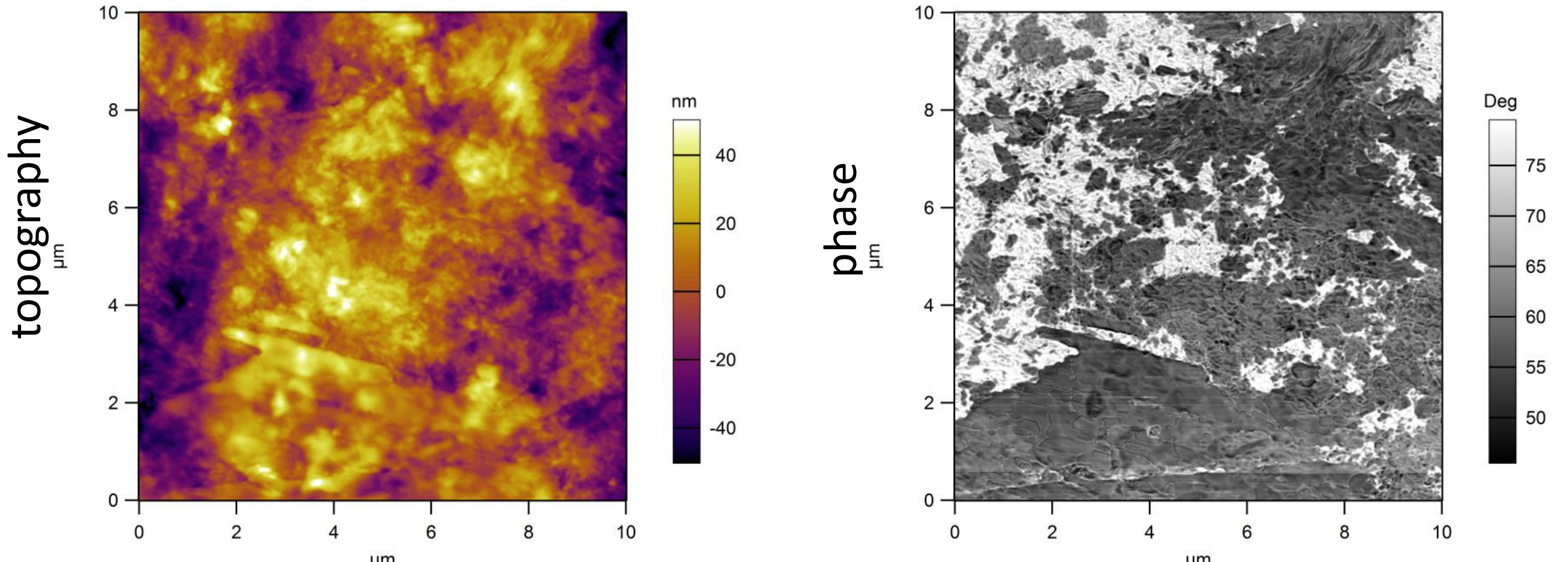
Qualitative Comparison

	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)

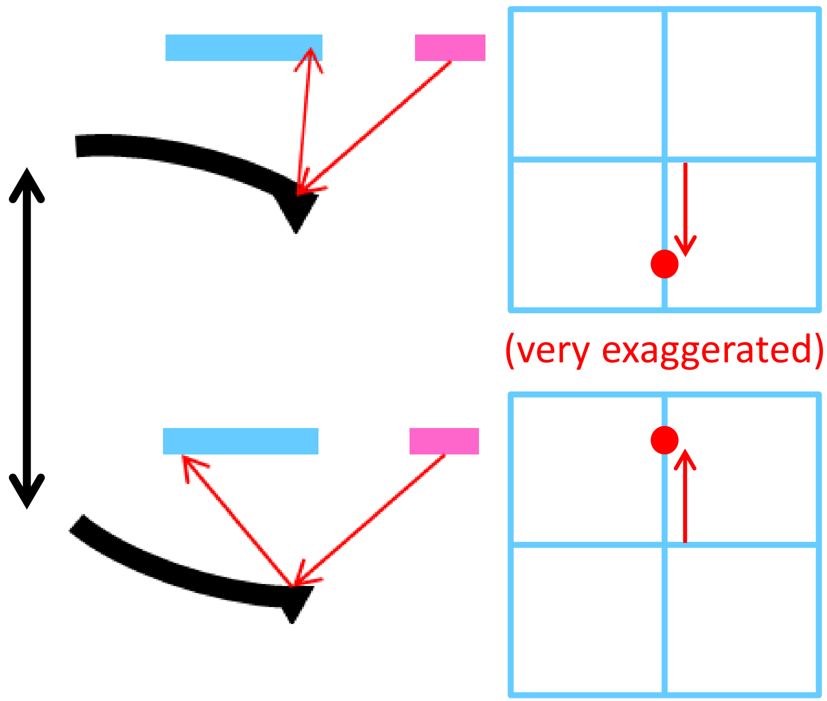


BOPP/PE blend (toothbrush wrapper)

Tapping Mode Imaging: Phase

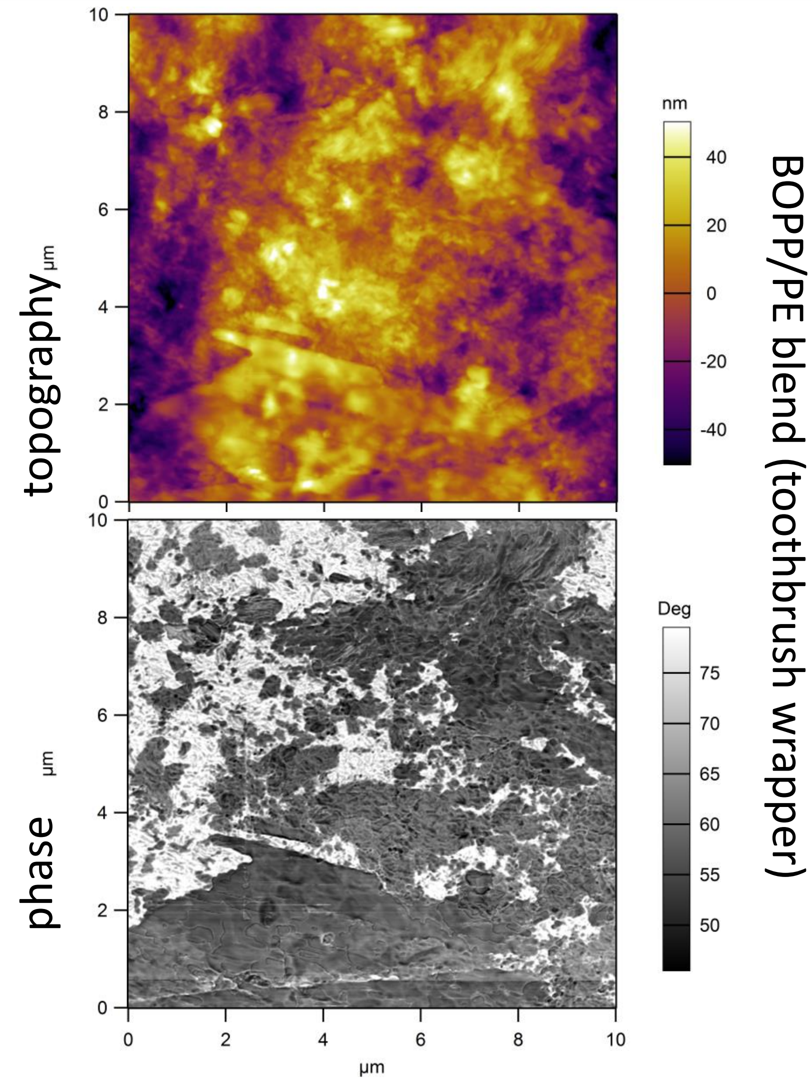
- Oscillating cantilever
- Tip—surface interactions affect oscillation
  - Cantilever driven to keep a constant amplitude
  - Dissipative interactions cause a phase lag (delay)
    - Viscous areas
    - Sticky areas

tip oscillates really fast
(tens of kHz to MHz)

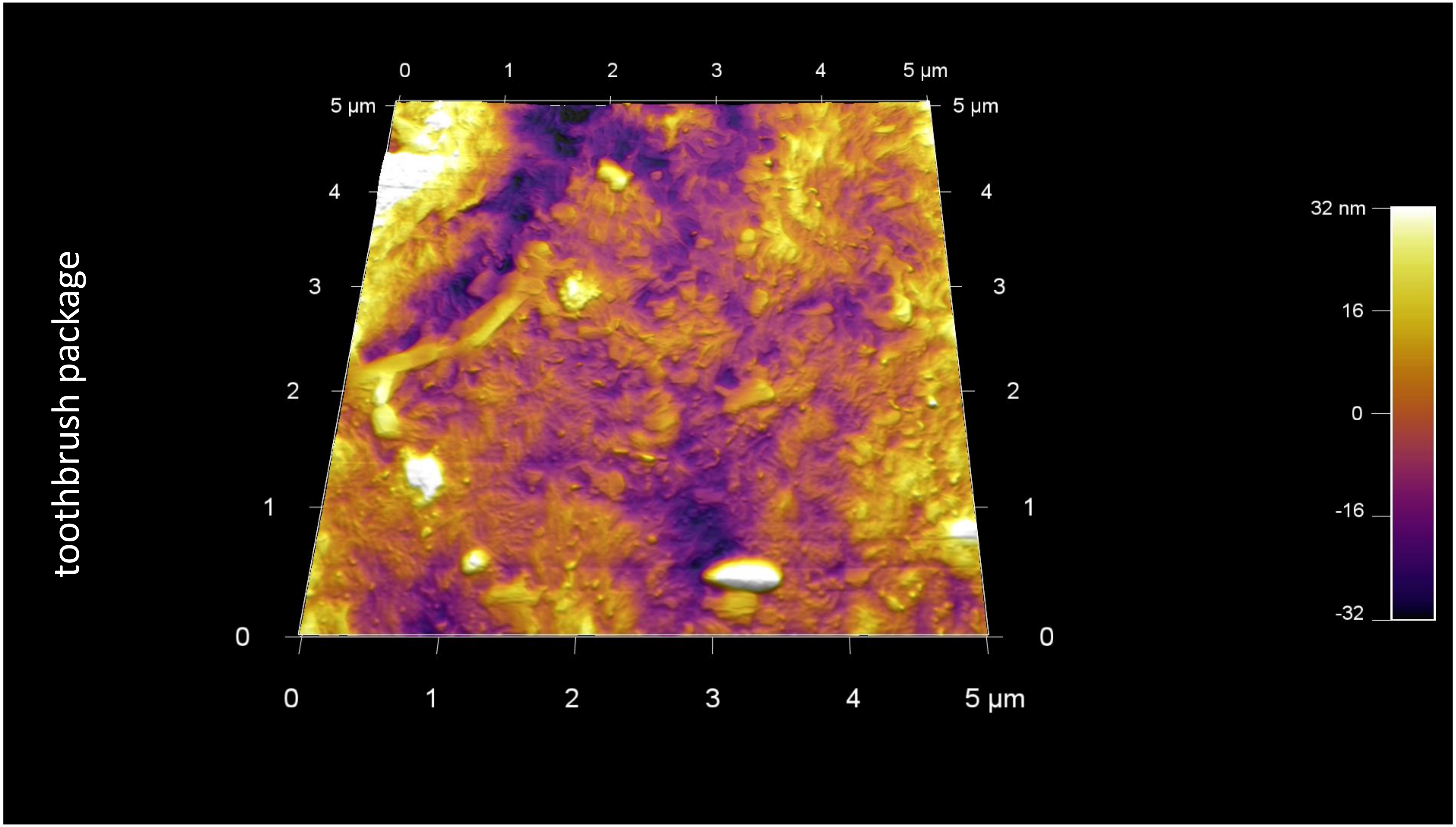


Phase (Qualitative)

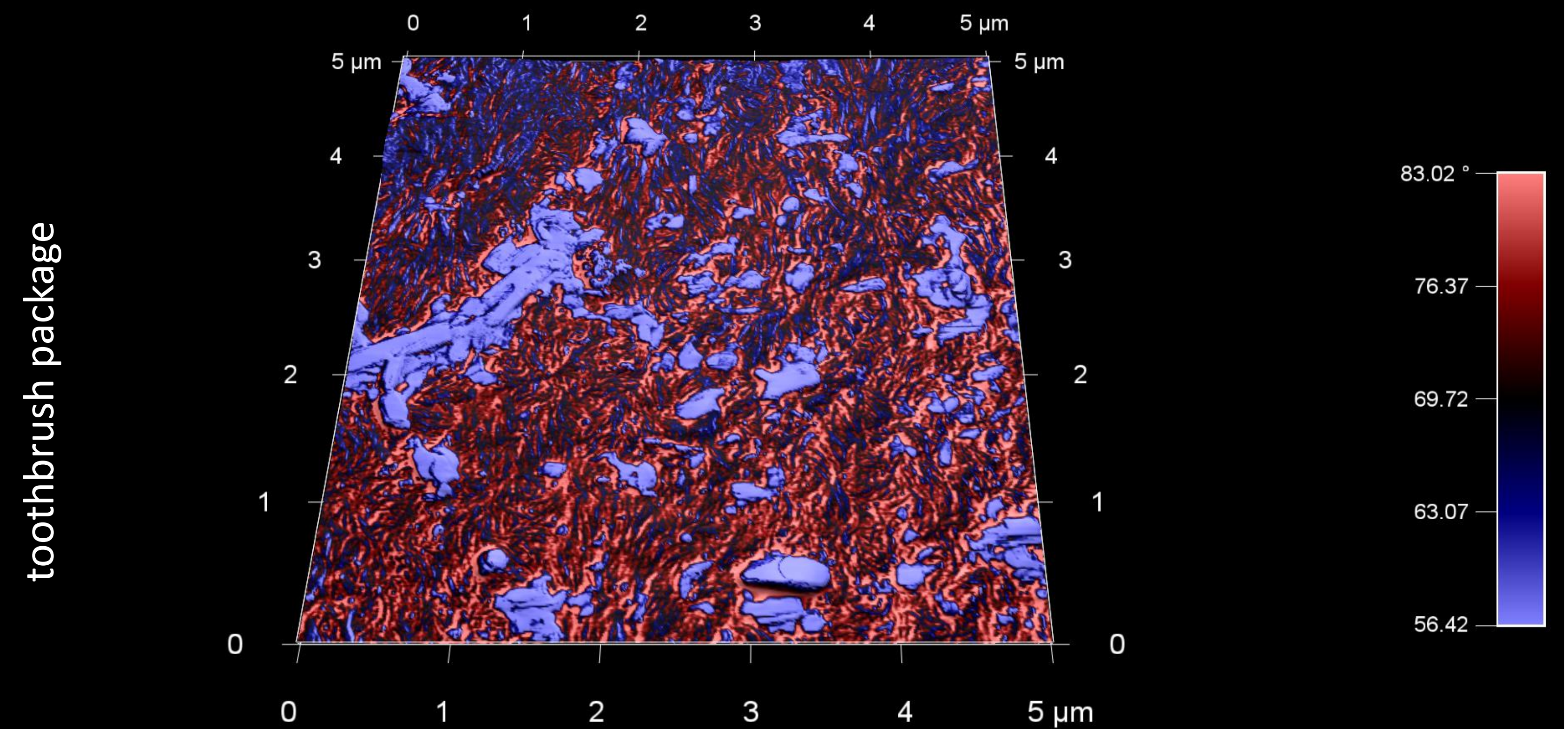
- Tapping mode imaging
- Contrast in phase image shows differences in mechanical properties
  - Qualitative, not quantitative
  - Great for mixtures
  - Great for soft materials deposited on hard surfaces



Topography

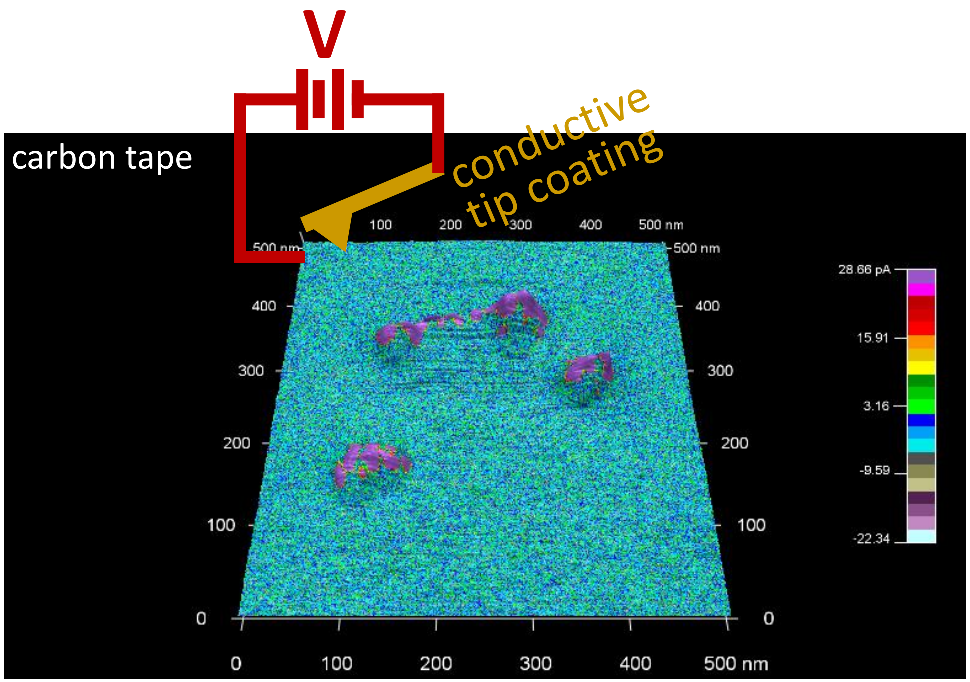


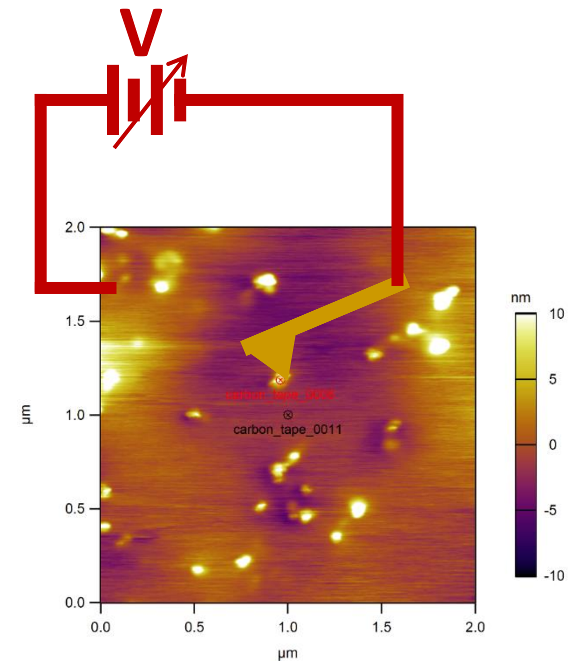
Topography with Colors from Phase

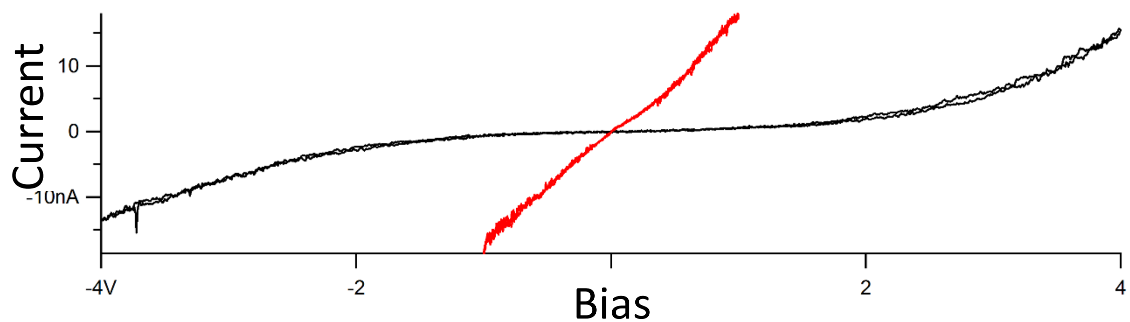


phase colorscale overlaid on 3D topography
redder areas are more dissipative

Application: Conductive AFM

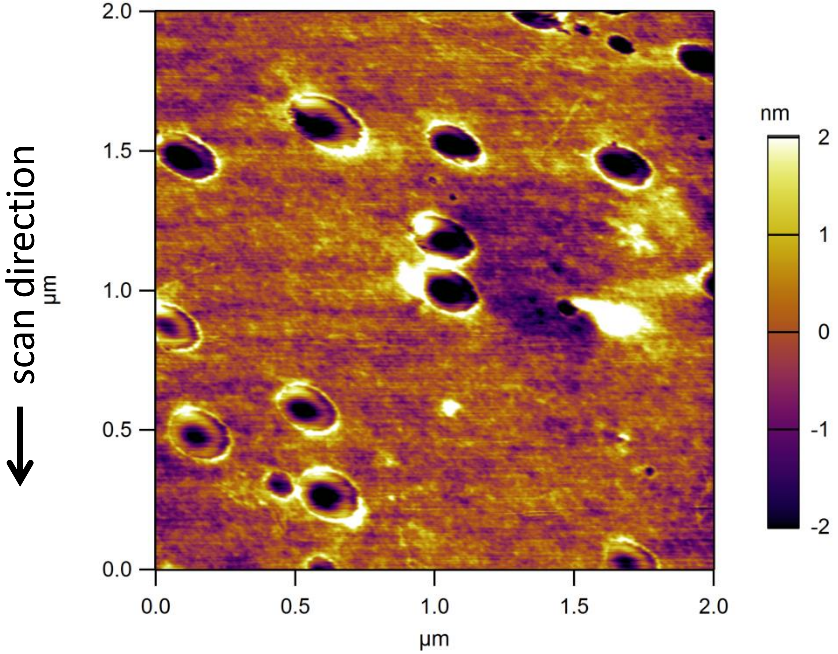




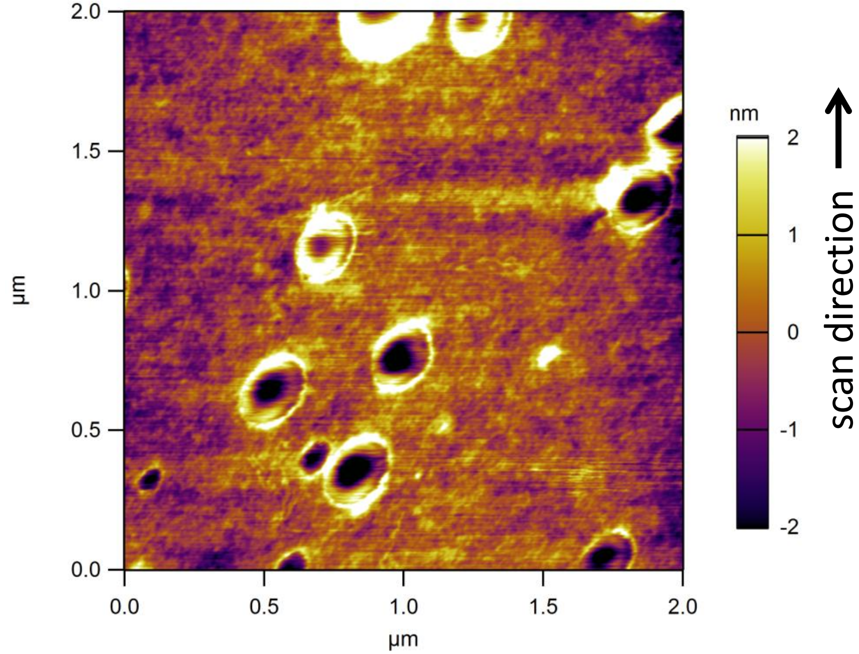


Sample Drift

Scanning downwards…



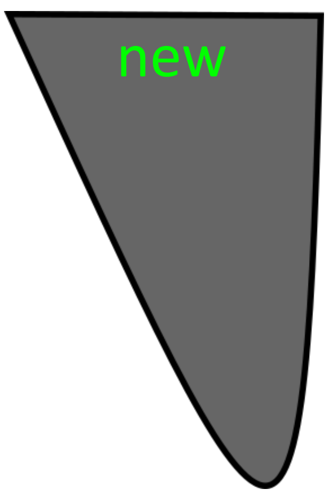
… then scanning upwards

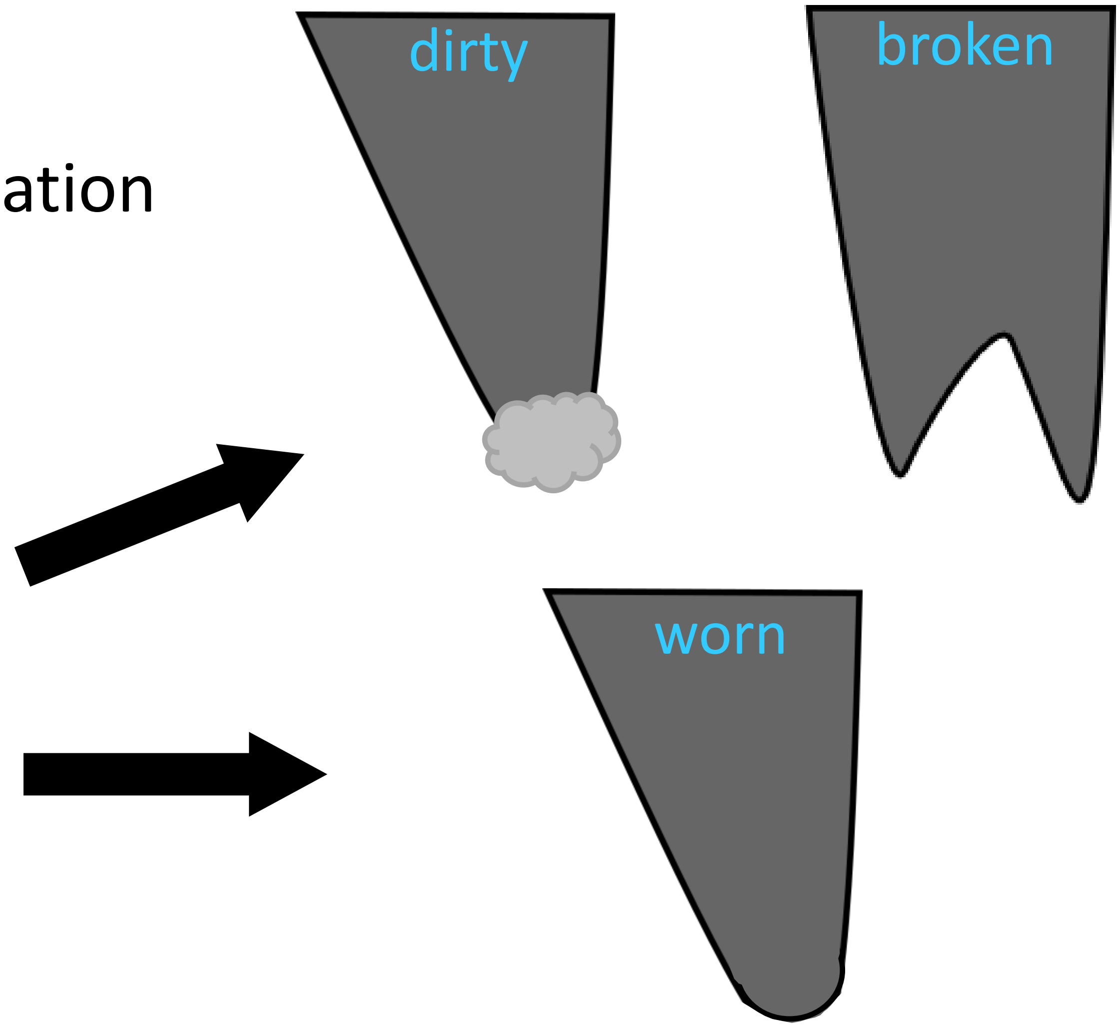


chewing gum

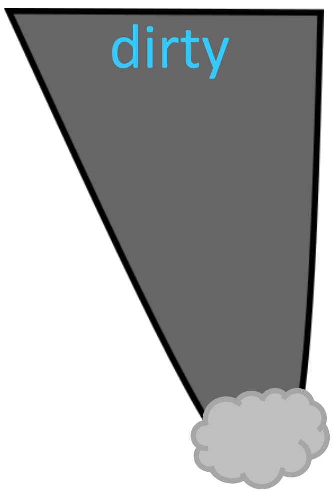
Tip Artifacts

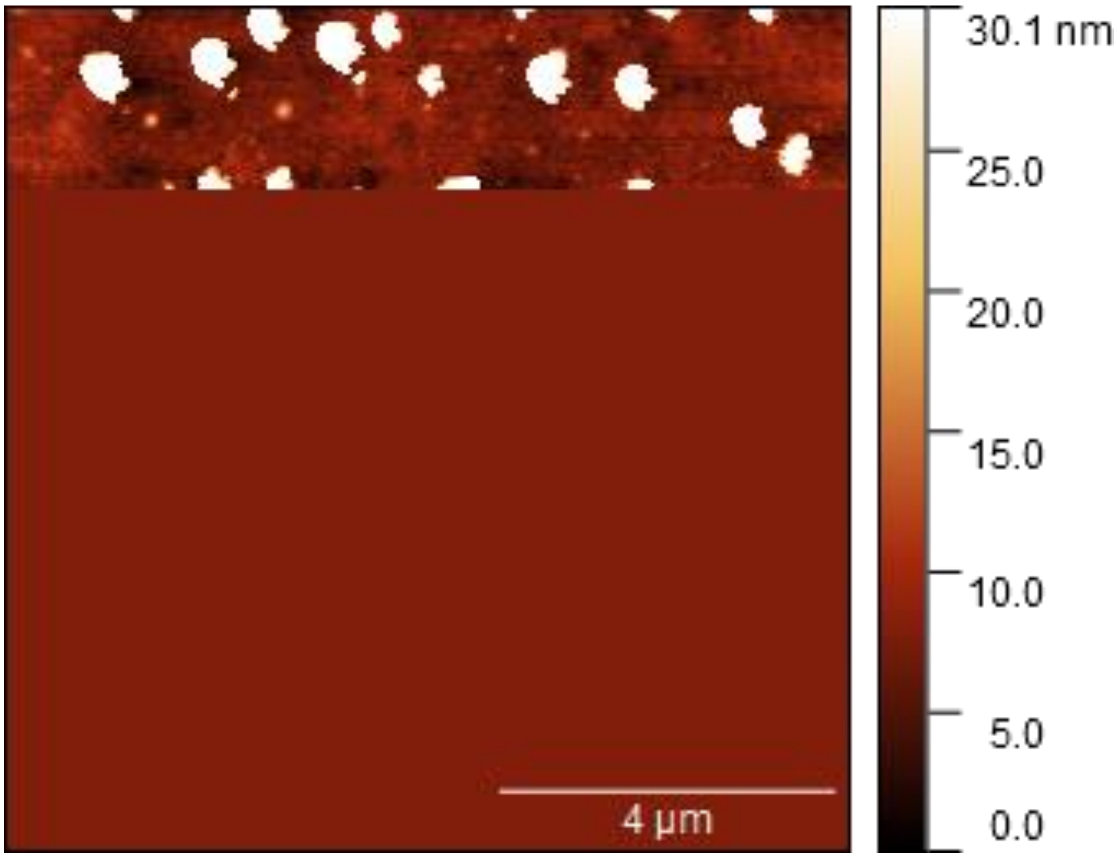
- Multiple tip
  - Tip contamination
  - Tip breaking
- Tip wear





Contaminated Tip

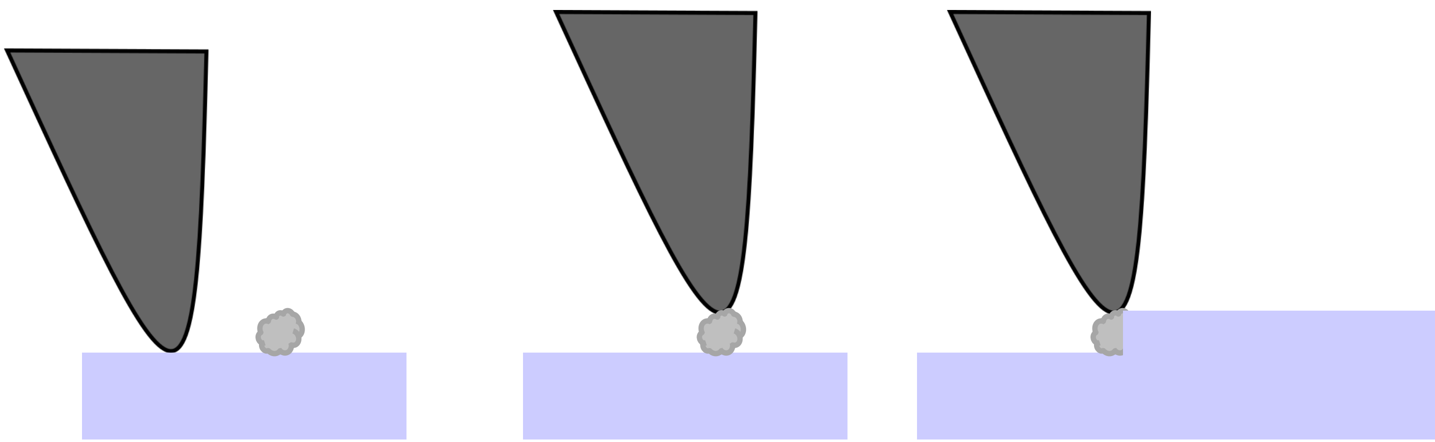




10μm partial scan

Line-by-Line Background Subtraction

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



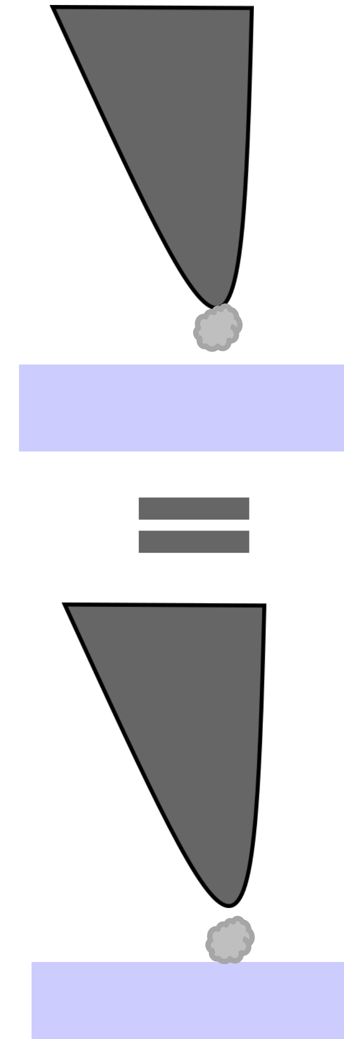


Image Processing

raw image

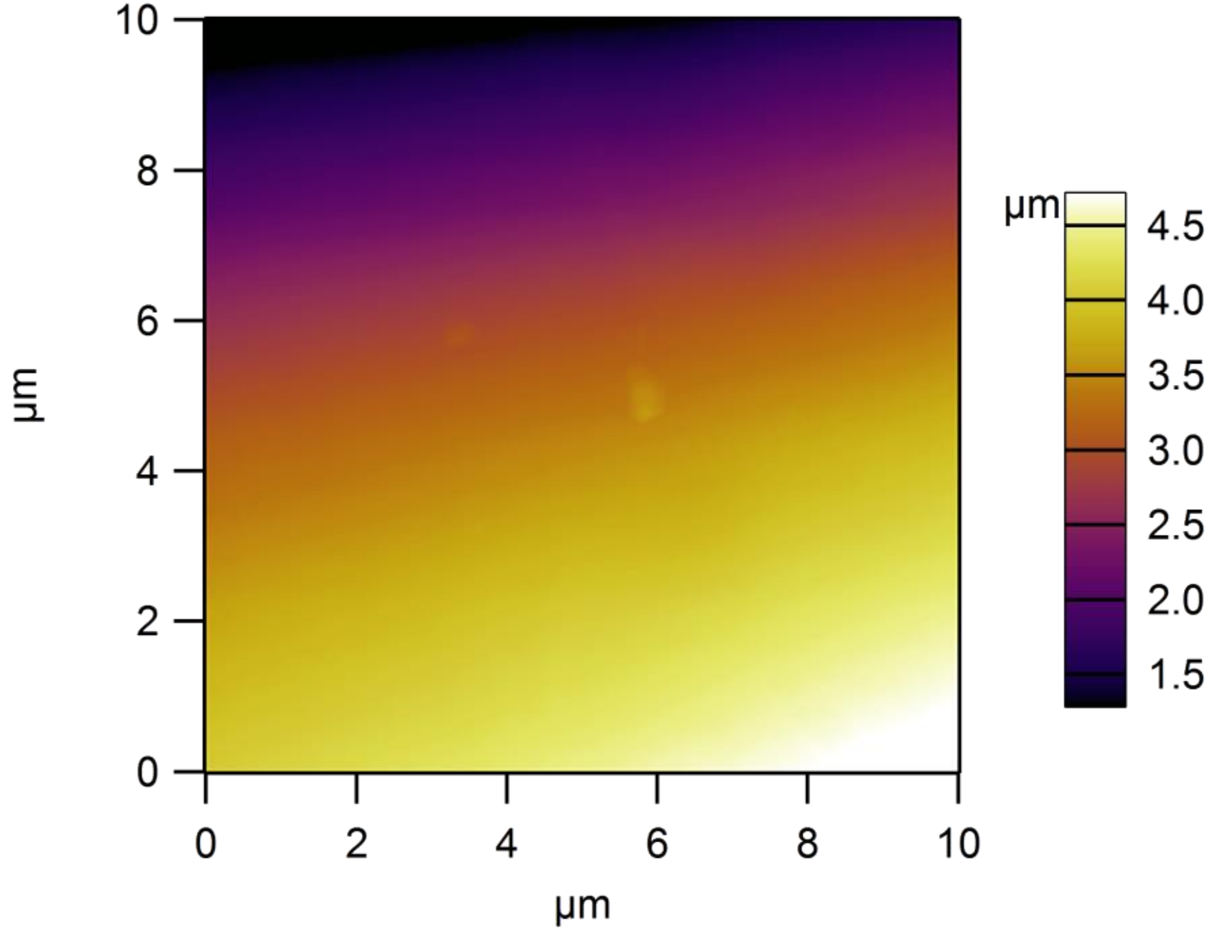


Image Processing

line subtraction

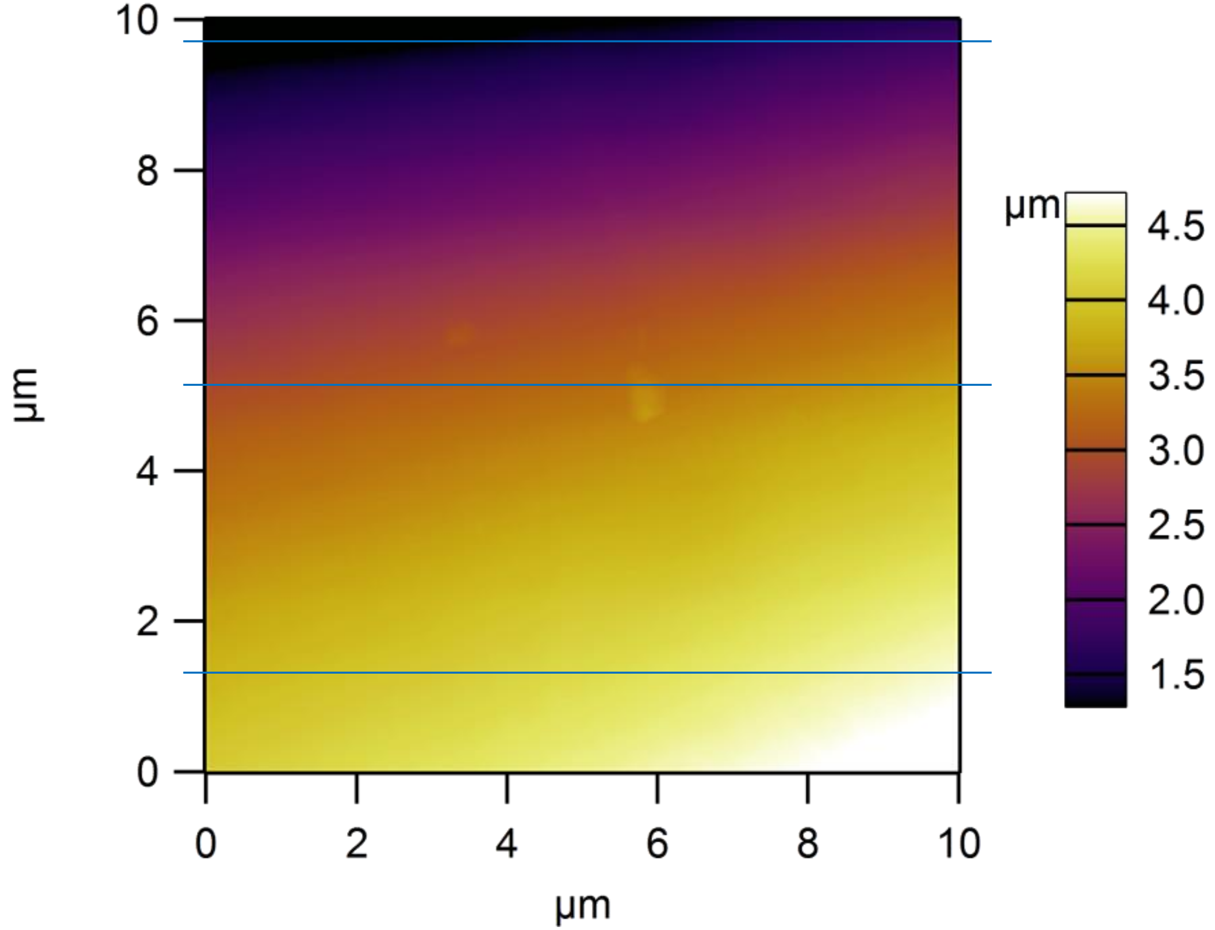


Image Processing

line subtraction

artifactually
lowered lines

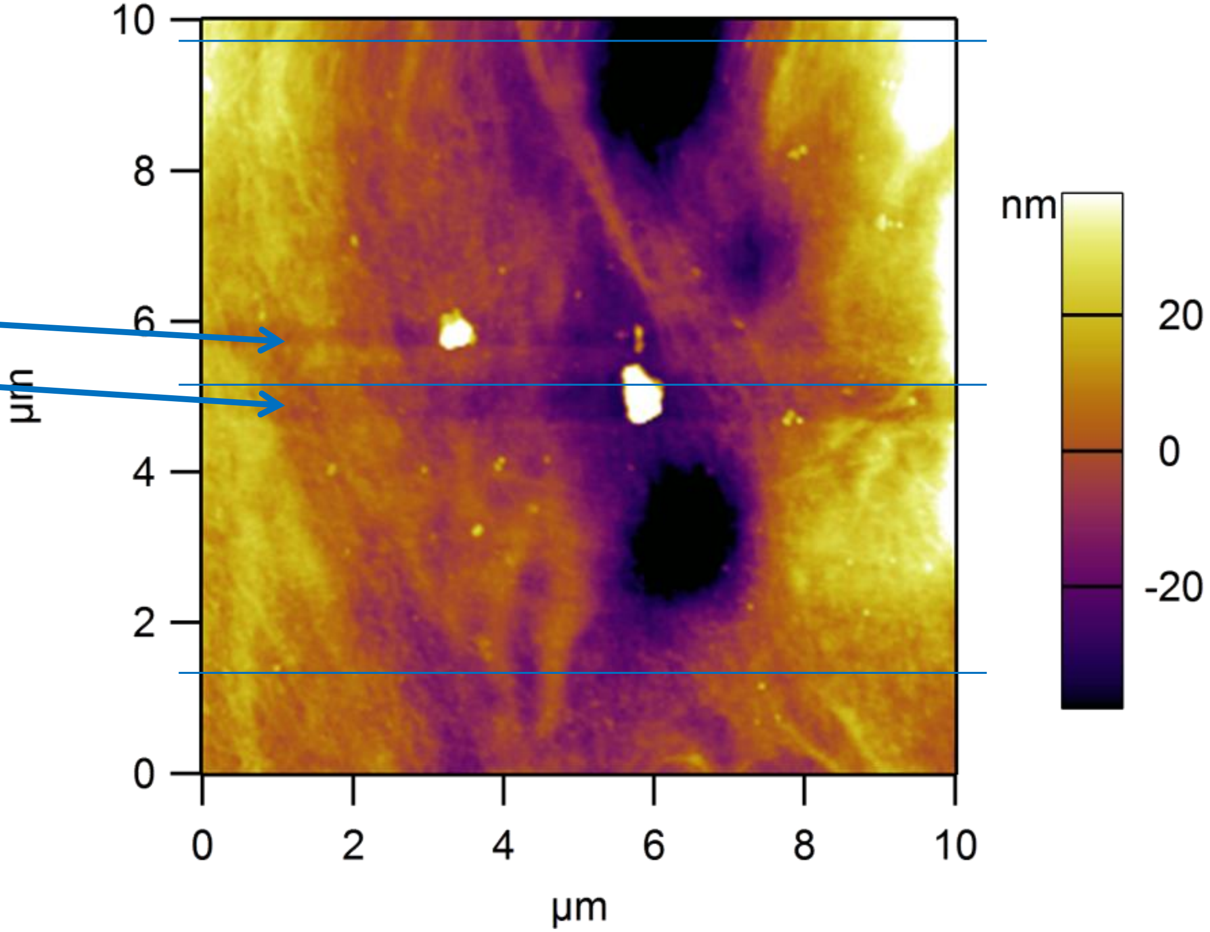


Image Processing

line subtraction:
mask outlier areas

areas to ignore
when processing

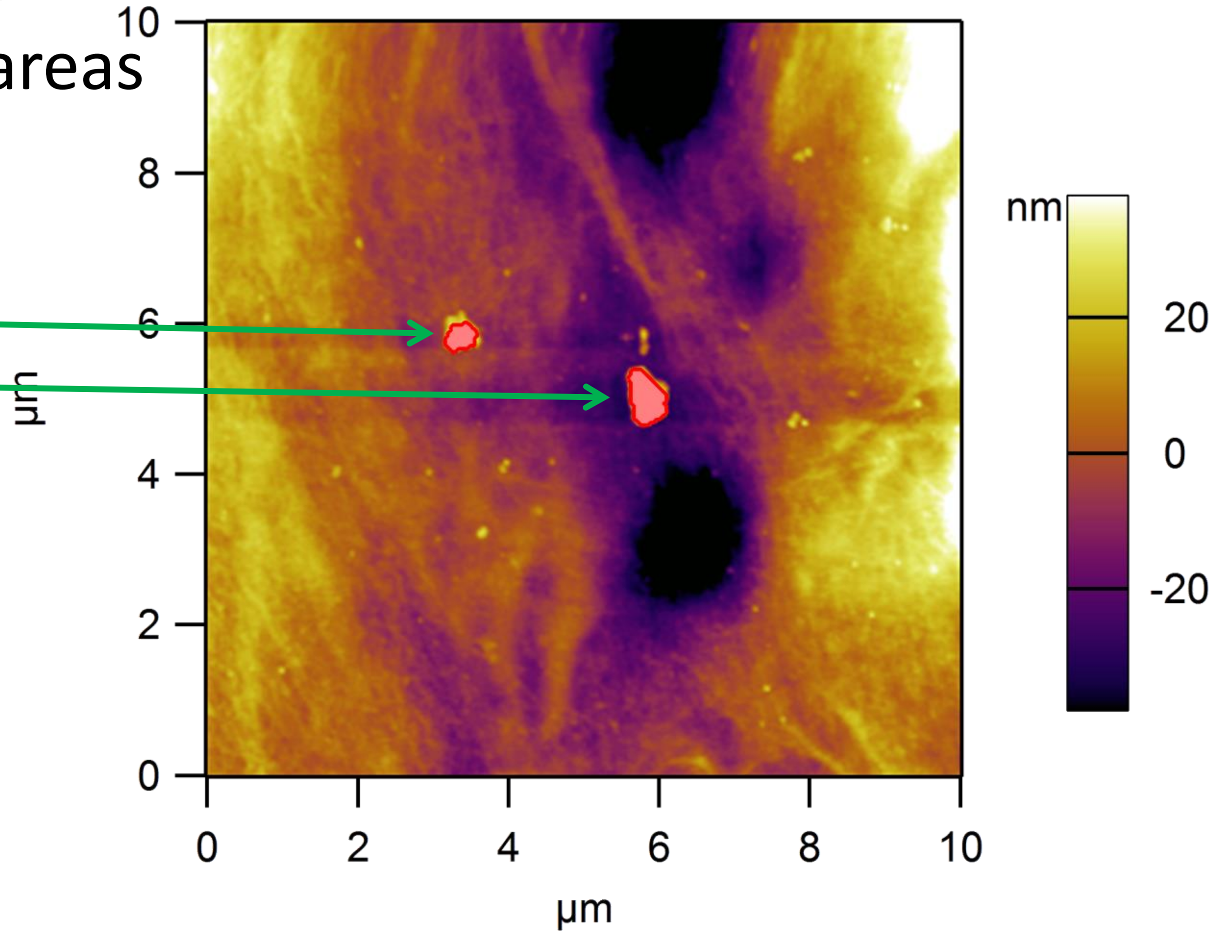
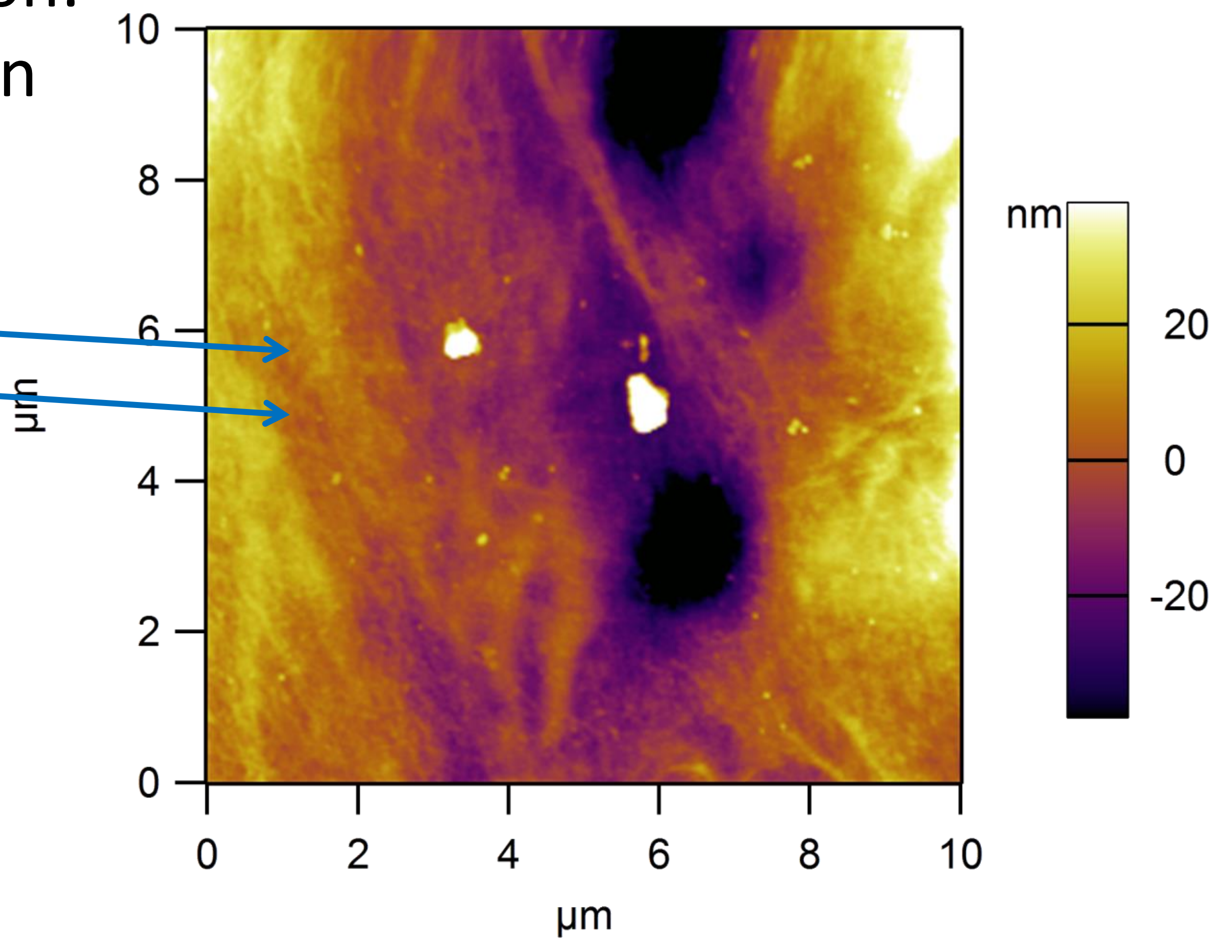


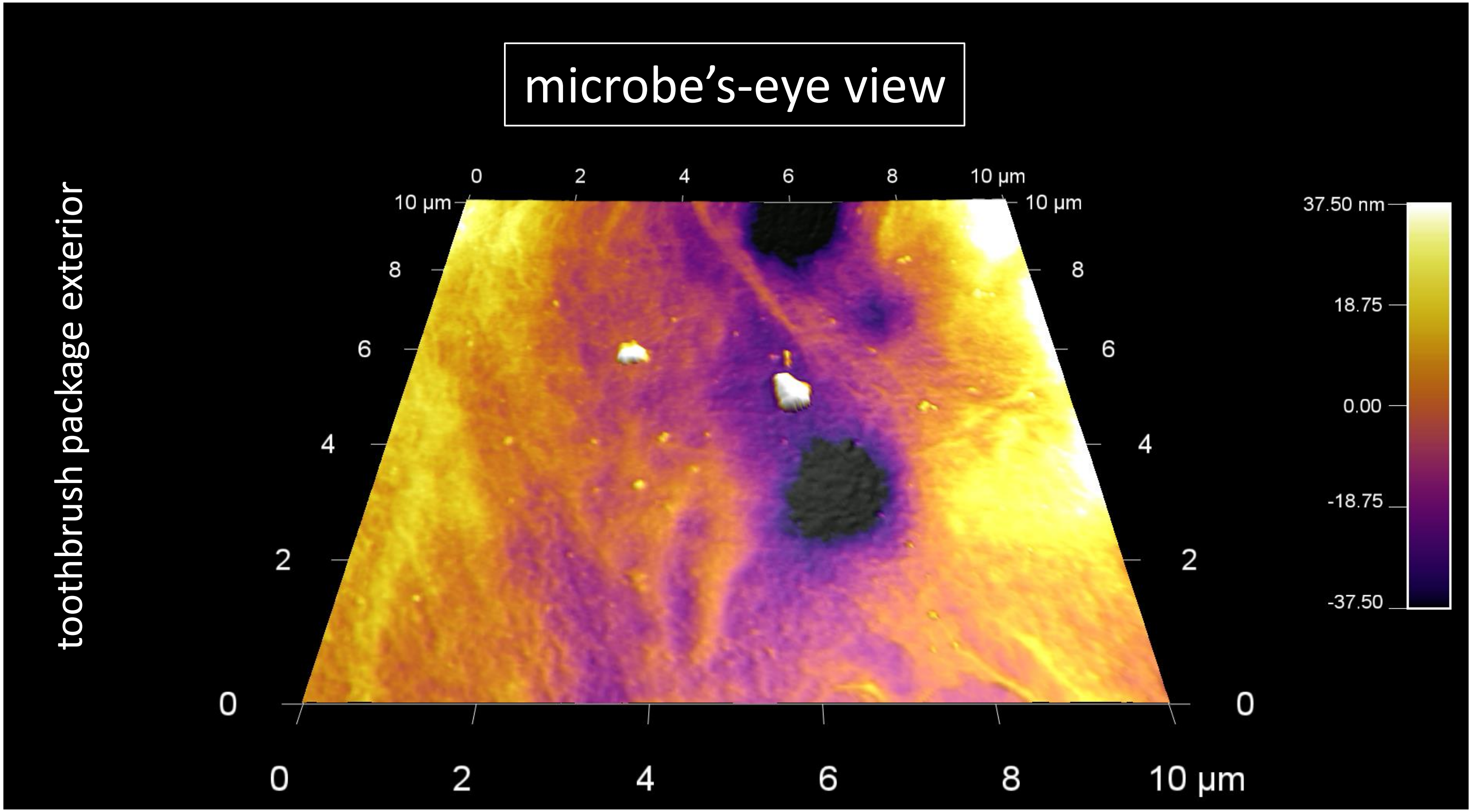
Image Processing

line subtraction:
masked flatten

no more
streaks

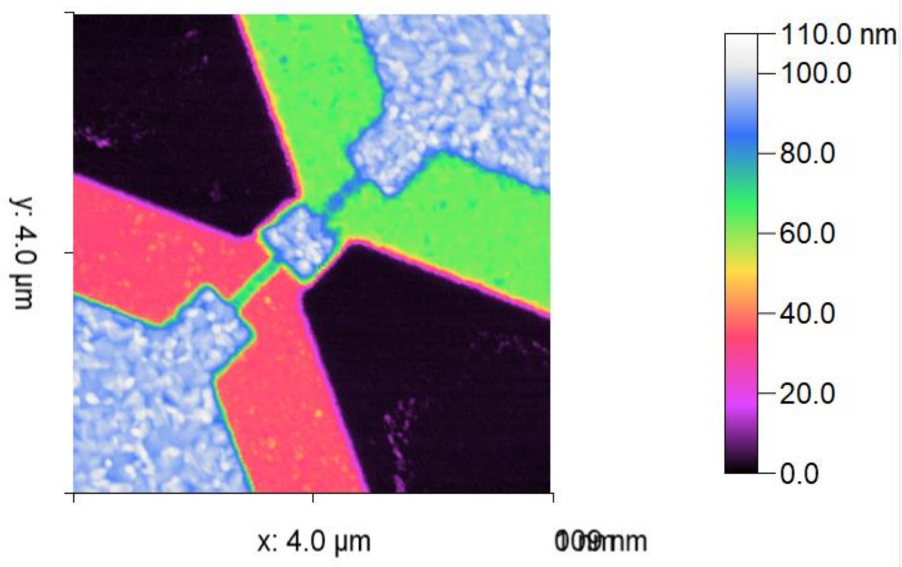


3D Display

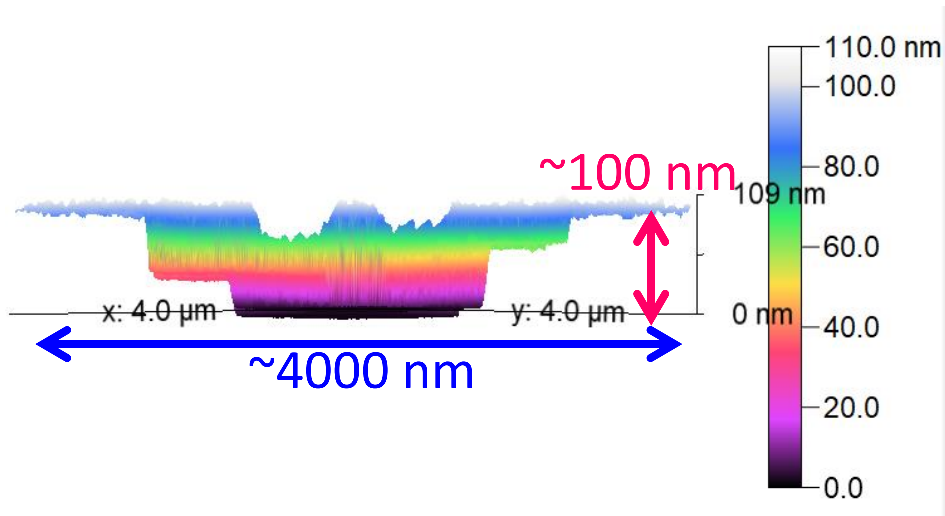

microbe's-eye view
toothbrush package exterior
0
2
4
6
8
10 µm
10 µm
37.50 nm
18.75
0.00
-18.75
-37.50

3D Display

face on



side view

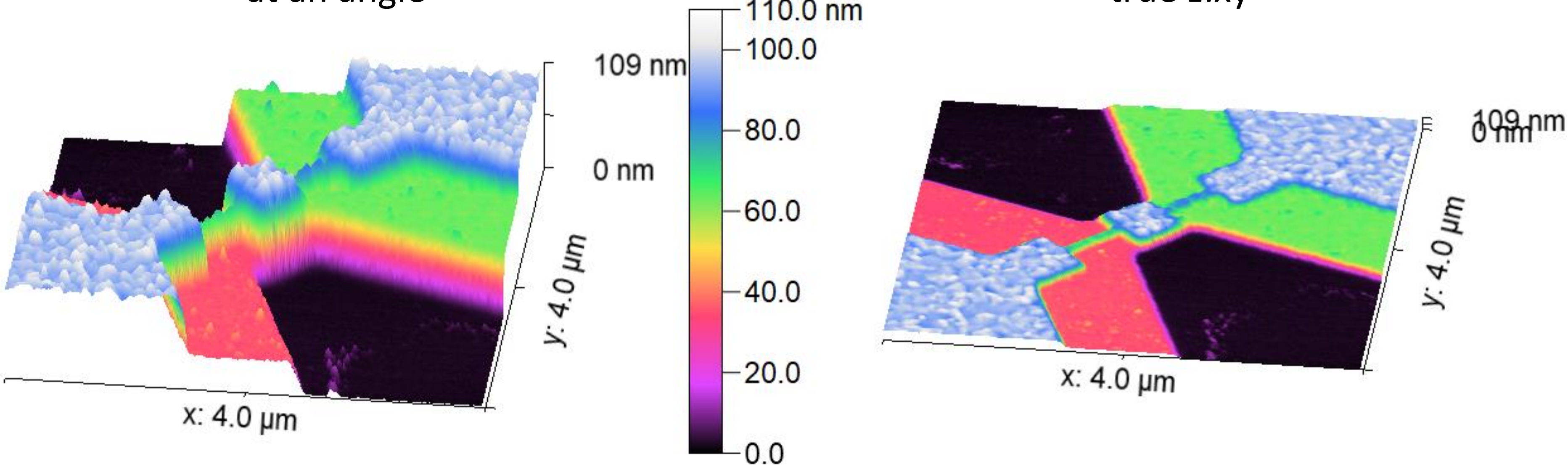


raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM

3D Display—z:xy

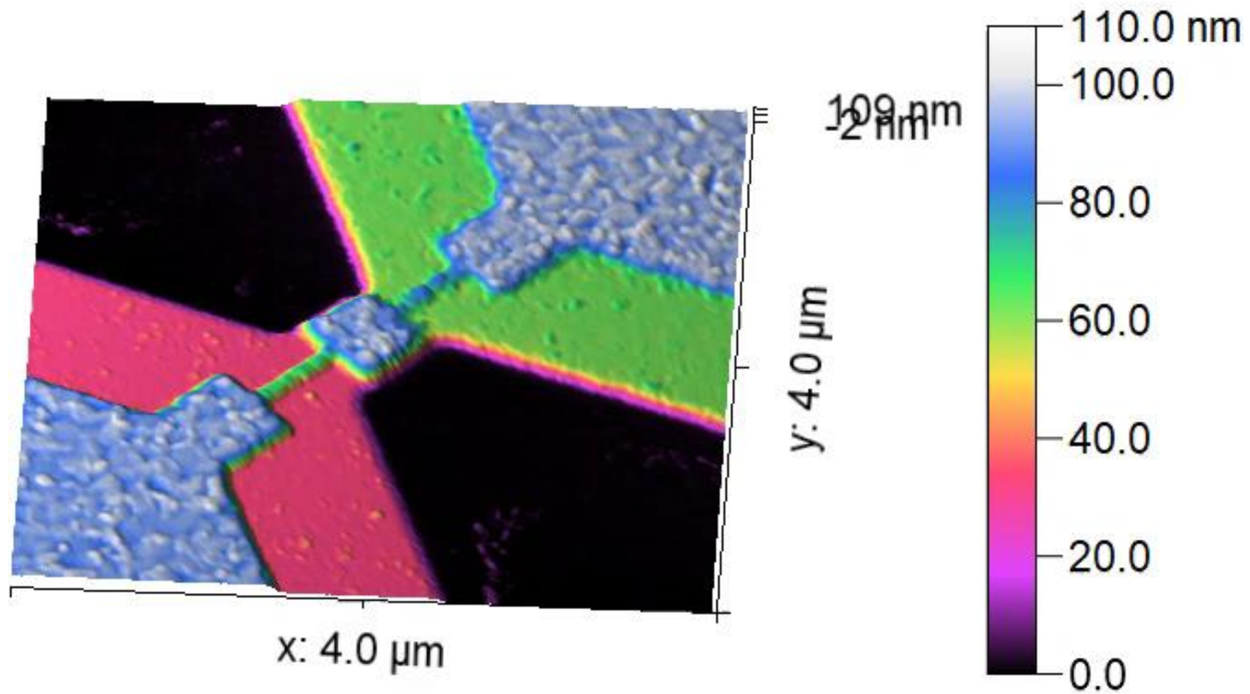
at an angle

true z:xy



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

3D Display—Lighting Angle



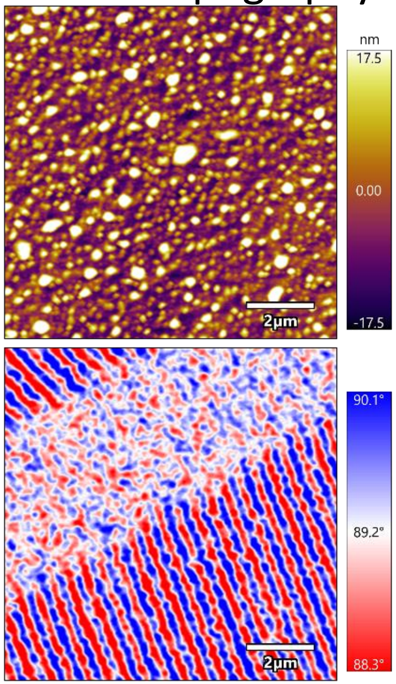
raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM

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)

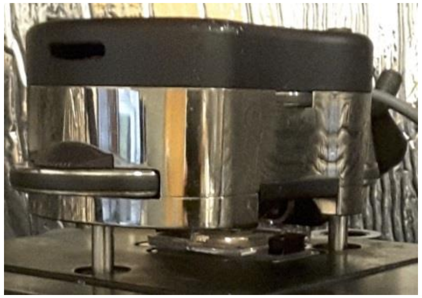
topography



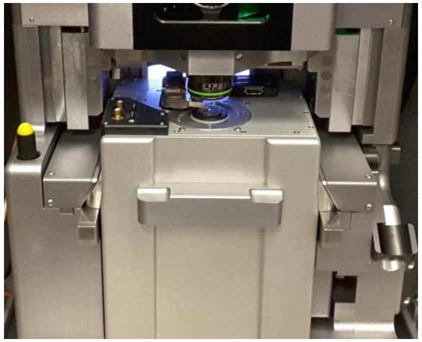
MFM phase

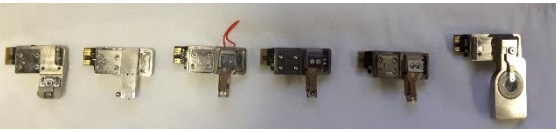
Attachments on the MRL AFMs

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



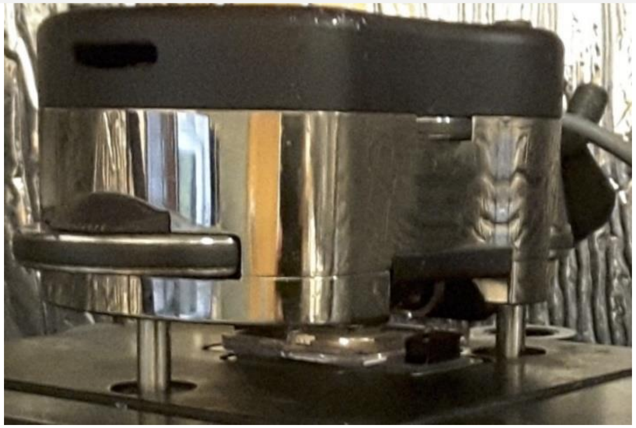
- blueDrive Photothermal Excitation
- Fast Force Mapping
- Dual-Gain ORCA Conductive AFM
- Piezoresponse Force Microscopy (HV-PFM)
- Contact Resonance Viscoelastic Mapping Mode
- AM-FM Viscoelastic Mapping Mode
- Scanning Tunneling Microscopy (STM)
- Air Temperature Controller (ATC)
- Droplet Cantilever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



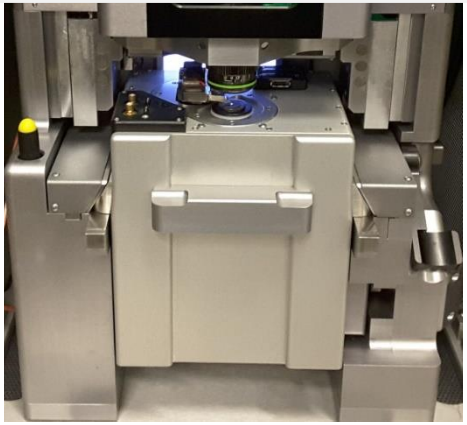


MRL AFMs—B12 MRL

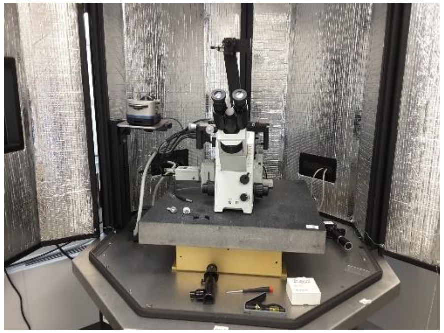
Asylum Research MFP-3D-SA (2 of these)
15µm z range, 90µm x 90µm scan size



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

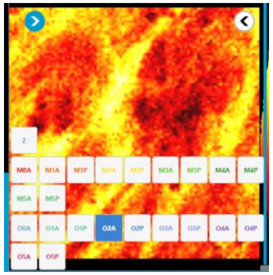


Coming Soon: Asylum Research MFP-3D-Bio
on an inverted optical fluorescence microscope

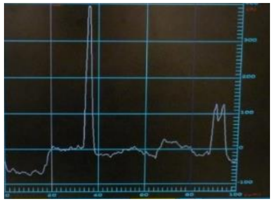


Related Instruments at MRL

- Neaspec Nano-IR
  - AFM + infrared
  - Highly localized chemical information
- Horiba TERS/TEPL
  - Tip-enhanced Raman spectroscopy


Julio Soares, MRL

- 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