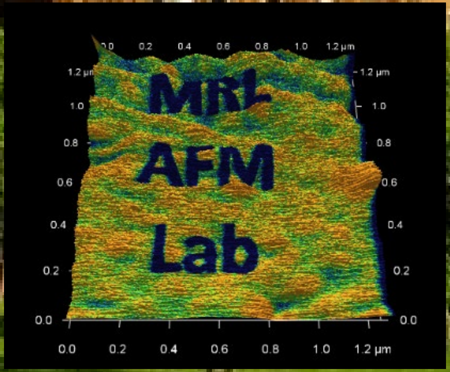
Grainger College of Engineering
UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

Atomic Force Microscopy

Kathy Walsh
Senior Research Scientist
Scanning Probe Microscopy

Materials Research Laboratory
Central Research Facilities

Physics 403
10/29/24



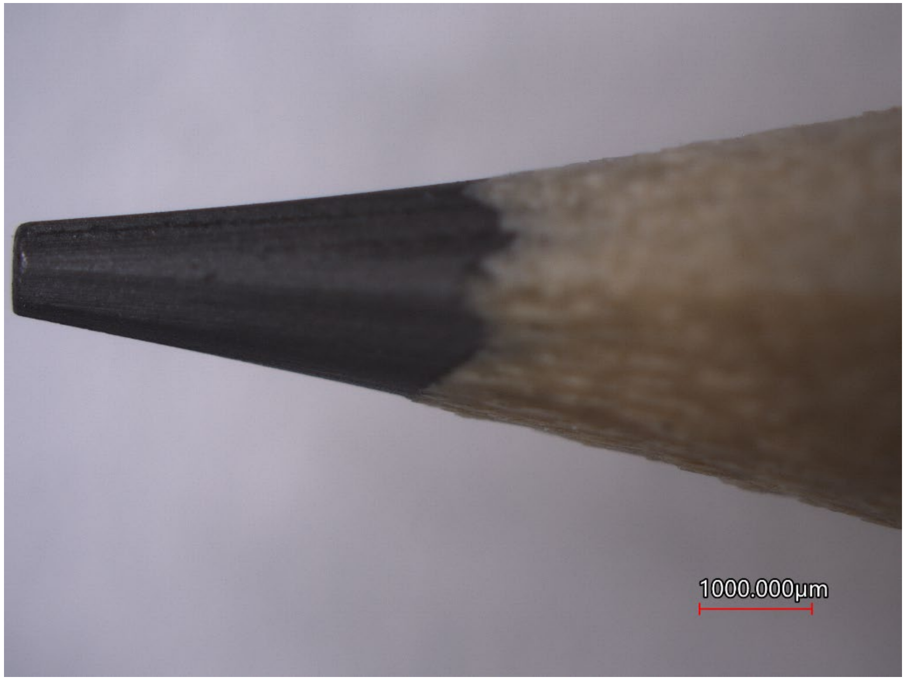
Illinois Materials Research Lab Central Research Facilities

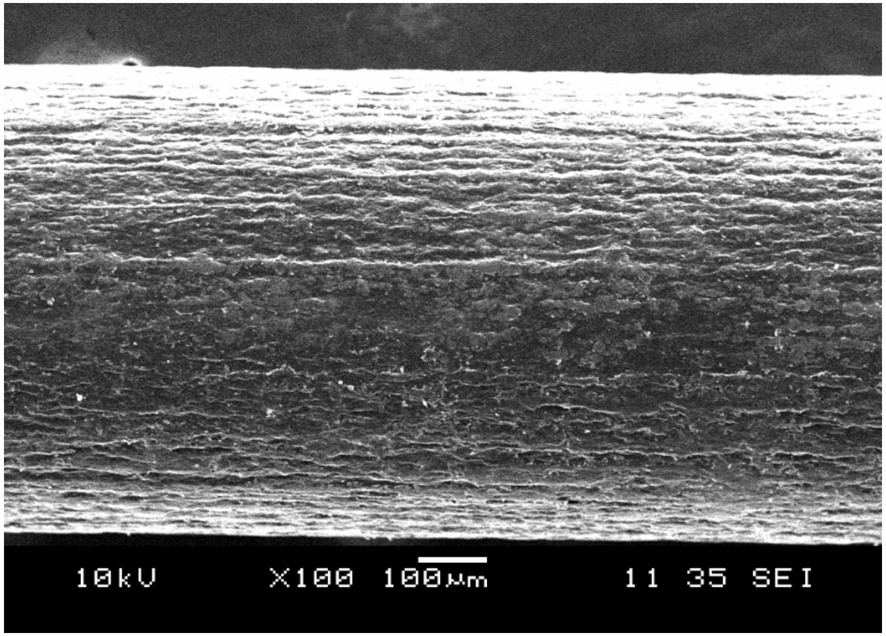
- User facility—anyone can be trained
    - UIUC and non-UIUC researchers welcome
    - Undergraduate researchers welcome
    - Staff collaboration or analysis available
- mrl.illinois.edu/facilities
- mrl-facilities@illinois.edu

Looking at Surfaces

Optical Microscopy

Scanning Electron Microscopy

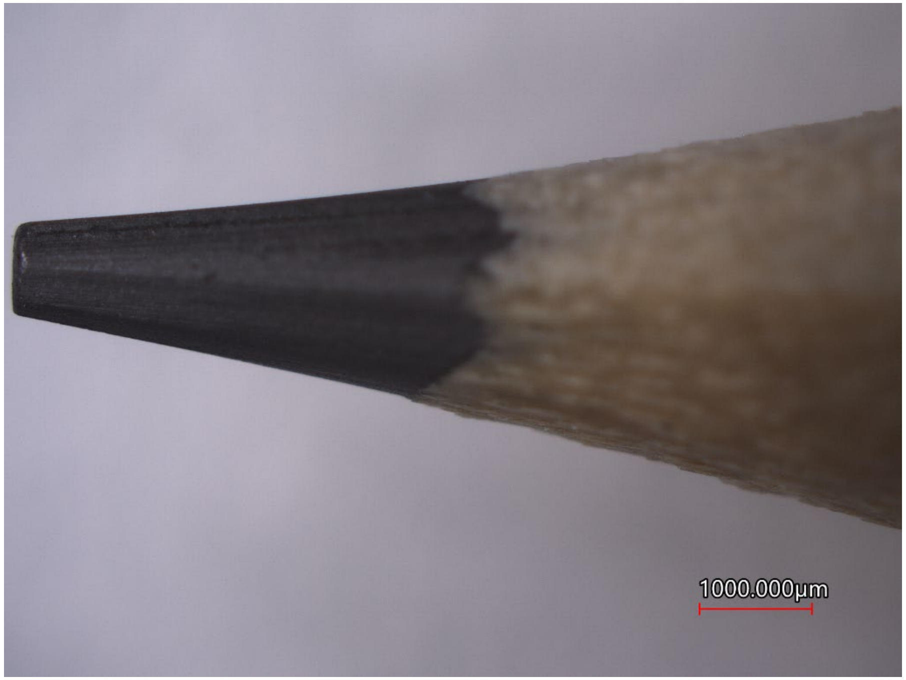




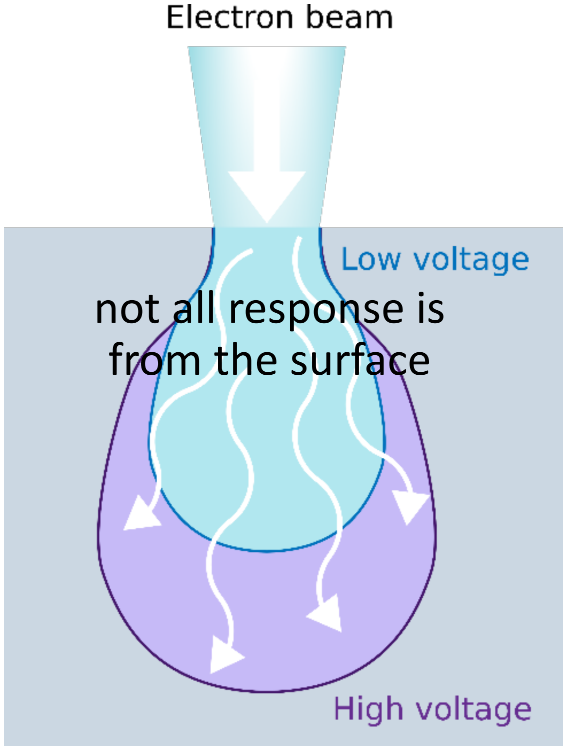
mechanical pencil “lead”

Looking at Surfaces

Optical Microscopy



Scanning Electron Microscopy

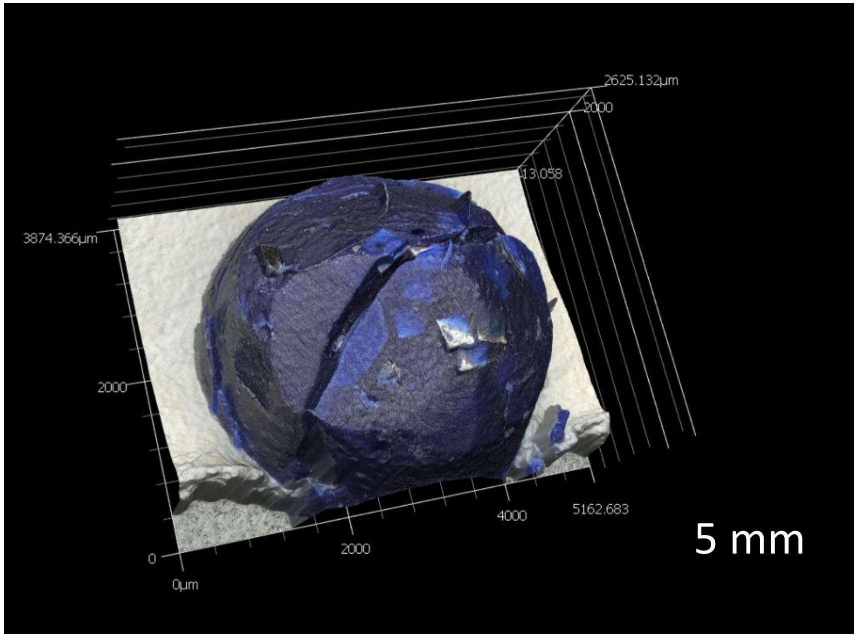


not all response is from the surface

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

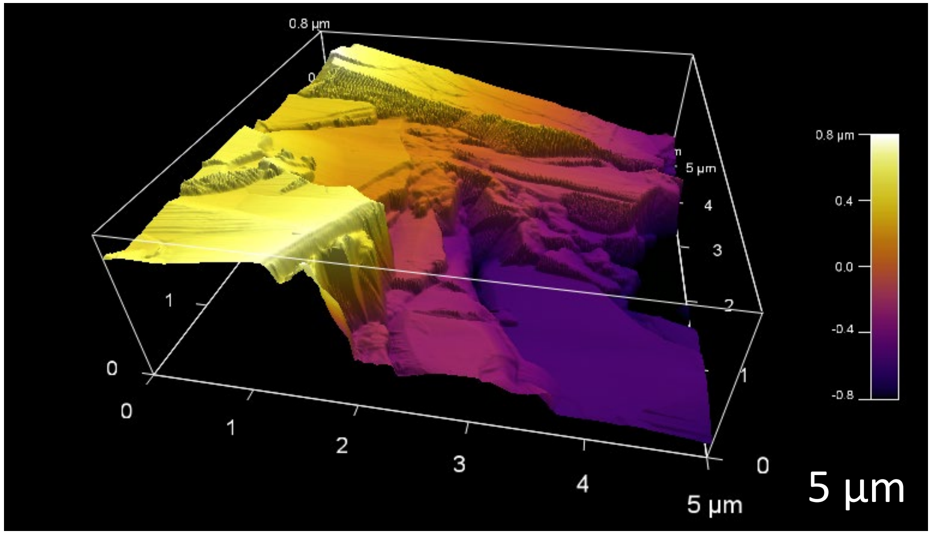
Surface XYZ Coordinates Needed

3D Optical Profilometry



blue glitter crayon tip

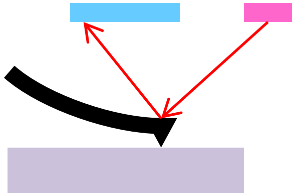
Atomic Force Microscopy

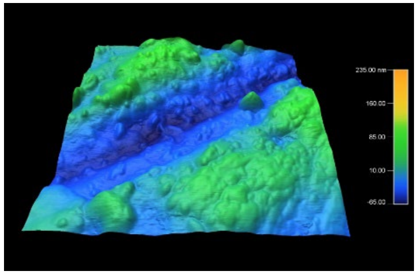


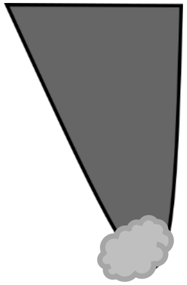
pencil “lead”

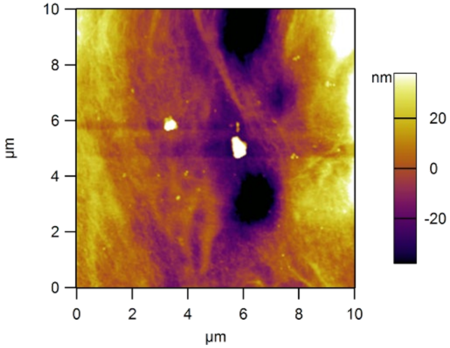
Topics for Today

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



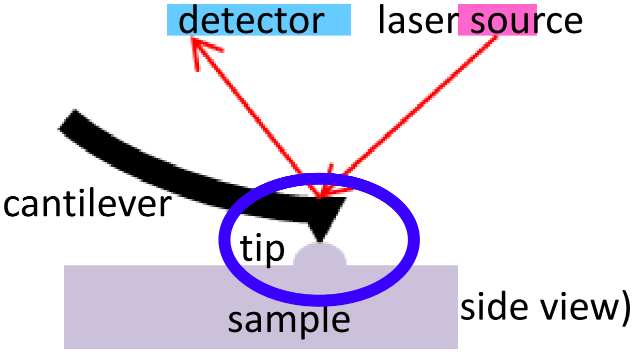






What’s an Atomic Force Microscope?

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


detector

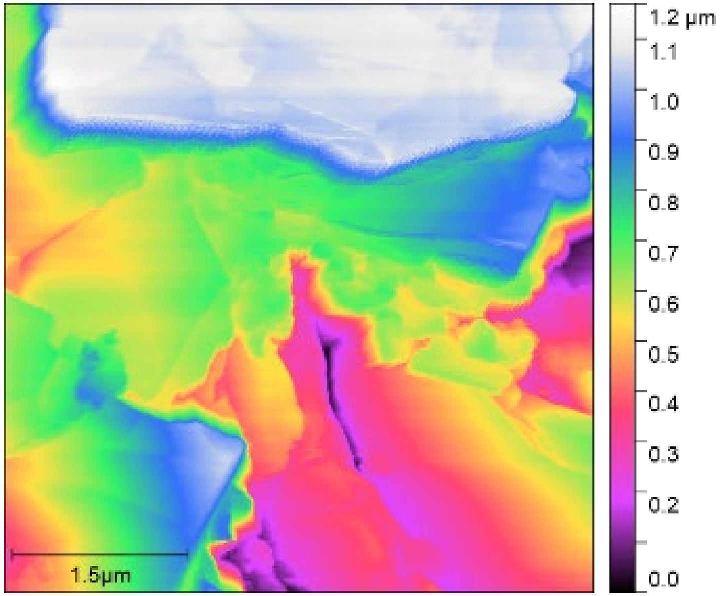
laser source

cantilever

tip

sample

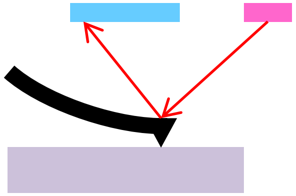
side view)

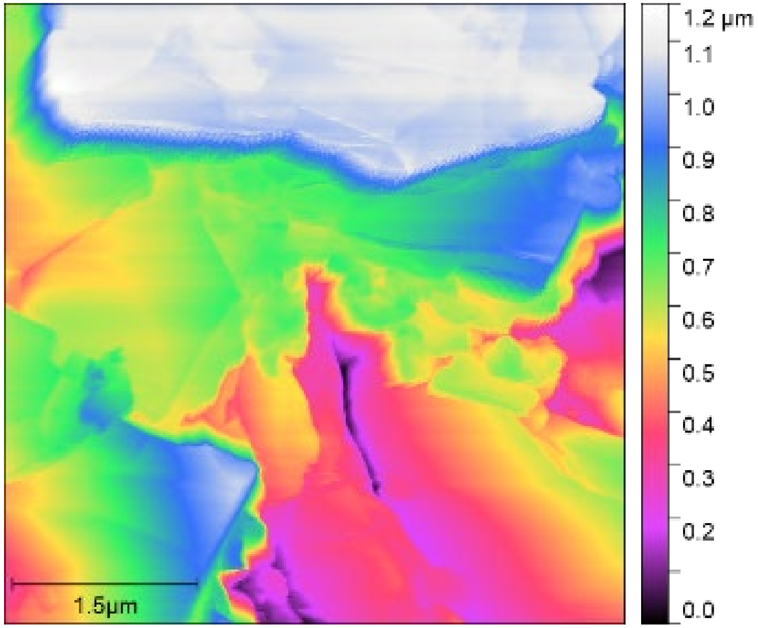


false-color surface topographs

What’s an Atomic Force Microscope?

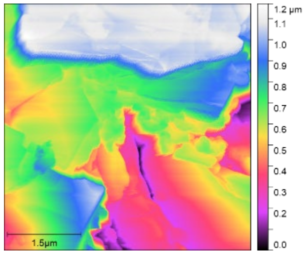
- “Atomic Force”—interactions between tip and sample
  - Not actual atomic resolution (usually)
  - Nanoscale lateral resolution (depends on tip)
  - Sub-angstrom vertical resolution
- “Microscope”—surface topograph (false color)

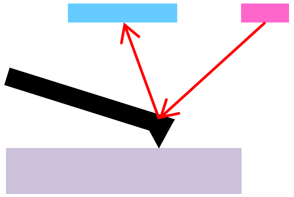




What’s an Atomic Force Microscope?

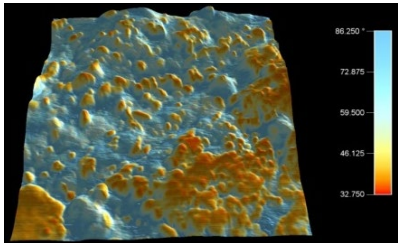
- “Atomic Force”—interactions between tip and sample
  - Sub-angstrom vertical resolution
  - Not actual atomic resolution (usually)
  - Nanoscale lateral resolution (depends on tip)
- “Microscope”—surface topograph (false color)





- 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

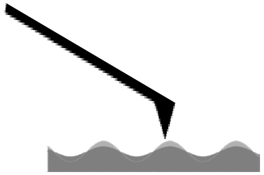


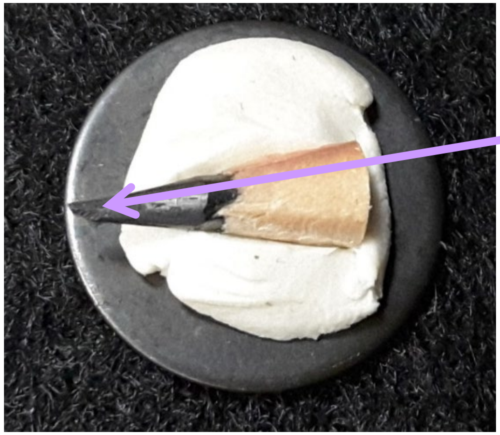
Turquoise, 1µm x 1µm
color overlay: phase

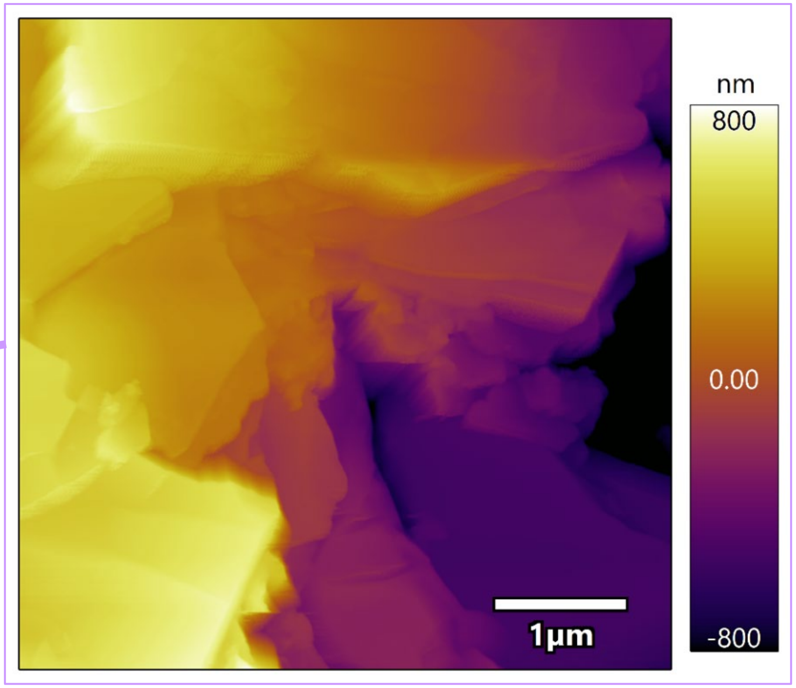
Typical AFM Scales

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

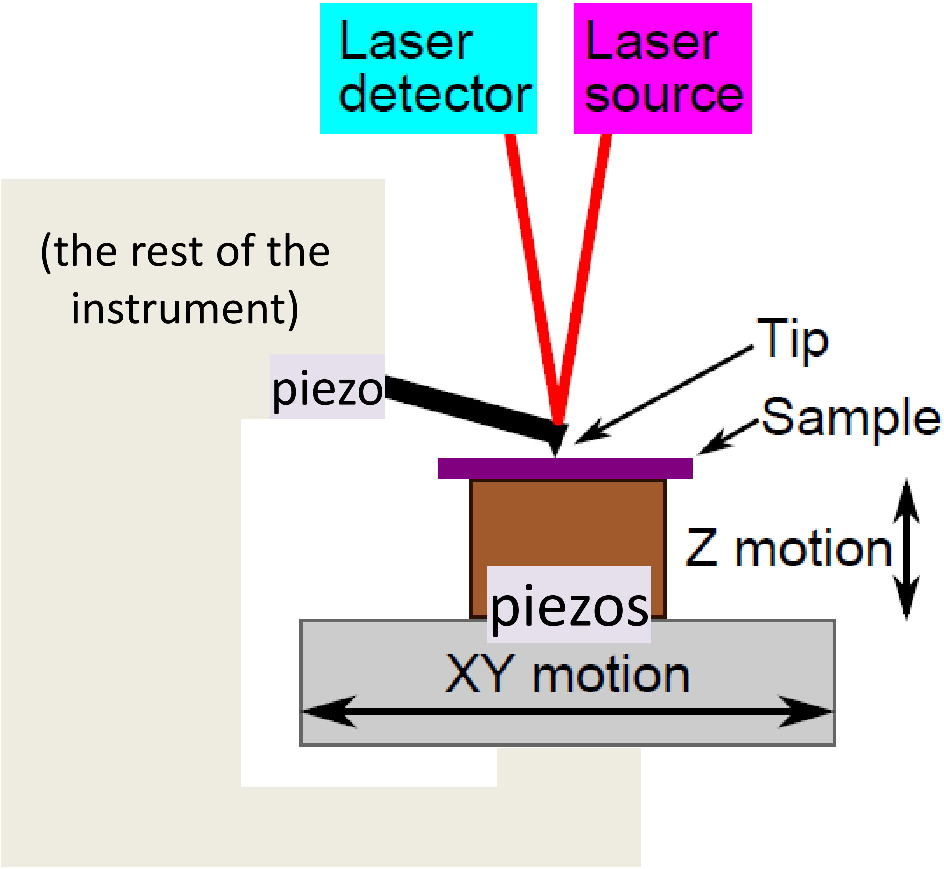
- Image sizes -- few to tens of μm^2
- Feature peak-to-valley -- Å to µm
- Sample sizes -- mm to cm
- AFM measures surfaces

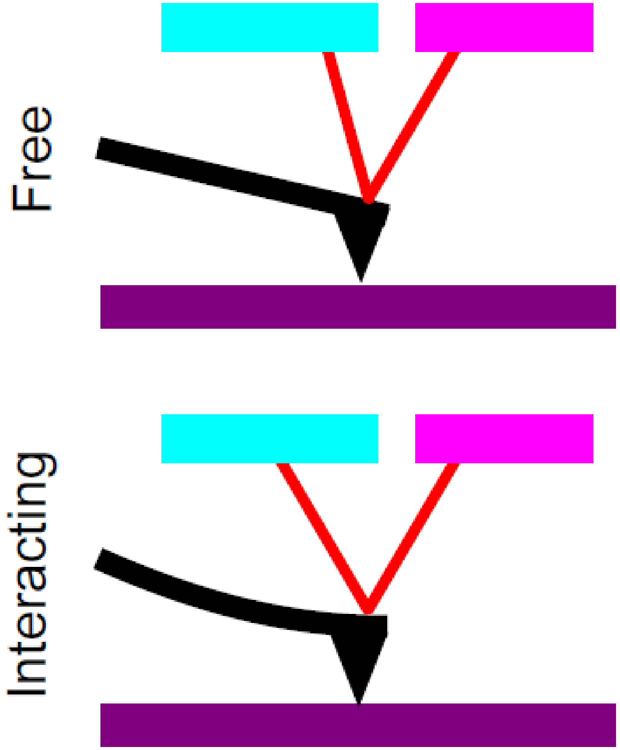






AFM Schematic

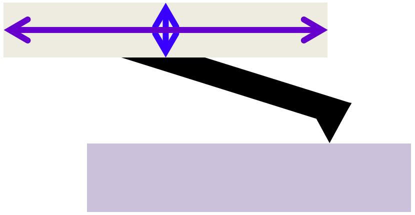




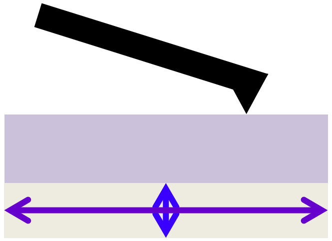
Scanners

scanning probe microscopy

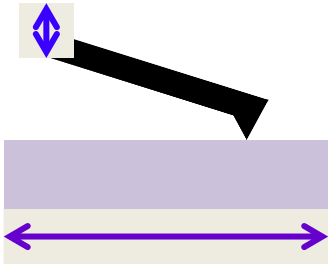
tip scanning



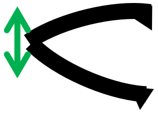
sample scanning



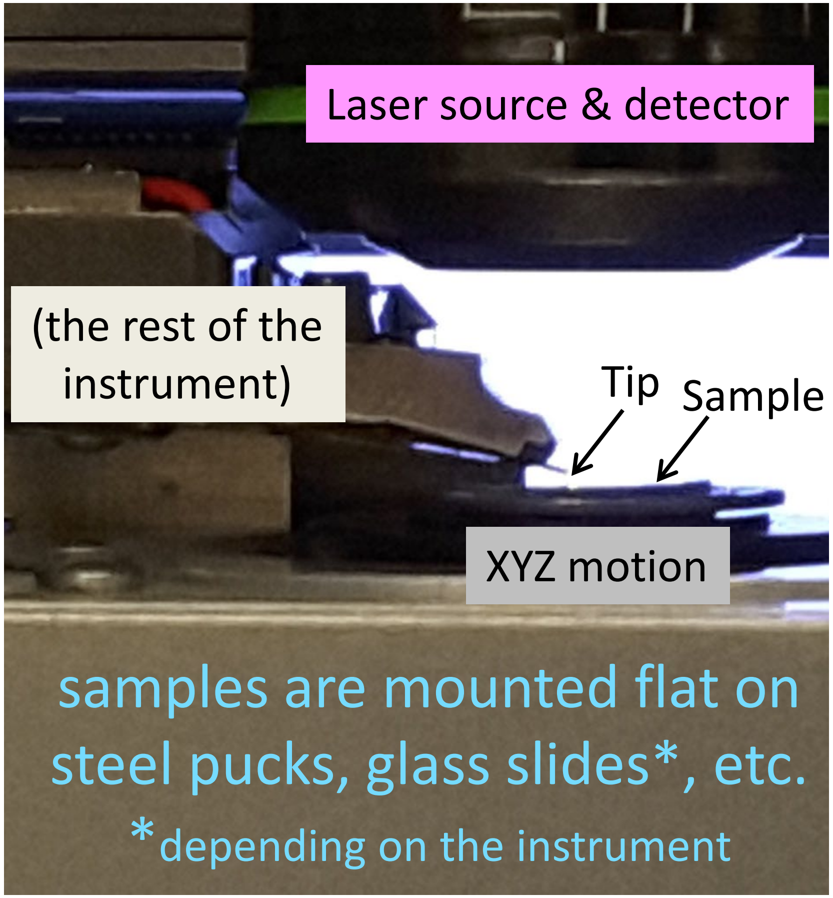
decoupled scanning



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



AFM Instrument

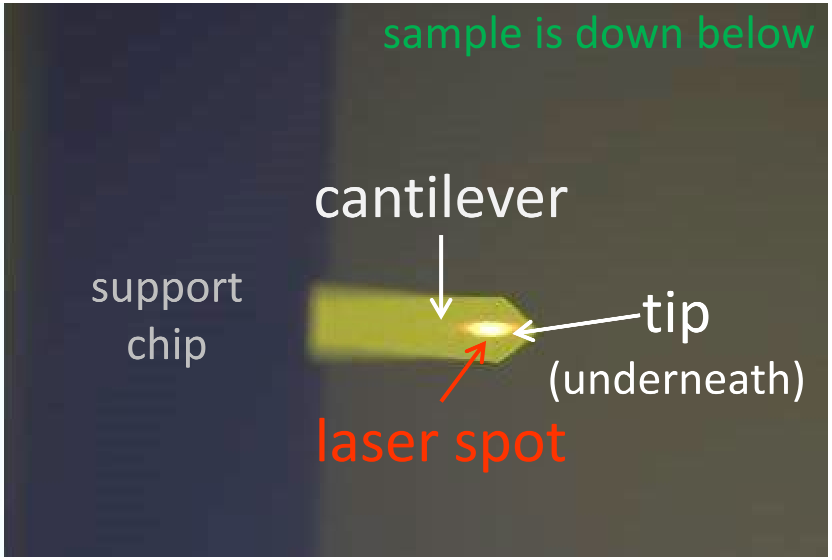


samples are mounted flat on steel pucks, glass slides*, etc.

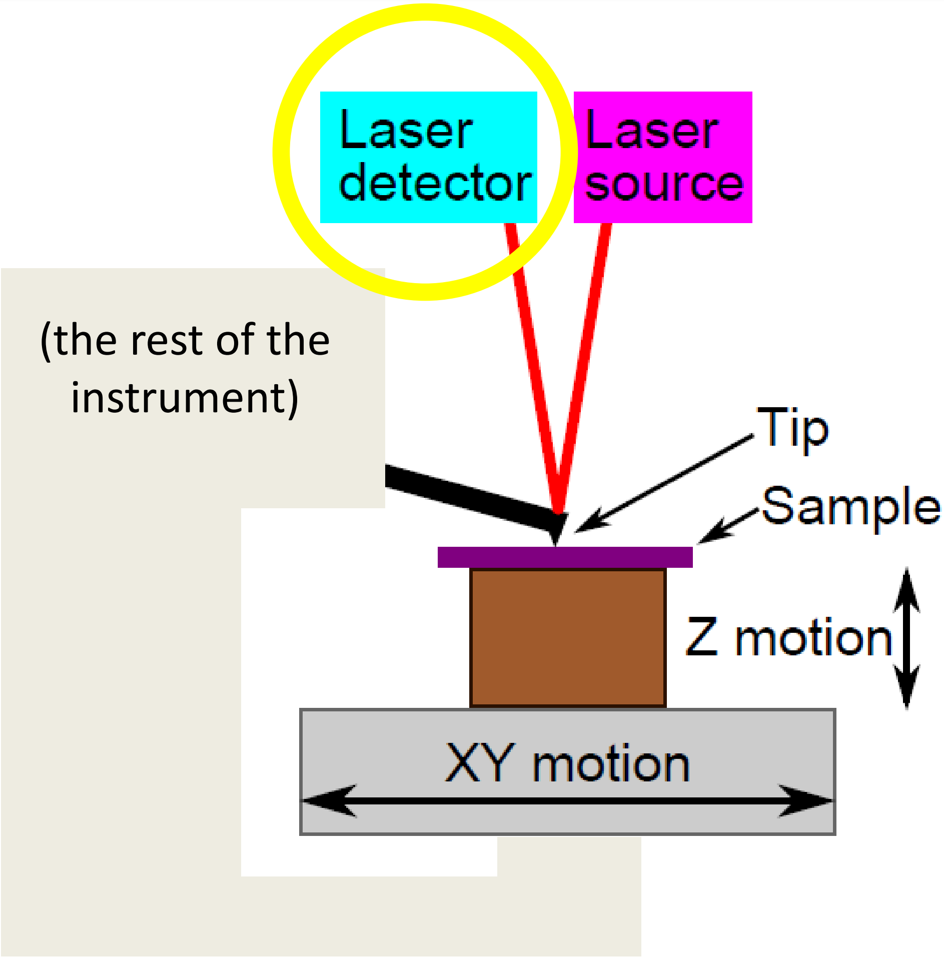
*depending on the instrument

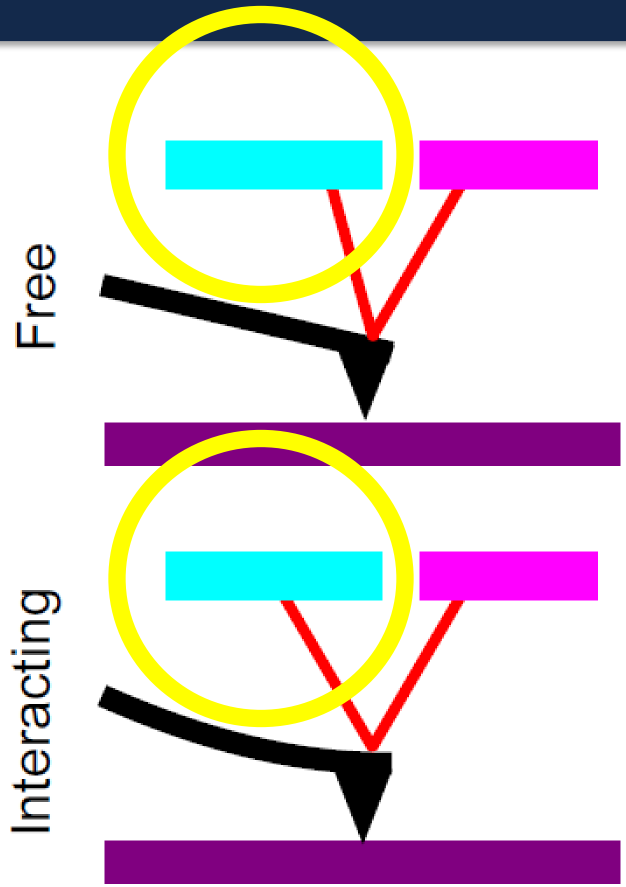
side view

top view



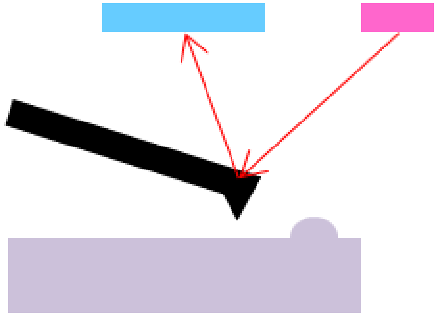
AFM Schematic



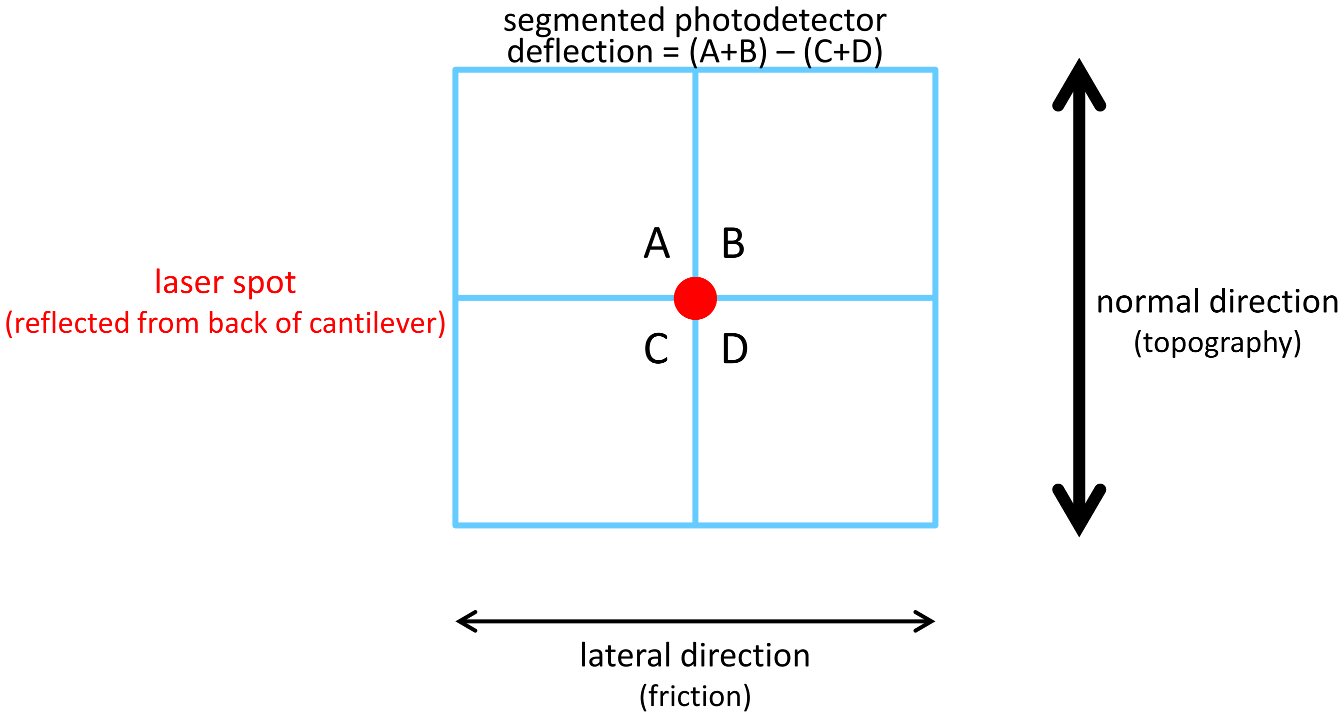


Laser Detection

non-interacting



(side view)



segmented photodetector
deflection = (A+B) – (C+D)

laser spot
(reflected from back of cantilever)

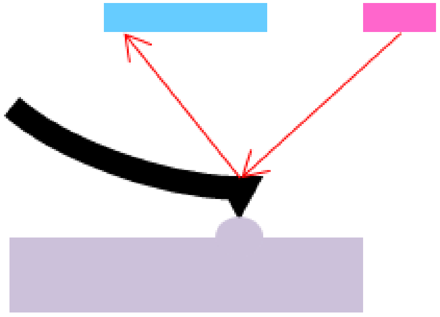
normal direction
(topography)

lateral direction
(friction)

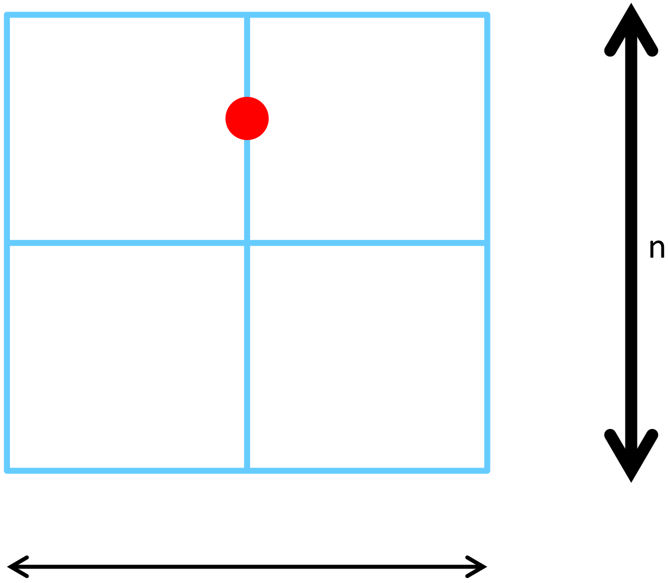
(exaggerated schematic)

Laser Detection

sample pushing up



(side view)



segmented photodetector

laser spot
(reflected from back of cantilever)

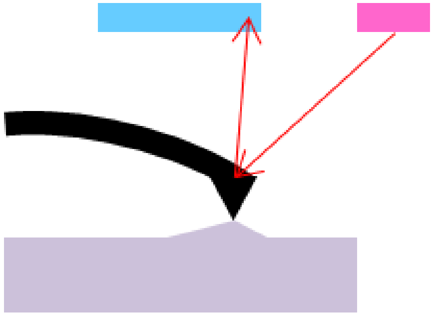
normal direction
(topography)

lateral direction
(friction)

(exaggerated schematic)

Laser Detection

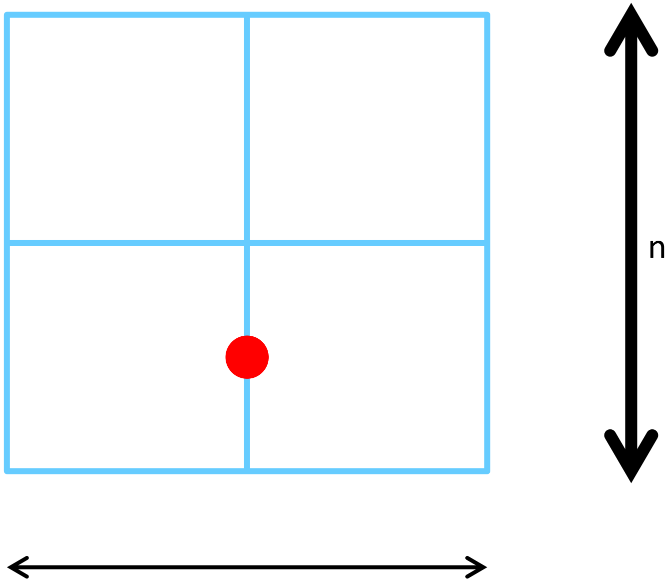
sample pulling down



(side view)

segmented photodetector

laser spot
(reflected from back of cantilever)



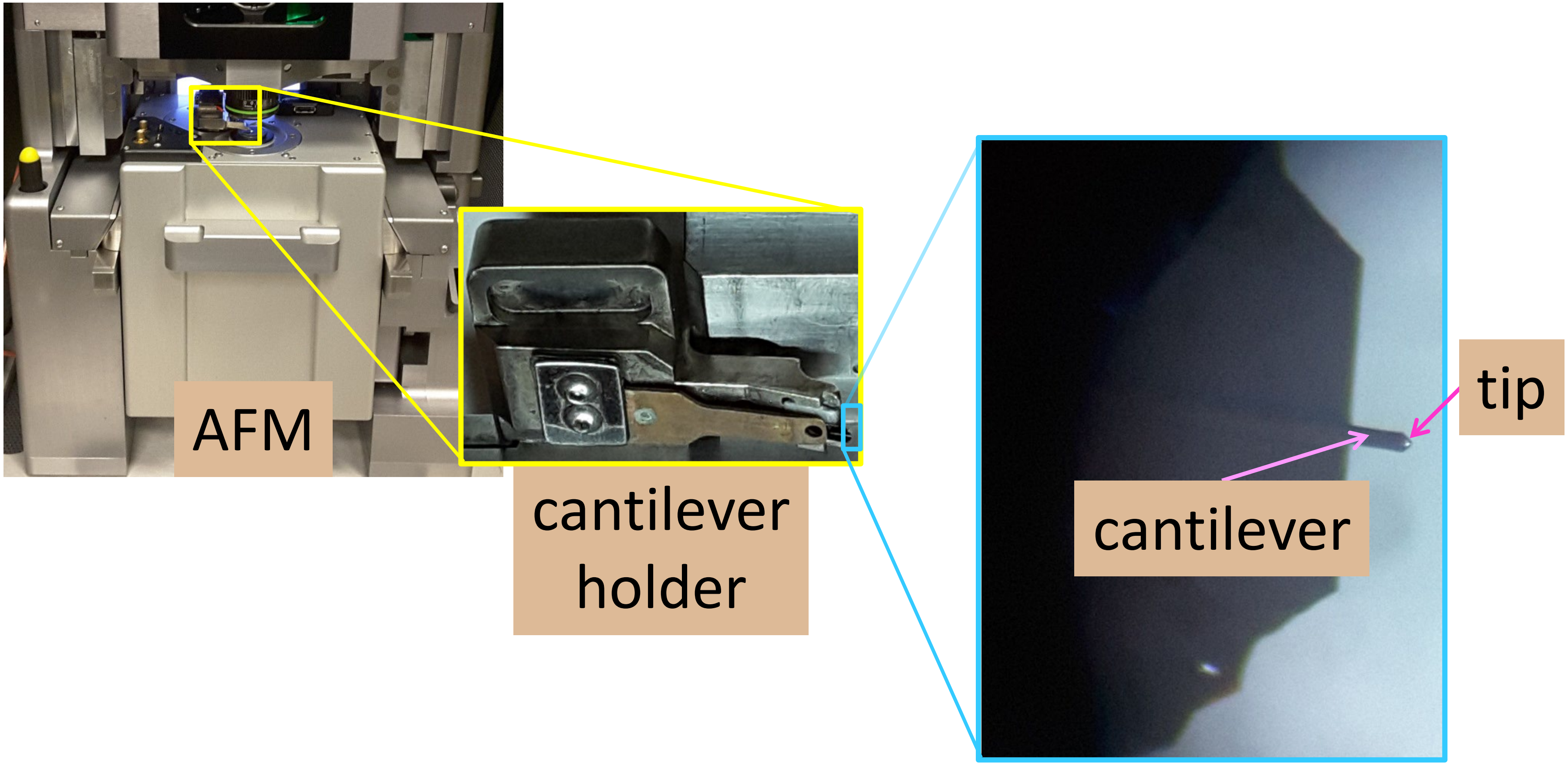
normal direction
(topography)

lateral direction
(friction)

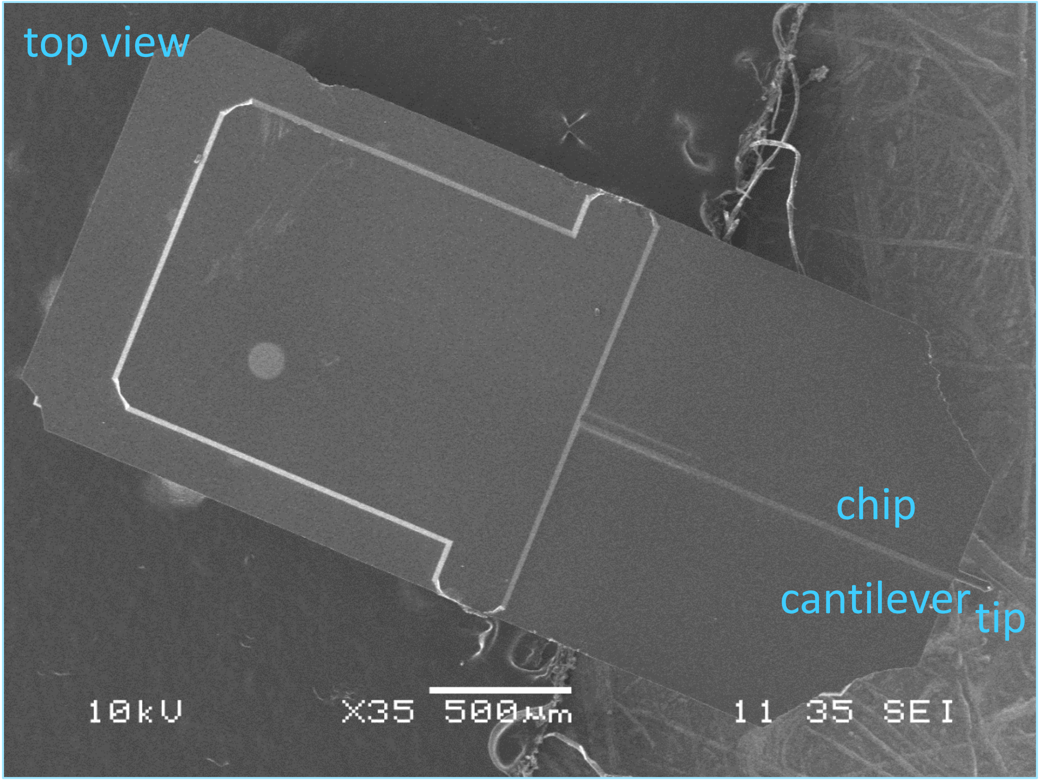
(exaggerated schematic)

AFM Tips

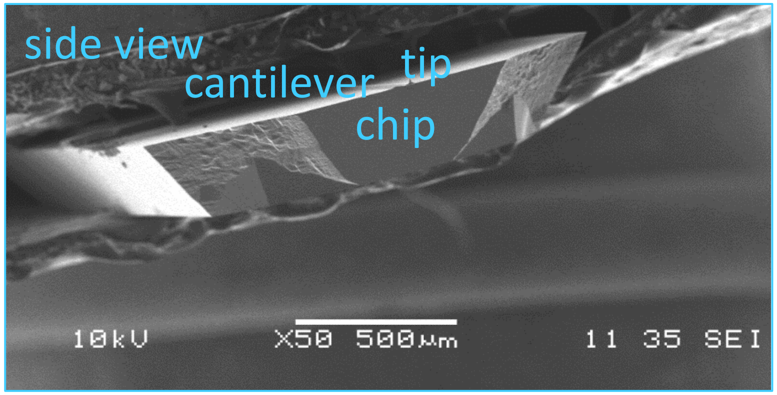
scanning *probe* microscopy



Tip Terminology



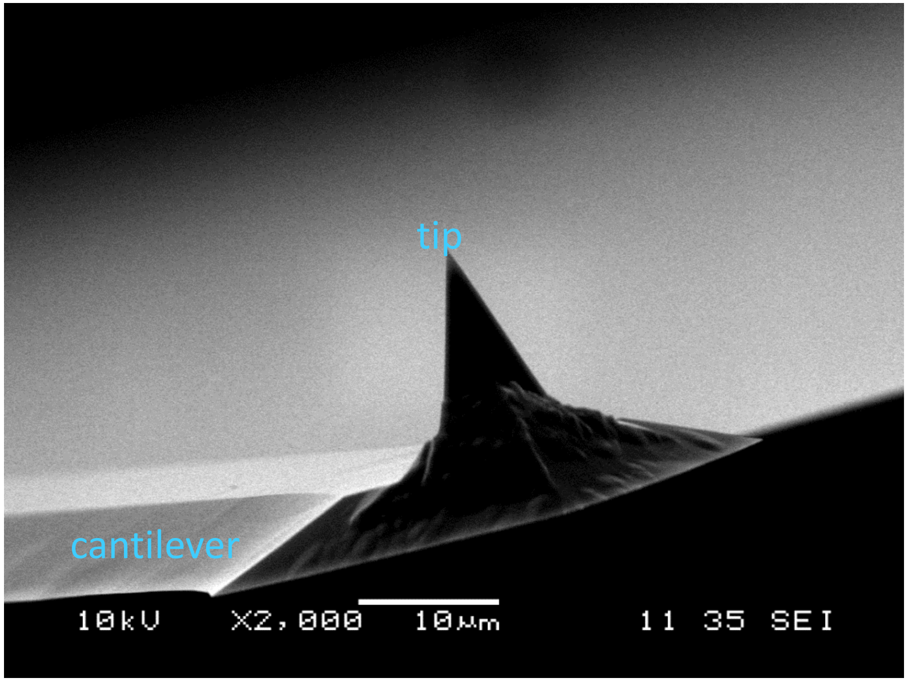
SEM images taken using MRL's JEOL 6060LV



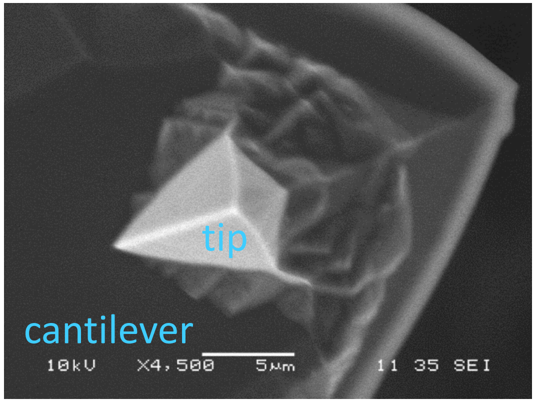
"probe"

tips point upwards in the box

Typical Tip

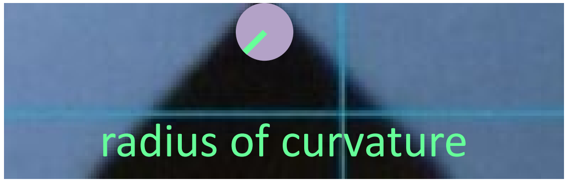


SEM images taken using MRL's JEOL 6060LV



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
  - 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 sphere-tips 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

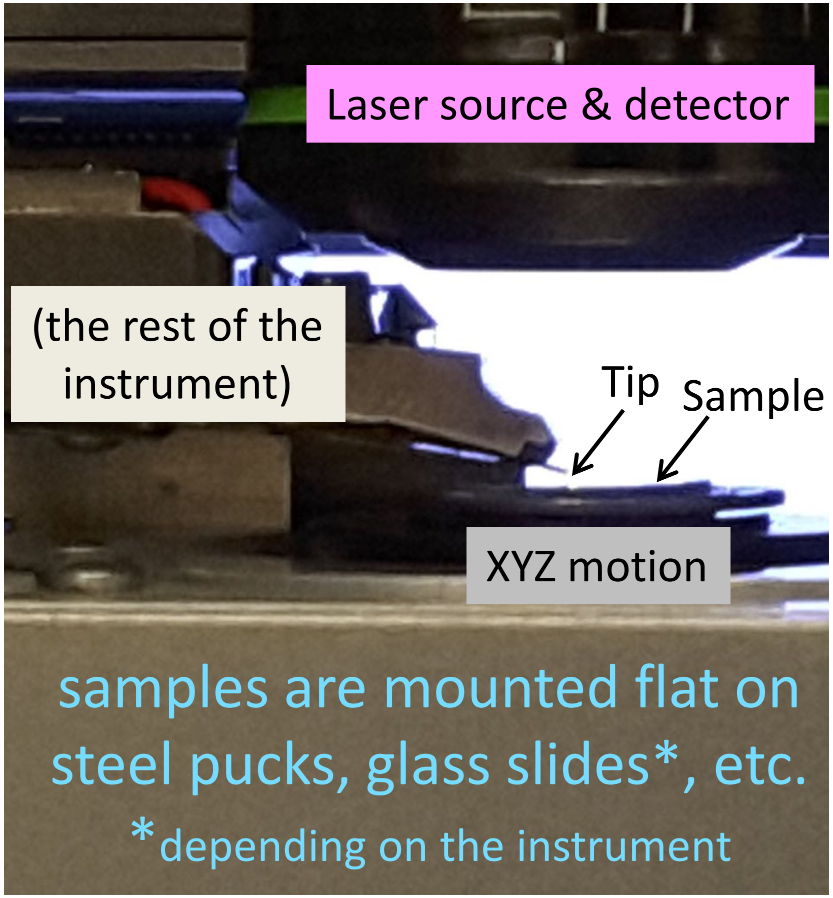
NANOANDMORE USA
The Nanotech Facilitator

“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)

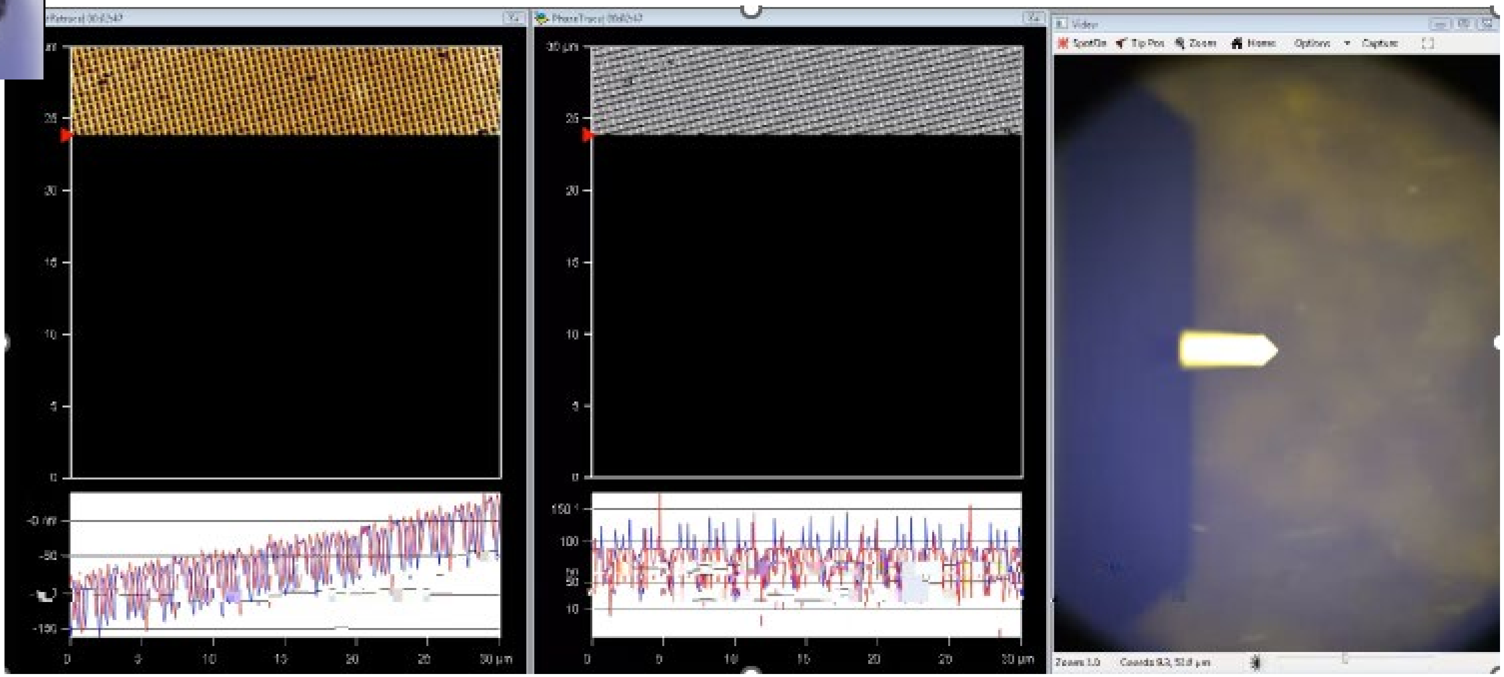


samples are mounted flat on steel pucks, glass slides*, etc.

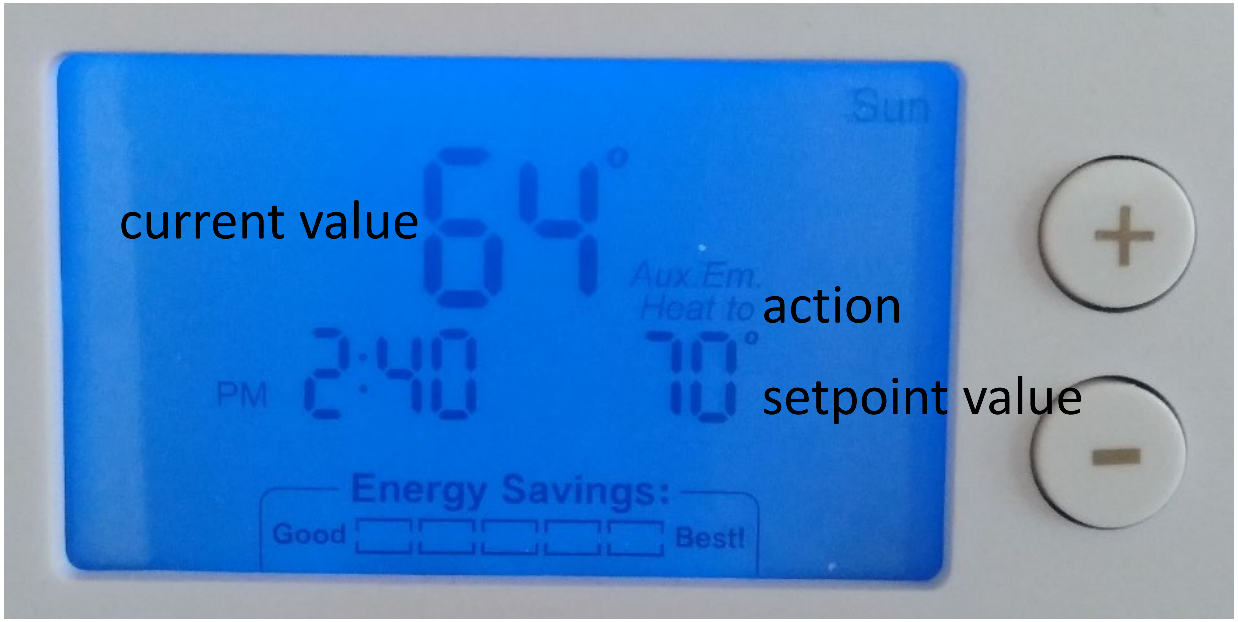
*depending on the instrument

Raster Scanning

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



Feedback



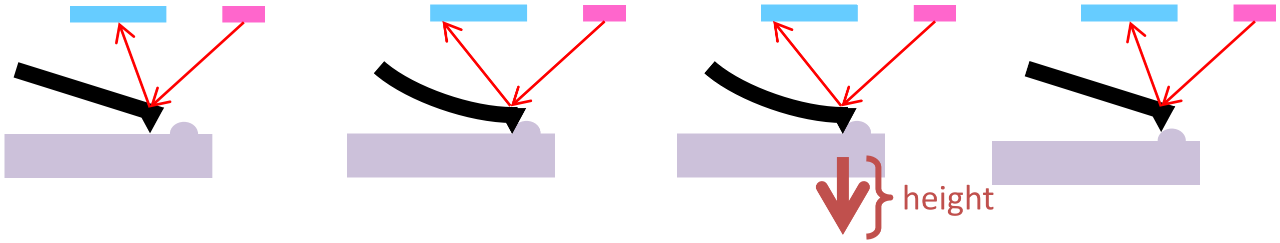
current value

action

setpoint value

Feedback

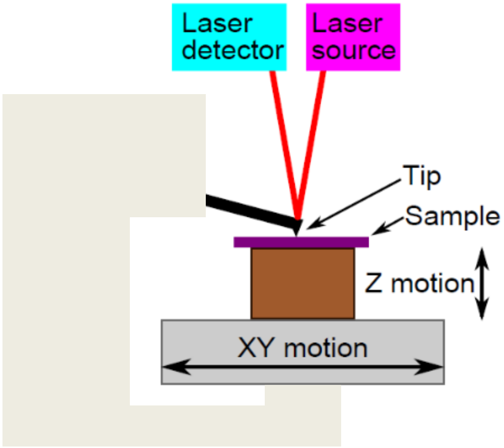
- z piezo extension adjusted to keep feedback signal equal to setpoint
  - too much force—move away
  - too little force—move closer
  - deflection for contact mode, usually amplitude for tapping mode
- distance extended or retracted describes the height of the feature

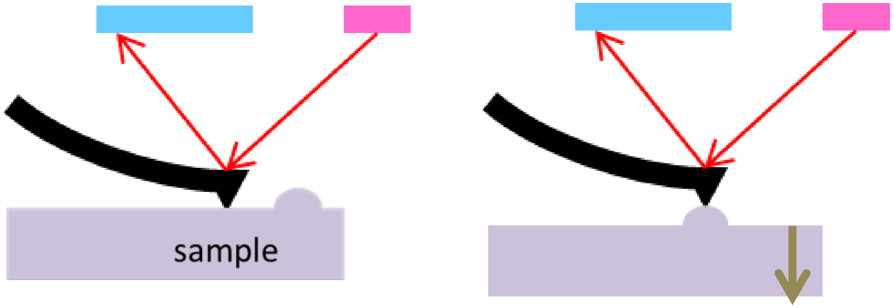


Contact Mode Imaging

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

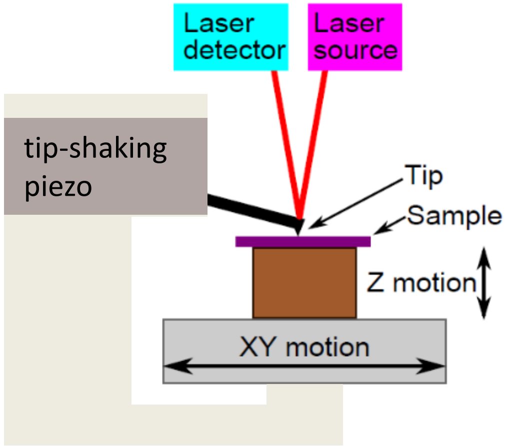
- Adjust tip—sample separation to keep cantilever deflection constant
  - Traces sample topography
  - Some AFMs move tip; some move sample



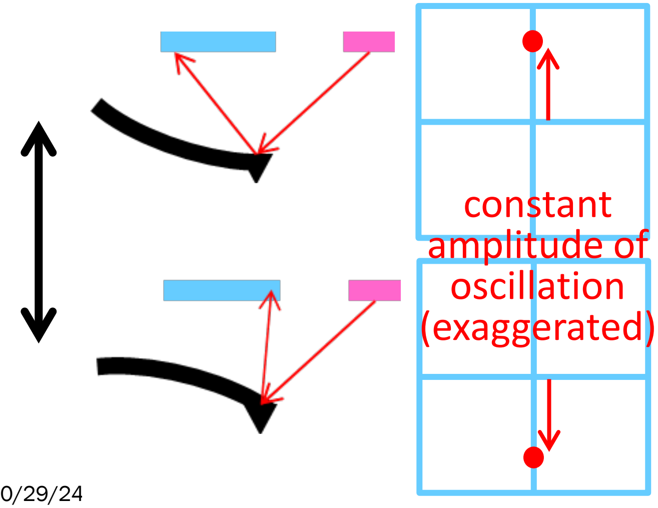


Tapping Mode Imaging

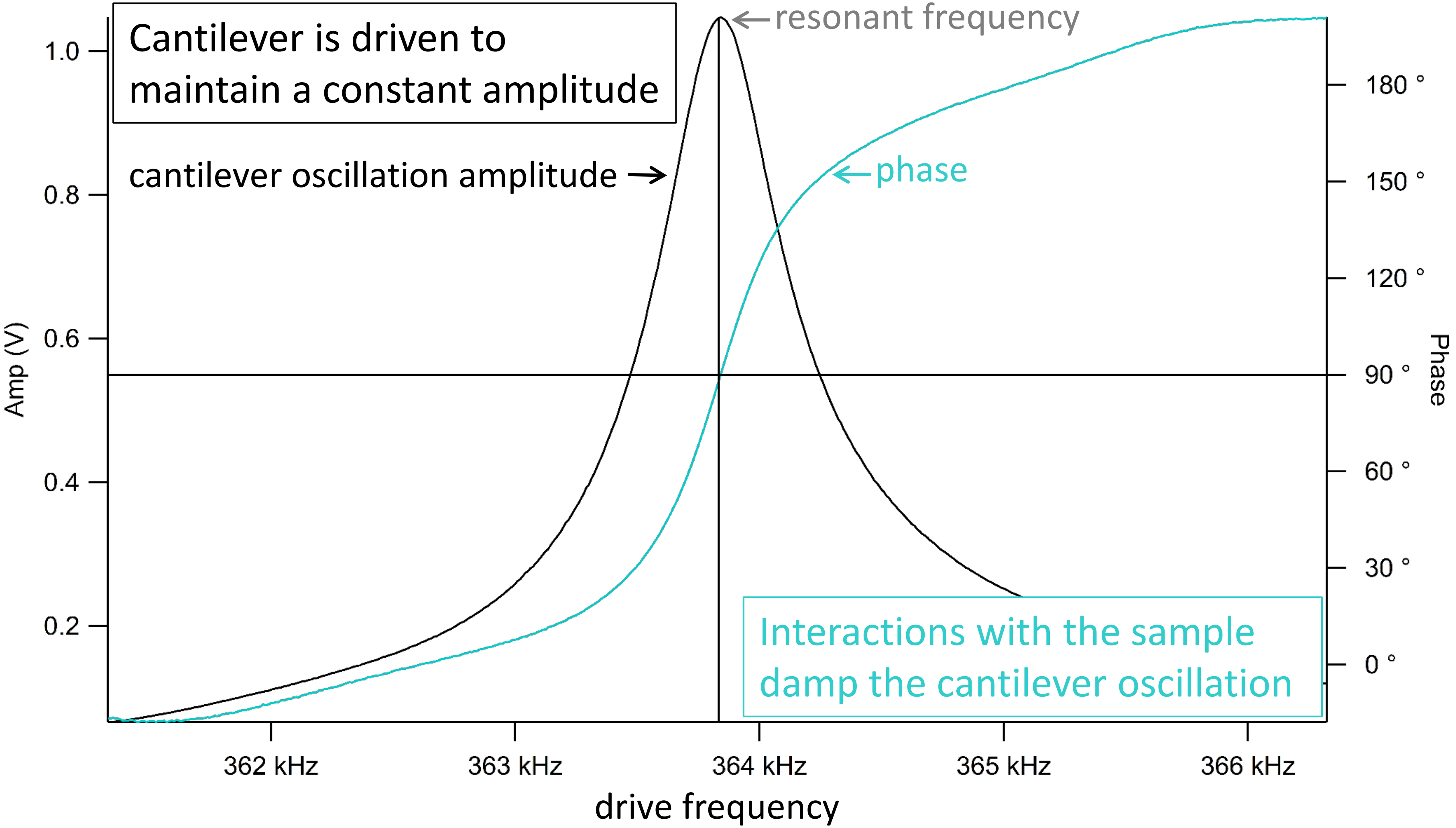
- Standard mode for AFM topography
- Intermittent contact, tapping, AC, amplitude modulation mode
- Not constantly in contact with the surface
- Driven, oscillating cantilever
- Tip—sample interactions affect oscillation



tip oscillates at tens of kHz to MHz



Tuning the Cantilever



Cantilever is driven to maintain a constant amplitude

cantilever oscillation amplitude →

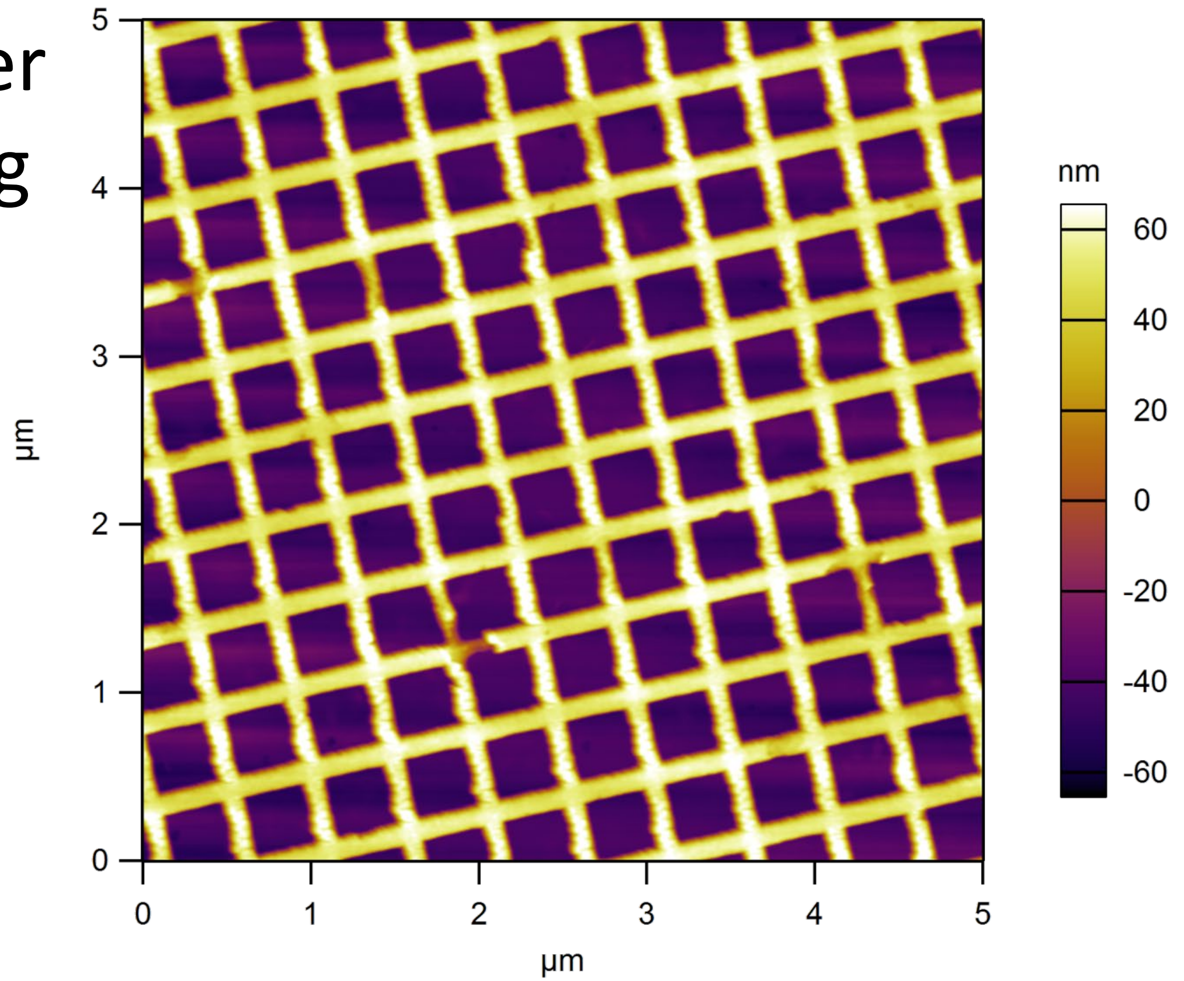
←resonant frequency

←phase

Interactions with the sample damp the cantilever oscillation

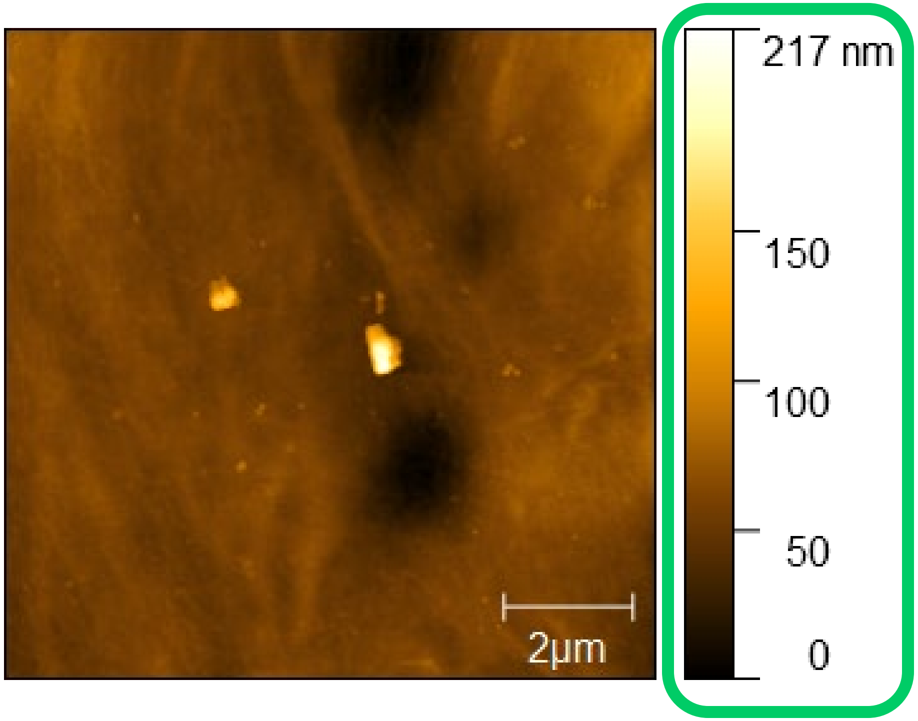
Application: Imaging

Polymer Grating



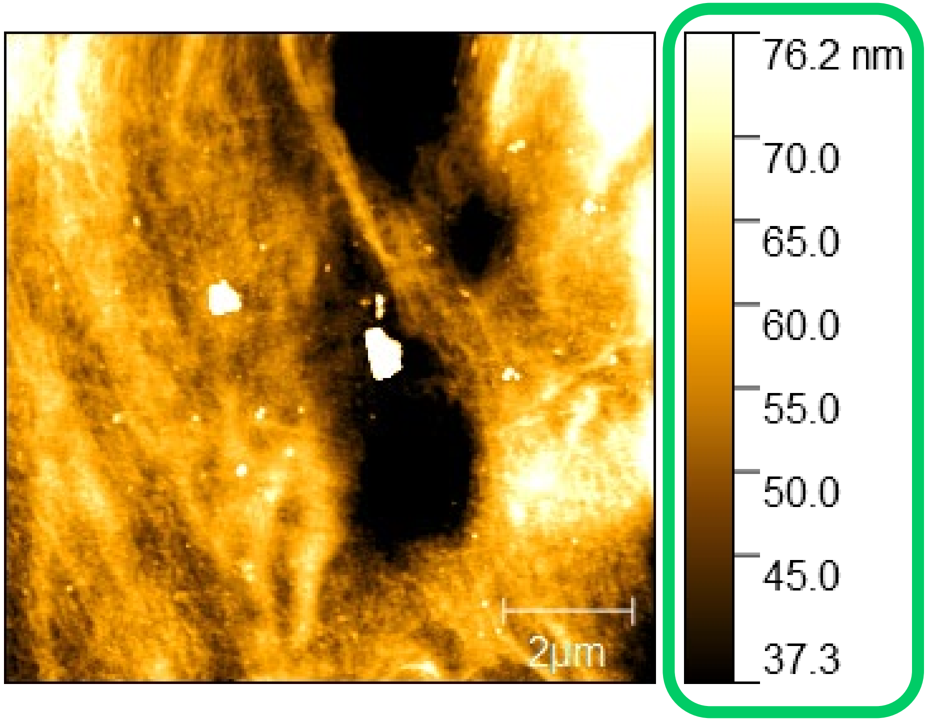
range of **colors, not heights** in the image

Reading the Colorscale



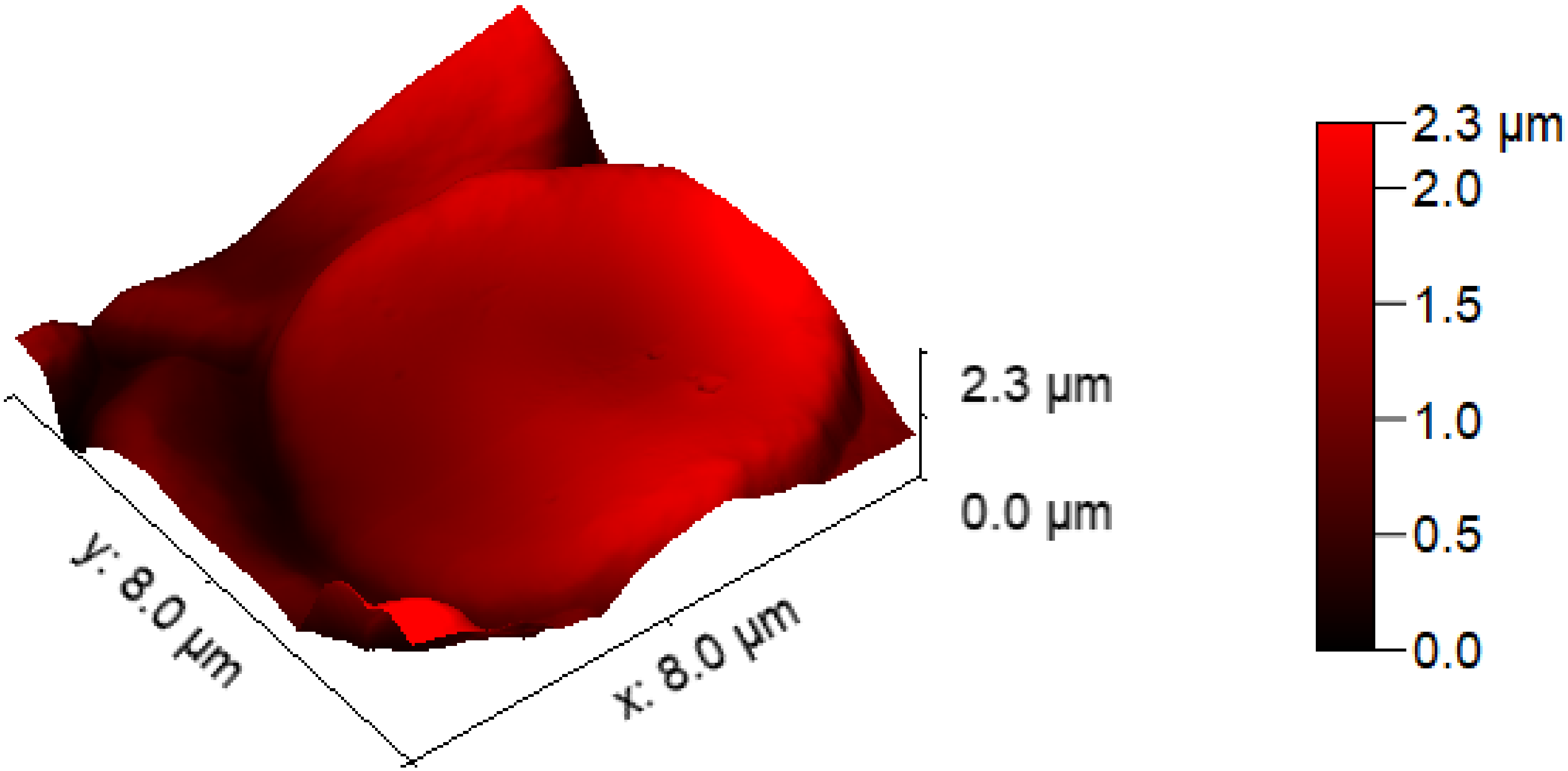
same image, different color ranges

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



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

Interpreting 3D Images

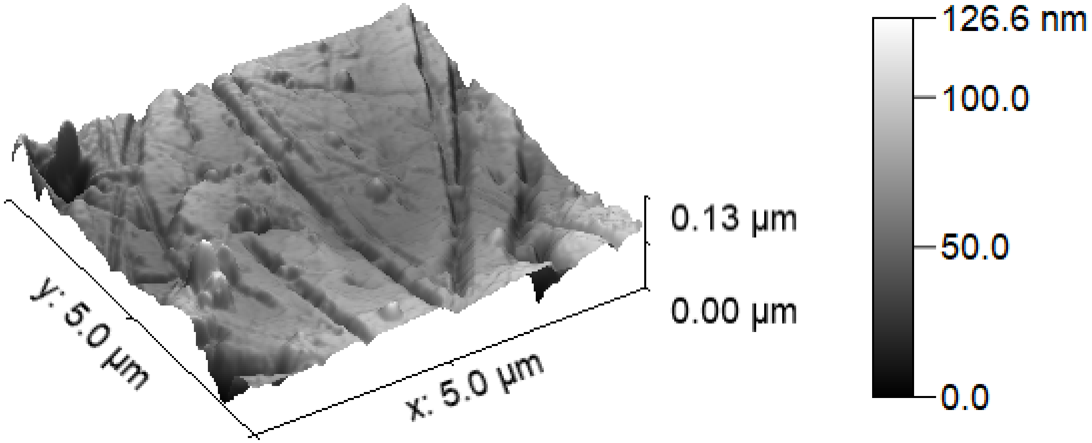


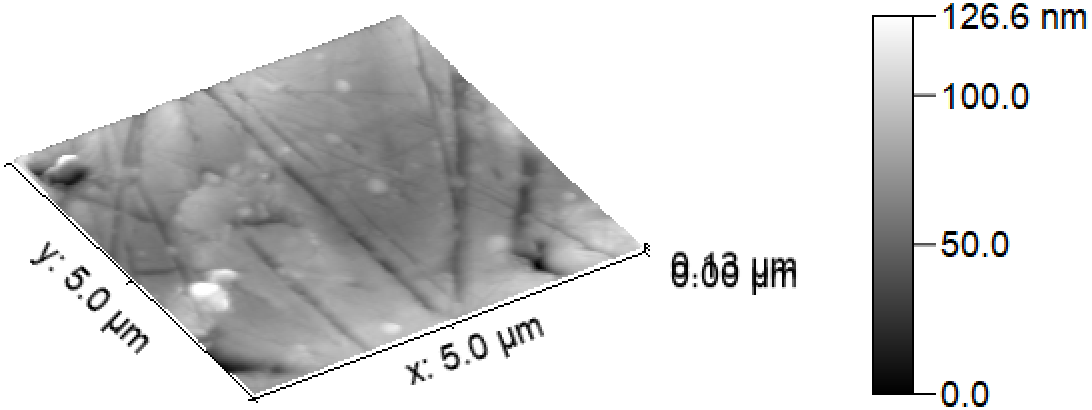
blood cells, 8µm x 8µm AFM topograph

Interpreting 3D Images

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

z often exaggerated compared to xy to **convey texture information**



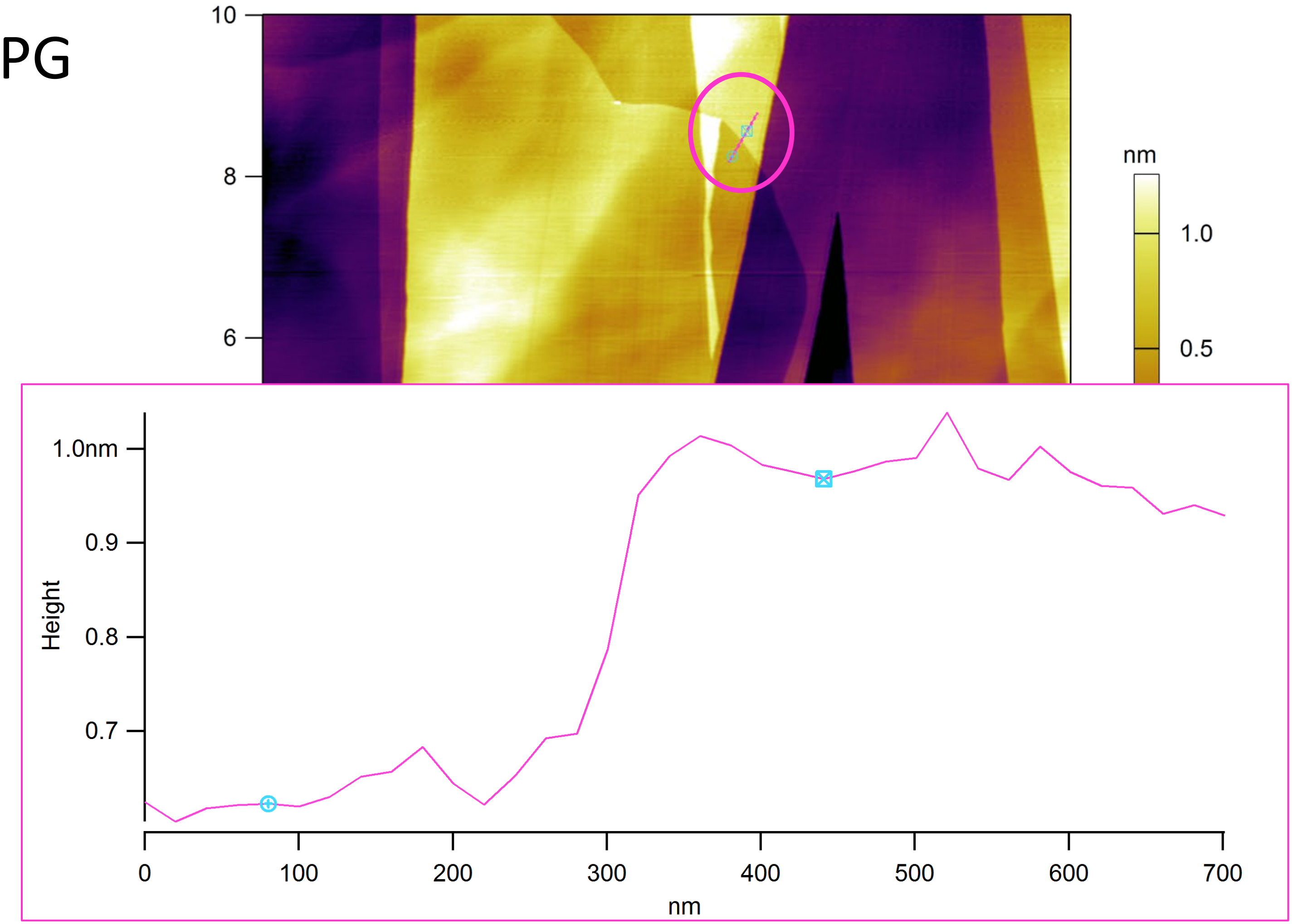


steel sample disk
5µm AFM topograph

(samples courtesy of
Physics 403 Lab 2022)

Application: Step Heights

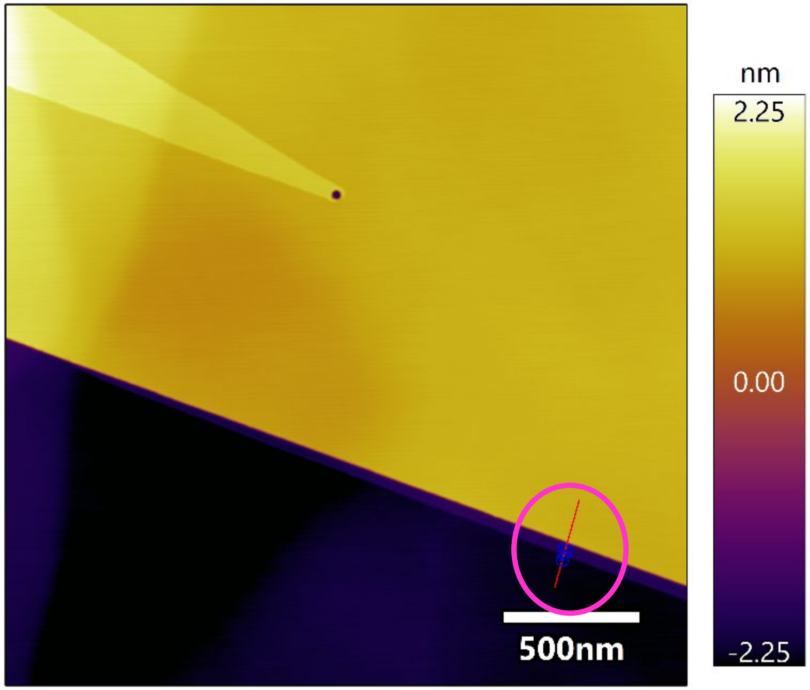
HOPG

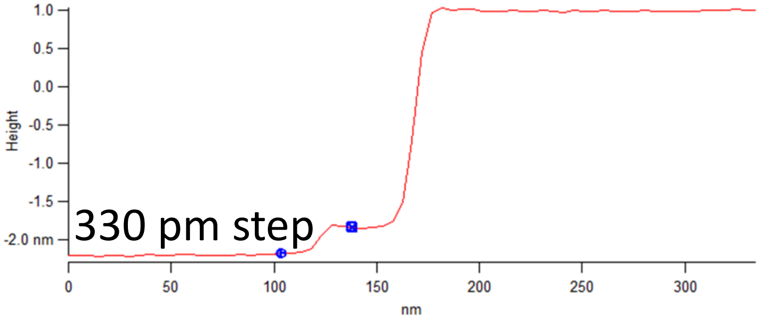


Application: Step Heights

HOPG

(highly oriented pyrolytic graphite)



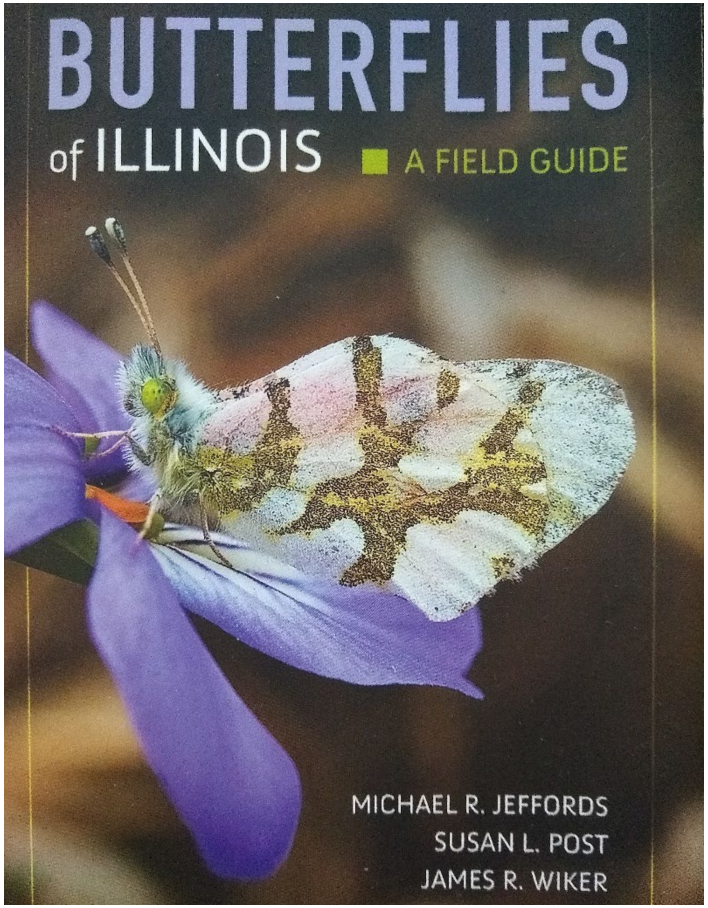


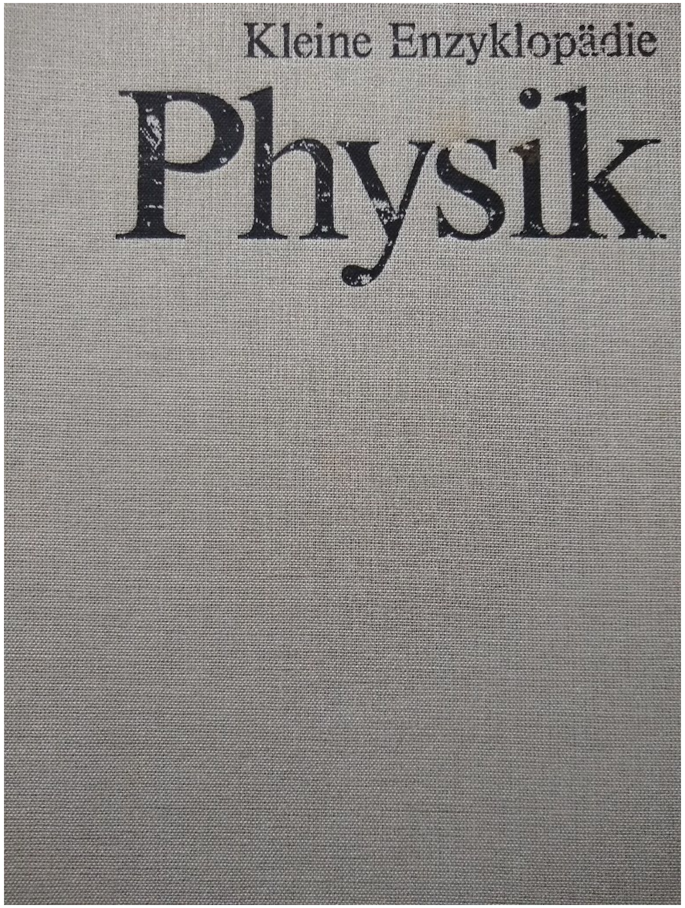
HOPG_0006HeightTraceMod0

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

Step Heights and Thicknesses

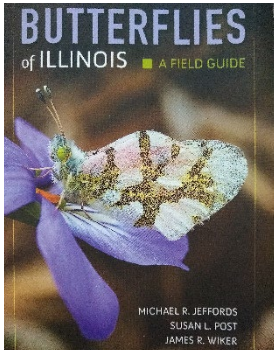
Which book is thicker?

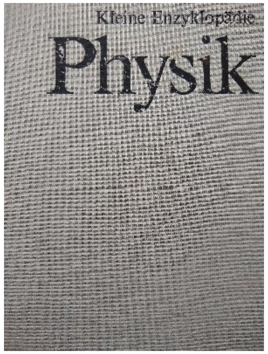


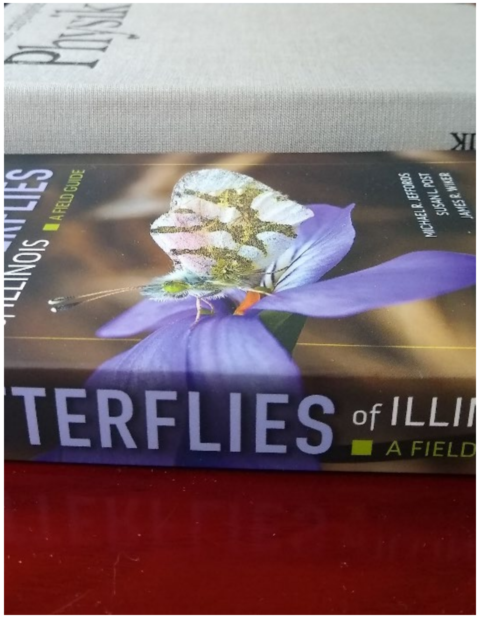


Step Height: Relative Height

- 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

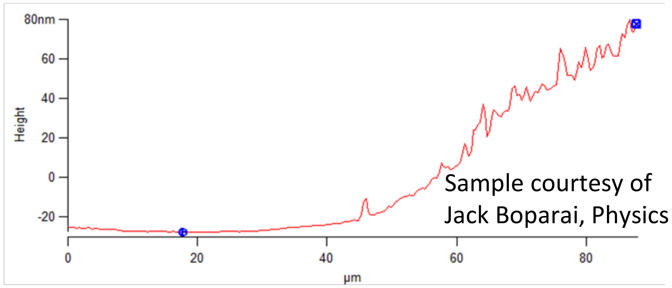






Step Height/Film Thickness: Complementary Techniques

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



Sample courtesy of Jack Boparai, Physics

- Stylus profilometry
- 3D optical profilometry

Need a height difference (step) like AFM

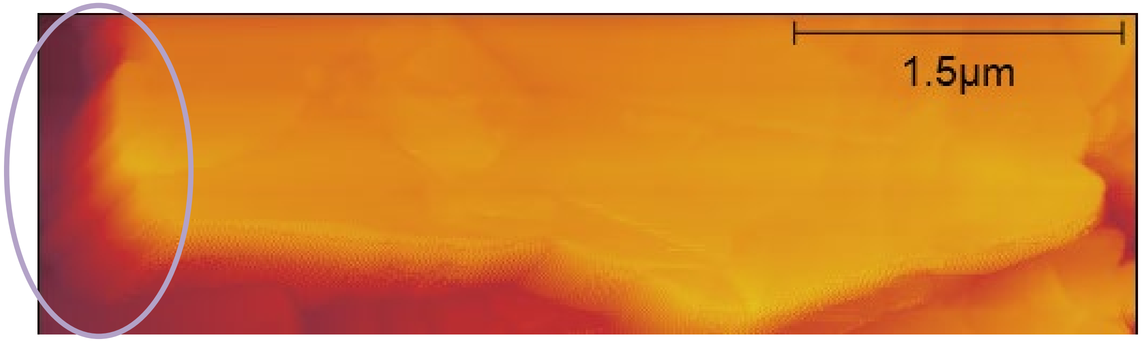
- X-ray Reflectivity (XRR)
- Rutherford Backscattering Spectrometry (RBS)

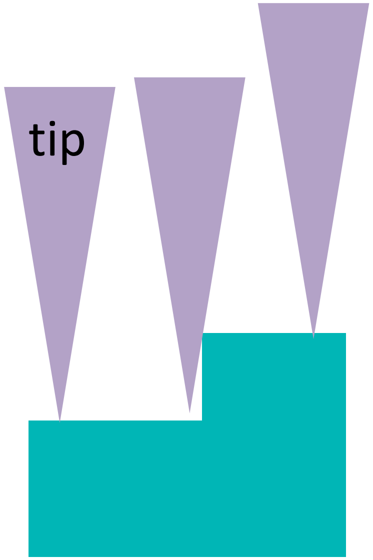
Continuous film (no steps)
May need to know density

Width Measurements

Beware of tip shape convolution

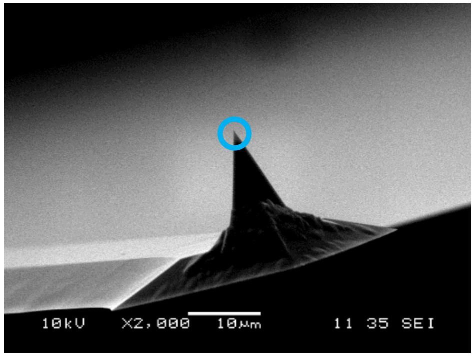
- As depth increases, tips get broader
- Steep drop-offs look less sharp
- High aspect ratio tips are available

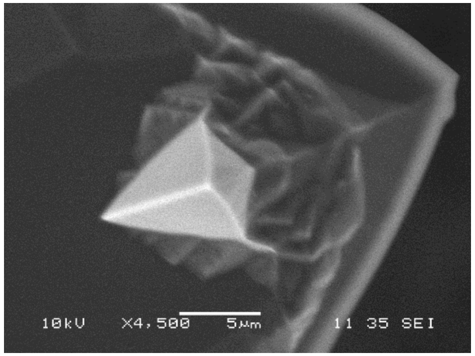


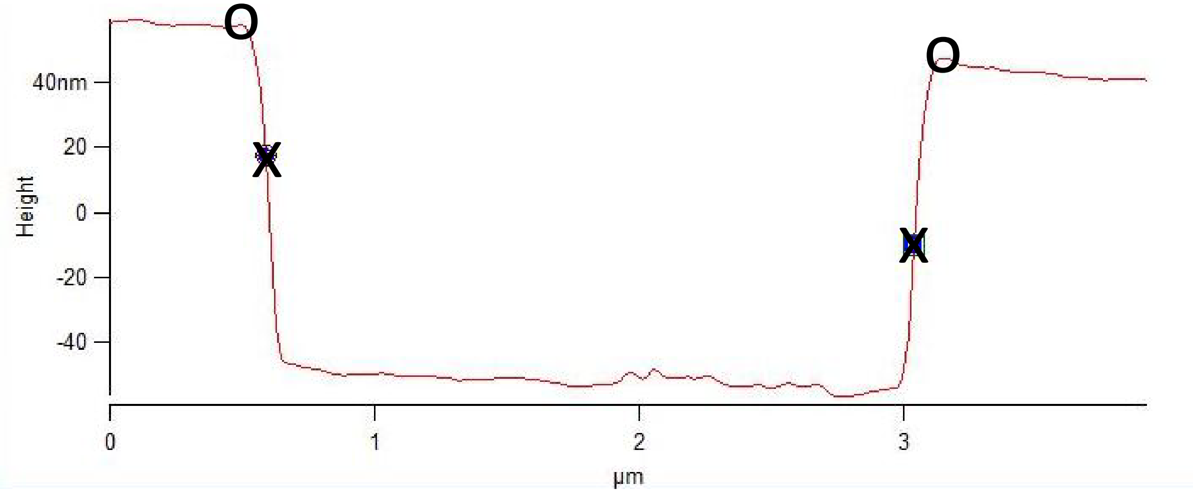


Width Measurements

- 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?

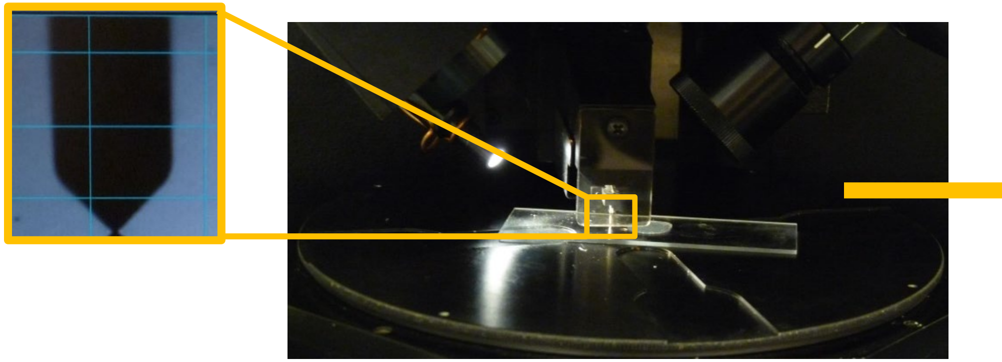
Michael Jeffords and Susan Post, University of Illinois Prairie Research Institute
https://photojournalingm-s.smugmug.com/Colorado-and-Kansas/i-3tJ3DZk/A

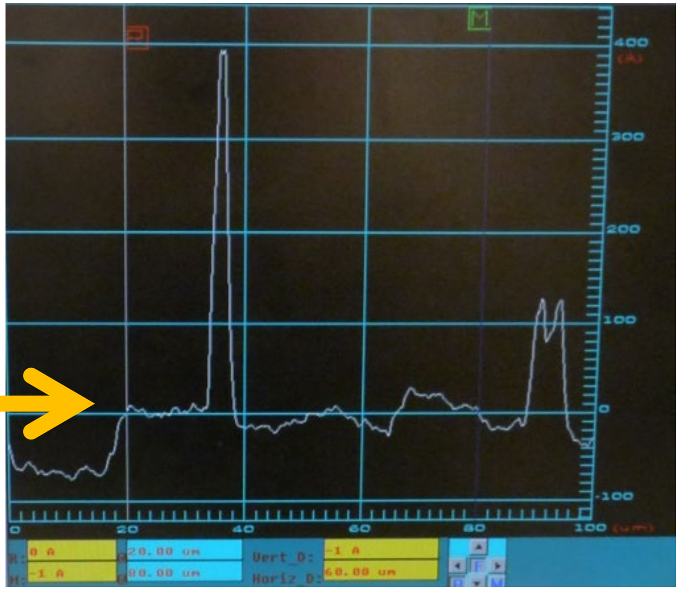
Complementary: Stylus Profilometry

2D stylus profilometry

(line profiles)

(diamond tip)

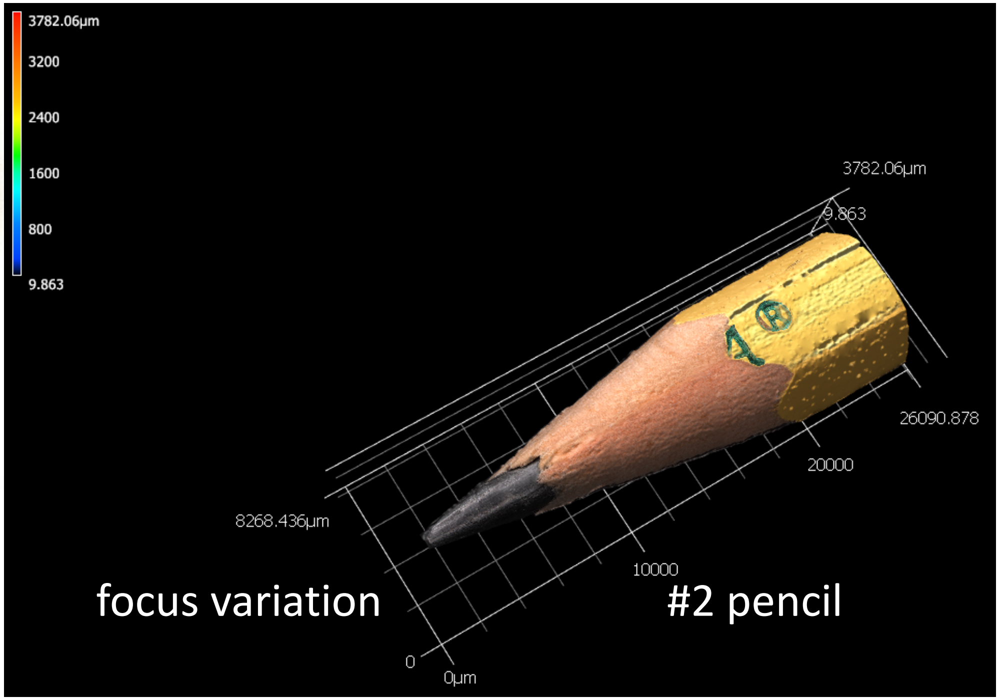




Complementary: Optical Profilometry

go.illinois.edu/MRL3DOpticalProfilometry

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



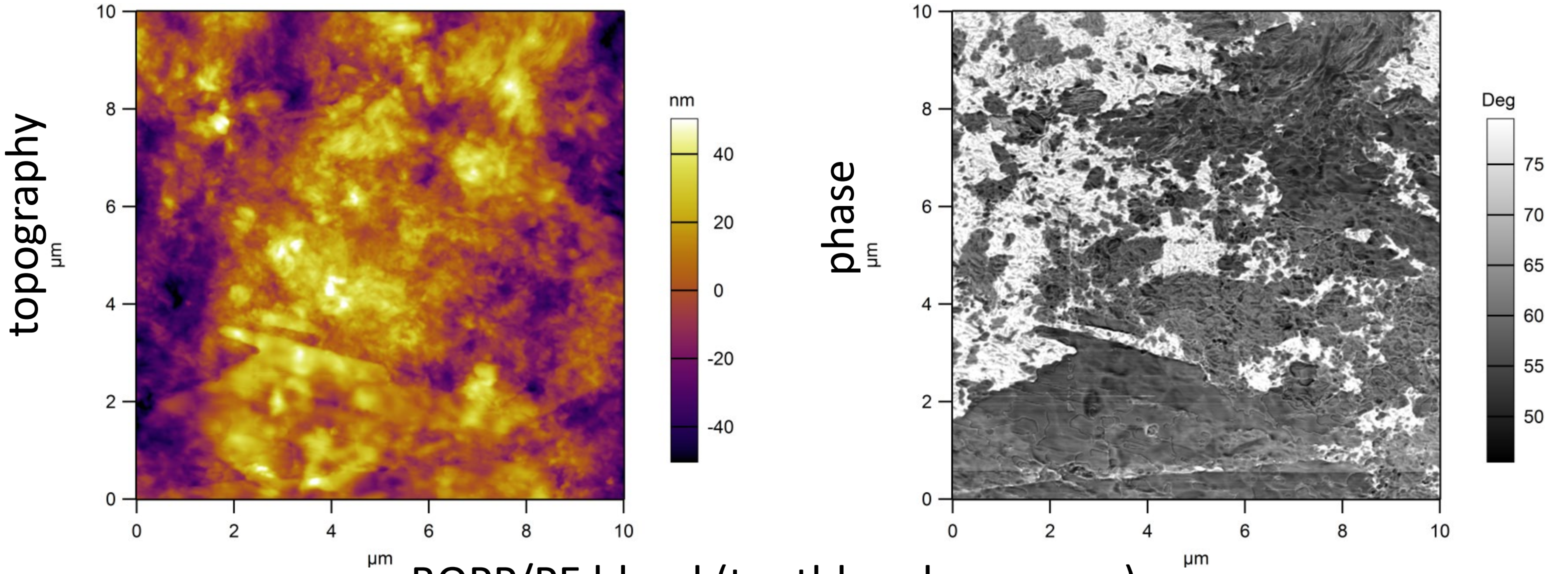
Qualitative Comparison

	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)

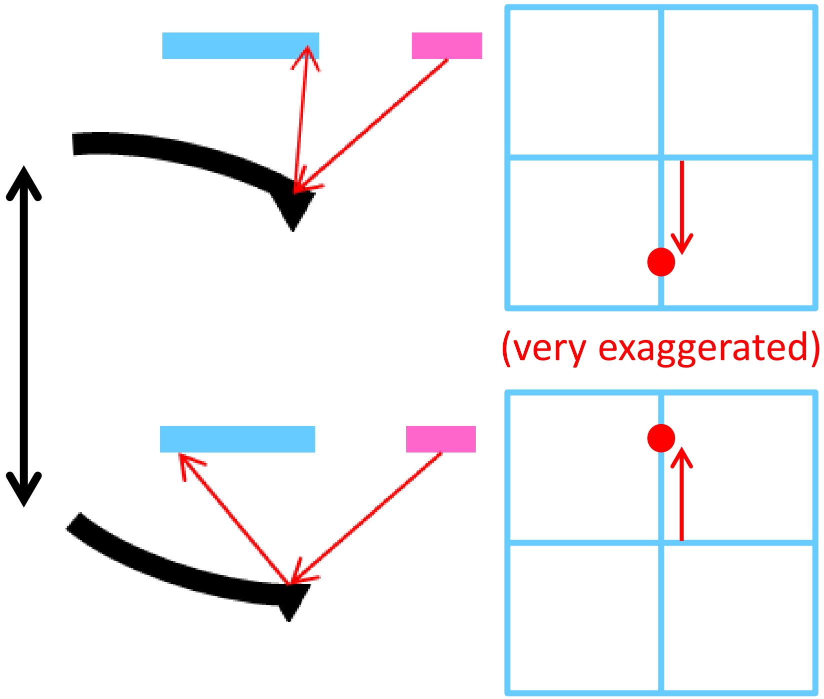


BOPP/PE blend (toothbrush wrapper)

Tapping Mode Imaging: Phase

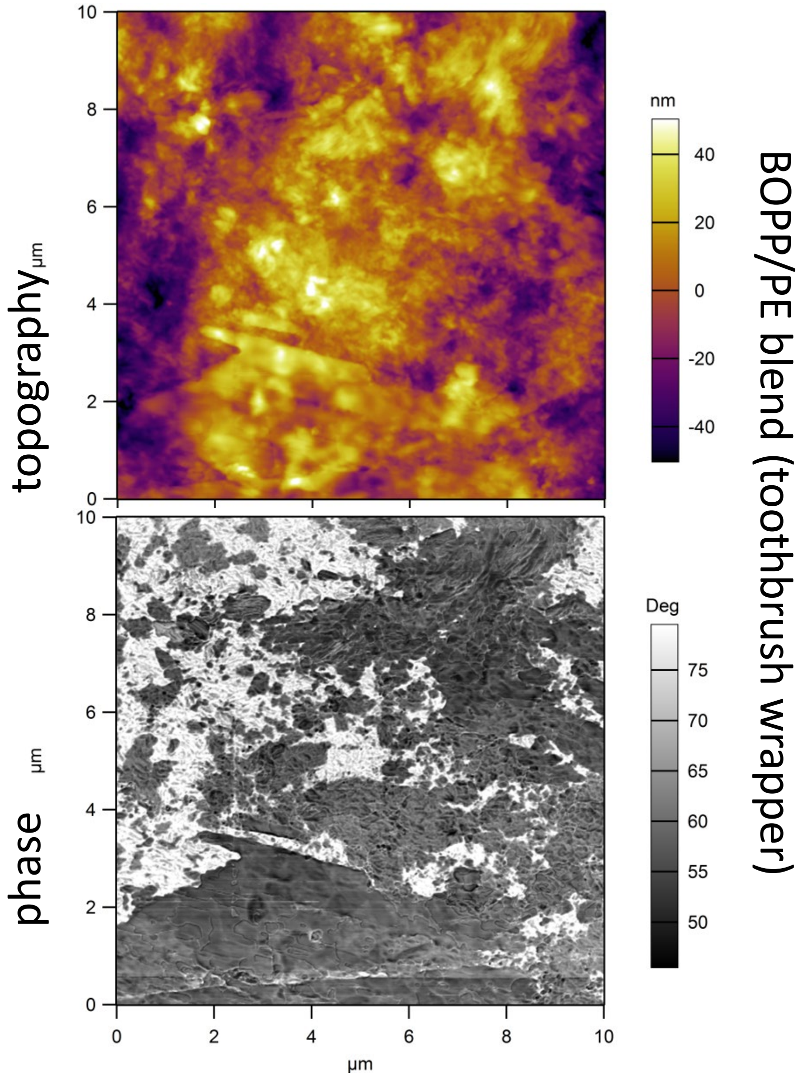
- Oscillating cantilever
- Tip—surface interactions affect oscillation
  - – Cantilever driven sinusoidally to keep a constant amplitude
  - – Dissipative interactions cause a phase lag (delay)
    - Viscous areas
    - Sticky areas

tip oscillates really fast (tens of kHz to MHz)



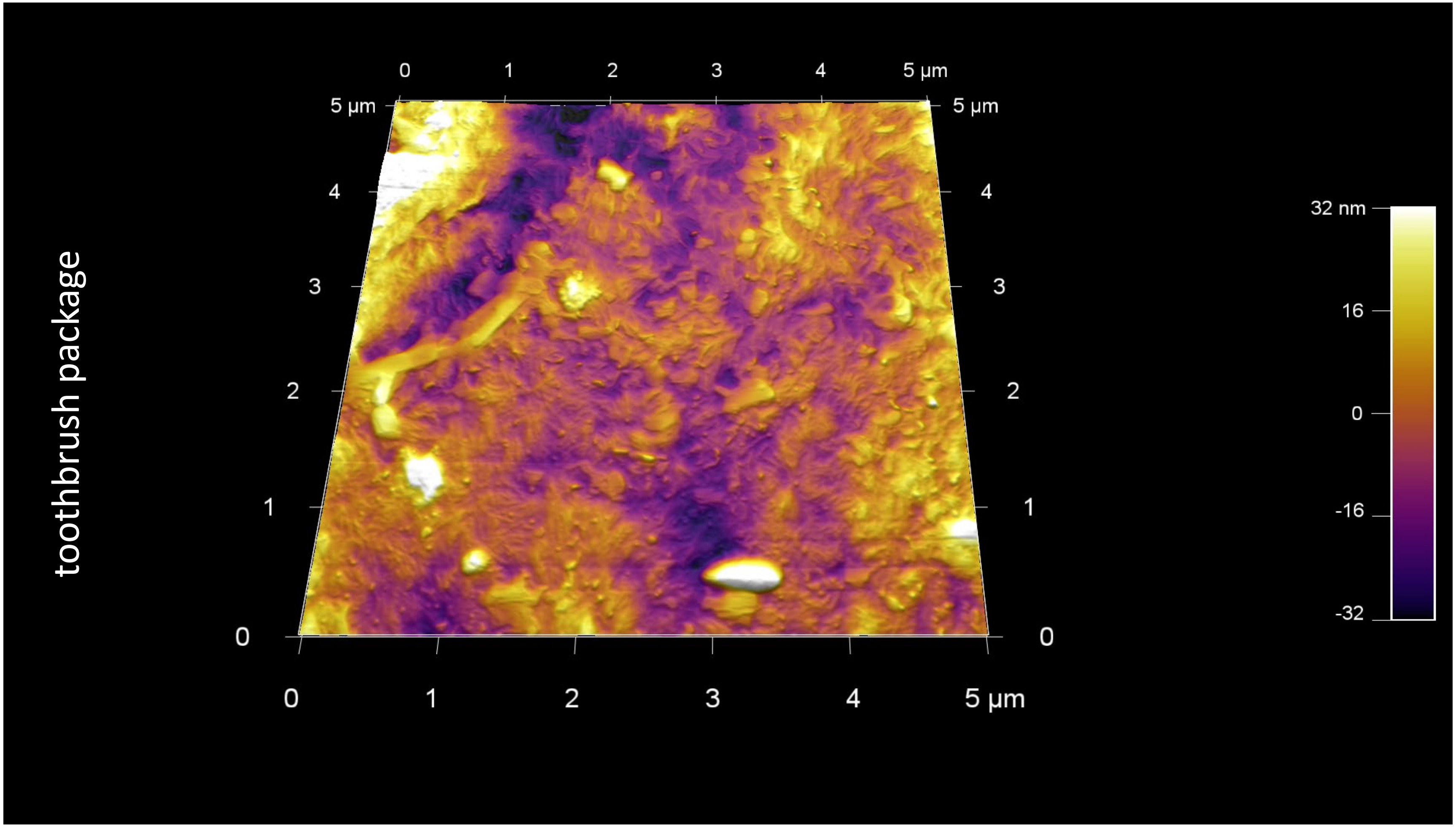
Phase (Qualitative)

- 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



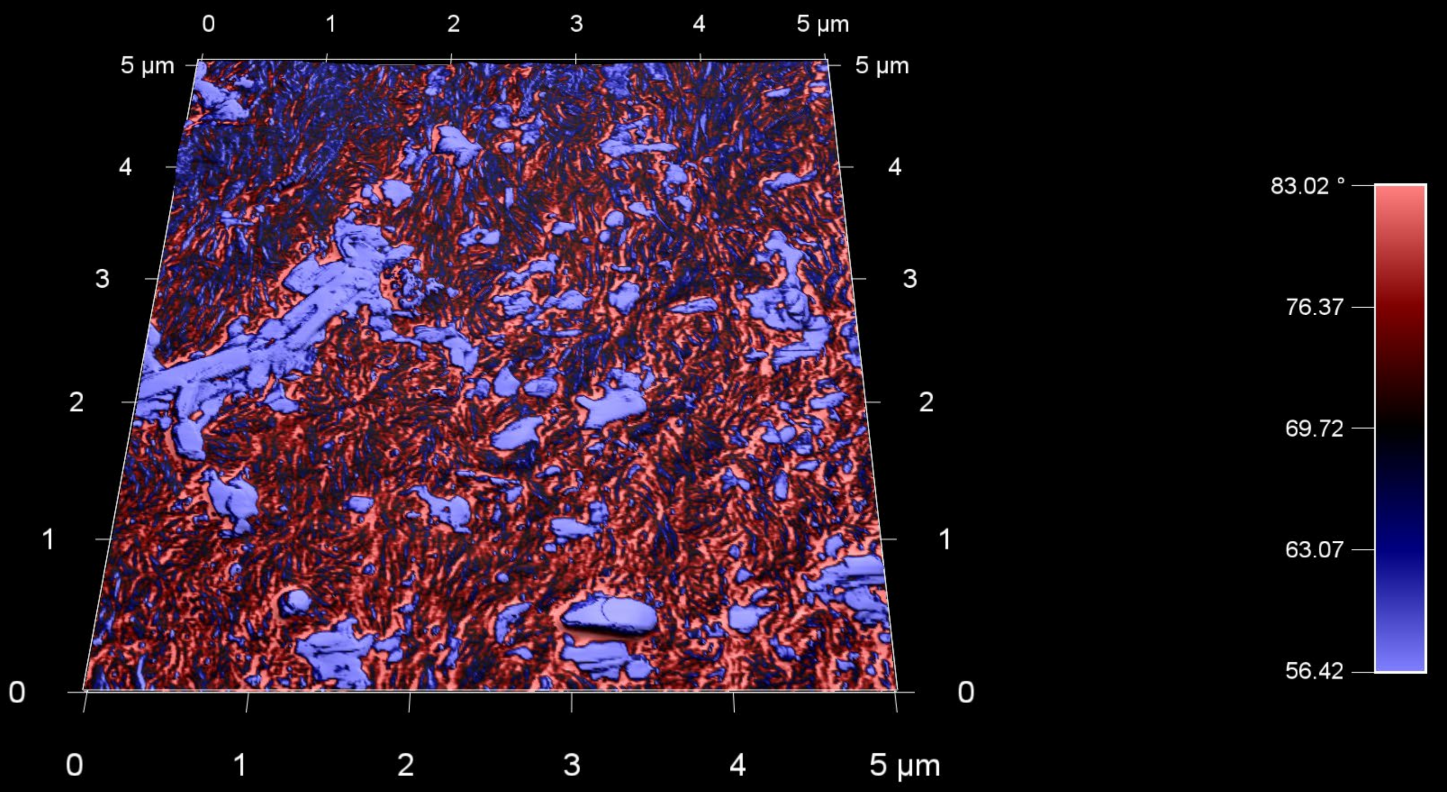
Kathy Walsh, Atomic Force Microscopy, Physics 403, 10/29/24

Topography


toothbrush package
0
1
2
3
4
5 µm
0
1
2
3
4
5 µm
5 µm
0
1
2
3
4
5 µm
0
1
2
3
4
32 nm
16
0
-16
-32

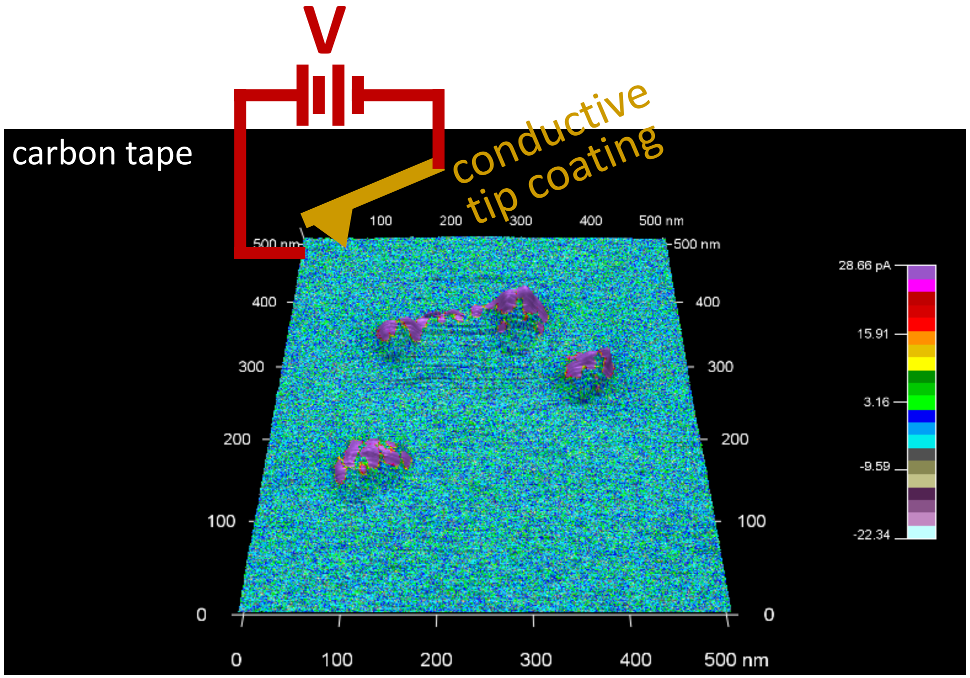
Topography with Colors from Phase

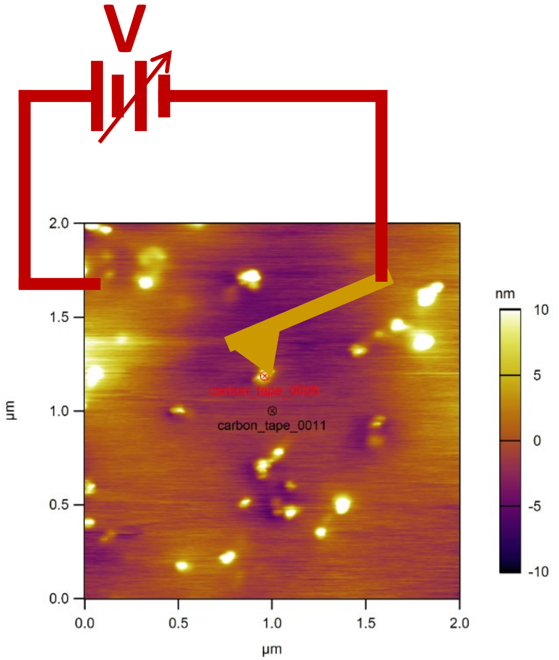
toothbrush package

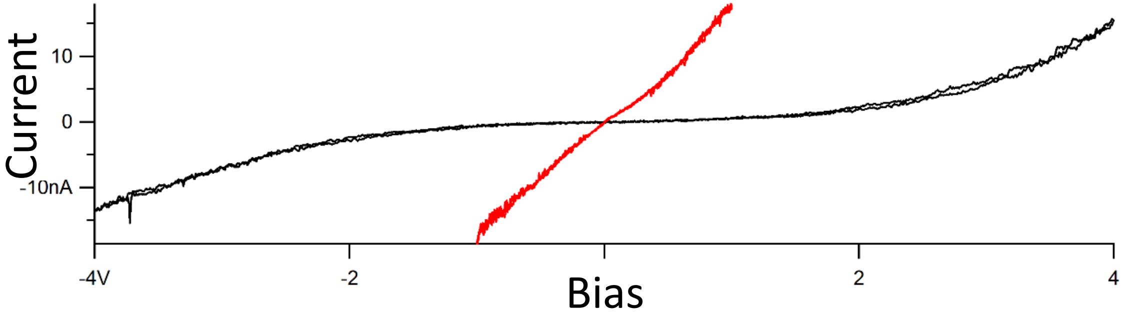


phase colorscale overlaid on 3D topography
redder areas are more dissipative

Common Application: Conductive AFM







Artifacts and Image Processing

- Recognize what is (or is not) informative about the sample

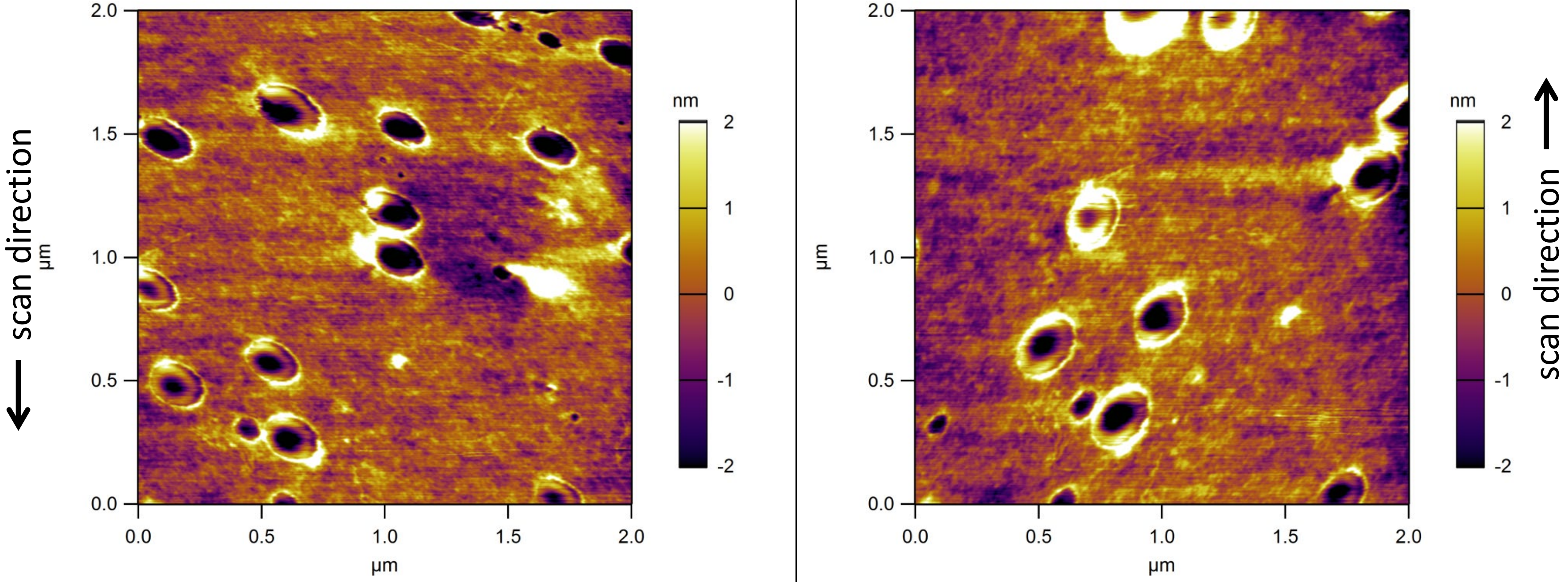
- Images may not always exactly represent the sample: sample drift, tip condition/geometry

- Display data in an informative way
  - Correct for sample tilt, etc.

Sample Drift

Scanning downwards…

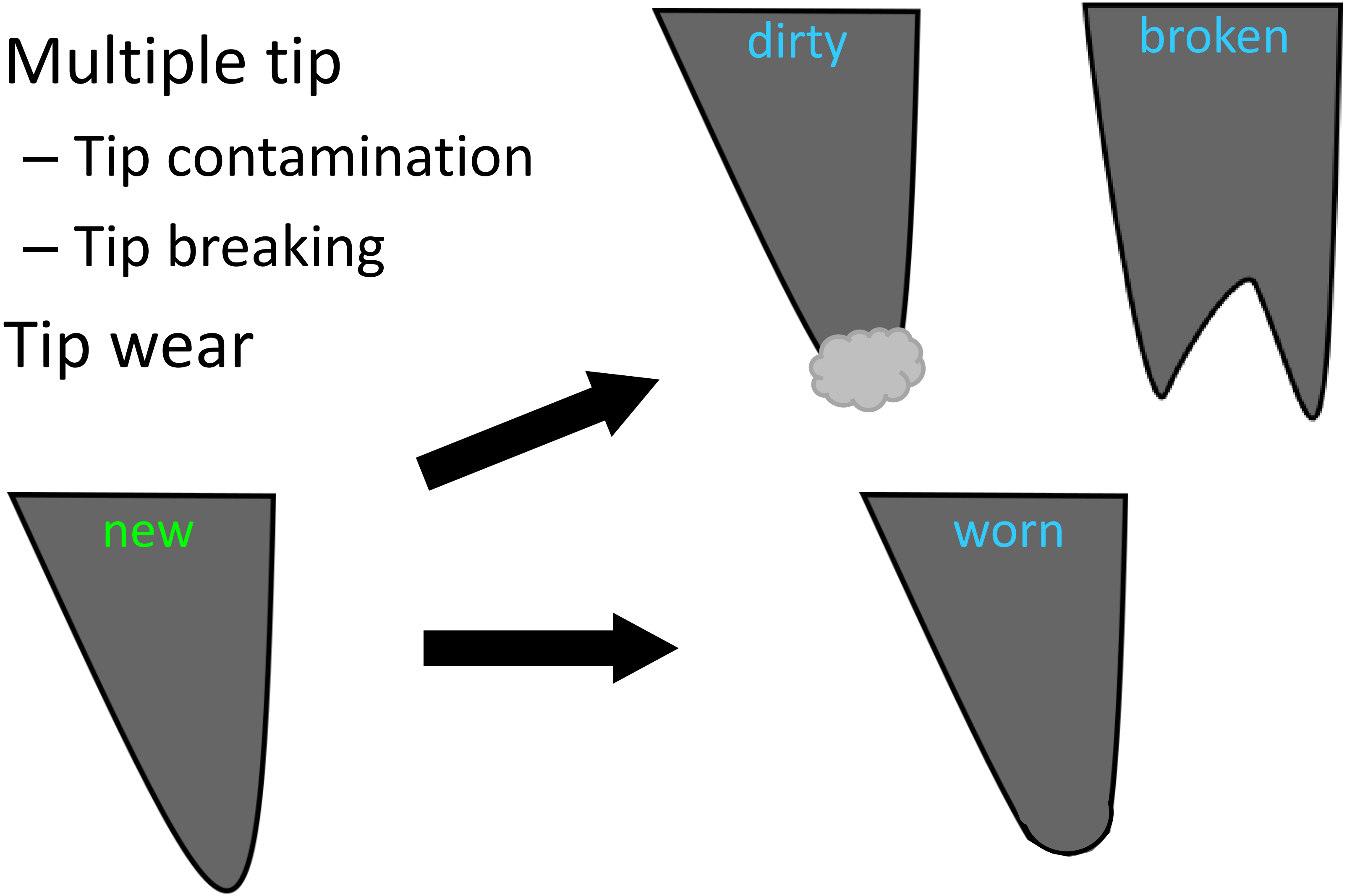
… then scanning upwards



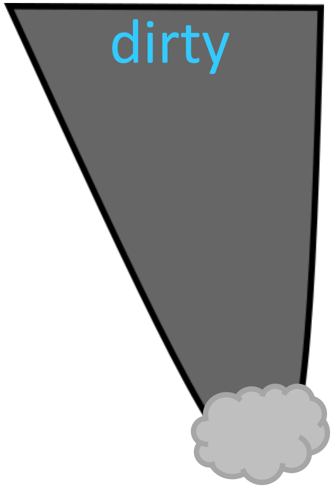
chewing gum

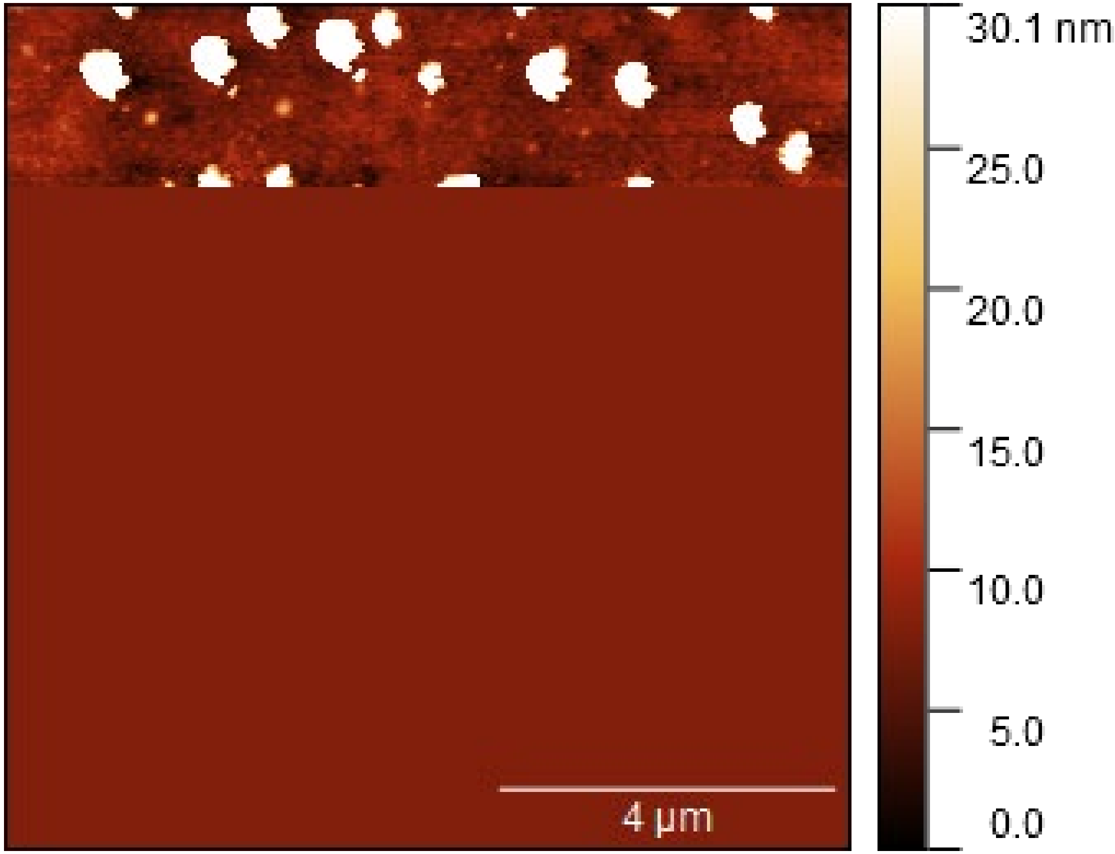
Tip Artifacts

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



Contaminated Tip

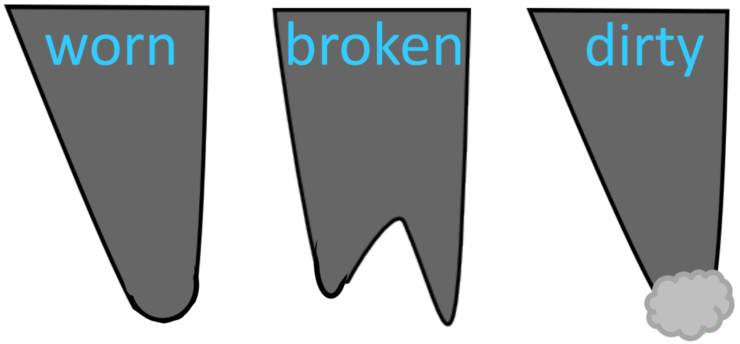




10μm partial scan

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

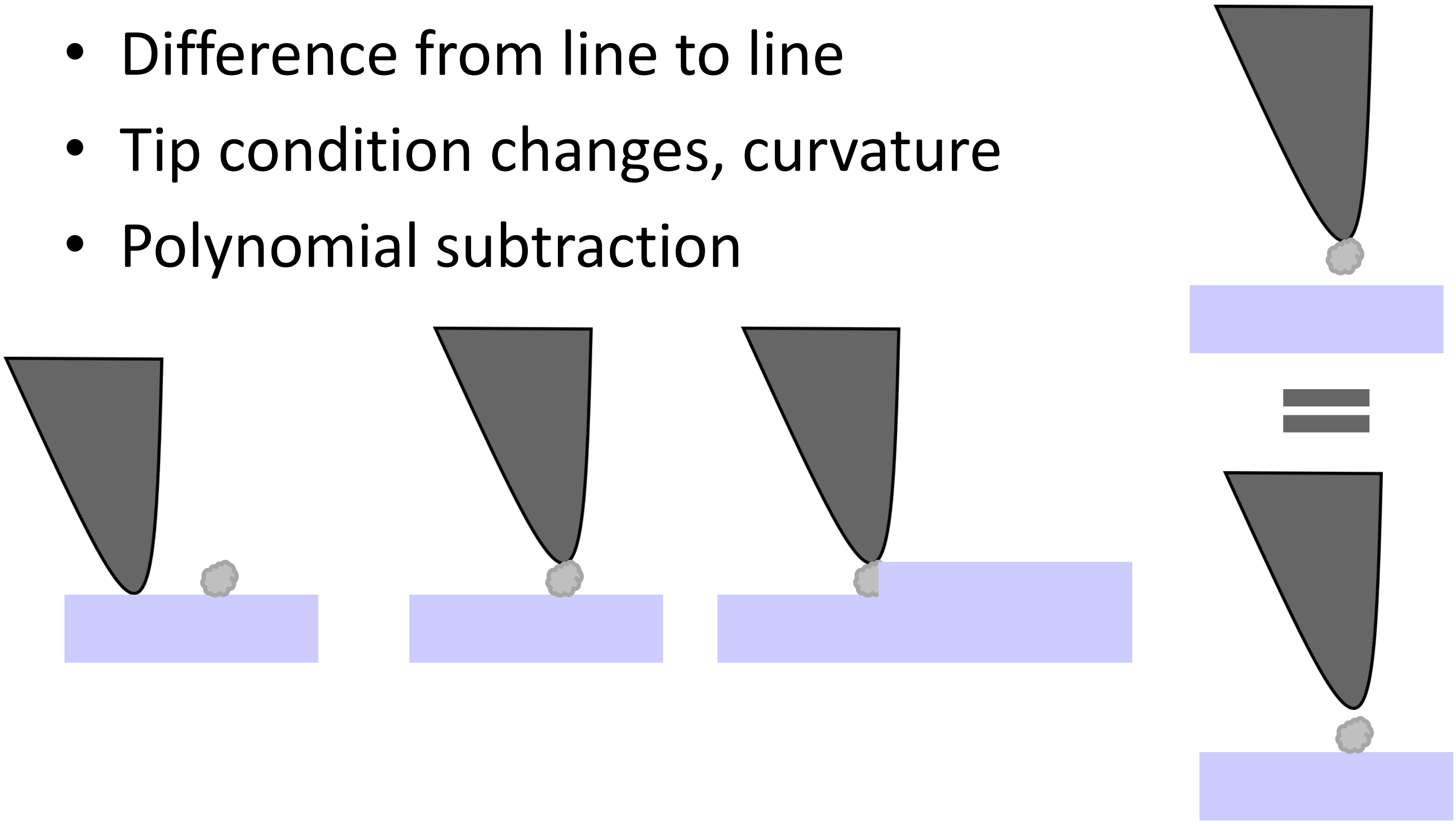


Image Processing

raw image

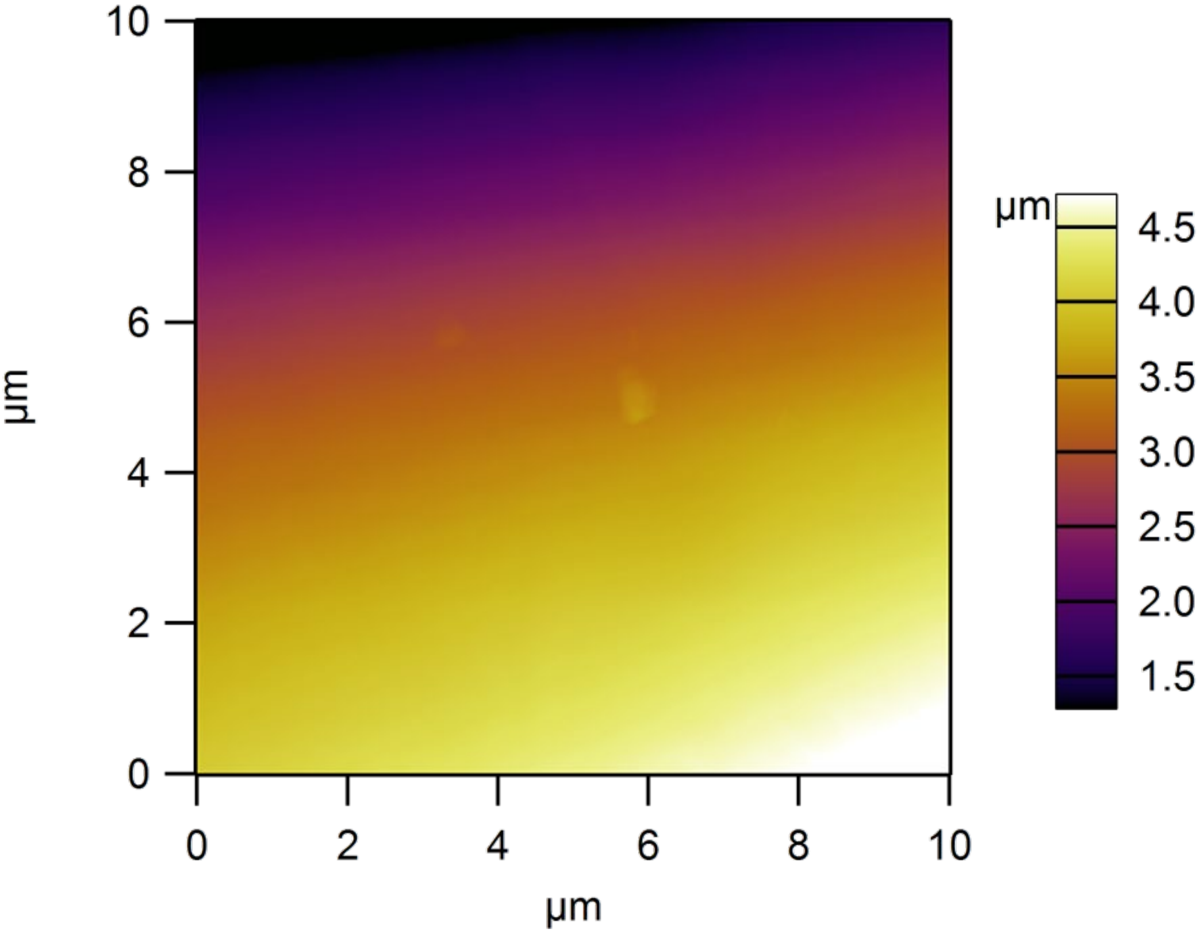


Image Processing

line subtraction

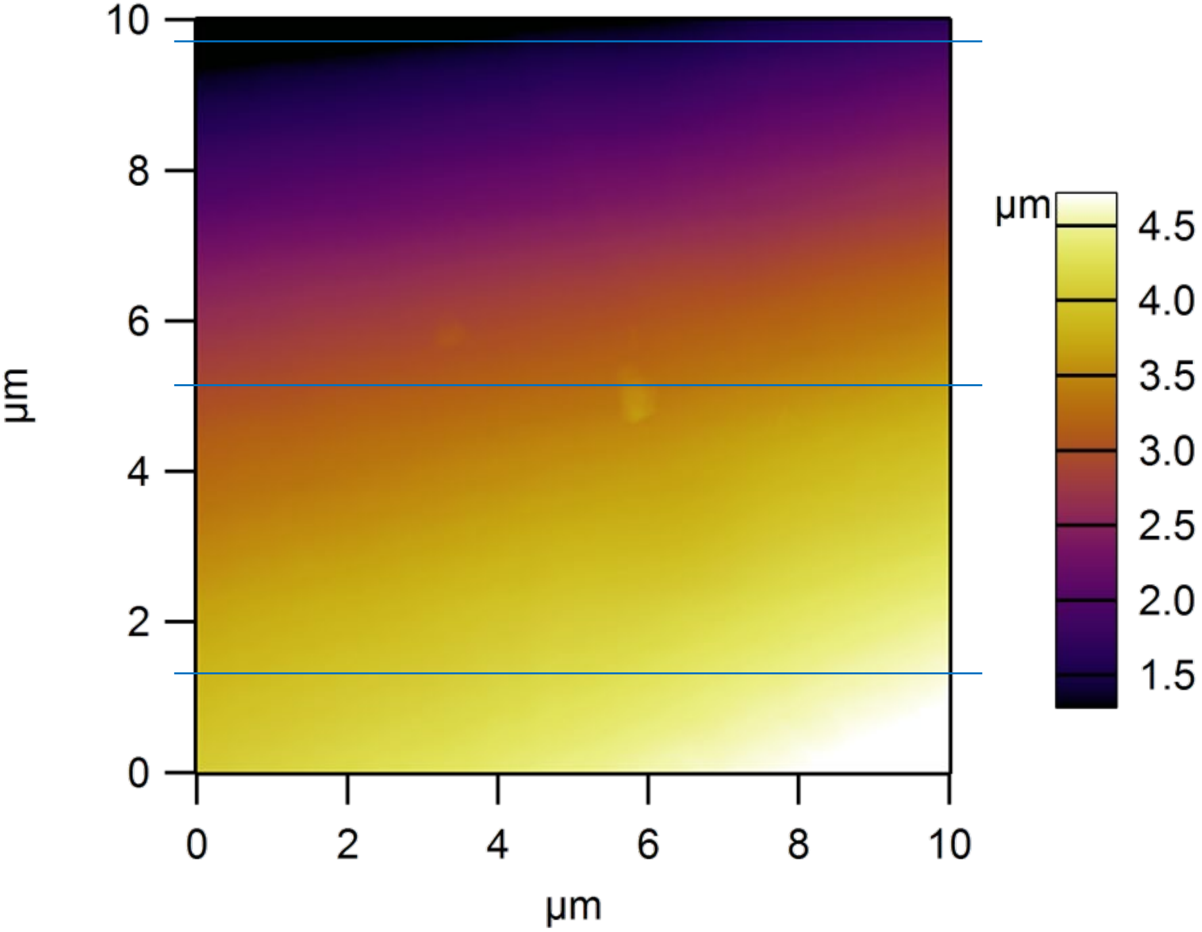
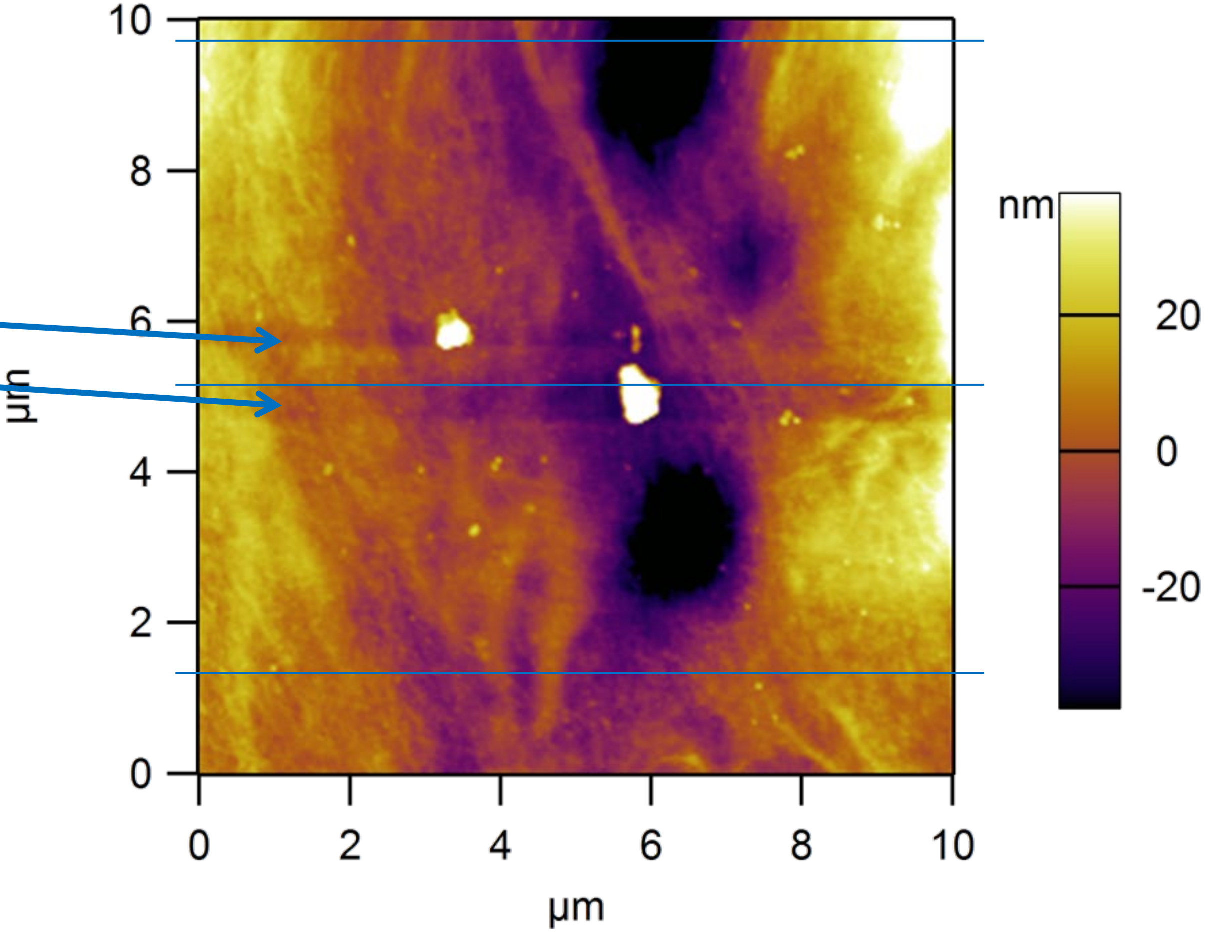


Image Processing

line subtraction



artifactually lowered lines

Image Processing

line subtraction:
mask outlier areas

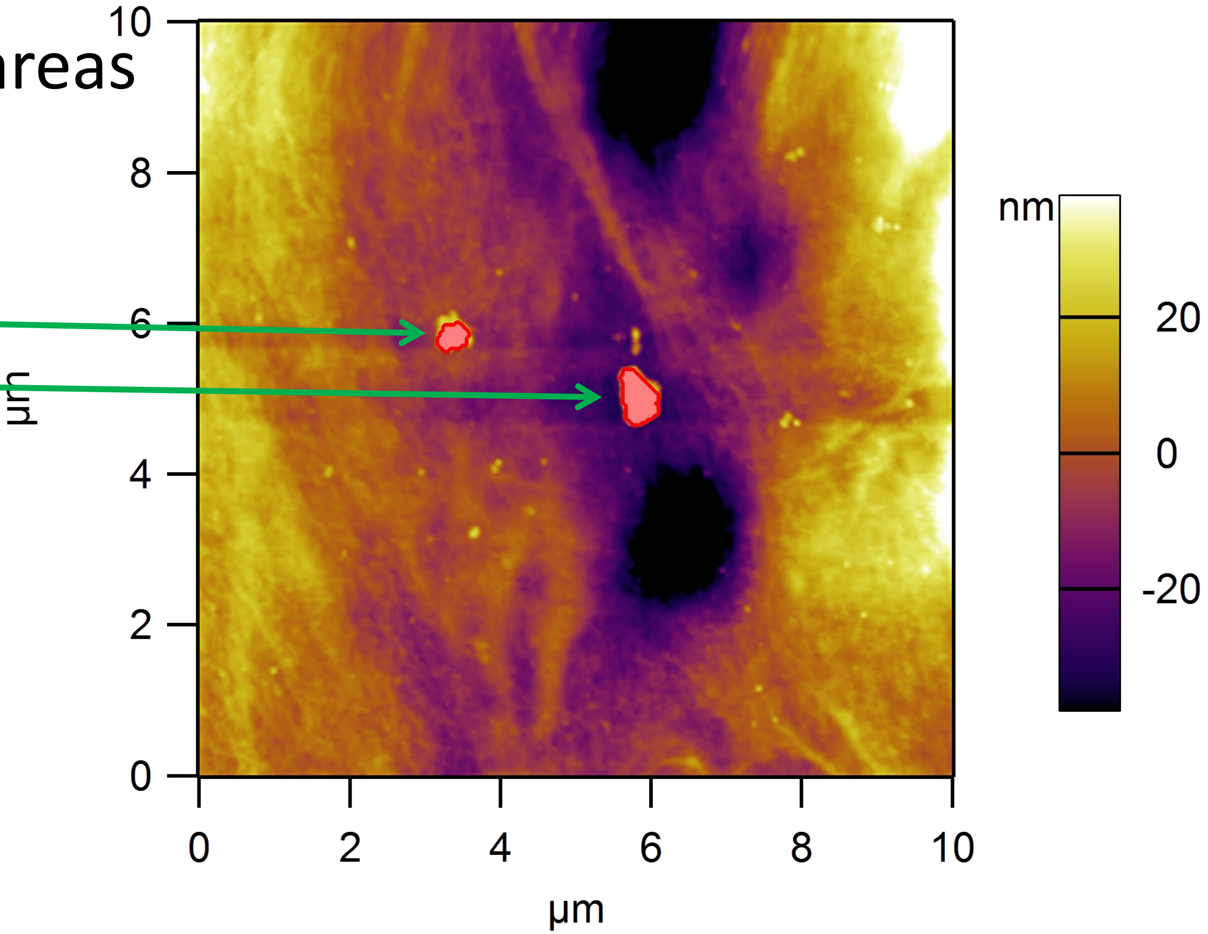
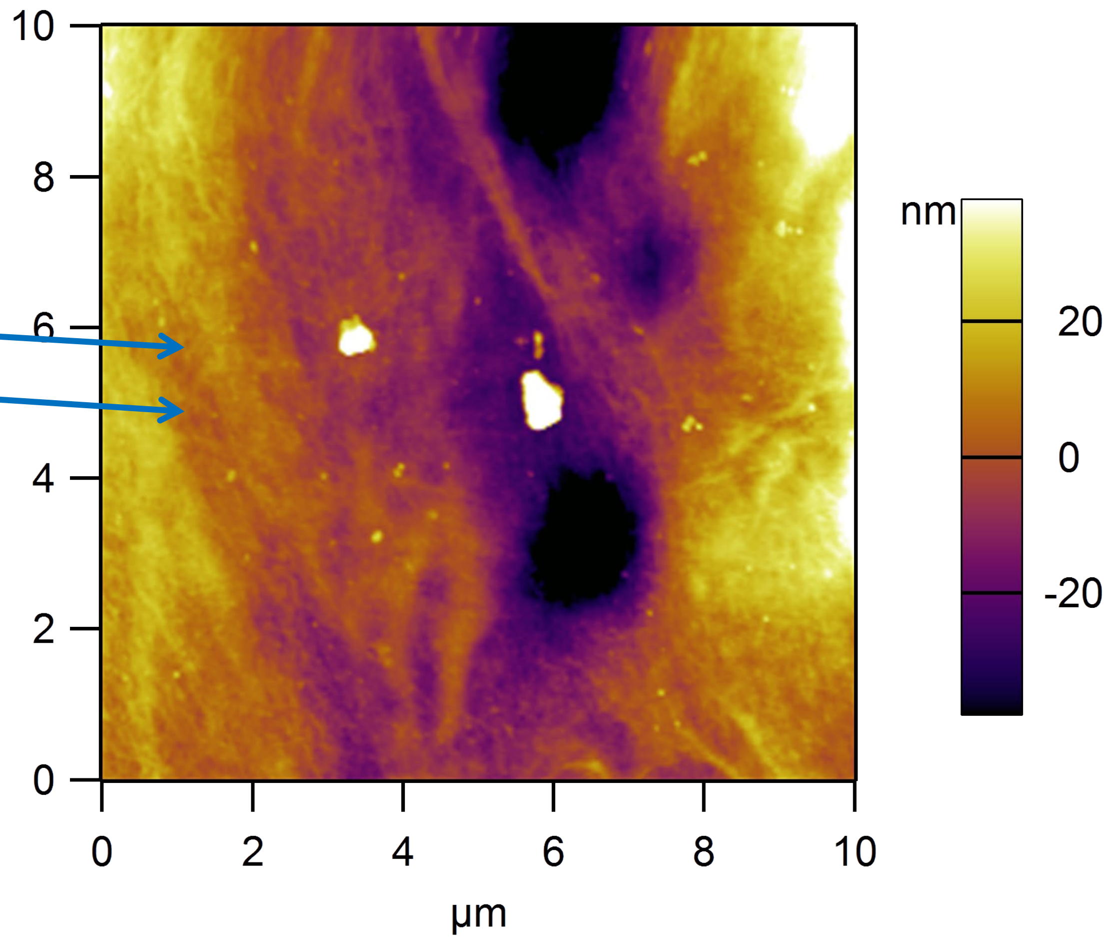


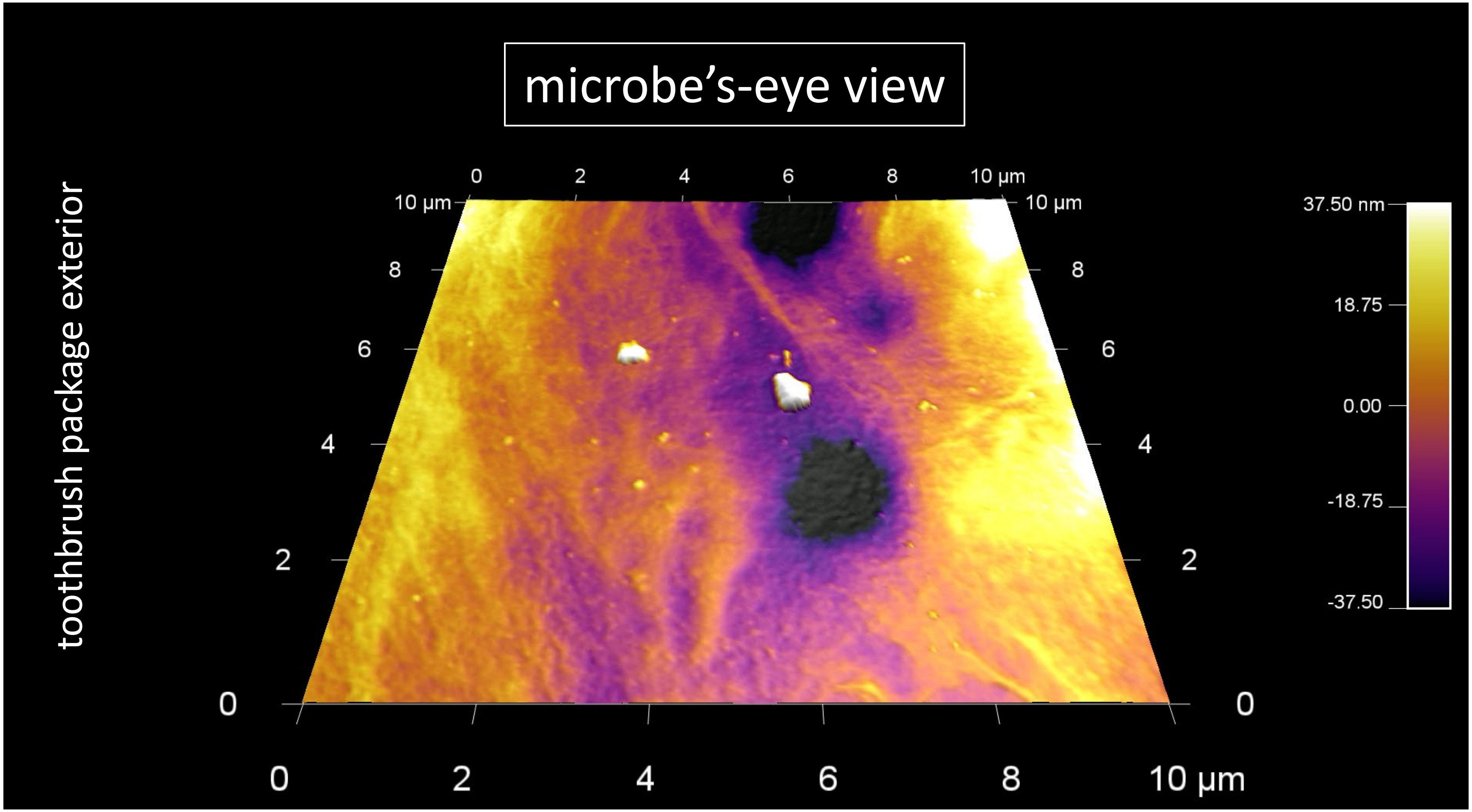
Image Processing

line subtraction:
masked flatten



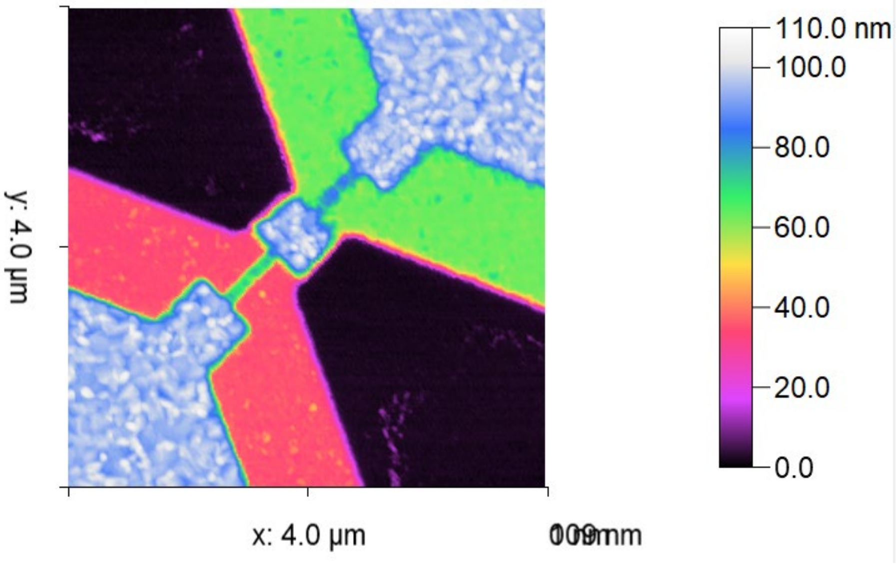
no more
streaks

3D Display

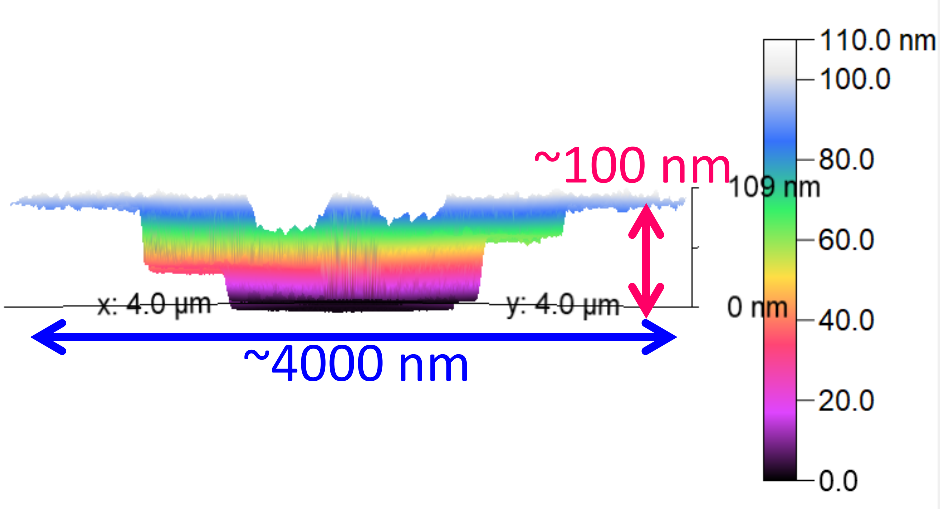

microbe’s-eye view
toothbrush package exterior
0
2
4
6
8
10 µm
10 µm
8
6
4
2
0
10 µm
8
6
4
2
0
0
2
4
6
8
10 µm
37.50 nm
18.75
0.00
-18.75
-37.50

3D Display

face on

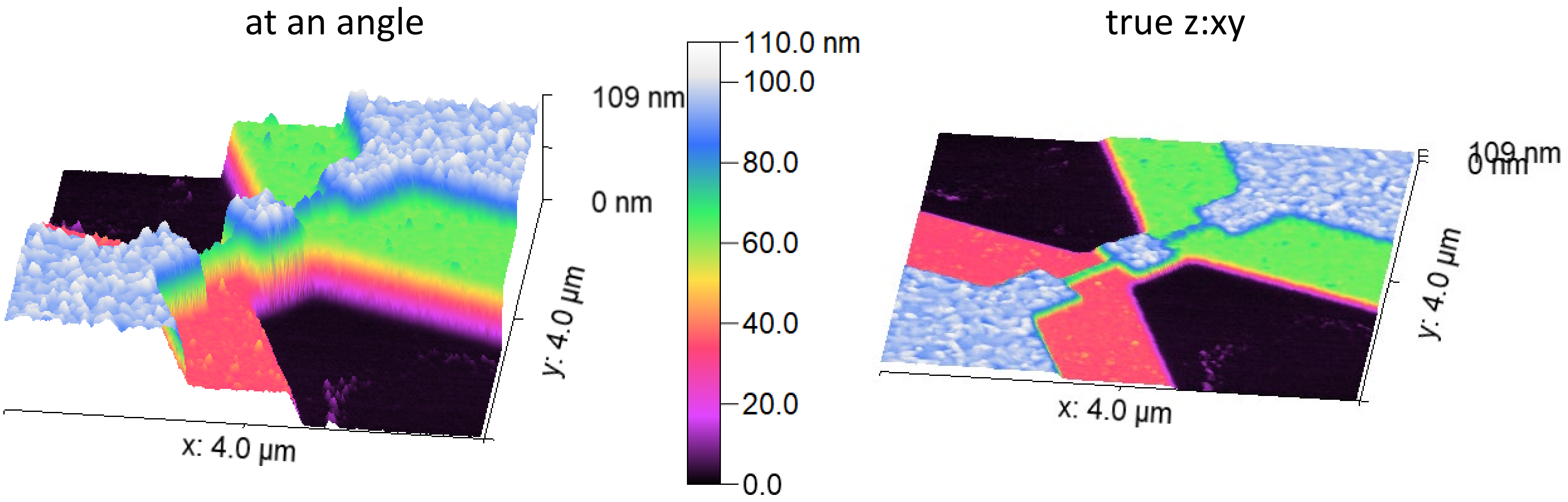


side view



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

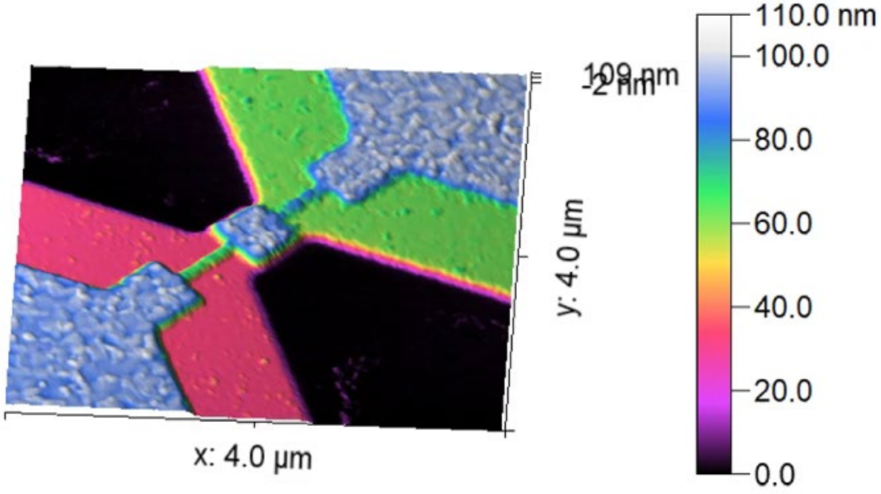
3D Display—z:xy

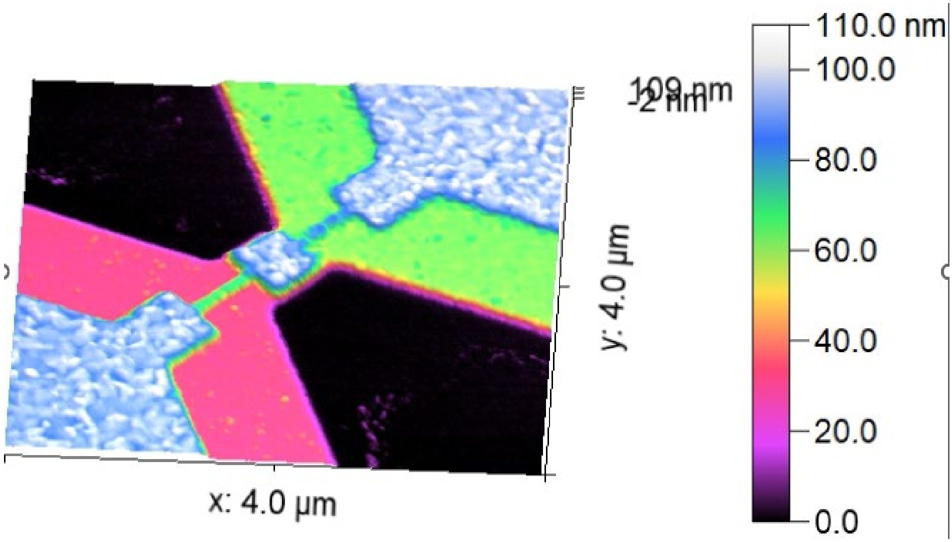

at an angle

true z:xy

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

3D Display—Lighting Angle





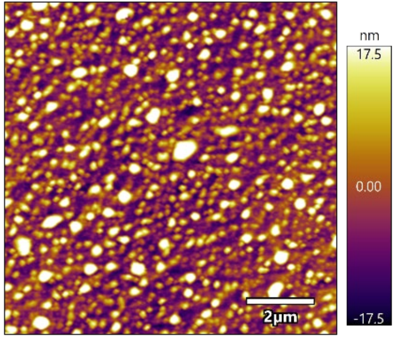
raw data courtesy of Ale Baptista,
Anton Paar Tosca 400 AFM

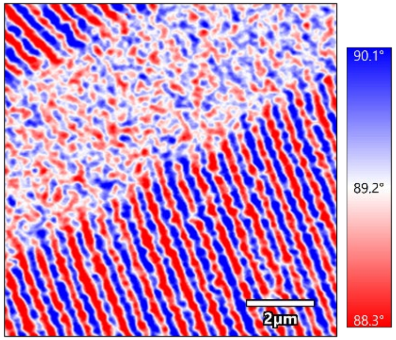
Many Other Applications

- 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



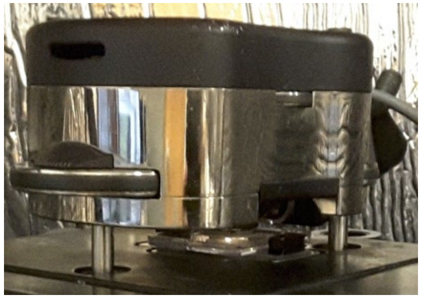


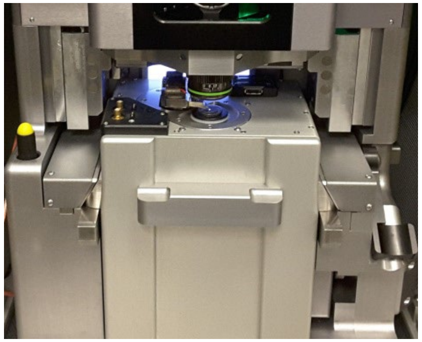
MFM phase

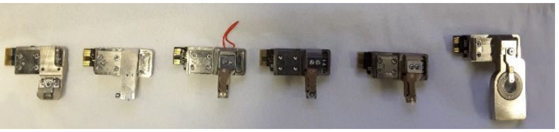
Attachments on the MRL AFMs

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

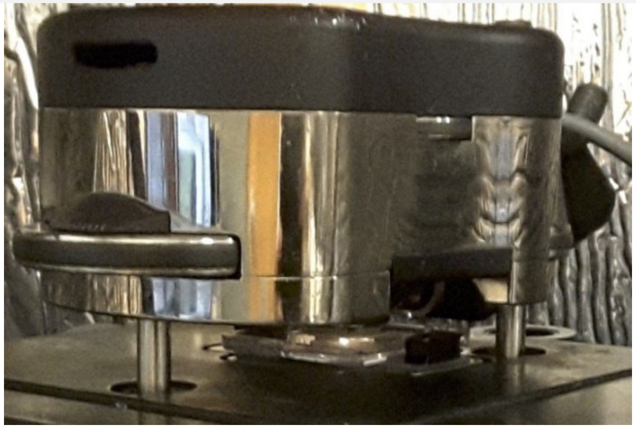




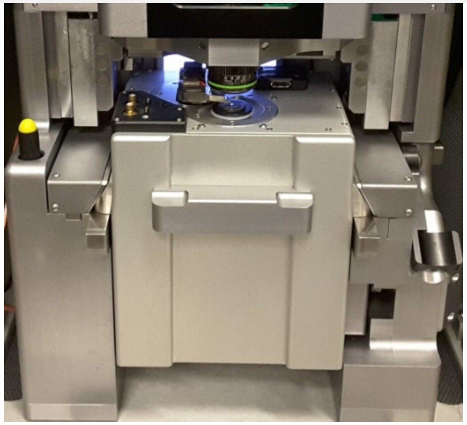


MRL AFMs—B12 MRL

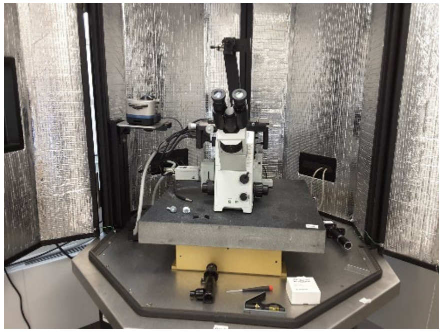
Asylum Research MFP-3D-SA (2 of these)
15µm z range, 90µm x 90µm scan size



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



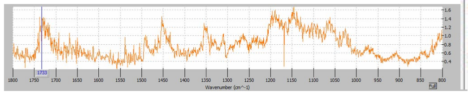
0026 Supercon: Asylum Research MFP-3D-Bio
on an inverted optical fluorescence microscope

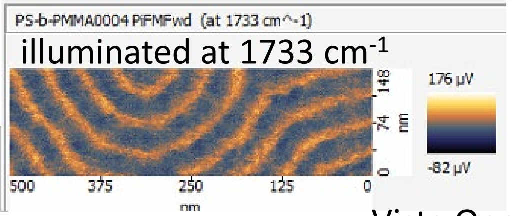


Related Instruments at MRL

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





Vista One

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