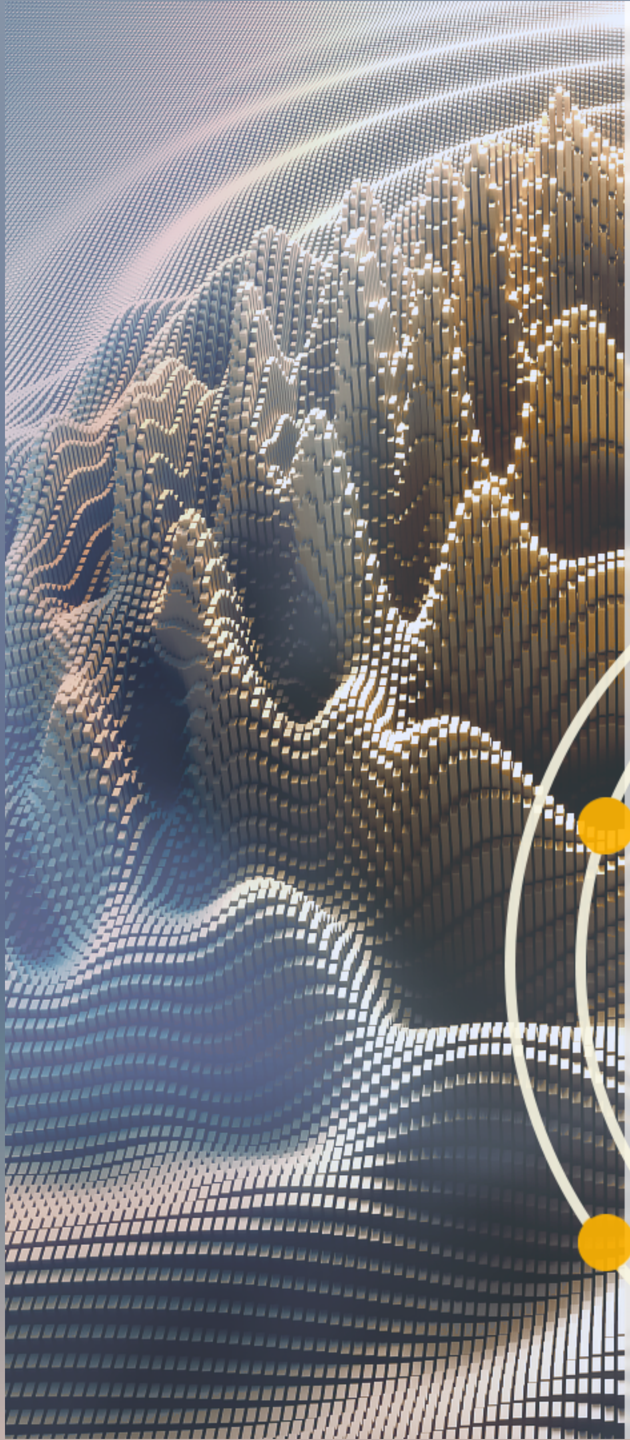


STYLUS PROFILOMETRY / ATOMIC FORCE MICROSCOPY (AFM)

Techniques for High-Resolution Surface Metrology & Imaging: A User's Guide

COVALENT ACADEMY

INTRO TO SURFACE CHARACTERIZATION PART 3 / 3

RSVP at:
https://bit.ly.covalent04

Covalent Metrology

- **Founded 2016**
- **Testing, measurement & characterization Platform**
- **30 team members (13 PhDs)**
- **9,500 ft² lab in Sunnyvale**
- **1-Stop-Shop Source for Answers**
  - 30 instruments in-house
  - 6 partnerships with instrument makers
  - 11 partner labs
  - 6 corporate "tool shares"
- **>275 customers** (80% repeat)

Covalent Technical Groups and Organization

hello@covalentmetrology.com

Electron & Scanning Probe Microscopy

(SEM/TEM, AFM, EDS, EELS, Nanoindent, acoustic microscopy)

Surface Analysis

(XPS, Auger, ToF-SIMS, dSIMS)

Optical Microscopy & Spectroscopy

(Laser Confocal, White Light, Chromatic Dispersion, Spectral Ellipsometry, UV-Vis-NIR Spectroscopy)

Materials/ PC Board FA

(Cross Sections, Inspections, Dye & Pry)

X-Ray Characterization

(XRD/XRR, XRF, µCT)

Chemical Analysis

(ICPMS, GCMS, FTIR, Raman, Solid & Solution NMR, 2D NMR)

Nanoparticle Analysis

(DLS, PSA, Particle Zeta Potential)

Bulk Properties Characterization

(DSC, TGA, DMA, Rheometry, Tensile Test, Pencil Test, Surface Zeta Potential)

Austin Barnes, Ph.D. Member of Technical Staff

Austin Barnes is an expert physical chemist with over 8 years of experience in scanning probe microscopy of ionomer materials and organic semiconducting nanostructures. He completed a PhD at the University of California in Santa Barbara, where his research incorporated specialized nanomechanical and conductive AFM probe measurements. He has published multiple articles in the field of charge transport of nanomaterials. Austin brings extensive knowledge of a wide range of scanning probe methodologies to his leadership of Covalent's stylus metrology team

Find Austin on LinkedIn

https://www.linkedin.com/in/austin-barnes-84012953

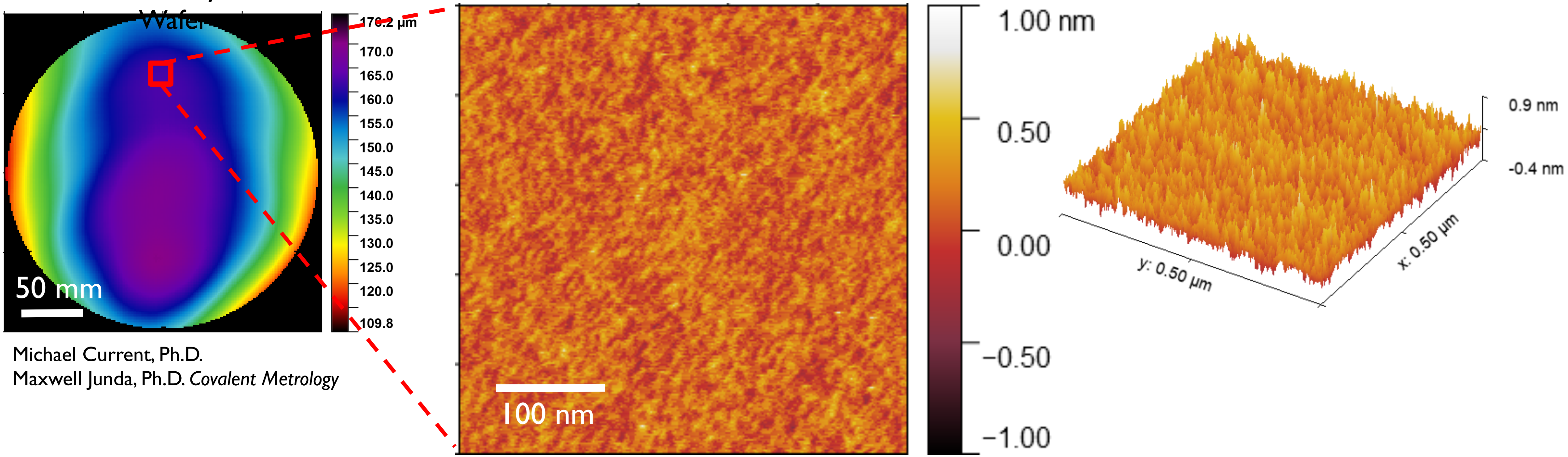
Episode 4 Overview

- Stylus Profilometry: The Pros and Cons
- Background AFM
- How to Choose the Right Tip?
- Common Scan Artifacts and How to Fix Them
- What Scan Size Should I Choose?
- What AFM should I use?

Narrowing the Scope:
AFM Looks at a Small FOV with Atomic Resolution

Chromatic Dispersion of CVD Oxide Layer on Si Wafer

AFM Topographic Map



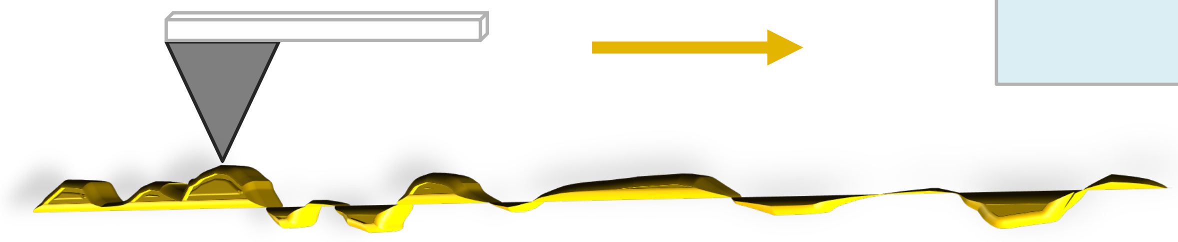
Michael Current, Ph.D.
Maxwell Junda, Ph.D. *Covalent Metrology*

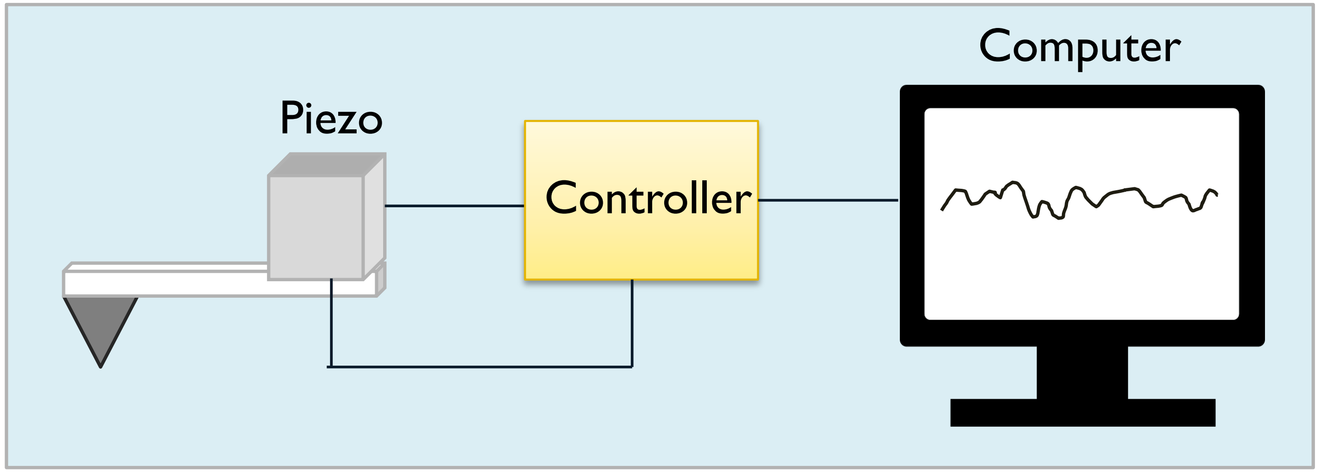
- Longer height variations can be seen in optical techniques over a large field of view.
- AFM looks at very small scale (atomic) height variations over a small FOV.

Stylus Profilometers Have Been Around for Many Years…

- Stylus profilometers have been around and is probably a familiar tool.
- It's the gold standard for quick 1-D step height and roughness measurements
- Capable of mapping as a "poor man's AFM". Low cost option compared to AFM.

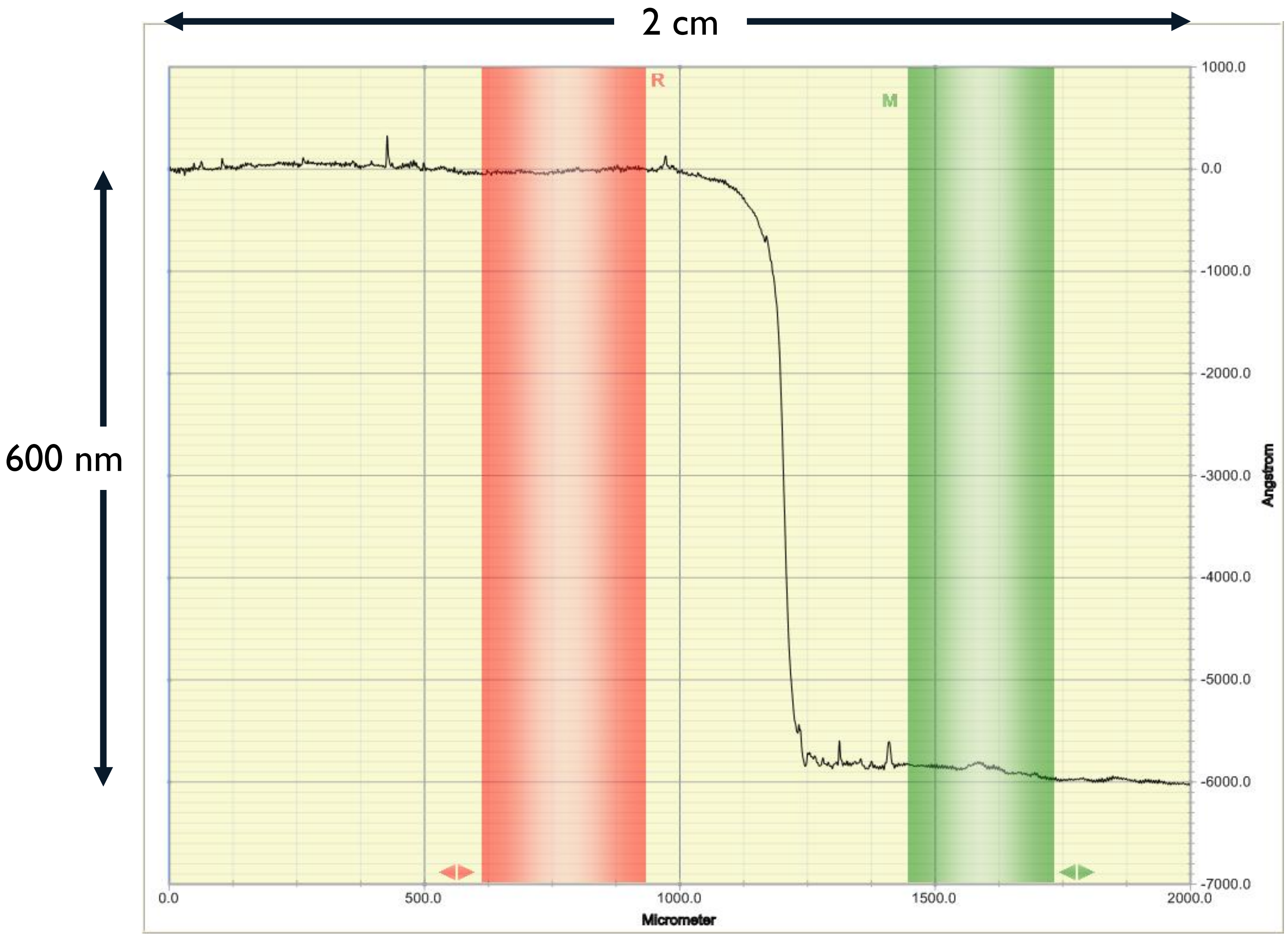
Stylus (Line) Profilometry Overview





- A constant contact force is applied while the stylus moves laterally.
- The feedback controller lifts the stylus tip up or down to maintain constant force
- Tips are usually diamond. The tip radius dictates the lateral resolution.
  – Tip radius ranges from 50 nm to 200 microns

Stylus Profilometry Excels At Long Scan Lengths and Hard Materials
Copper Film on Silicon



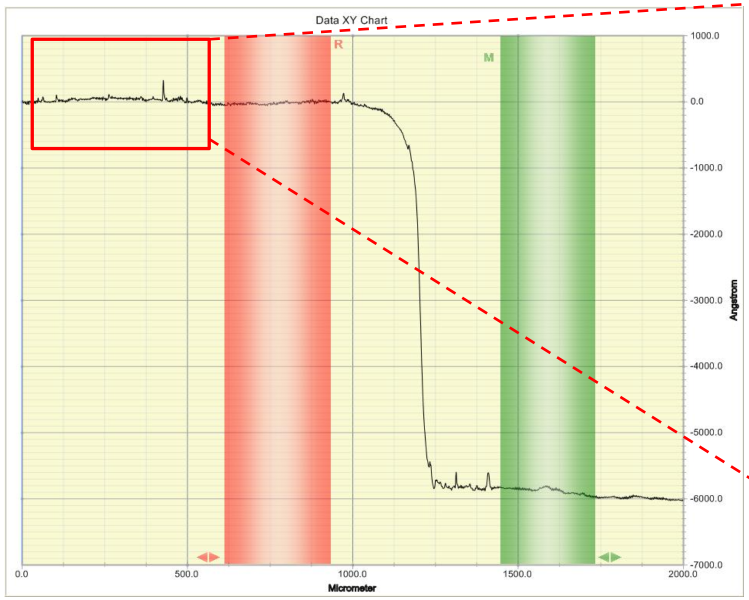
R = Reference

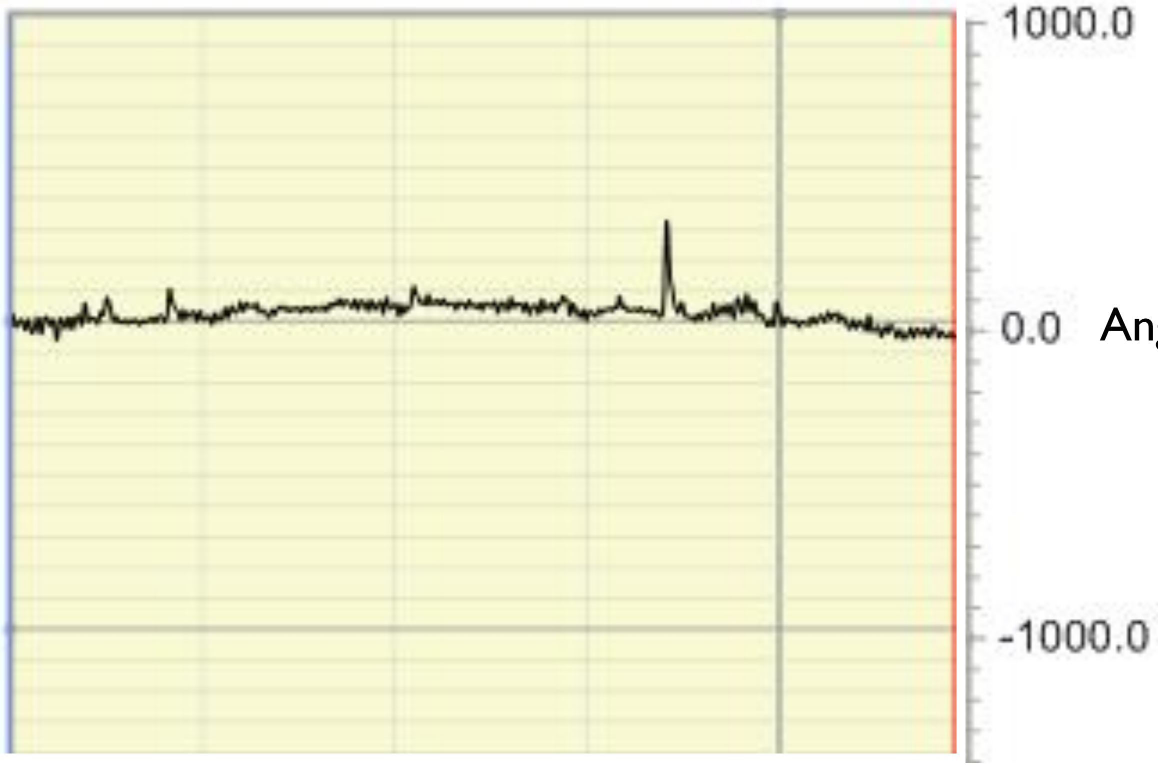
M = Measure

Height = Averaged over the 2 regions

Film Thickness = R - M

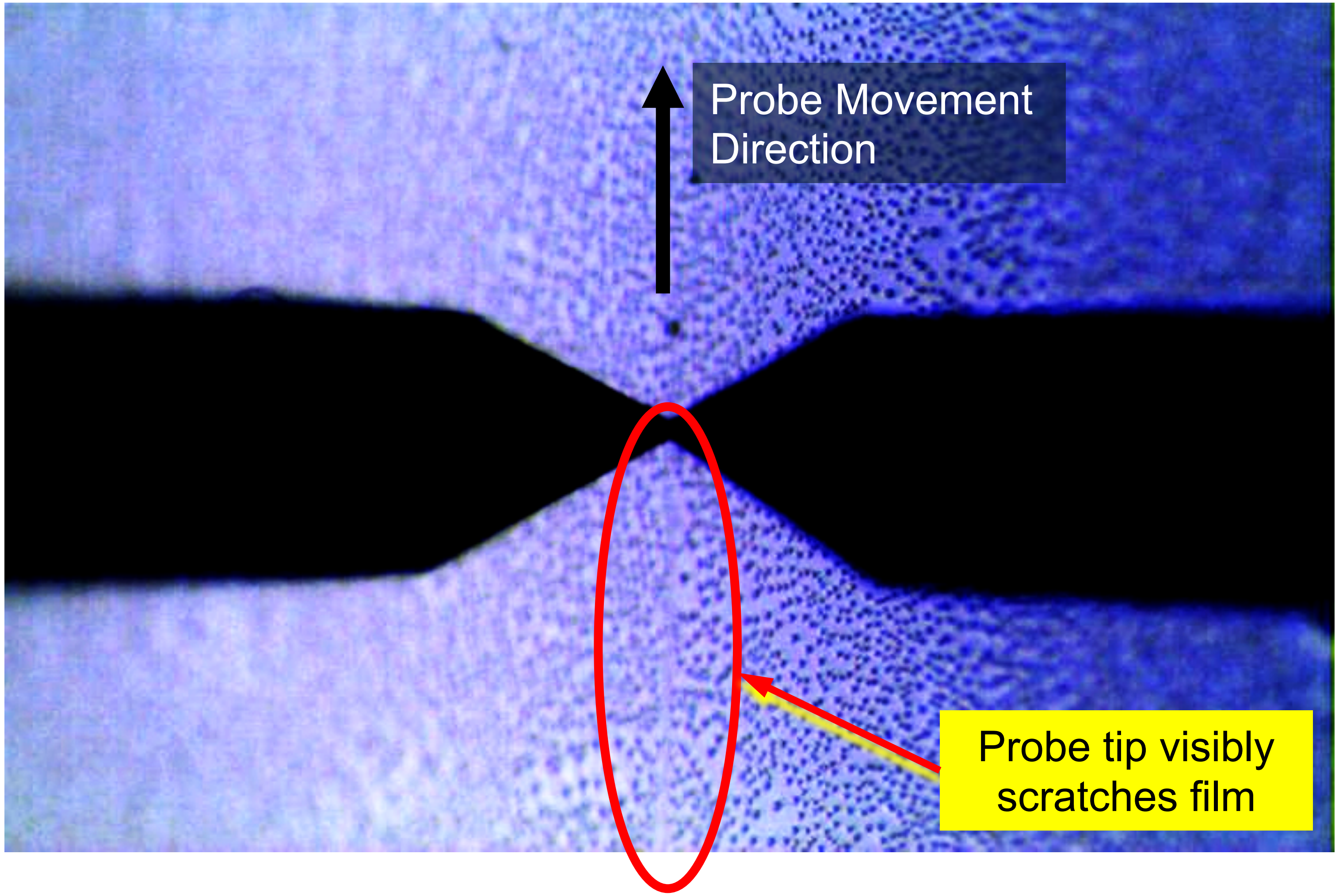
Stylus Profilometry for Line Roughness



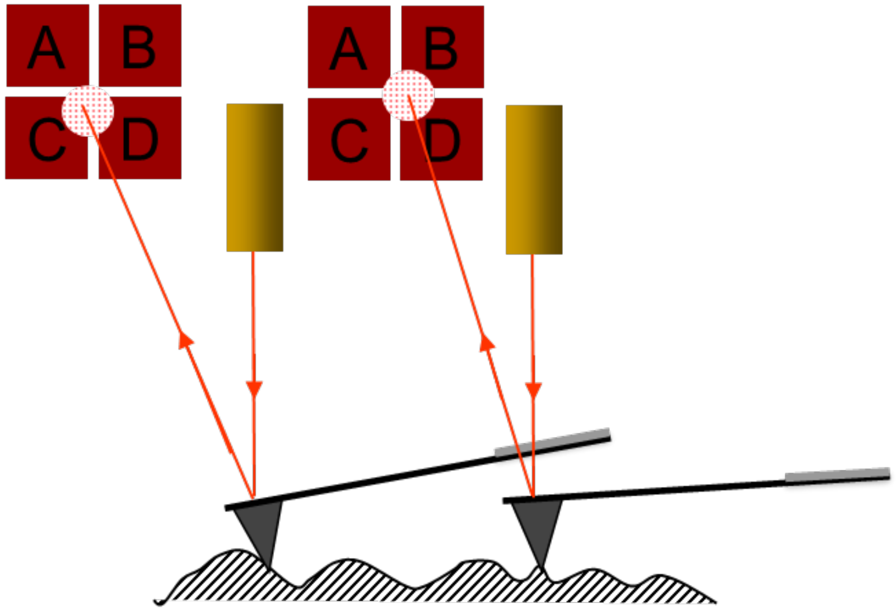


- Can provide a measure of line roughness. Average line roughness ~10 nm.
- Lateral and vertical resolution limitations.
  - tip dimensions, piezo vertical range (sub nanometer vertical often not achievable)

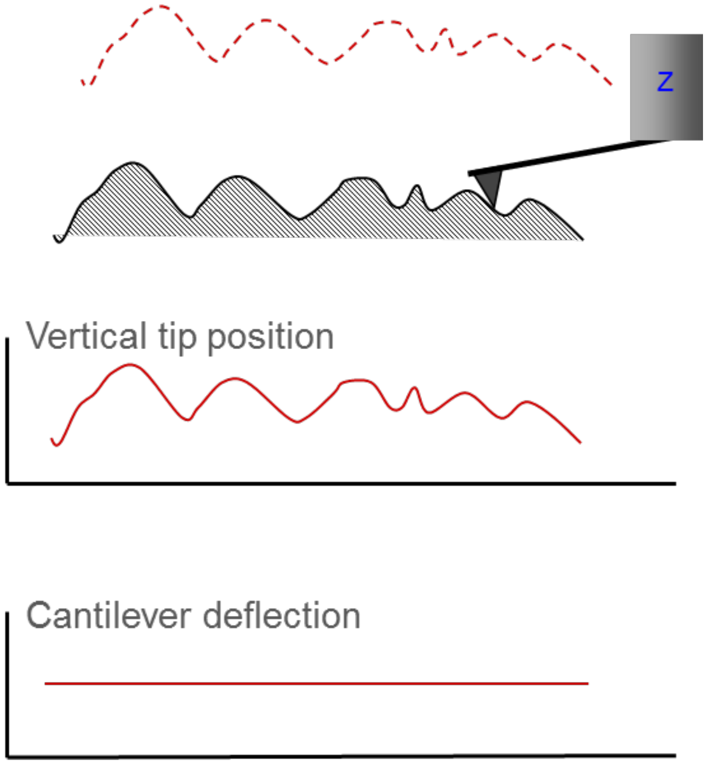
Suffers for Soft Coatings



Atomic Force Microscopy (AFM)
2 Methods to Measure Topography: Contact Mode

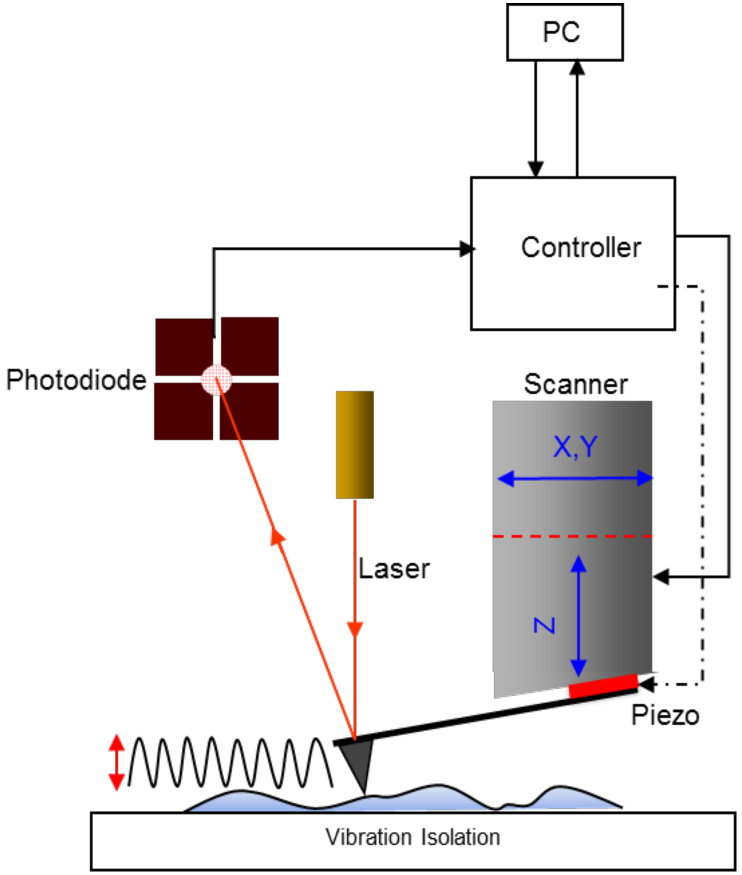


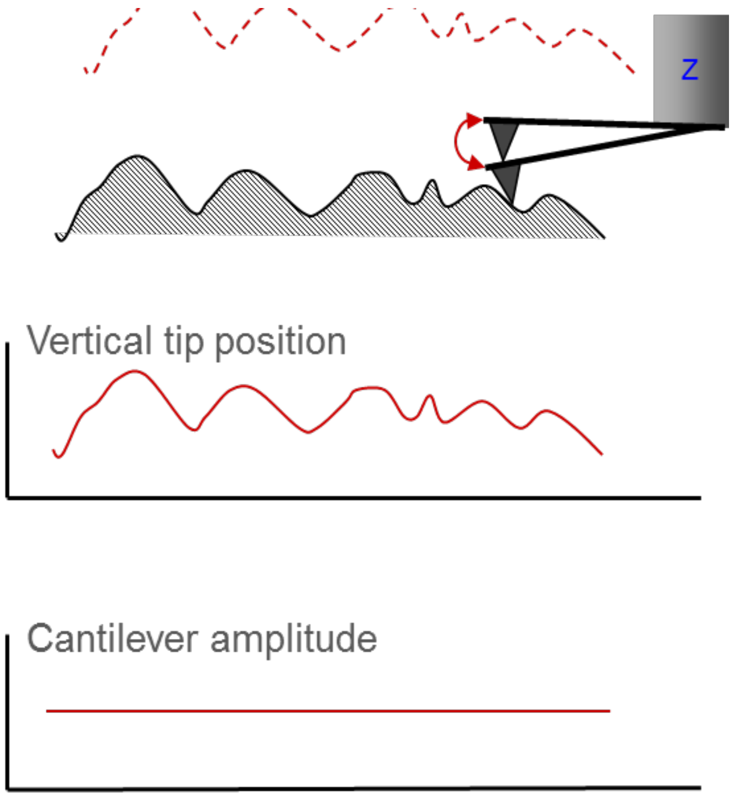
Deflection: $\dfrac{(A+B)-(C+D)}{(A+B)+(C+D)}$



- Keep lever deflection constant. Tip is “dragged” across the sample
- Base for other advanced modes: conductive AFM, contact resonance, …

Tapping Mode (AC-Mode)





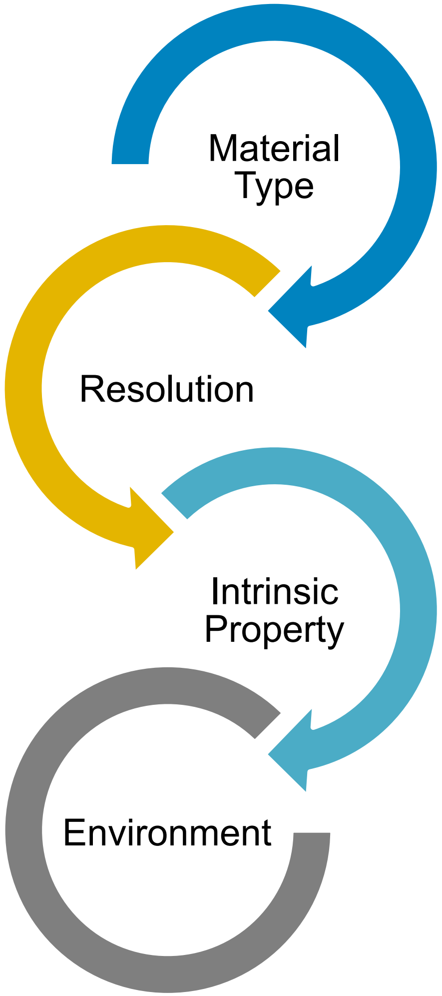
AFM User Controls…

- Choice of tip
- Amplitude setpoint (tip-sample distance)
- Integral gain
- Scan speed
- Scan size

- Cantilever is driven at its resonant frequency by a piezo.
- The amplitude is monitored by laser deflection.
- Constant amplitude is maintained by adjusting the z piezo up or down.

How to Choose the Right Tip?

- *What's my sample/material?*
  - Is the sample hard (e.g. metals, ceramics, semiconductors) or soft (polymers, gels, biomaterials)
- *What lateral (x,y) resolution do I need?*
  - Smaller than 7-10 nm?
- *Surface properties other than topography?*
  - Mechanical (stiffness/modulus), conductivity, magnetic domain orientation, charge state,…
- *Environmental conditions?*
  - Sample needs to be imaged in liquid?



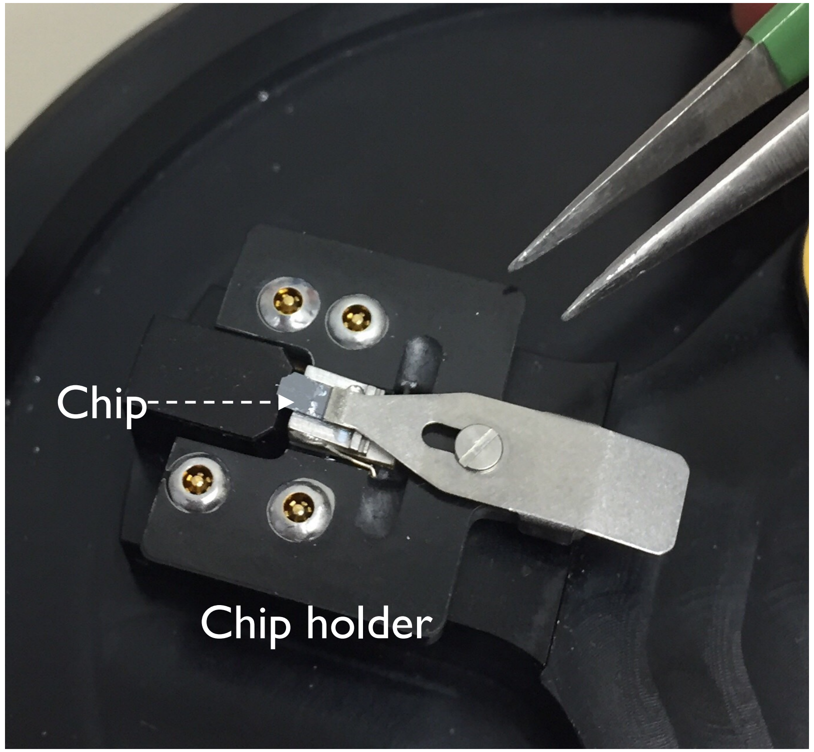
<u>Boils down to a few key properties:</u>

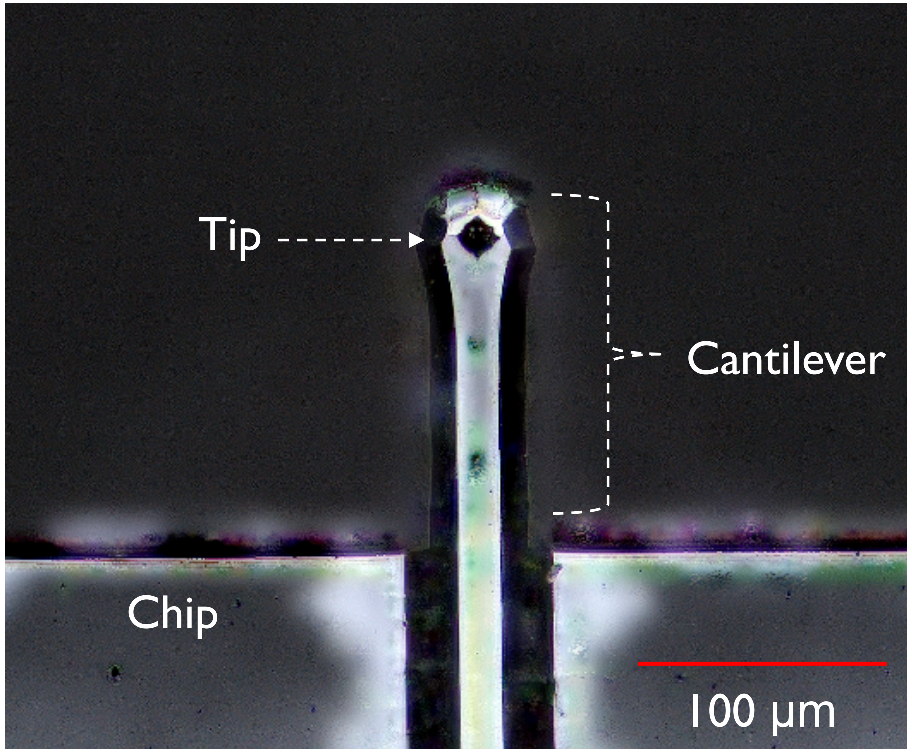
Spring constant (k) … tip force

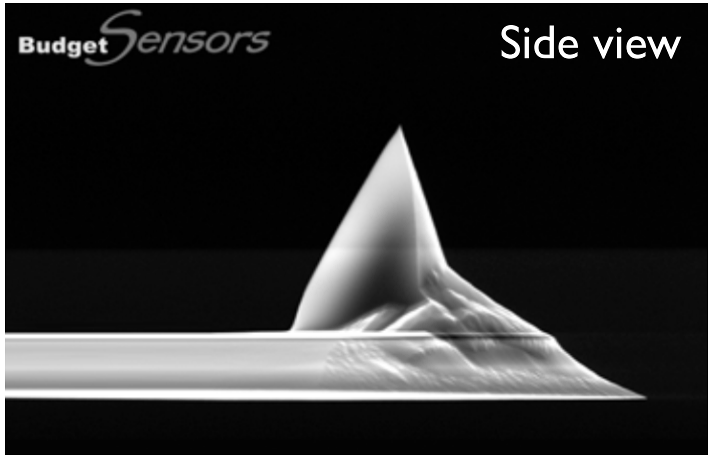
Tip sharpness (r) … lateral resolution

Resonant frequency and Q-factor … imaging speed (will be discussed in a later webinar)

Anatomy of the AFM Probe



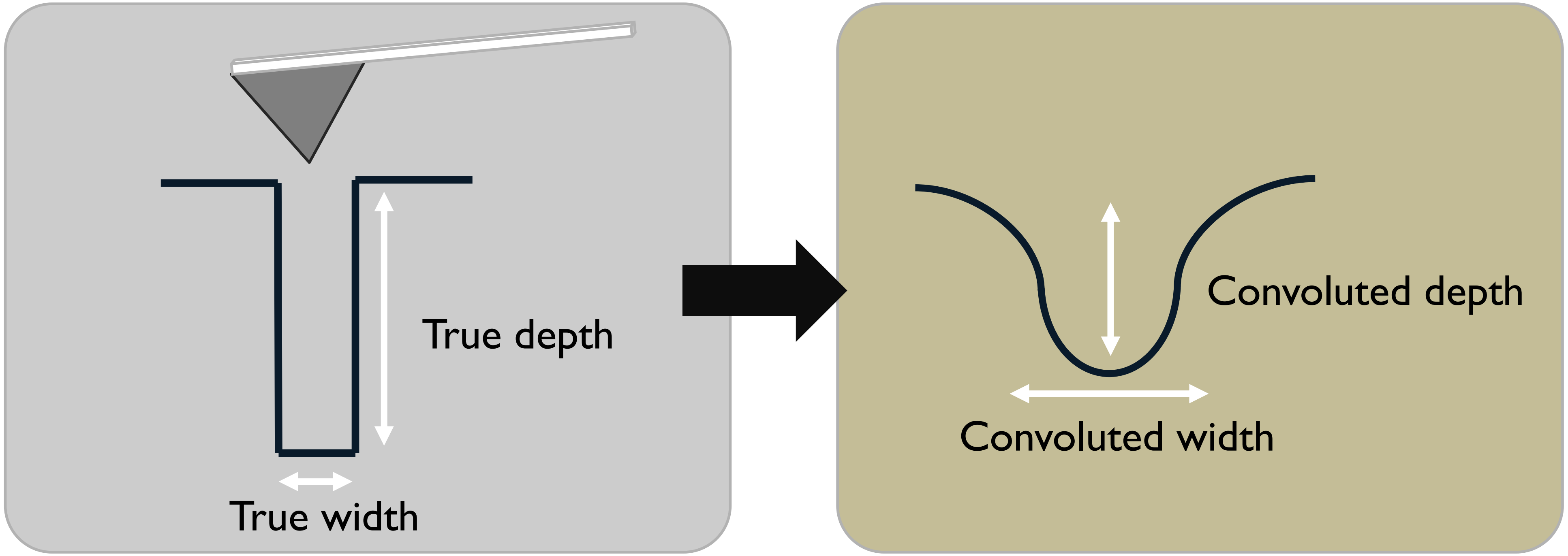




- Probes are typically made from Silicon or Silicon Nitride. Backside of cantilever usually has a reflective Al coating
- Tip radius, probe material, cantilever length can be the difference between good AFM data and bad
- **Key probe manufacturers**: NANOSENSORS, NanoWorld, BudgetSensors, MikroMasch,App Nano,NuNano.

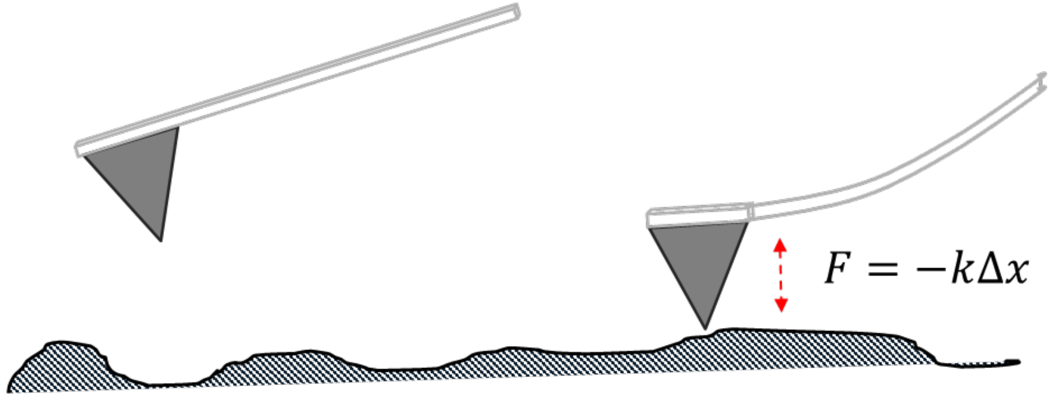
Tip Radius and Convolution Effects

- Radius of curvature of the tip defines the lateral resolution.
  – Not only leads to convoluted x,y dimensions, can caused convoluted depth… effects surface roughness!



Cantilever Spring Constant and Tip Sample Force

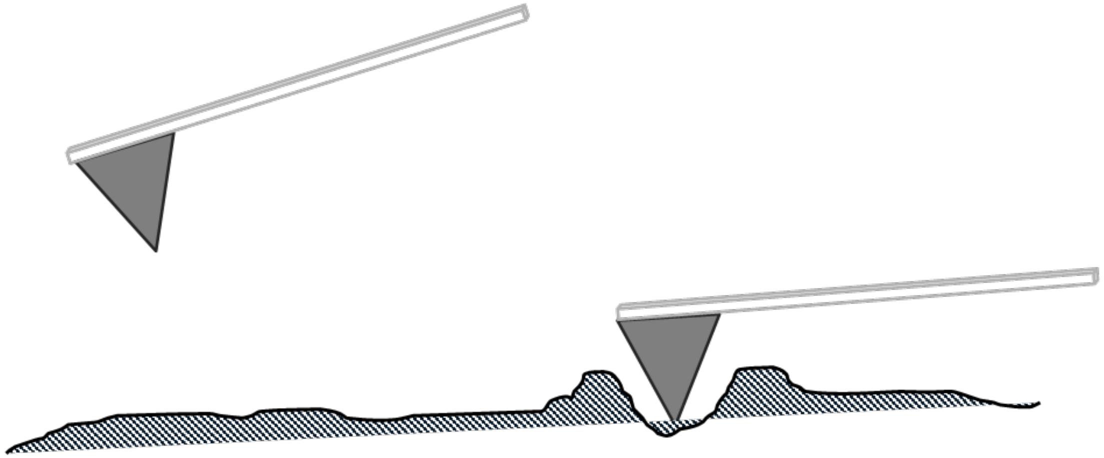
Case 1: $K_{sample} >> k_{probe}$



- **Think of the probe as a mass on a spring**

  Δx is related to the deflection or amplitude
- This case represents hard materials (ceramics, metals, semiconductors).
- Almost any choice of tip spring constant won't induce sample deformation
- Need to know if surface is sticky. Cantilever upswing is necessary for tracking

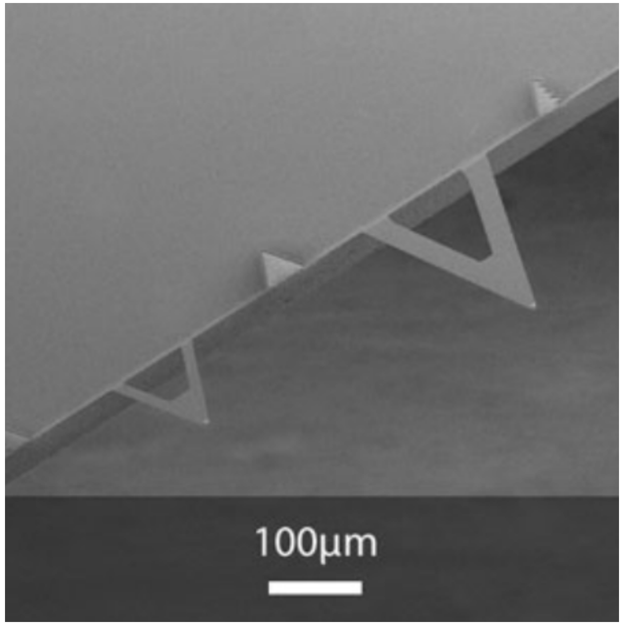
Case 2: $K_{sample} << k_{probe}$



- **Tip can indent or poke through the sample**
- This case represents soft materials (polymers, gels, membranes)
- Require a tip in which k_{probe} is less than or comparable to K_{sample}

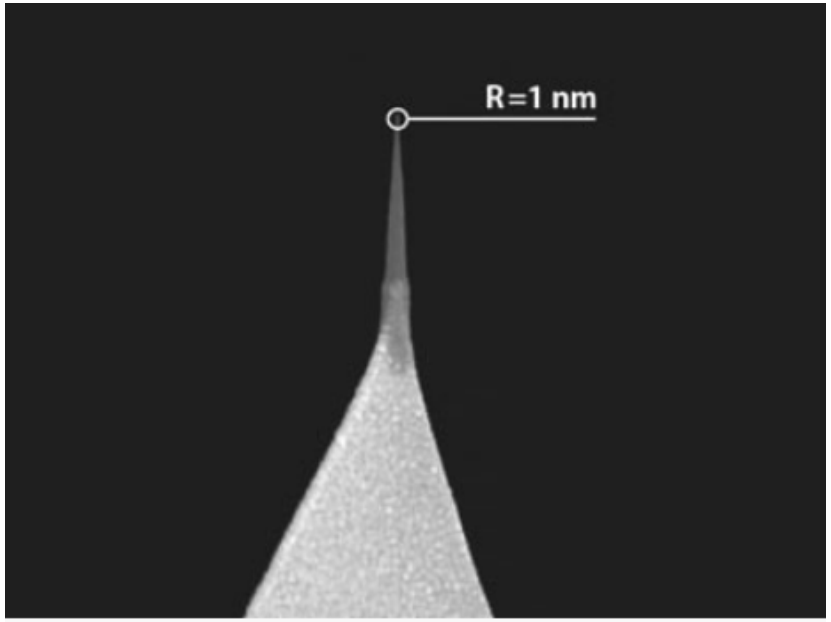
AFM Probes Have Evolved Into a Variety of Species

XNC12/Cr-Au by



Soft cantilevers, low frequency
Imaging in liquid, soft materials

SHR150 by Budget Sensors



Super sharp tip
High lateral resolution

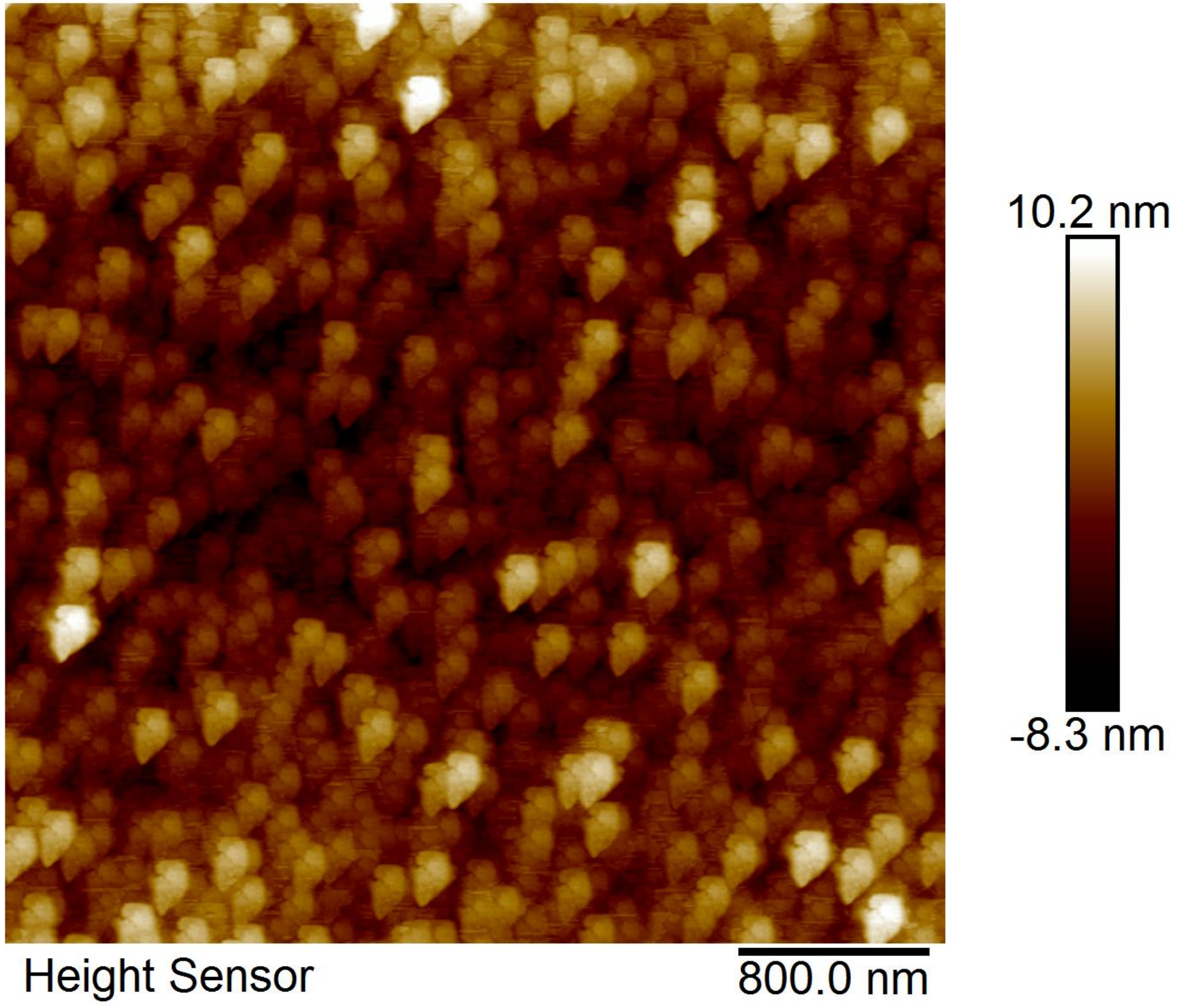
EBD-NCH by Nanotools

Diamond-like electron beam deposited (EBD)
Cone shaped, hard contact, wear resistant

Images courtesy of NanoAndMore

Tip Wear and Tip Contamination

- Don't bother cleaning or sharpening tips. They are consumables.



Tip-Check Samples

COVALENT METROLOGY

<u>Wear-resistant coating on silicon</u>

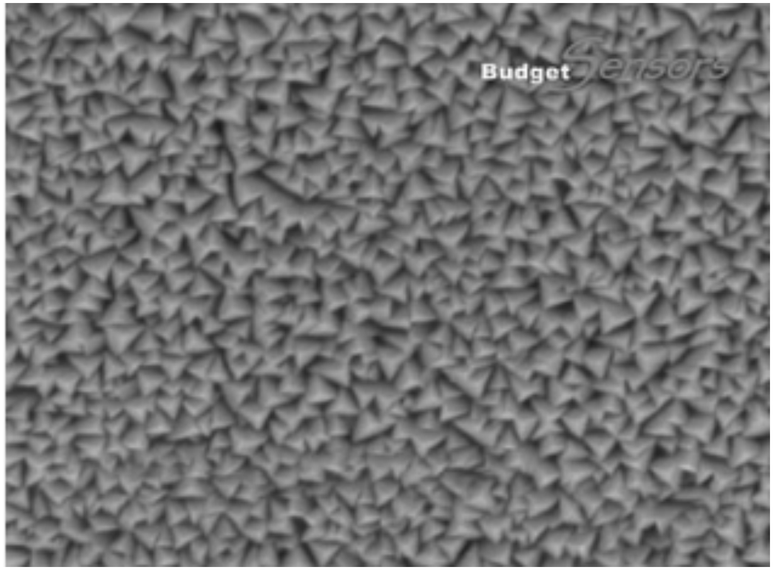
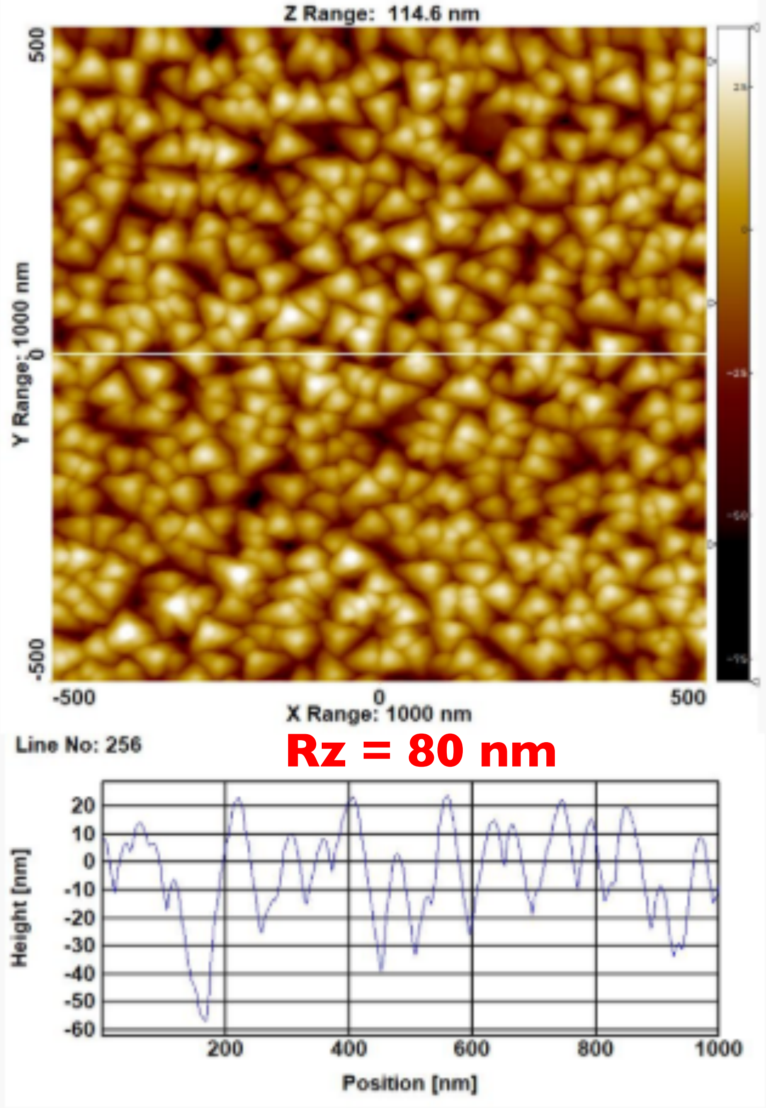
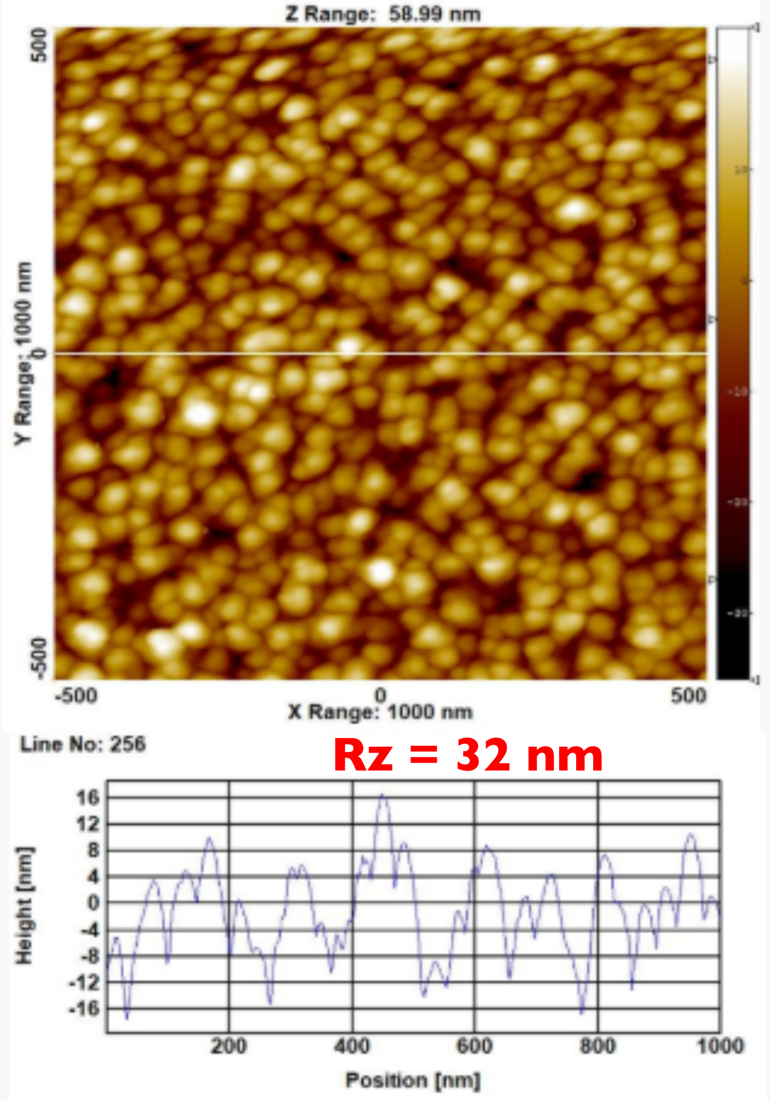


Image Courtesy BudgetSensors and NanoAndMore

Sharp Tip



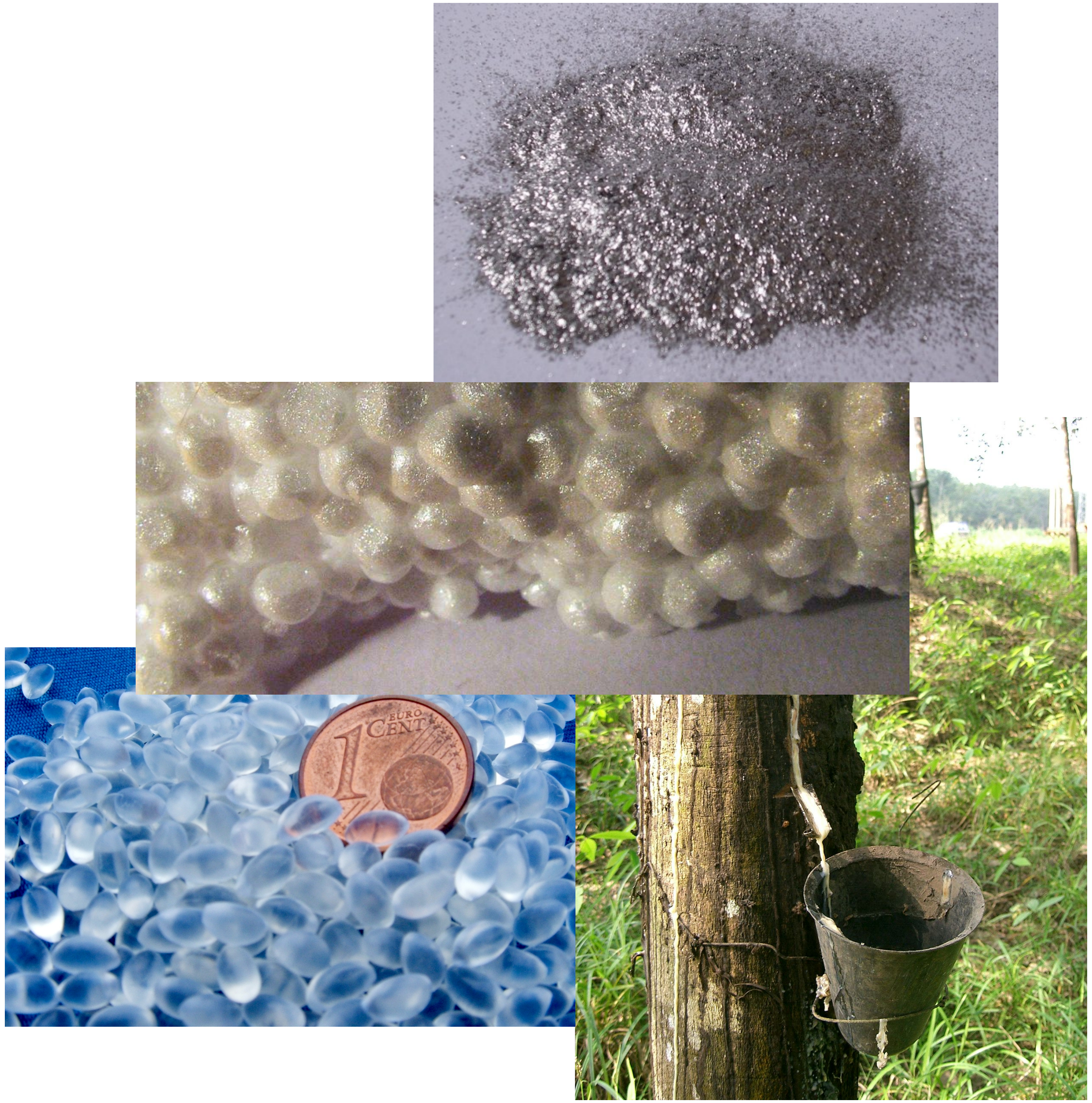
Worn tip



Differences in peak to valley (Rz) roughness can be seen

Sample Preparation

- Does your sample or surface change under ambient conditions?
  - Ambient conditions fluctuate seasonally
  - Hygroscopic samples? e.g. in some cases polymers, salts, powders, woody biomass
- AFMs can be operated under controlled temperature and humidity



Sample Mounting for Scanning

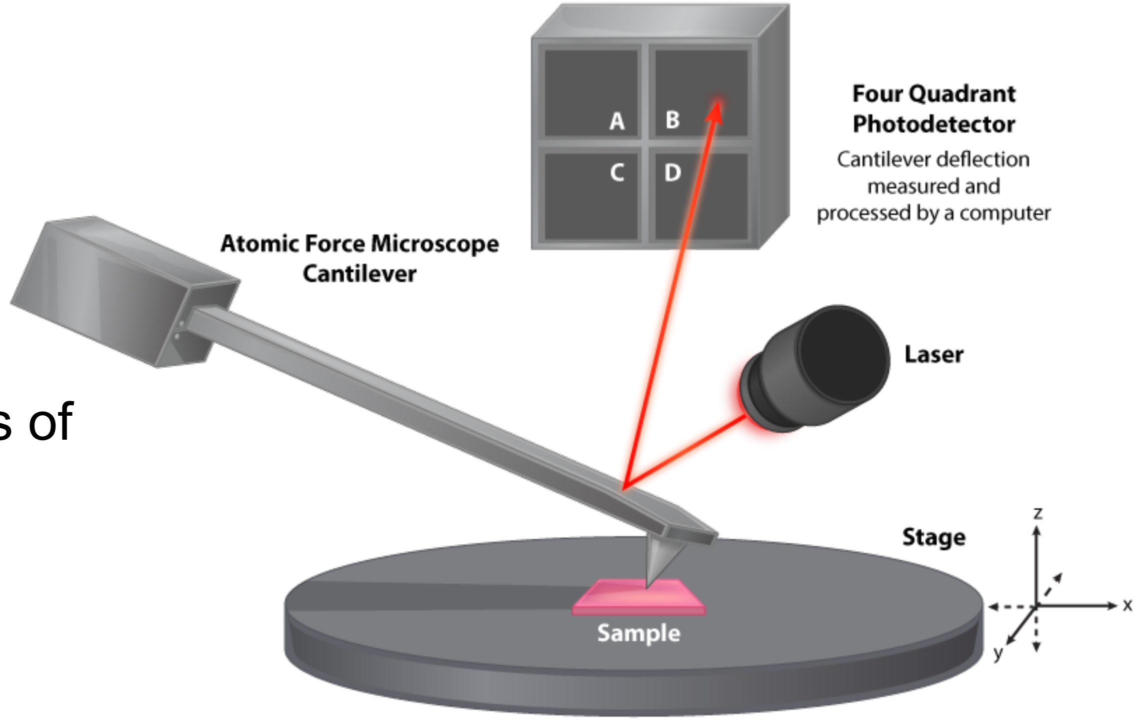
- Sample needs to sit still during scanning

✓ Flat and flush on the stage

✓ Magnets work great for mounting sample

✓ Epoxy also works wells for thin or brittle sheets of material to mount on a glass slide.

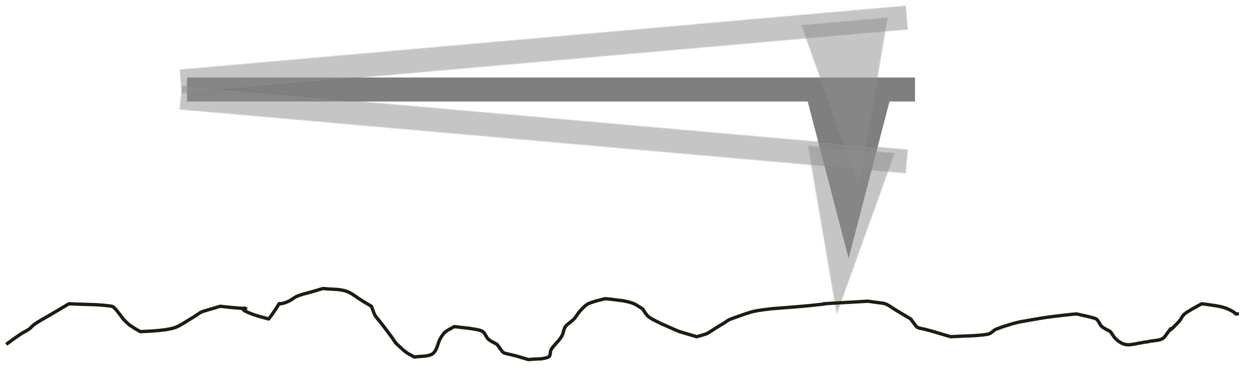
✗ Tape tends to cause drift during the scan

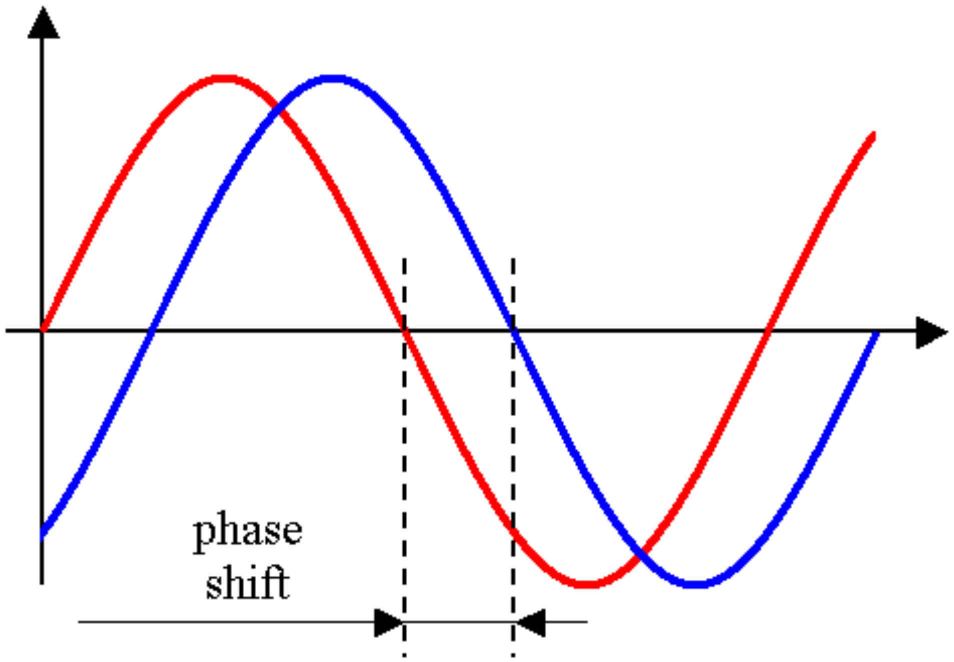


Engaging the Tip on the Sample: Optimizing Sensitivity while Maintaining Tracking

- Monitor the change in amplitude and phase signal
- Choice of setpoint amplitude (engaged) relative to free air (not engaged) amplitude
  - Is your sample soft? Eliminate tip-induced damage, or false height readings.
- Integral gain and scan speed

Interpreting the Phase Signal

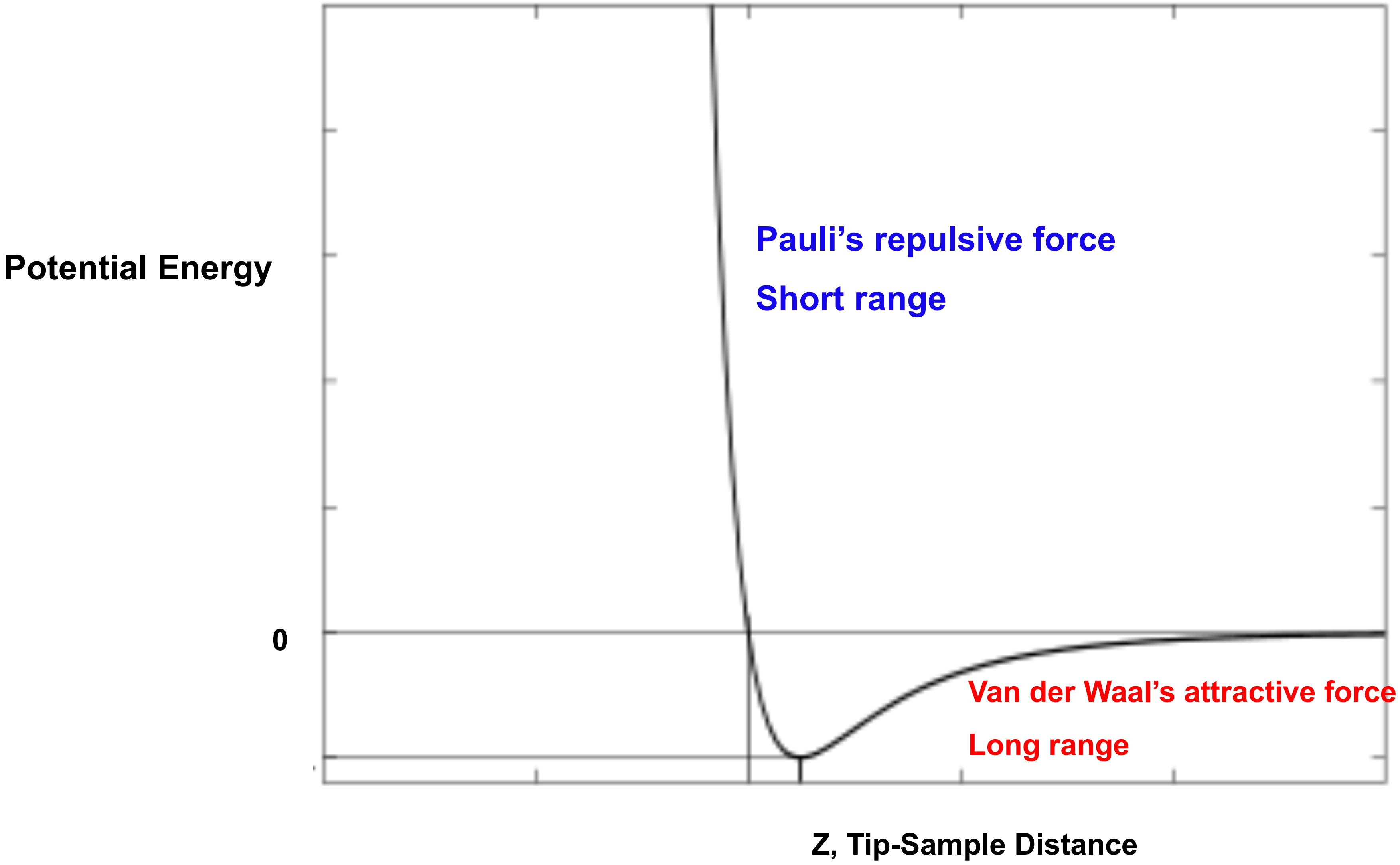




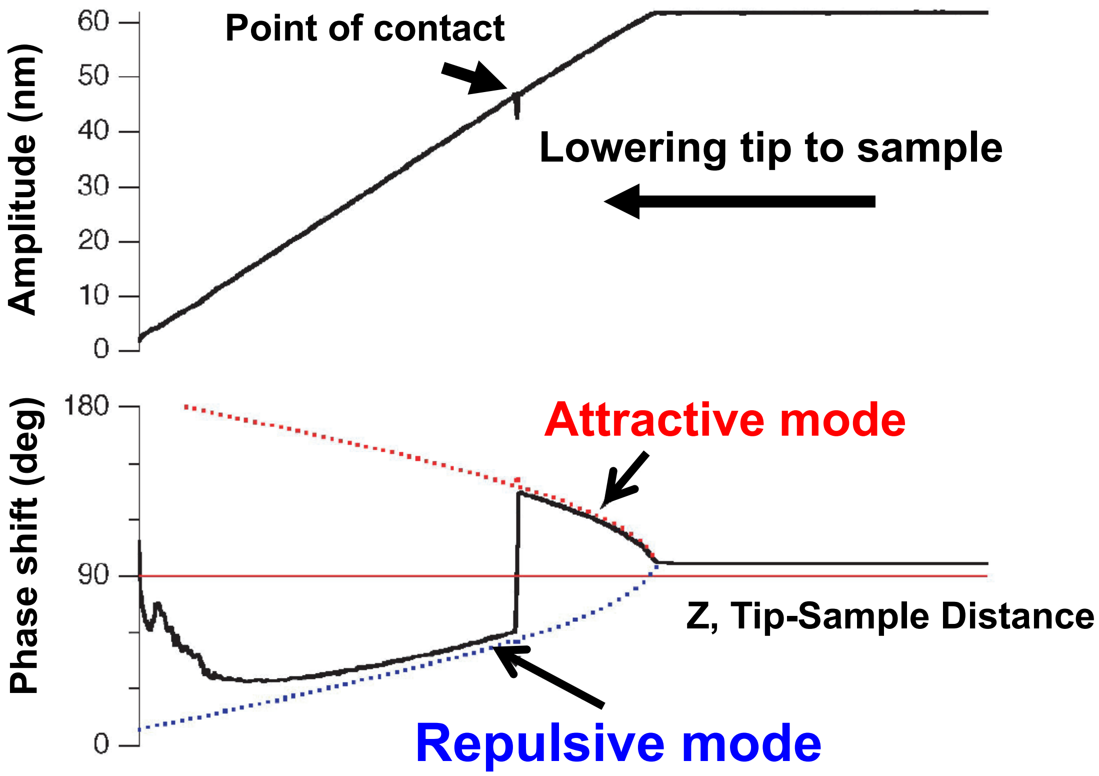
- Phase shifts is the deviation of the output signal from the drive signal.
- Phase shifts can be caused by sharp changes in height and/or differences in material compliance
- Positive/negative shifts are caused by differences in tip-sample force…

Interaction Potential Between Tip and Sample vs. Distance

Lennard-Jones Potential



Monitoring Amplitude and Phase as the Tip Is Lowered to the Sample



In **attractive mode**, the tip turns around before contacting surface, while in **repulsive mode**, it makes intermittent contact with surface.

Attractive mode much less invasive (favorable for soft samples)

Phase 90-180deg: Long-range attractive forces dominate tip-sample interaction (attractive mode)

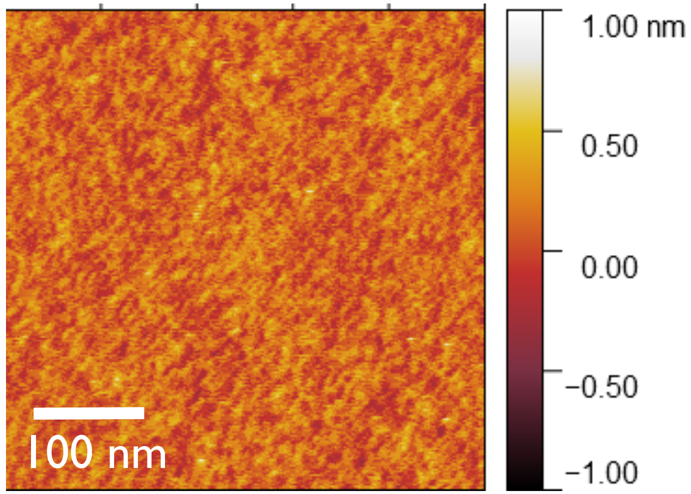
Phase 0-90deg: Short range repulsive forces dominate (repulsive mode)

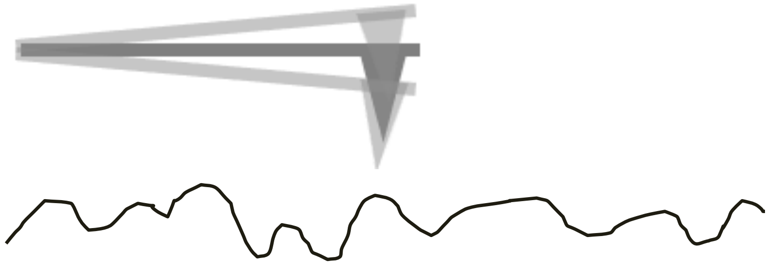
J. Cleveland and R. Proksch, Asylum Research

CVD Oxide Film on Silicon Wafer
Comparison of Attractive and Repulsive Imaging Modes

Attractive mode	***Repulsive mode***

Attractive mode

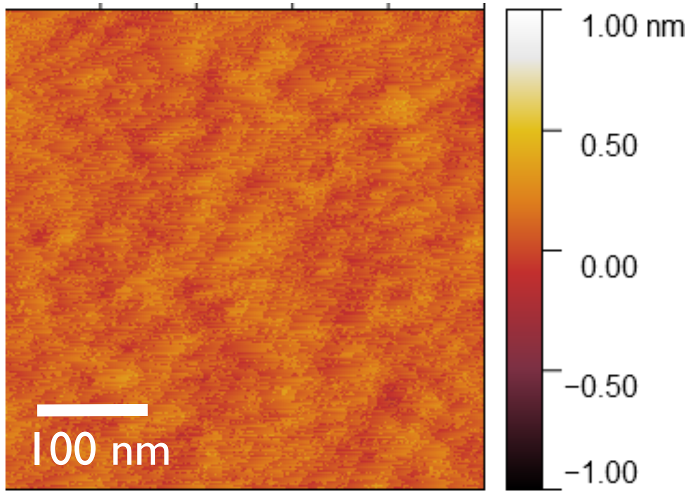


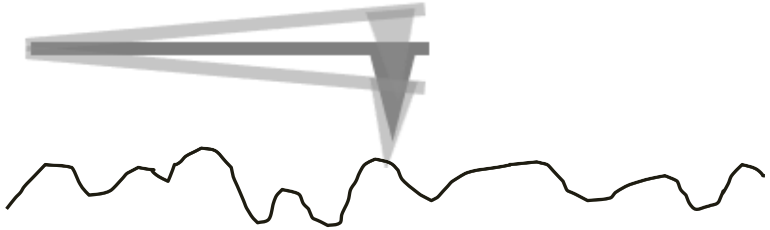


Dominate tip force is attractive

Gentle, "non-contact"

Repulsive mode

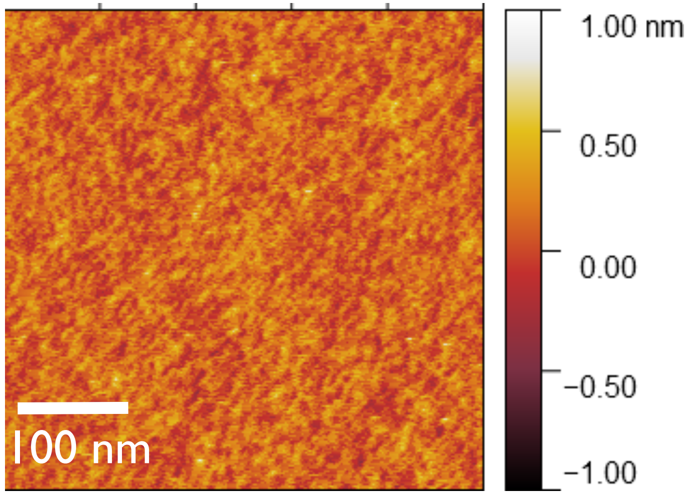
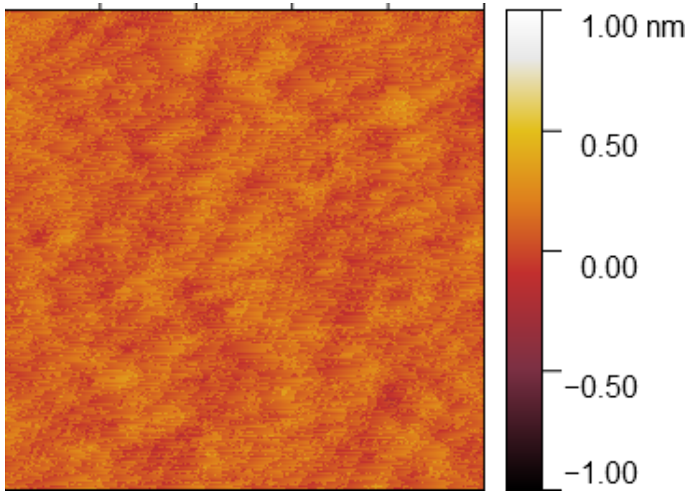




Dominate tip force is repulsive

Tip is hammering at surface

Effect on Surface Roughness

Attractive mode	Repulsive mode
	
Sq = 0.145 nm	Sq = 0.086 nm

0.06 nm difference in RMS surface roughness significant?

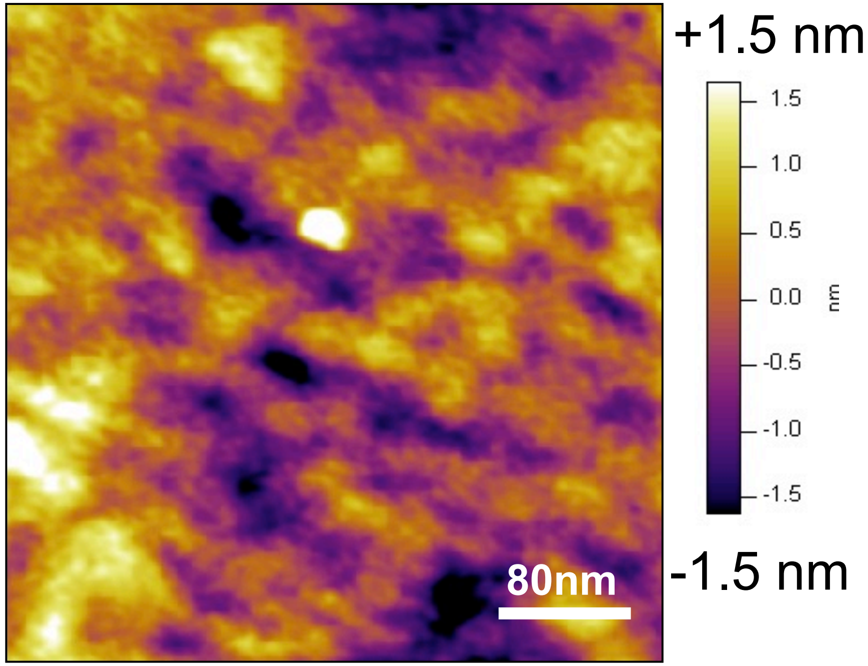
Yes, if noise floor (~0.01 nm) is only variable.

Variability in tip, sample, mechanical/electrical noise, etc.

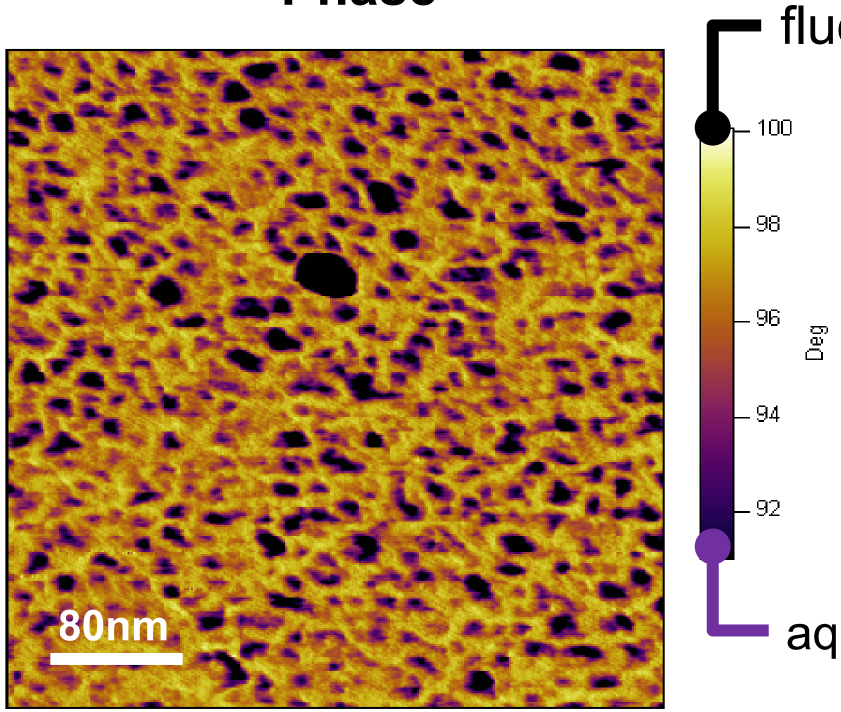
Sub-angstrom resolution relevant to your materials/process?

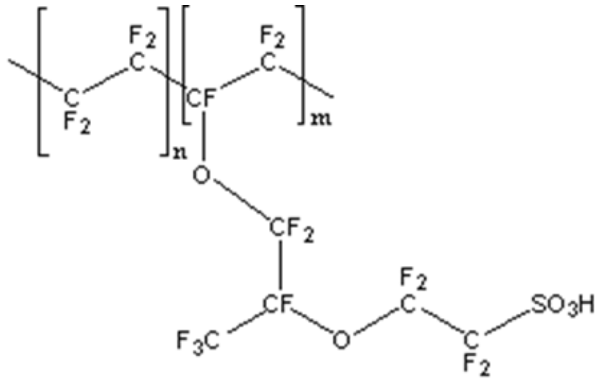
Phase Imaging for Nanomechanical Characterization Proton Exchange Membranes

Height



Phase



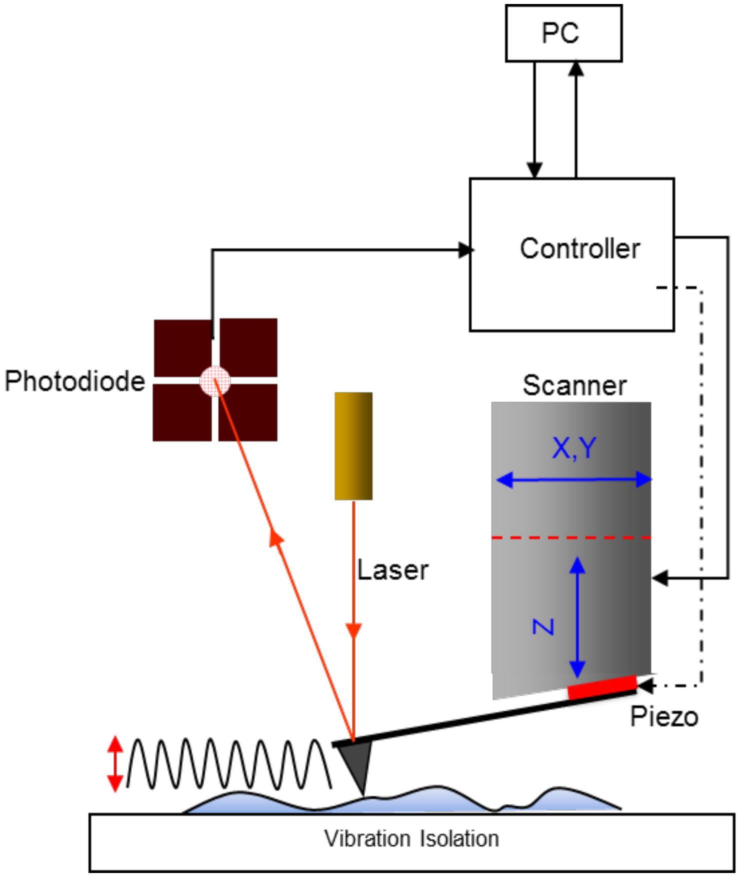


- Sometimes the topography is not meaningful
- Phase contrast is related to chemical heterogeneity to the surface

19.4% hydrophilic
Average domain size = 94 nm^2
10.9 nm diameter

O'Dea, J.R., Buratto, S.K. *J. Phys. Chem. B* **2011**, *115*, 1014–1020

Choice of Integral Gain and Scan Speed to Improve Surface Tracking



- Gain is related to the controller – controls speed of feedback
  - Integral gain is the integrated error over time

- **Low Gain:** Never reach setpoint amplitude. False height readings.

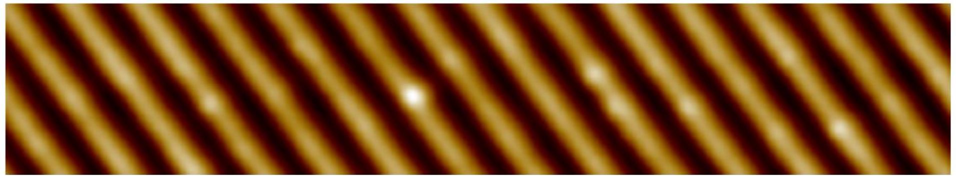
- **High Gain:** Ringing in trace and retrace scans. Adds noise

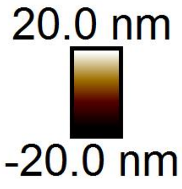
- **Scan speed:** 1 Hz scan rate typical. Can lose tracking from surface, tip can parachute off a large feature if too fast.

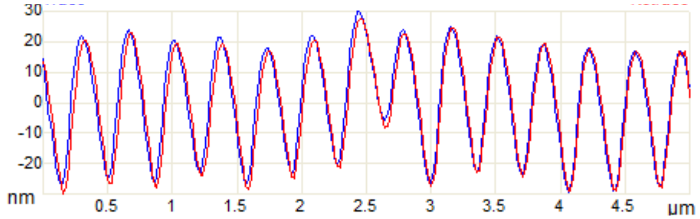
Calibration Grating
Effect of Integral Gain and Scan Speed on Surface Tracking

Trace --▸ ◂-- Retrace

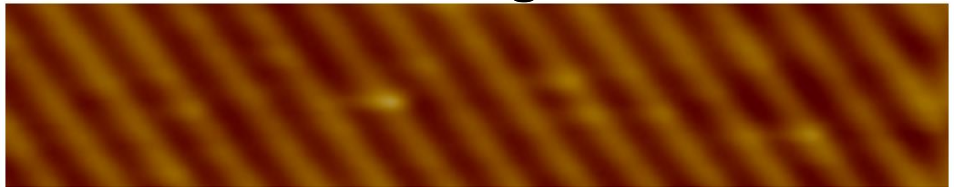
Clean Scan

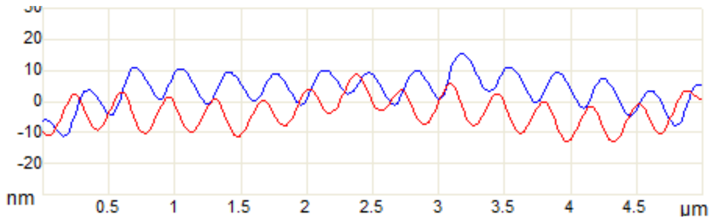




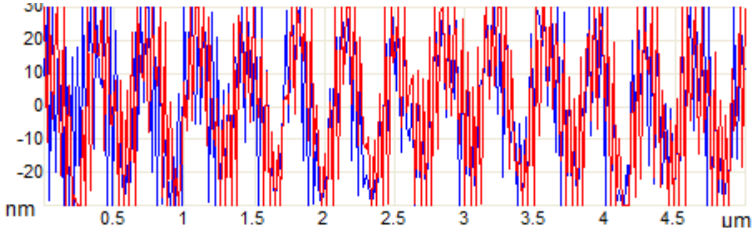


Low Integral Gain

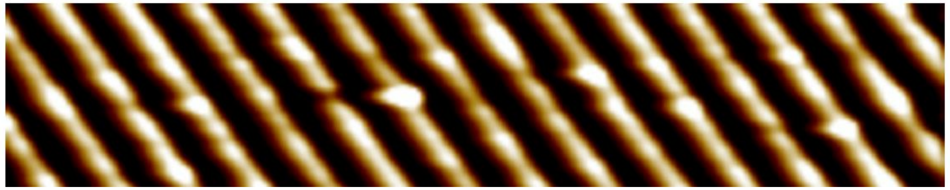


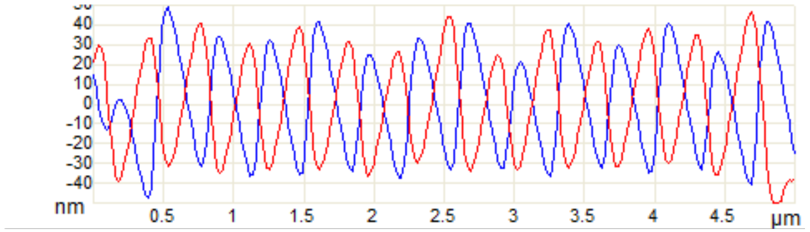


High Integral Gain



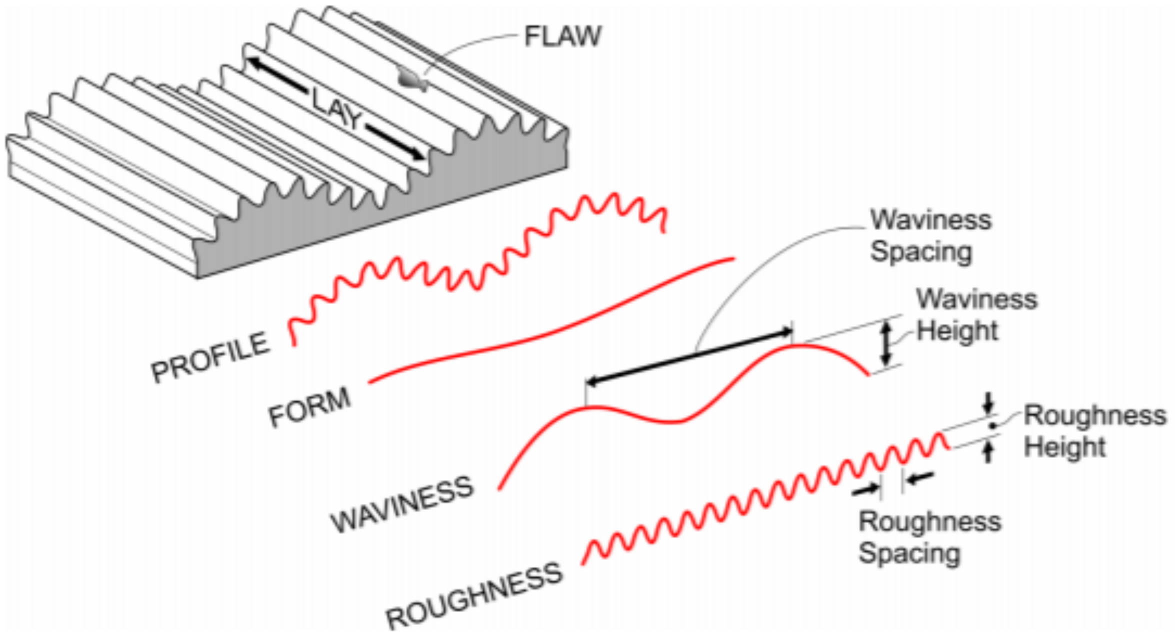
High Scan Speed





What Scan Size Do I Choose for Surface Roughness?

Zygo Mx Surface Texture Parameters

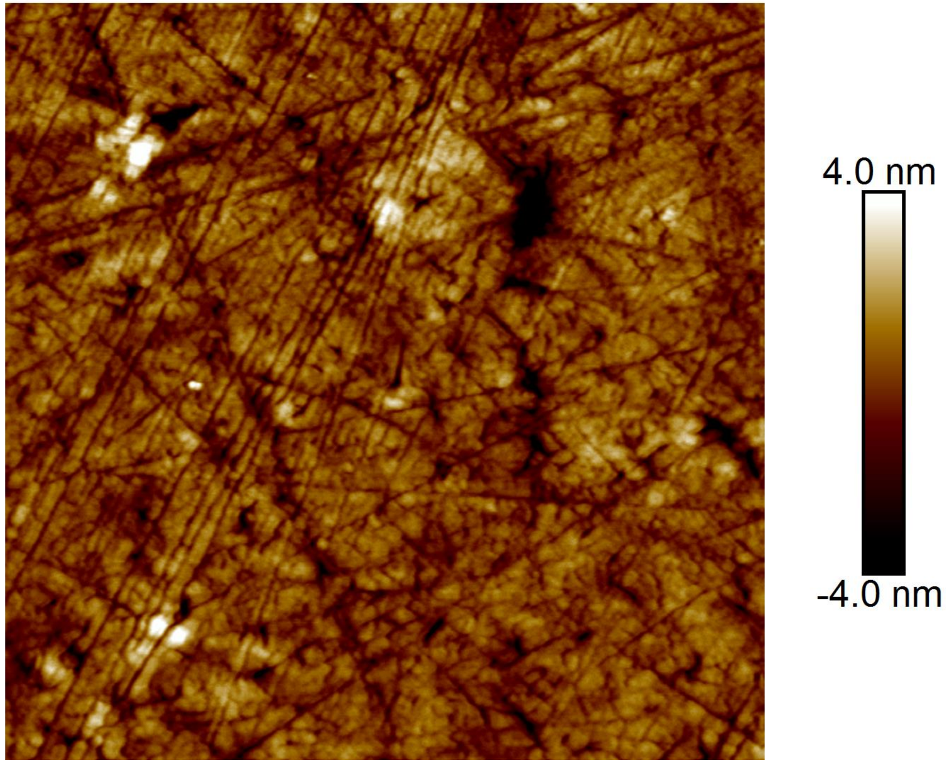


- Roughness depends on frequency
  - lowest frequency = size of the image, highest frequency = pixel size.
- Pixel resolution typically fixed (512 pixels/line)
  - Oversampling if pixel size is less than tip radius

- *Could you take a maximum scan size image with a pixel size 10 nm?*
  - Yes, but the scan would take an eternity.
- Better alternative is to take an image at a small scan size (2x2 micron) and a larger scan size (10x10 micron)

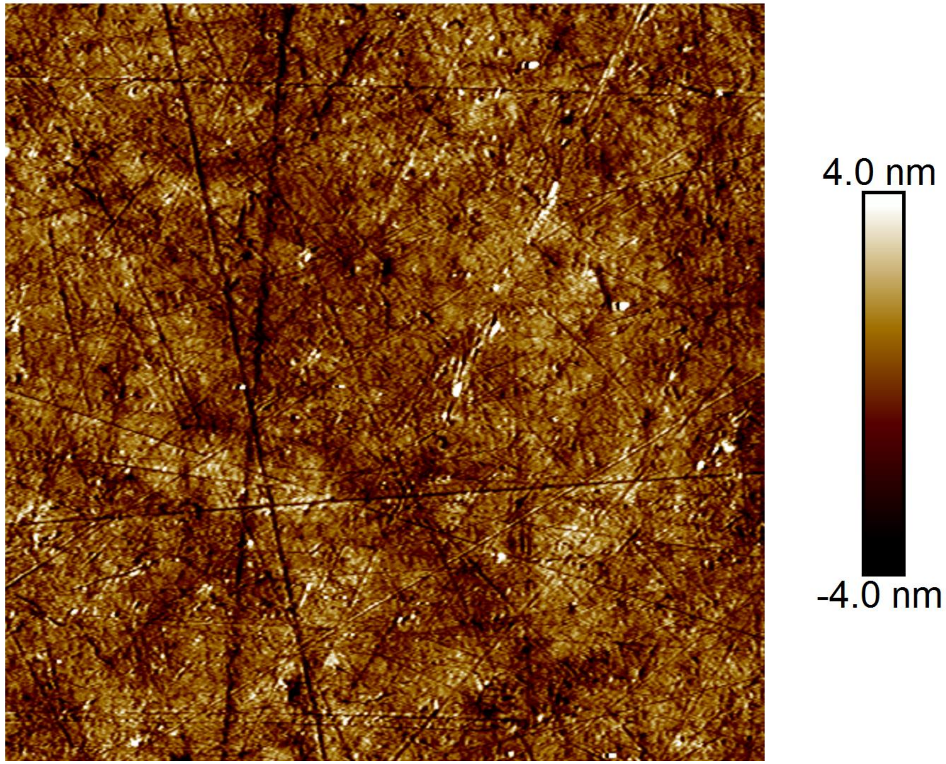
Topography of Steel 304:
Surface Roughness on Different Length Scales

2 µm x 2 µm



Avg. Surface Roughness Sa = 0.70 nm
RMS Surface Roughness Sq = 0.97 nm
Peak-Valley Surface Roughness Sz = 20.41 nm

10 µm x 10 µm

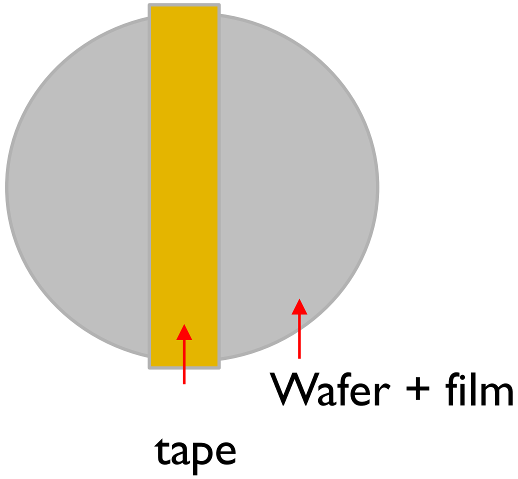


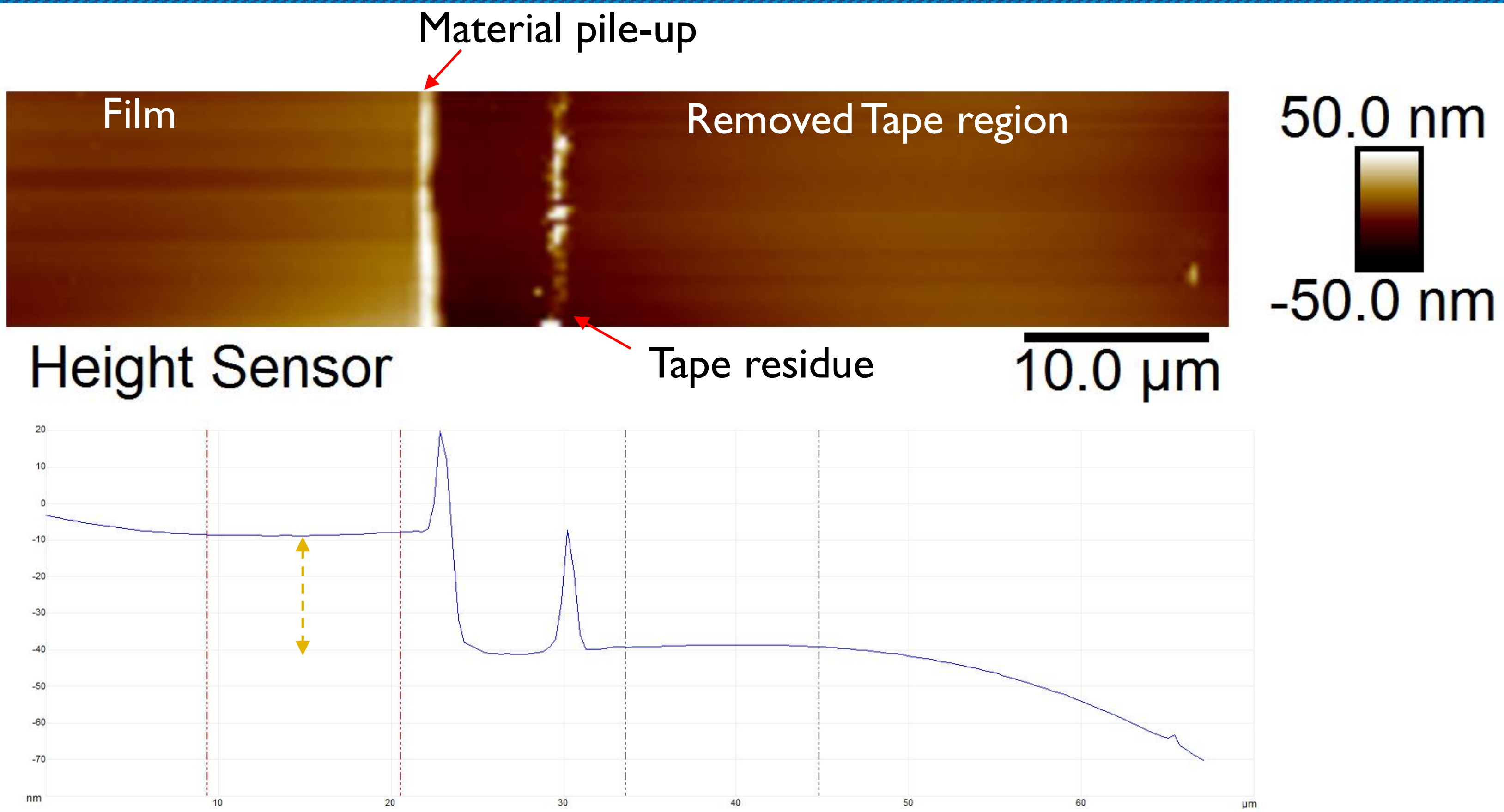
Sa = 0.94 nm
Sq = 1.28 nm
Sz = 53.03 nm

Which set of roughness parameters represents the surface?

- Material/Intrinsic length scales relevant to you
- Larger FOV -> Sz, waviness, but sacrificing resolution

Bow Artifacts Over Large Scan Lengths

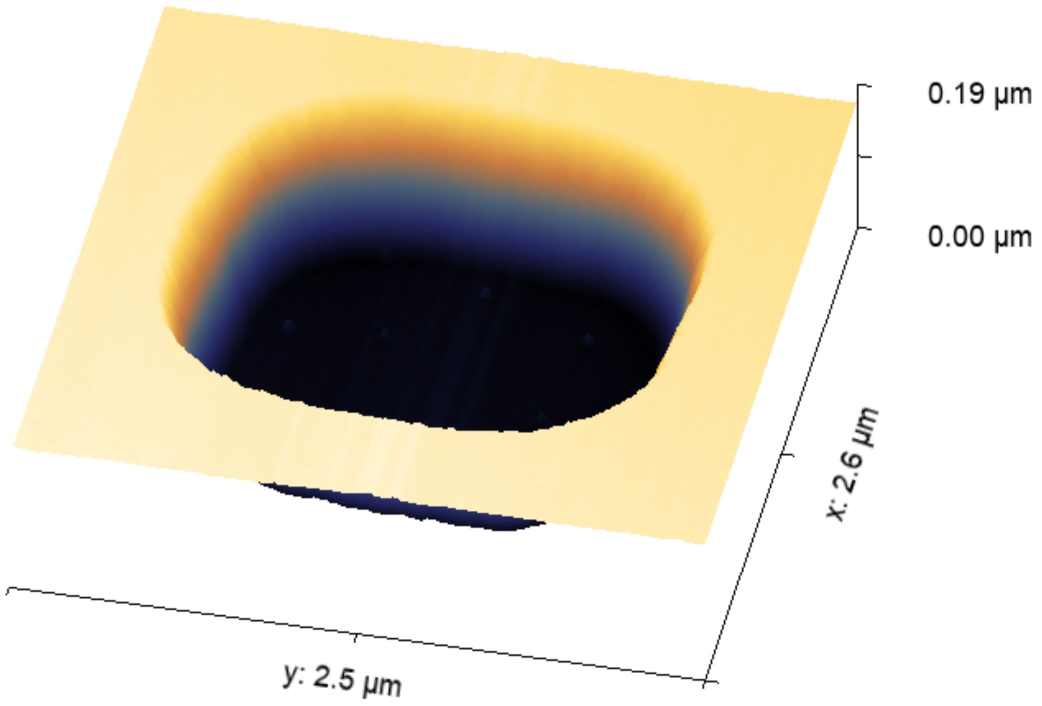




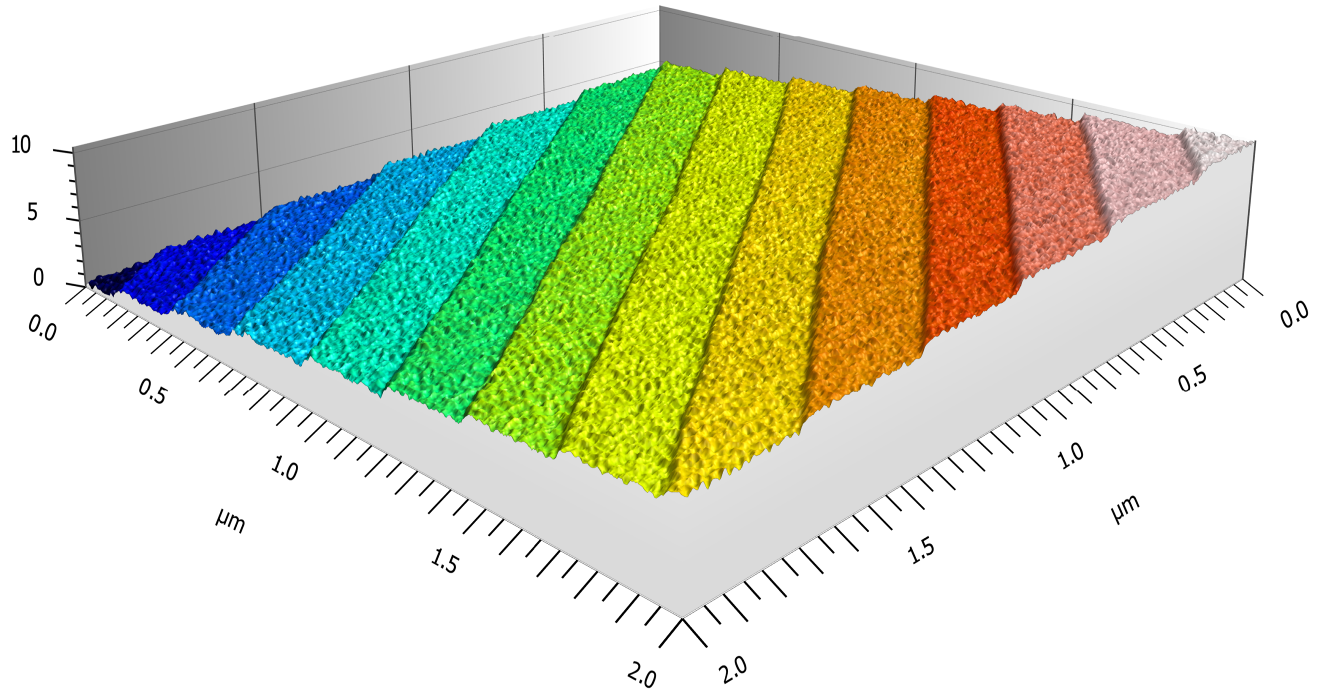
- Bow artifacts are caused by the non-linearity of the piezo extension over large lengths
- This artifact is common with XYZ scanner tubes.
  - Sometimes this can be corrected with flattening or plane-fitting.
  - XY scanners decoupled from the z scanner can mitigate the artifact

Piezo Calibration: Use of Standard Samples

VLSI Standard Piezo Calibration sample



SiC sample collected on new Anton Paar Tosca 400



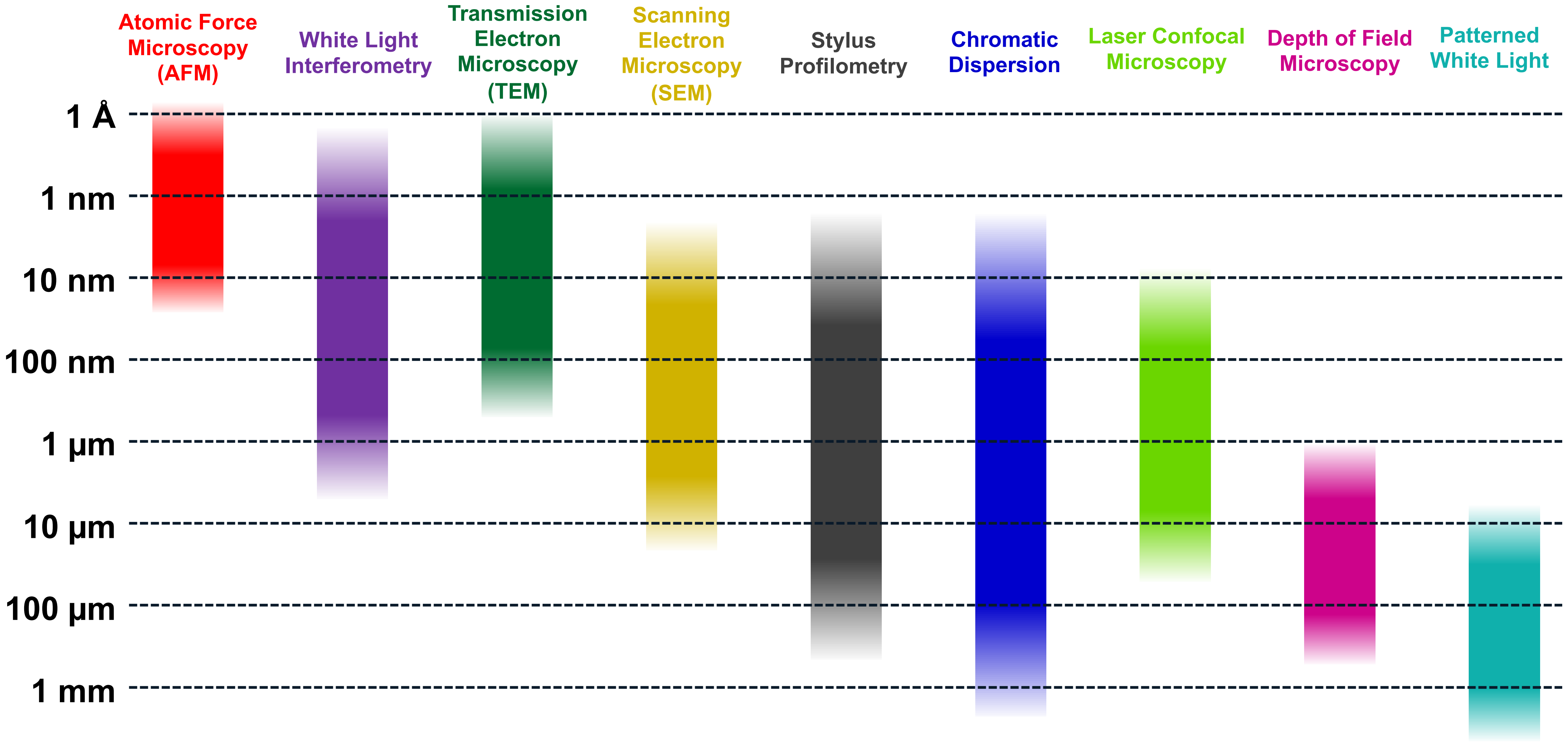
- X,Y, Z calibration can be achieved with a VLSI standard.
- Sub nm step heights (0.75 nm) can be calibrated against SiC samples.

Overview of Commercial AFM Companies

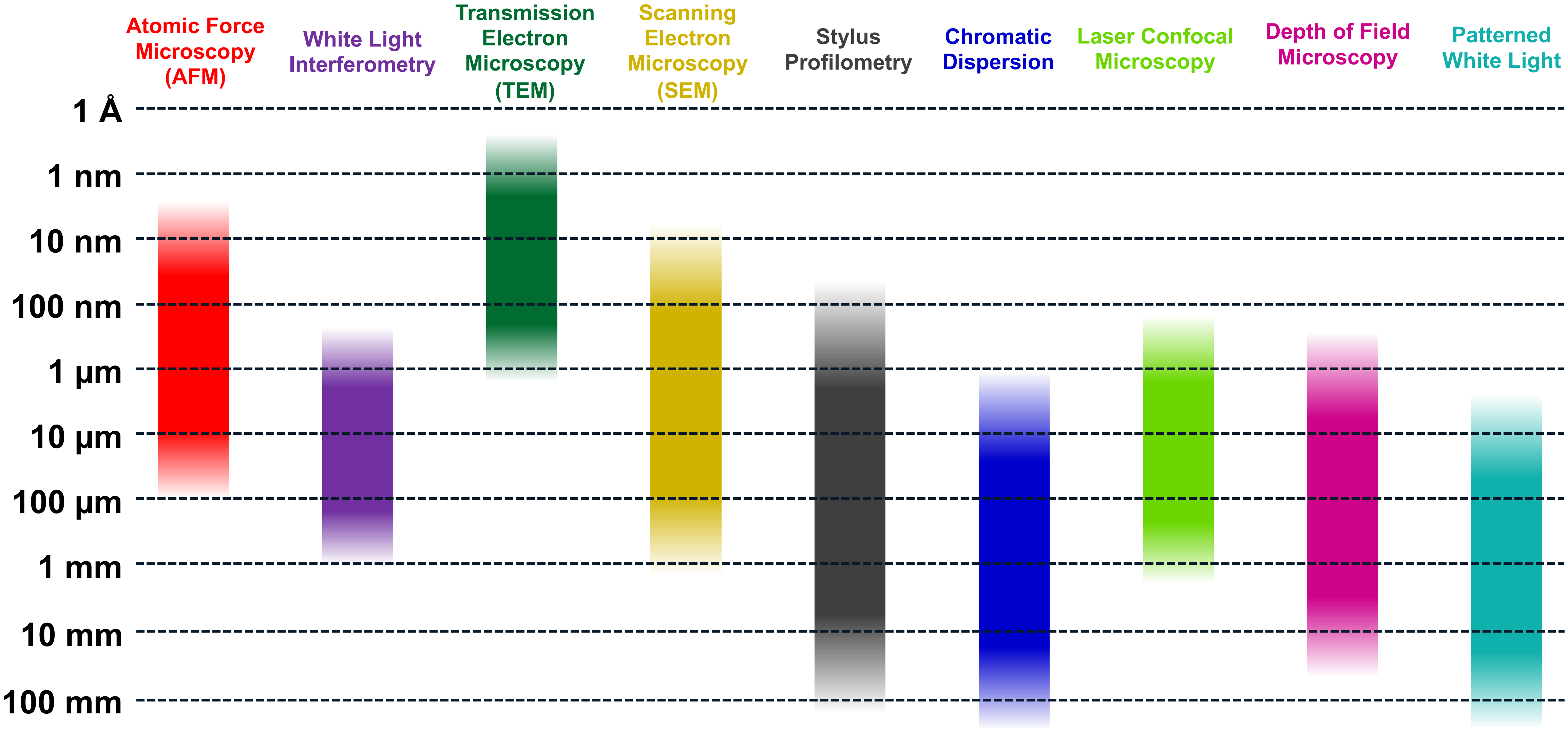
- **Bruker** – Leading manufacturer (acquired Veeco 10 years ago). Bruker Icon in-house at Covalent.
- **Park Systems** – Est. in the 90s as Park Scientific, dominant in industry.
- **Asylum Research** - Owned by Oxford Instruments, major player in academia.
- **Anton Paar** – new to AFM market, Tosca 400 AFM soon to be in-house at Covalent.

- All provide top of the line AFMs to handle large samples as well as offering low cost AFMs.

Summary of 3 Part Series
Roughness Scale (Vertical Range)



Summary of 3 Part Series
Roughness Scale (Horizontal Range)



Summary of 3 Part Series Product Catalog

COVALENT METROLOGY

Probe	"Stylus" Techniques		Optical				Electron Beam	
Technique	Atomic Force Microscopy	Stylus Profilometry	White Light Interferometry	Laser Scanning Confocal	Chromatic Confocal	3D White Light	Scanning Electron Microscopy	Transmission Electron Microscopy
You May Hear This Referred to As:	AFM or SPM	"Dektak"	Optical Profilometry	"Keyence"	"FRT"	“VR”	SEM or HRSEM	TEM
Key Manufacturers	Bruker	Bruker	Zygo	**Keyence**	FRT	**Keyence**	**ThermoFisher**	**ThermoFisher**
	Park Scientific	KLA	Bruker	Zeiss	Nanovea		JEOL	JEOL
	Asylum Research		PolyTec	Leica			Zeiss	Hitachi
	Anton Paar			Olympus			Hitachi	
	Molecular Vista			Nikon			Tescan	
	Many others						Nikon	
Substrate Size Limitations	200mm max to reach edges; 300m in center	Up to 300mm (Covalent @ 200mm)	Flexible, with fixtures	Flexible, with fixtures	Up to 300mm (Covalent @ 200mm)	Flexible, with fixtures	Most analytical services <200mm	Coupons
Field of View - Min to Max	200nm to 70µm	Not Applicable	160µm to 800µm	100µm to 1mm	Few um to 200mm	4mm to 24mm	1µm to 2mm	2nm to 10µm
Vertical Resolution	Å	nm	Å	nm	nm	Generally 3-5µm	nm	Å
Particular challenges	Sample should be smooth	No mapping	Reflectivity, +/- vertical ambiguity	Reflectivity, Steep Slopes	Steep Slopes	Resolution, shadowing, reflectivity	Non-conductive samples charging	Very small field of view
Cost @ Covalent	$175 / scan	$225 / hr	$275 / hr	$300 / hr	$300 / hr	$275 / hr	$275 / hr (standard res) $375 / hr (hi-res)	$900 / sample (standard sample)

Advanced AFM Modes

AFM Modes	Samples	Typical Data
Contact AFM	Surface roughness	Ra, Rq
Tapping Mode AFM	Surface roughness	Ra, Rq, Phase imaging
E-AFM (Electrostatic)	Metal/Semiconductor films	Electrostatic Potential
MFM (Magnetic)	Magnetic films	Magnetic Domain Stucture
Peak Force AFM	Thin films	Roughness, Surface Topography.
PF-QNM (Quantitative Nanomechanical)	Polymer coatings	Roughness, Surface Topography, Polymer Domains
AM-KPFM (Kevin-Probe, Amplitude Mod)	Thin films, semiconductors	Surface Potential
PFM (Piezo)	Piezo materials	Piezo Domain Structure
SCM (Scanning Capacitance)	Semiconductor films	Dopant profile, Relative Capacitance
FM-KPFM (Kevin-Probe, Frequency Mod)	Thin films, semiconductors	Surface Potential
Force Modulation	Polymer blends	Relative Hardness of Viscolastic Materials
Conductive AFM	Thin films, thin oxide	Leakage Currents, Variations in Conductivity
TUNA (Tunneling)	Thin films, thin oxide	Low Leakage Currents, Variations in Conductivity
PF-TUNA (Tunneling)	Thin films, thin oxide	Low Leakage Currents, Variations in Conductivity
SSRM (Scanning Spreading Resistance)	Semiconductor films	Dopant profile, Relative Resistance

- Many advanced AFM modes – surface property information other than topography/roughness.
- These techniques will be discussed in a later webinar.

Acknowledgments

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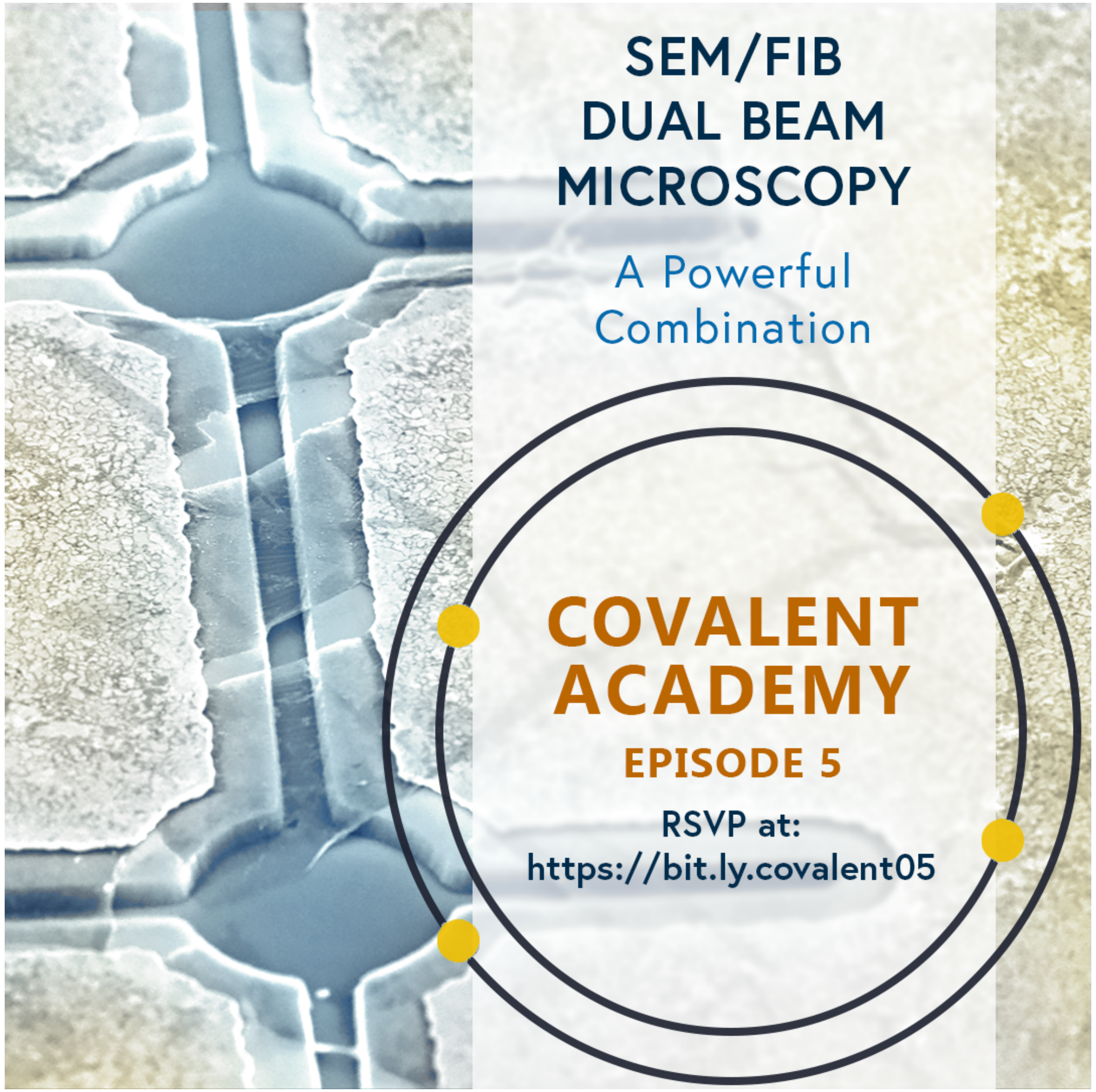
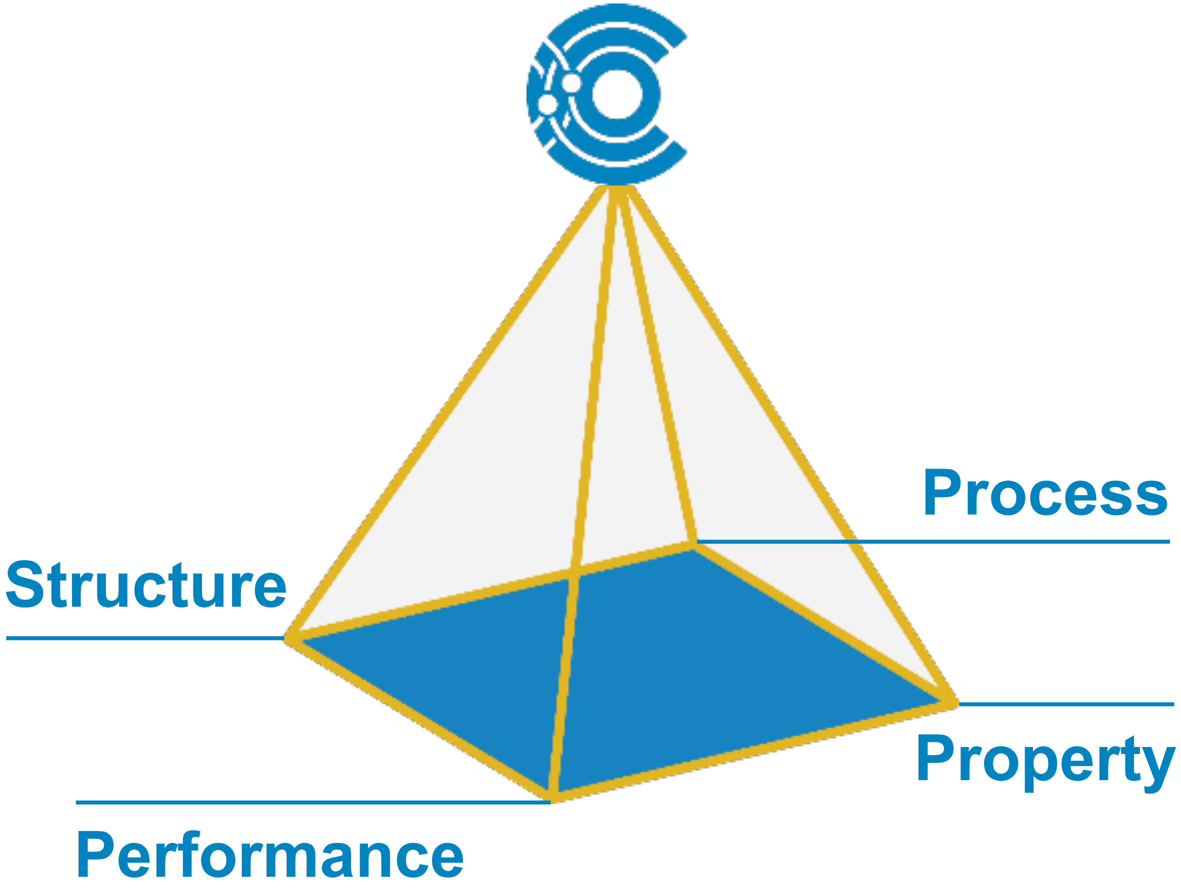
- Nicholas Schacher, *NanoAndMore*.
- Patrick O’Hara, *Anton Paar*.
- Maxwell Junda, *Covalent Metrology*.
- Matt Wong, *Covalent Metrology*.

Next Episode



Expertise You Can Trust



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www.covalentmetrology.com

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(408) 498-4611

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hello@covalentmetrology.com

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