ME597/PHYS57000
Fall Semester 2009
Lecture 20

Recommended reading

"AFM Image Artifacts" by West and Starostina

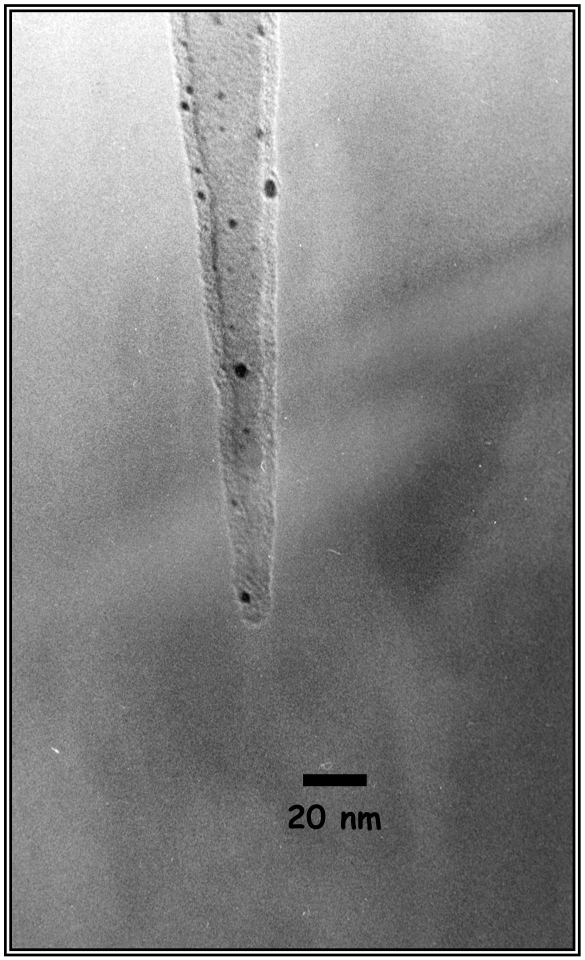
http://www.lot-oriel.com/site/site_down/pn_artifacts_deen.pdf

OUTLINE

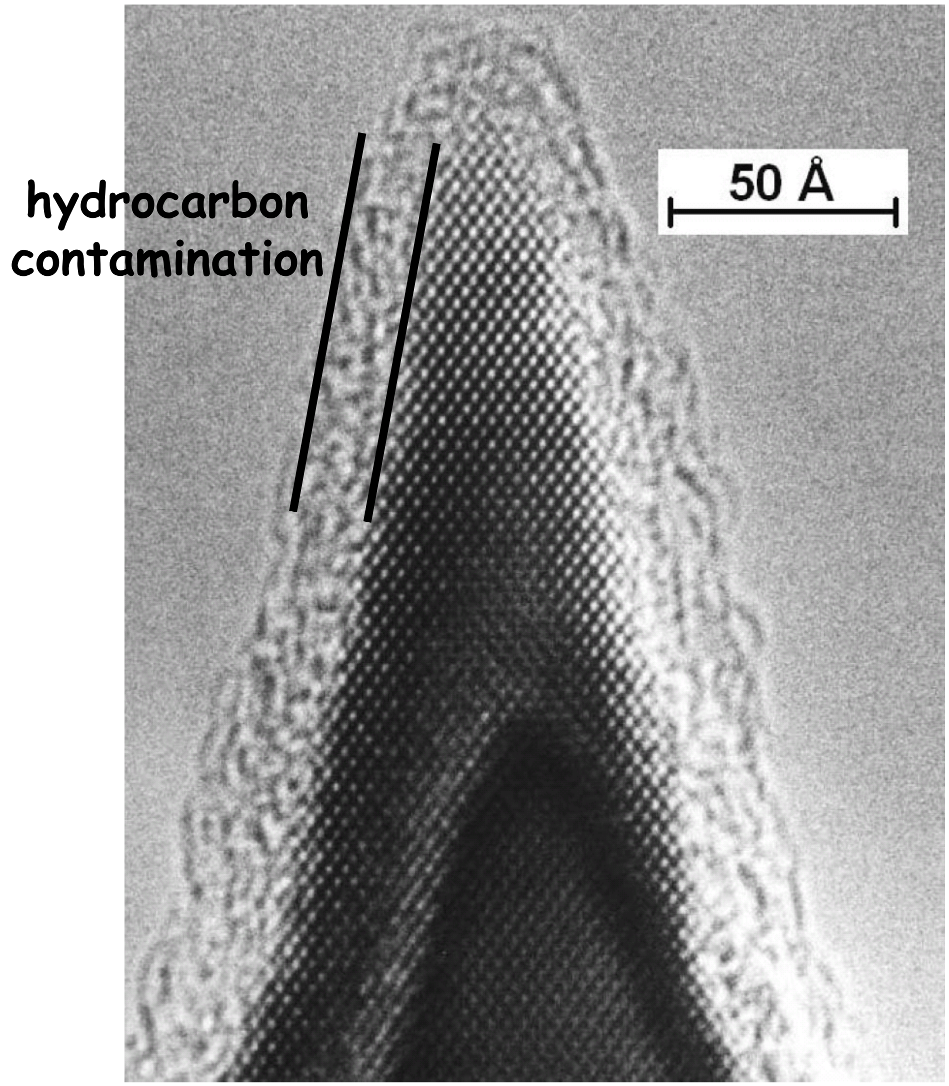
- Probe Tip Artifacts
- Instrumental Artifacts
- Large Force Artifacts
- Image Processing Artifacts
- Intrinsic Limitations
- Tip Cleaning

Probe Tip Artifacts

A Good Tip

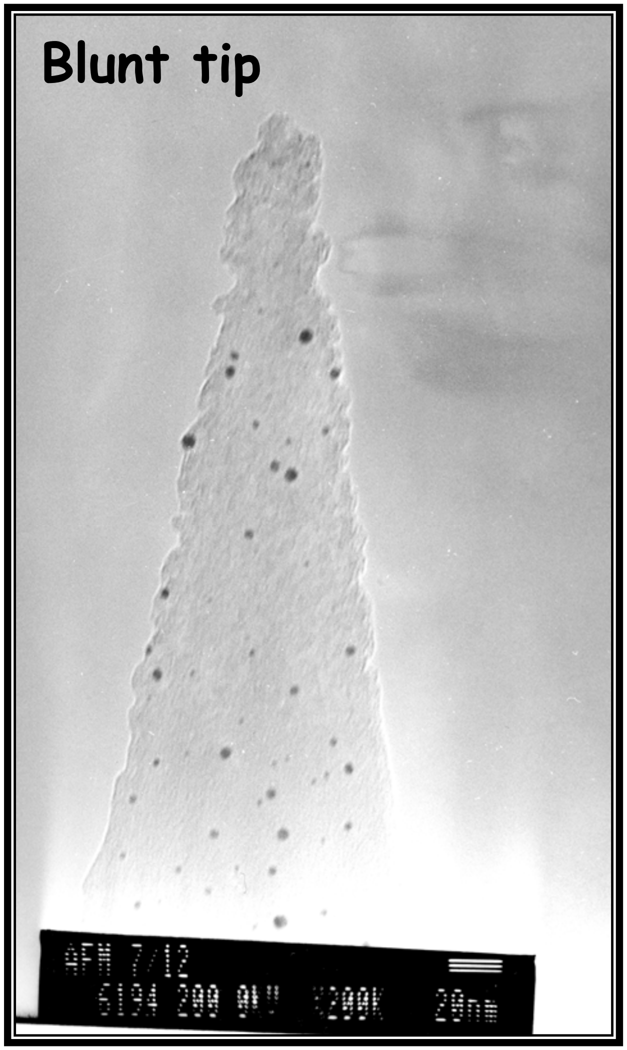


D. Schaefer, PhD Thesis, Purdue University (1993)



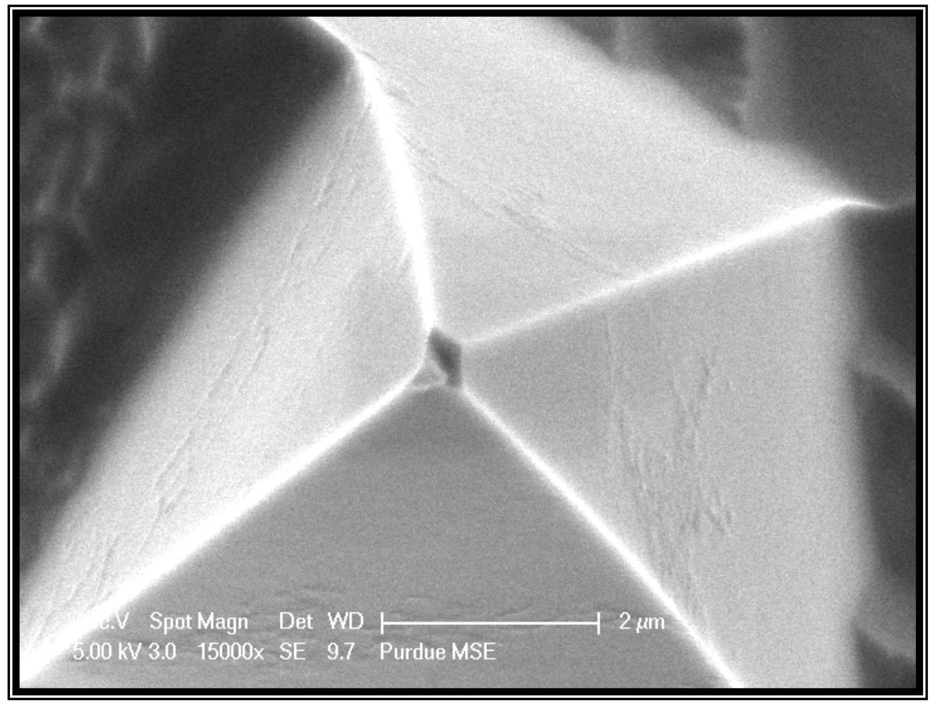
R. Marcus et al., Appl. Phys. Lett. 56, 236 (1990)

Real Tips



D. Schaefer, PhD Thesis, Purdue University (1993)

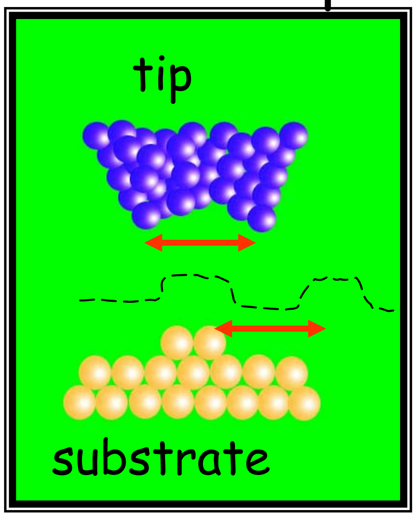
Broken tip



G. Prakash, PhD Thesis, Purdue University (2010)

Tip Imaging Artifacts

Double Tip



substrate feature repeated in image

Tip Dilation

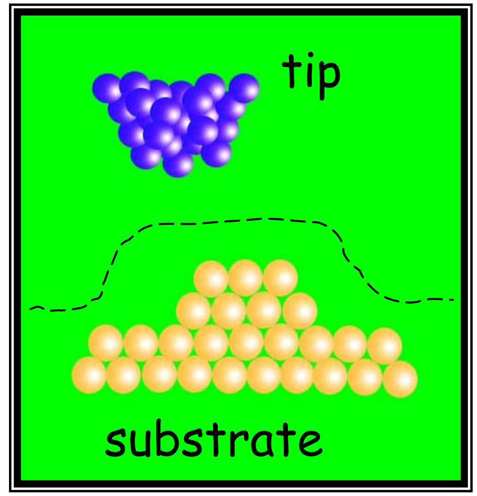
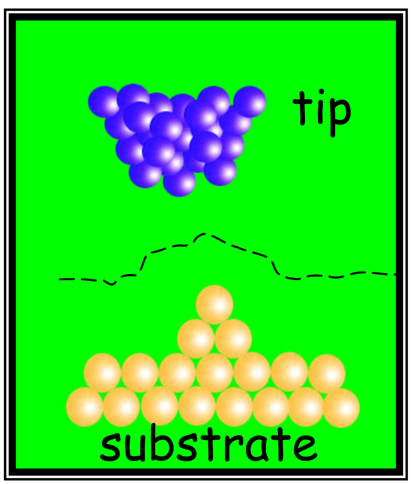


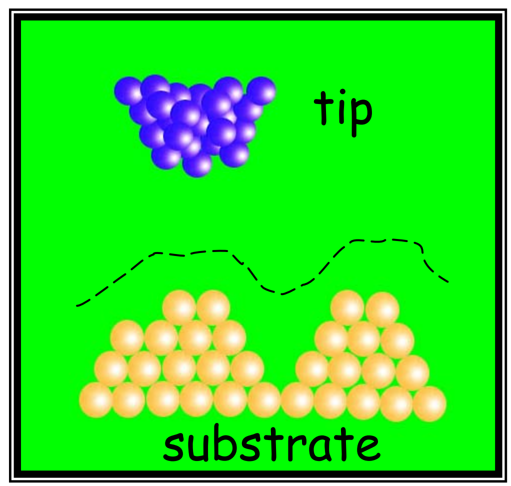
image of feature in substrate broadened by tip “radius”

Sharp Feature in Substrate



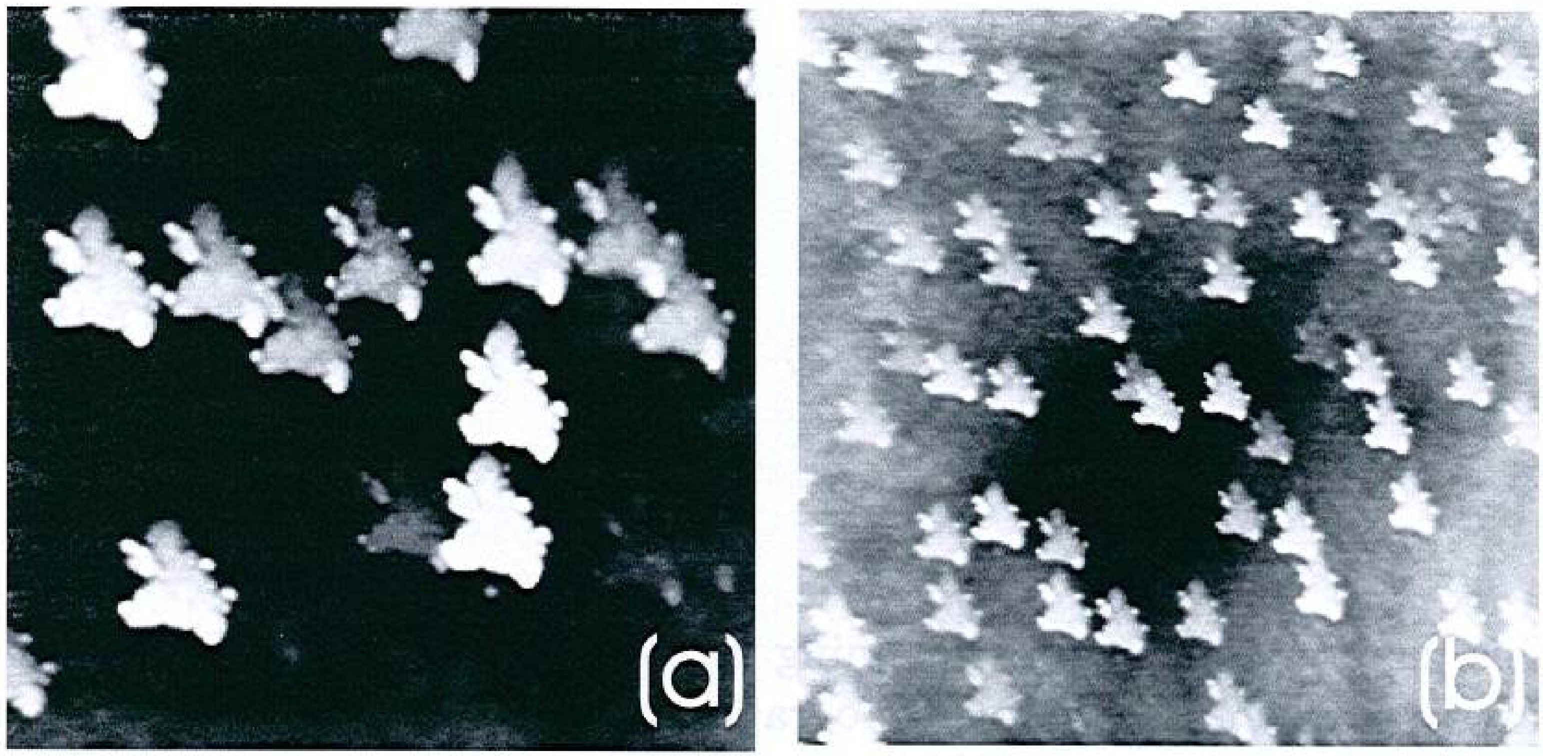
substrate feature images tip

Wide Tip



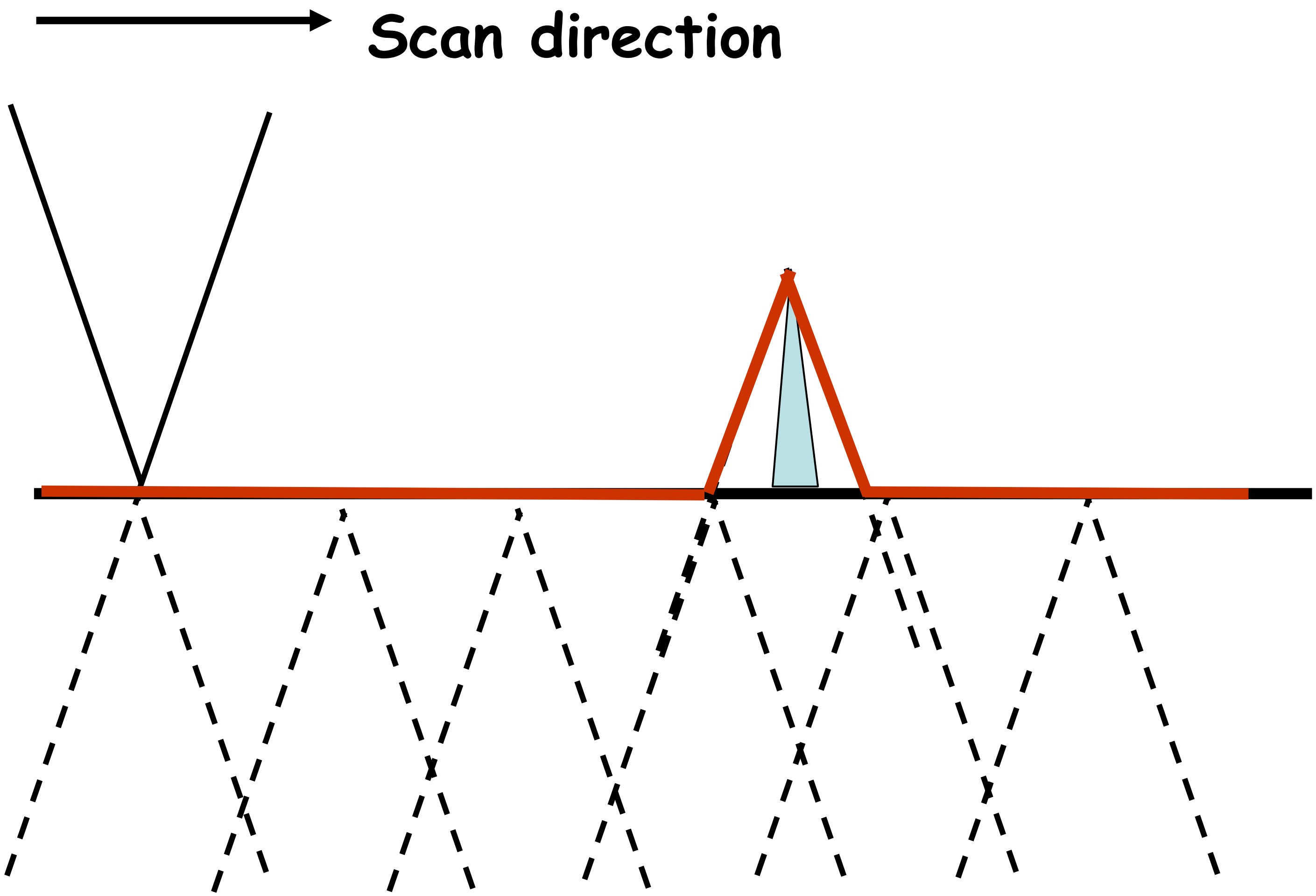
depth of feature in substrate inaccurate

Double Tip Image



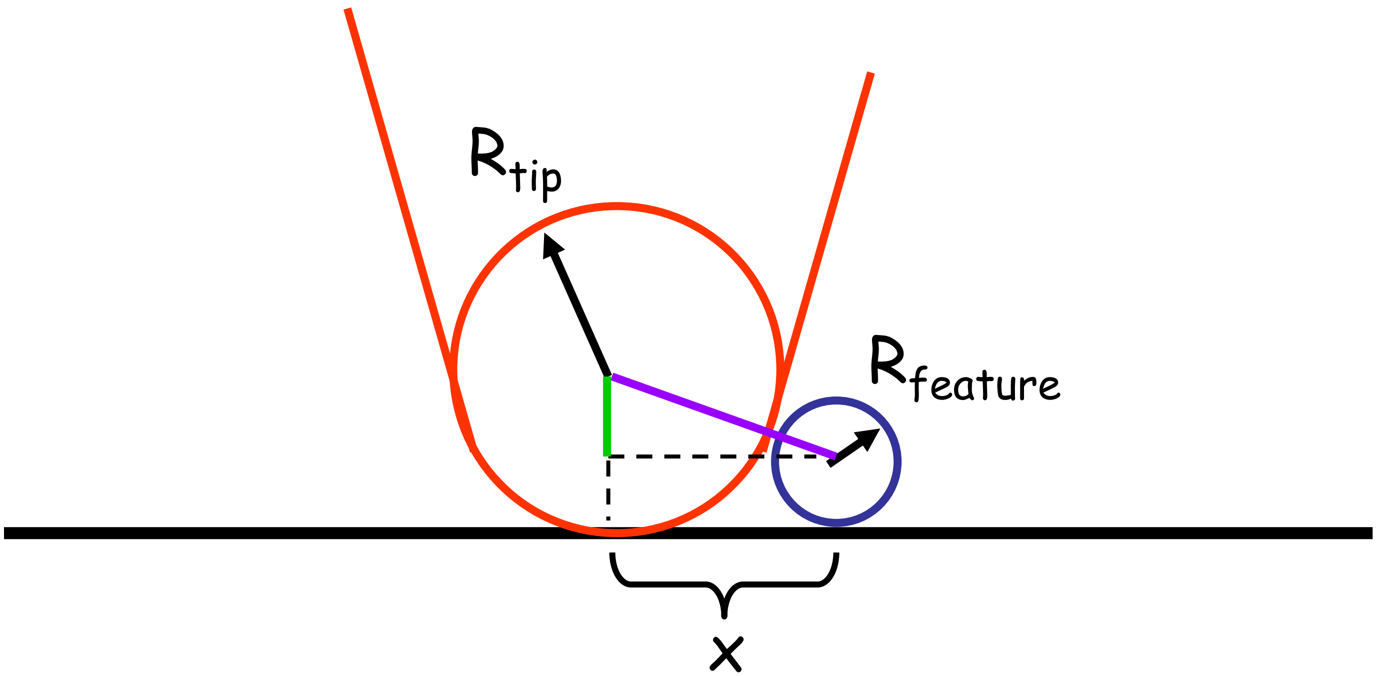
E. Meyer, H.J. Hug and R. Bennewitz, *Scanning Probe Microscopy*, Springer (2003).

The Tip Dilation Algorithm



J.S. Villarrubia, *Journal of NIST*, 102 425 (1997)

Apparent width of small object



$$x^2 = (R_{tip} + R_{feature})^2 - (R_{tip} - R_{feature})^2$$

$$x^2 = R_{tip}^2 + 2R_{tip}R_{feature} + R_{feature}^2 - R_{tip}^2 + 2R_{tip}R_{feature} - R_{feature}^2$$

$$x = 2\sqrt{R_{tip}R_{feature}}$$

$$apparent\ \ feature\ width \simeq 2x = 4\sqrt{R_{tip}R_{feature}}$$

Summary: Tip Artifacts

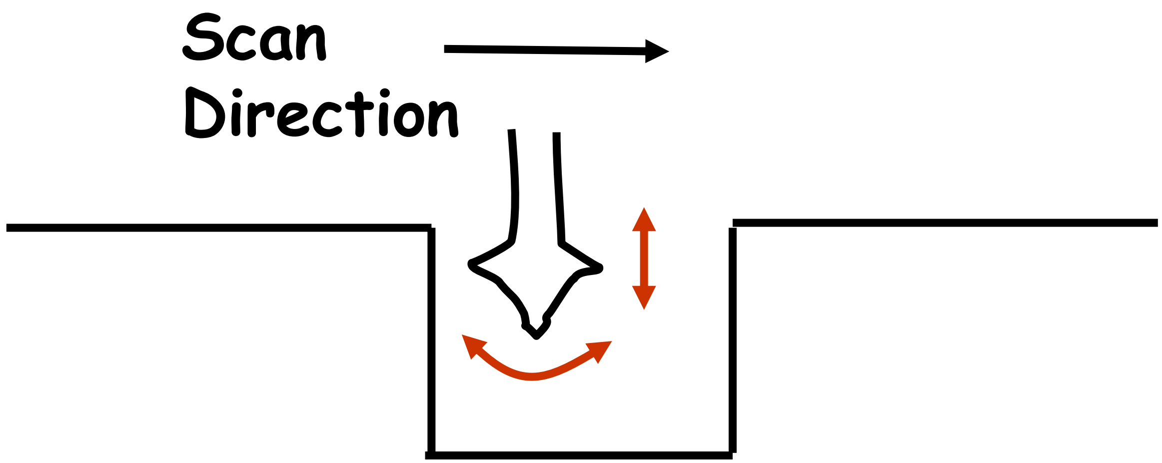
Rule of thumb – any feature with a radius of curvature less than radius of curvature of tip is not accurately imaged.

Lesson: Choose a tip shape/cantilever consistent with what you are trying to accomplish.

Mode	L(μm)	W(μm)	t(μm)	f_o (kHz)	k_c (N/m)
Contact	400	30	2	18	0.2
Non-Contact	150	30	5	350	50
Non-Contact	250	30	7	165	30
Lateral Force	250	30	1	25	0.1
Electrostatic	250	30	3	70	2.5
Magnetic Force	250	30	3	70	2.5
Under liquids	60	30	0.16	37	0.05

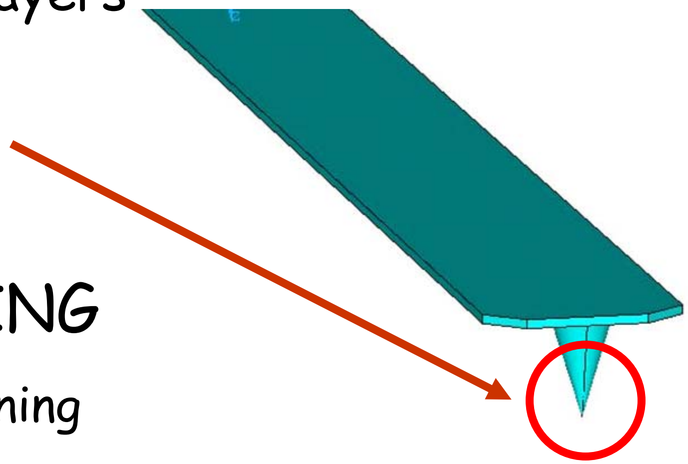
Approximate values

Sometimes, special tip shapes are required



Tip Care

- Thin organic layers
- Oxide layers
- Particulates



DRY CLEANING

- UV (ozone) cleaning
- Heating (pyrolosis)
- Argon/Oxygen/Air plasma (glow discharge)
- Sputtering (UHV)
- Indenting
- CO_2 "snow"

WET CLEANING

- Chemical Etching
- Ultrasonic cavitation
- Passivation (coating)

Tip Surface:

- SiO_2
- Si_3N_4
- Au-coated
- Pt-coated

Unknown microstructure at the nanoscale? (Au-coated, Pt-coated)

Tip Shape:

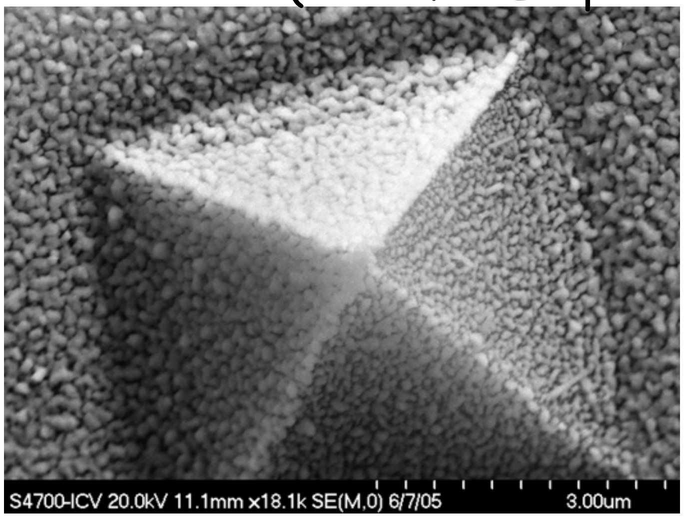
- Pyramidal
- Conical

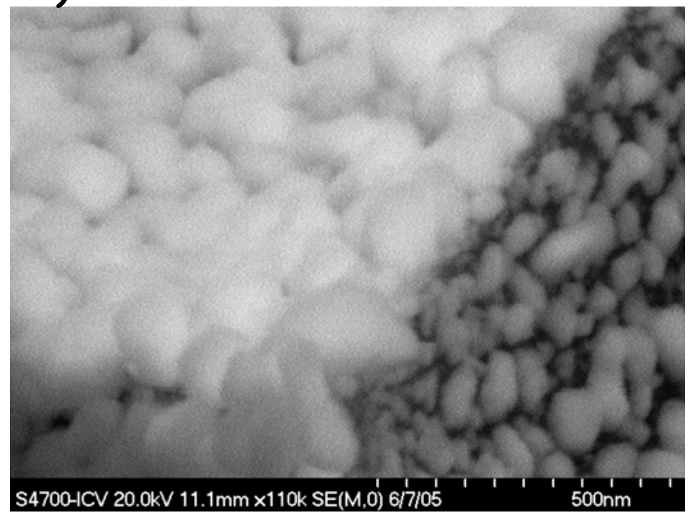
Unknown tip morphology at the nanoscale? (Pyramidal, Conical)

Assumption: Cleaning the tip is roughly equivalent to cleaning the whole cantilever.

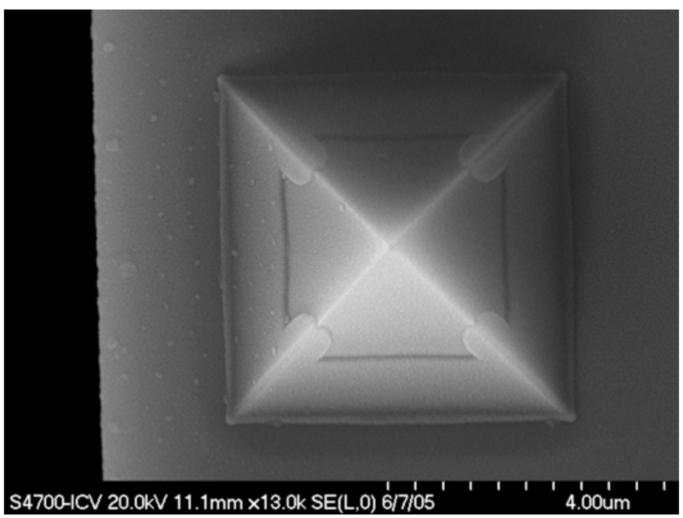
Microstructure of Metal Coating

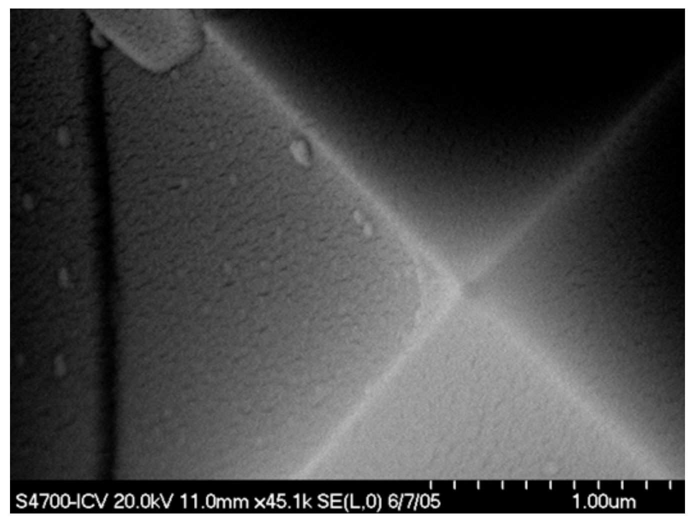
Gold coated (Thermal Evaporation):





Gold/Palladium coated (Sputtered):



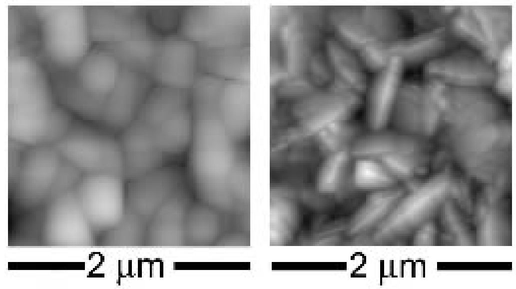


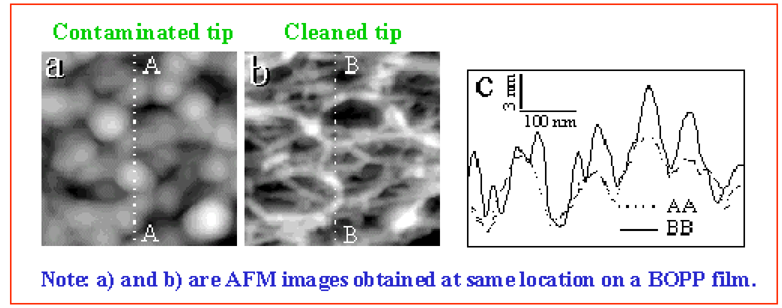
J. Gomez-Herrero, Univ. Autonoma de Madrid

The Problem

How do you know the tip is dirty?

1. Low resolution:





2. Strong adhesive force observed:

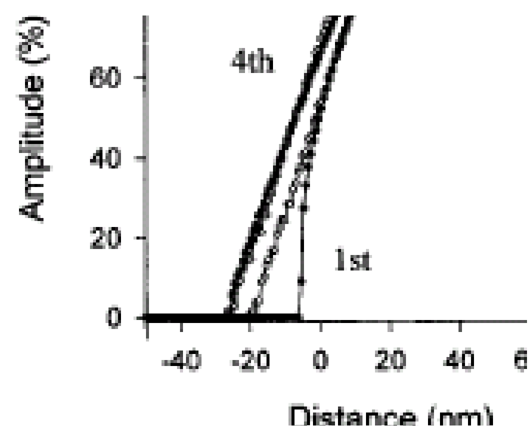
Adhesion due to water (typical):

$F=4\pi R\gamma\cos(\theta)$ – capillary force for sphere-plane geometry

$R=10\text{-}15$ nm; $\gamma=0.0073$ N/m; $\theta = 46^{\circ}$ (from chip) $\longrightarrow$ $F=6\text{-}10$ nN

Adhesive Force Histograms are a must!

3. Hysteresis in Amplitude vs. z data:



Tests for Scanning Artifacts
(The R^3C^2 Rule)

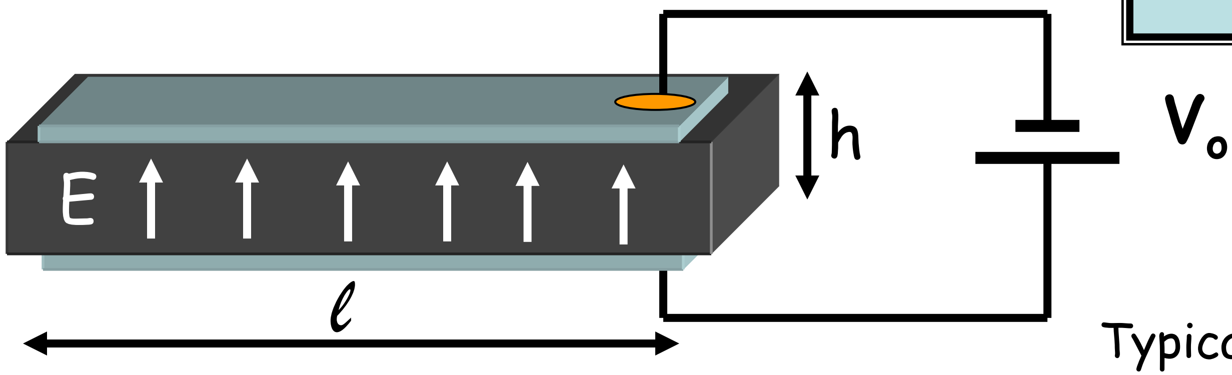
- Repeat the scan – does it look the same?
- Reverse the scan direction, does the new image look like the original one?
- Rotate the scan direction; do the features rotate as expected?
- Change the scan size; do the size of features scale properly
- Change the scan speed; do the features remain stationary?

Instrumental Artifacts

Ideal Piezos

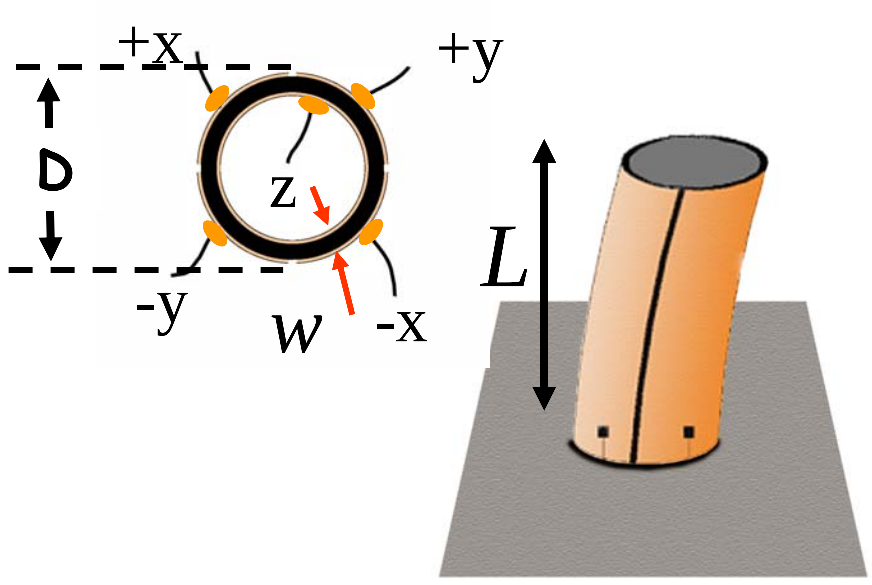
$$\Delta \ell = d_{31} \frac{\ell}{h} V_o$$

Piezoelectric Bar



Typically, $\Delta \ell$ ~ 0.5 nm/V

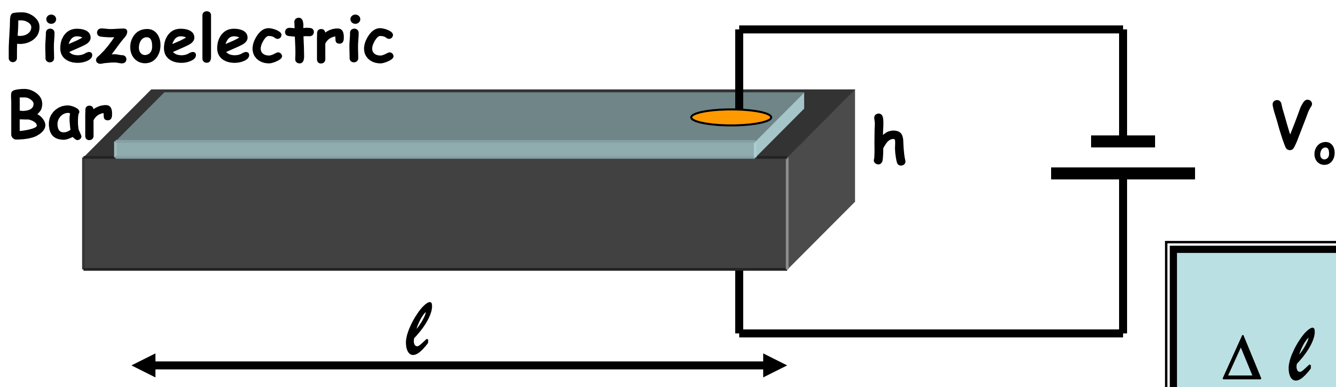
Quadranted Piezoelectric Tube



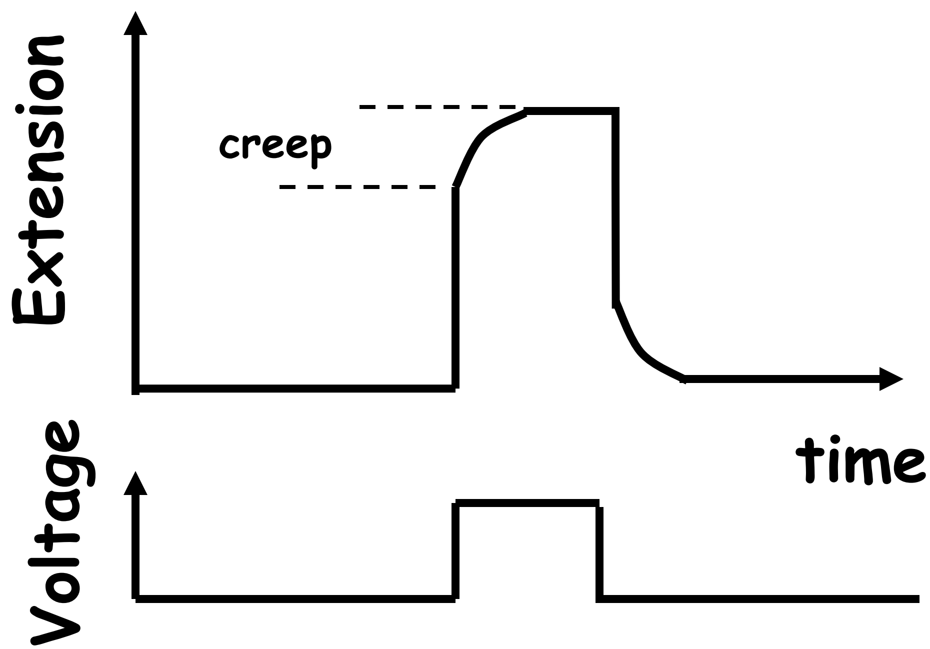
$$\Delta z = L \frac{d_{31}}{w} V_o$$

$$\Delta x \simeq \Delta y = \frac{2\sqrt{2}}{\pi D} \frac{L^2 d_{31}}{w} V_o$$

Limitation: Piezo Creep

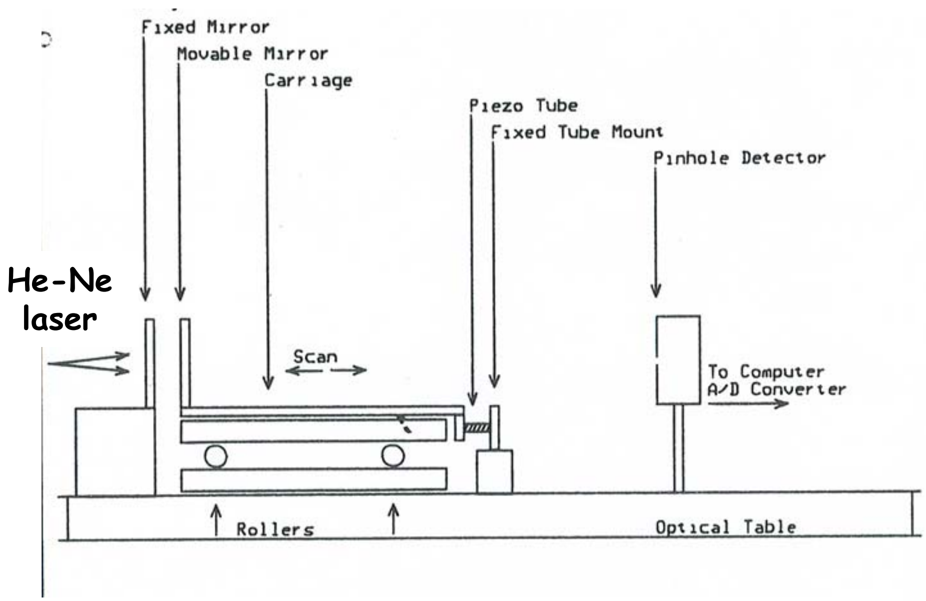


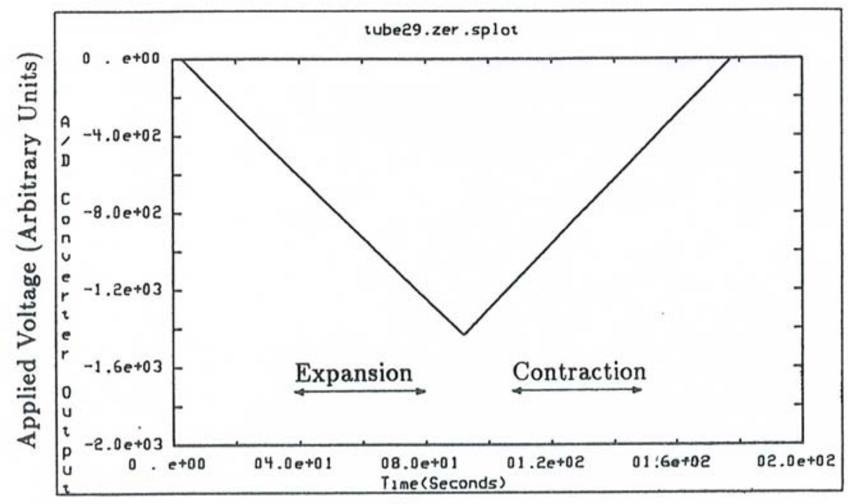
$$\Delta \ell = d_{31} \frac{\ell}{h} V_o$$



Limitation: Piezo Hysteresis

Calibrating piezo tubes





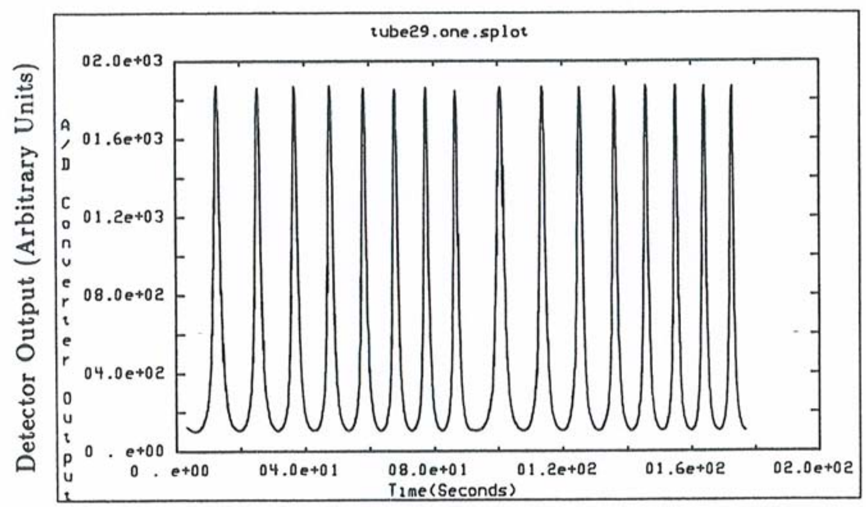
Fabry-Perot Interferometer

$$I_{\det} \propto \frac{I_{inc}}{1+\frac{4r^2}{\left(1-r^2\right)^2}\sin^2\left(\frac{\delta}{2}\right)}$$

$$\delta = \frac{4\pi d}{\lambda}$$

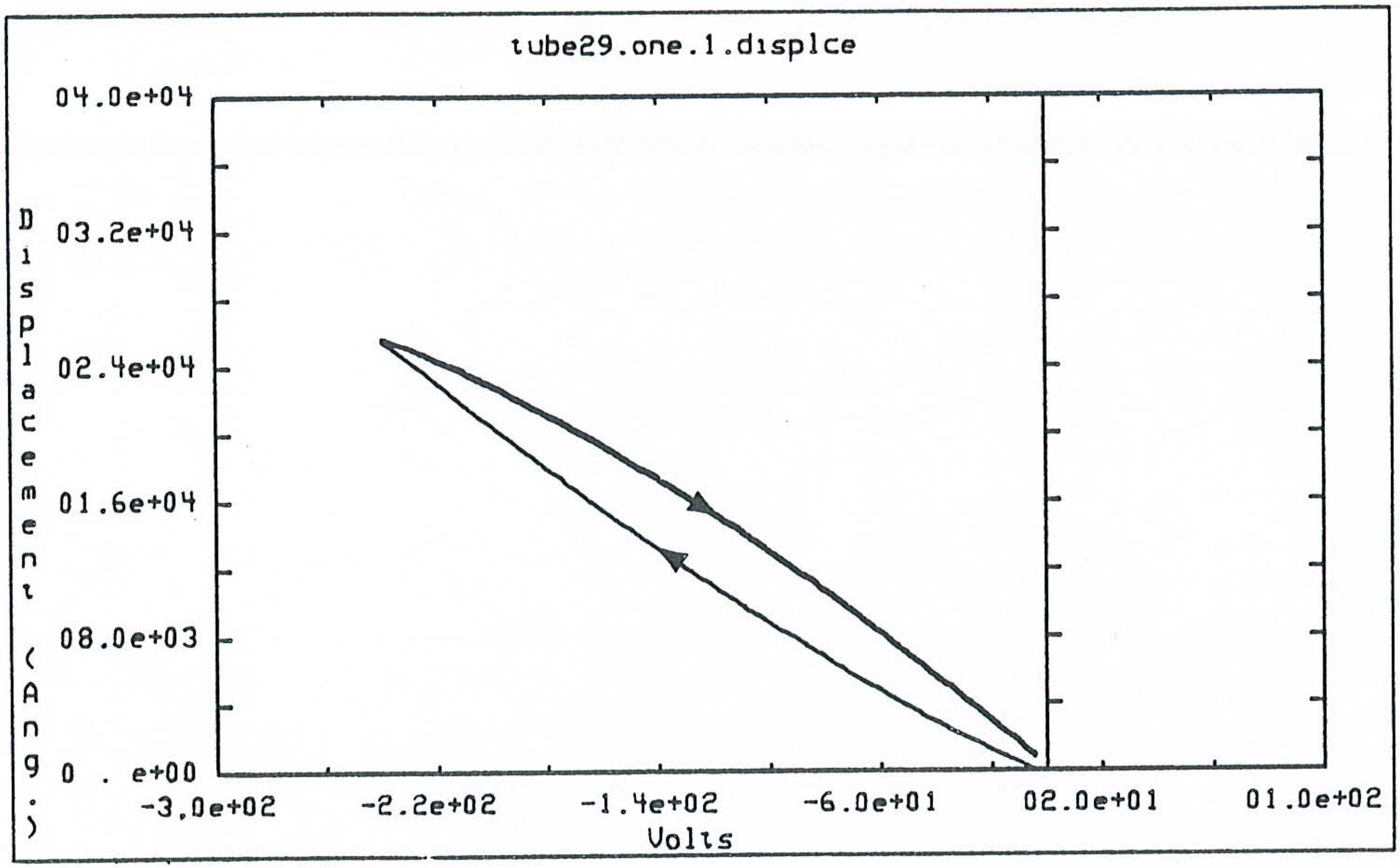
d is distance between mirrors

$$d(t) = d_o + V_{app}\left(t - t_o\right)$$



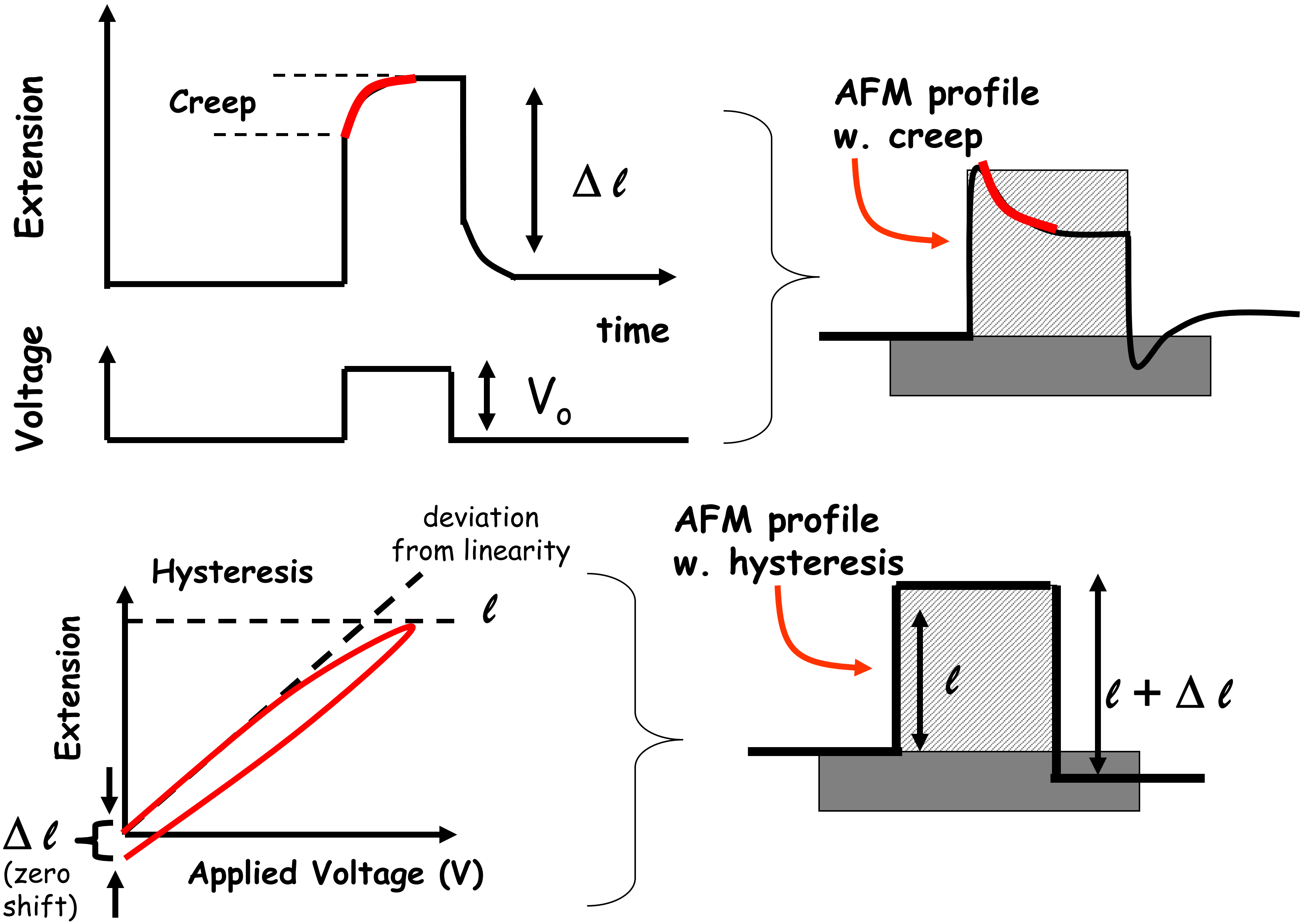
R. Piner, PhD thesis, Purdue University, (2000)

Limitation: Piezo Hysteresis



R. Piner, PhD thesis, Purdue University, (2000)

Effect of Piezoelectric Creep and Hysteresis



Overcoming the Limitations

- Use Closed Loop Scanners for X and Y motion -

• Flexure (hinge-like) design eliminates friction/stiction

• Feedback on absolute position with high resolution using strain gauges, capacitors or inductors (LVDTs)

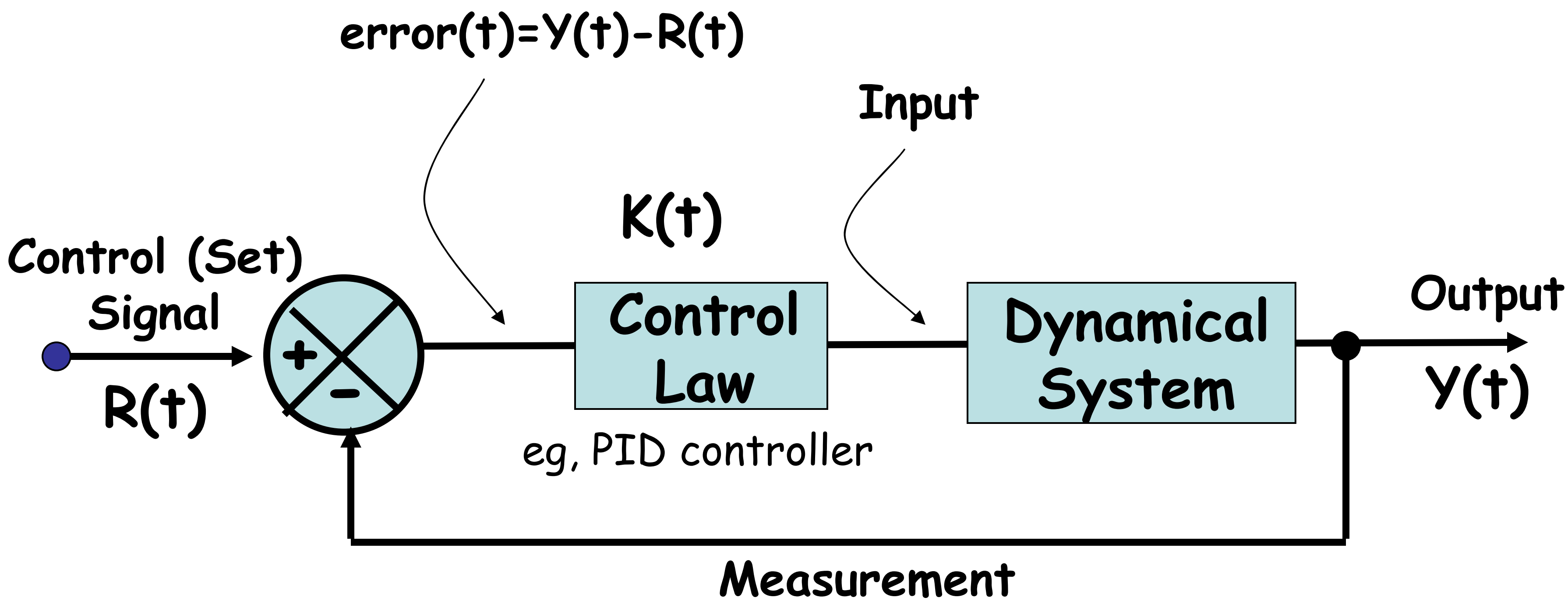
Advantages

• Absolute position monitored in real time

•Accuracy/repeatability traceable to optical interferometer calibration

Principle of Feedback Control

Goal: Make Y(t) follow R(t) as closely as possible



- K(t) tries to minimize error(t)
- Negative feedback!

The PID Controller

$$e(t) = Setpoint - Measurement(t)$$

$$P = K_p e(t) \qquad I = K_i \int_0^t e(\tau)\,d\tau \qquad D = K_d \frac{de}{dt}$$

0

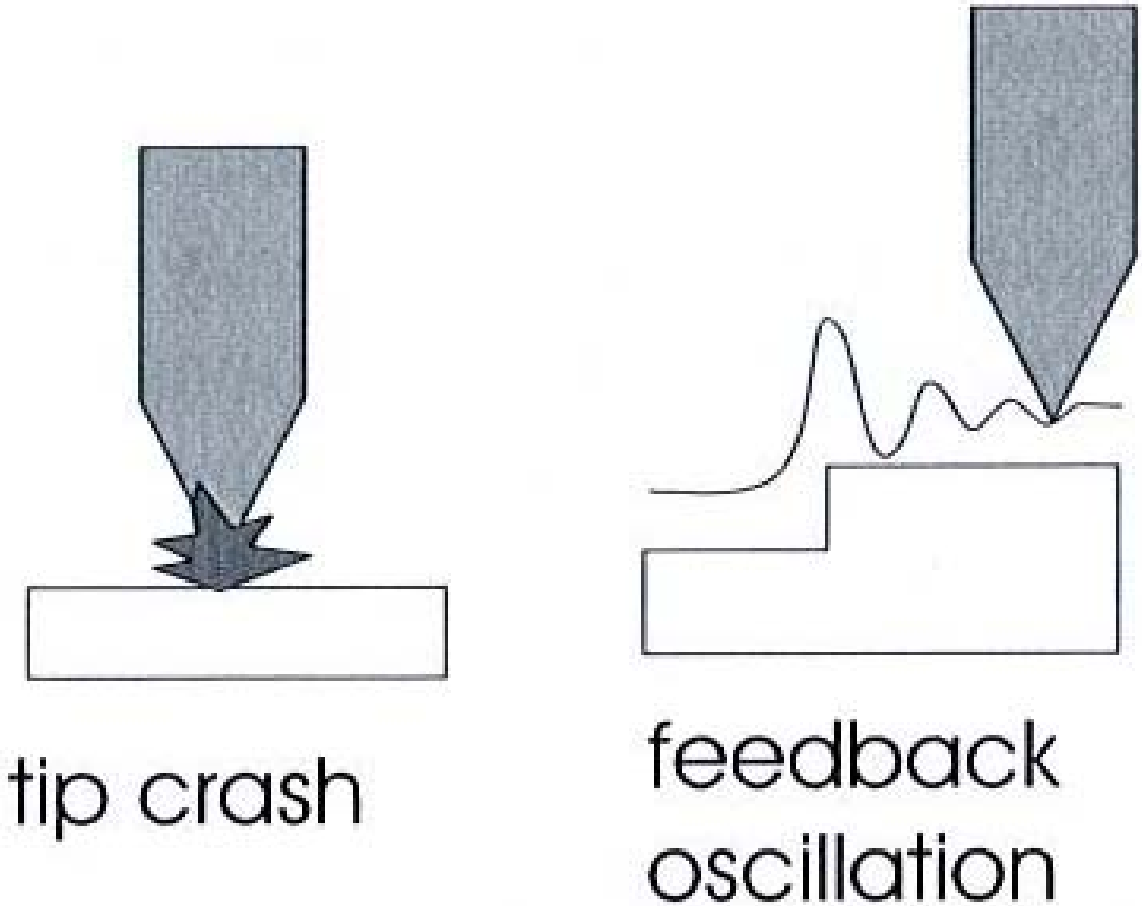
$$Output(t) = Y(t) = P + I + D$$

$$Output(t+\Delta) = Y(t+\Delta) = K_p e(t) + K_i Output(t) + K_d \left[e(t) - e(t-\Delta)\right]; \qquad K_i \equiv 1$$

Rules of thumb:

- **Larger K_p means faster response since the larger e(t), the larger the feedback.**
- **Larger K_i means steady state errors are eliminated quickly. The tradeoff is overshoot.**
- **Larger K_d decreases overshoot but slows down transient response. Usually set K_d = 0 if system is noisy.**

When feedback is not set properly



E. Meyer, H.J. Hug and R. Bennewitz, *Scanning Probe Microscopy*, Springer (2003).

Feedback Warning Signs

If feedback is too slow, blurred images result

If feedback is too slow, tip crashes result

If feedback is too fast, feedback oscillations or overshoots result

Typical Procedure:

- Set integral gain to zero (K_i=0)
- Set proportional gain (K_p) to ~2/3 value at which oscillations are observed
- Increase integral gain until very first signs of oscillations are observed

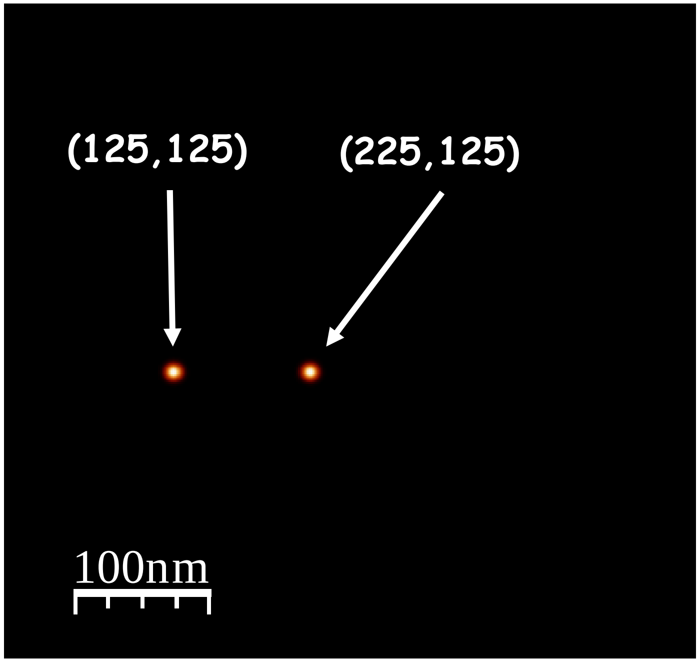
Image Processing Artifacts

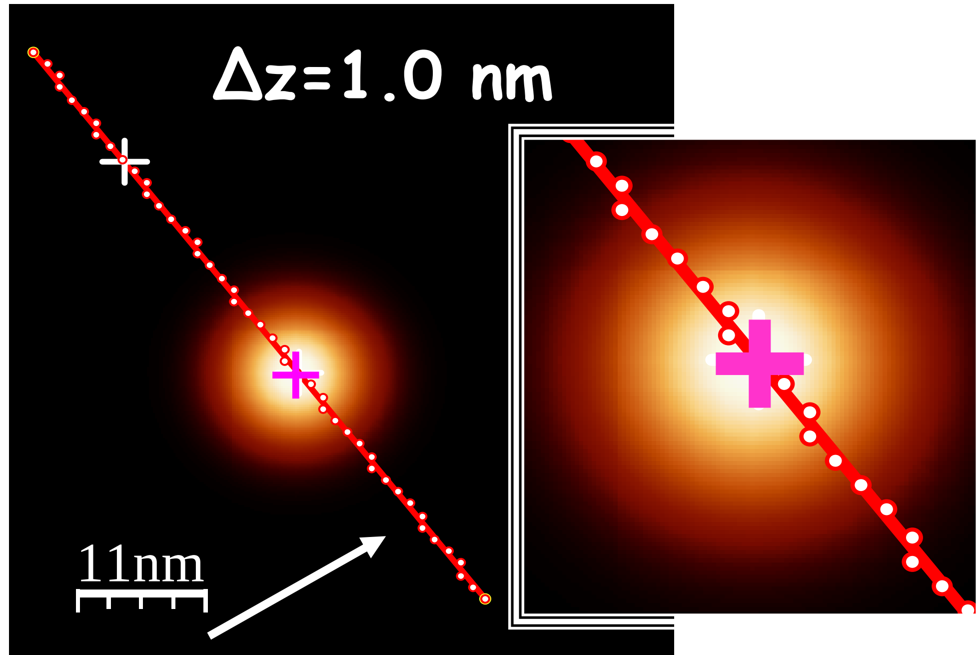
Why trust the image processing algorithms?

Check out image software by generating known images

Generating a known image

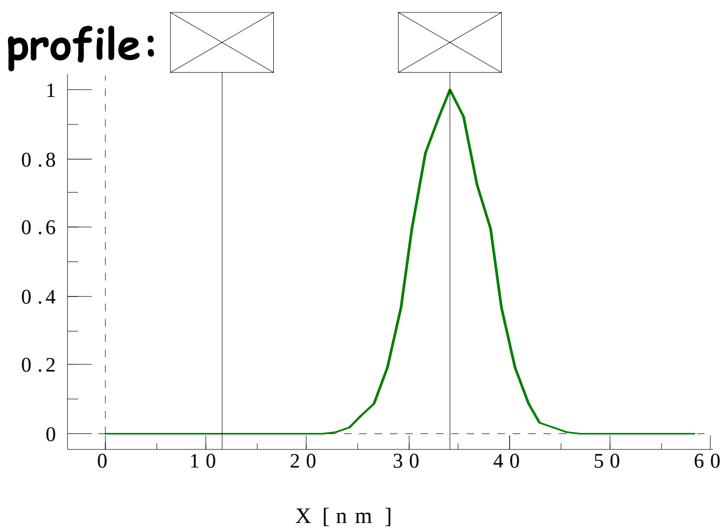
f(x,y)=exp(-0.04*(x-125)^2-0.04*(y-125)^2) + exp(-0.04*(x-225)^2-0.04*(y-125)^2)





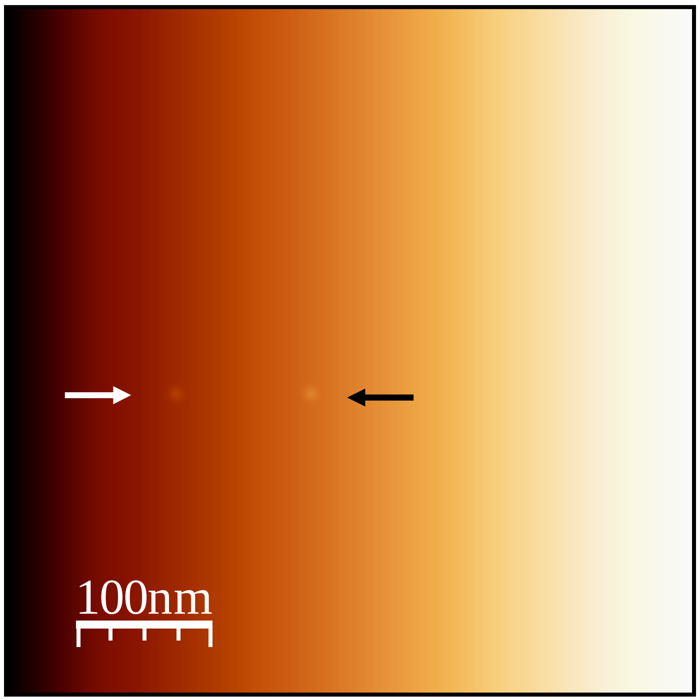
Can you see the individual data points?

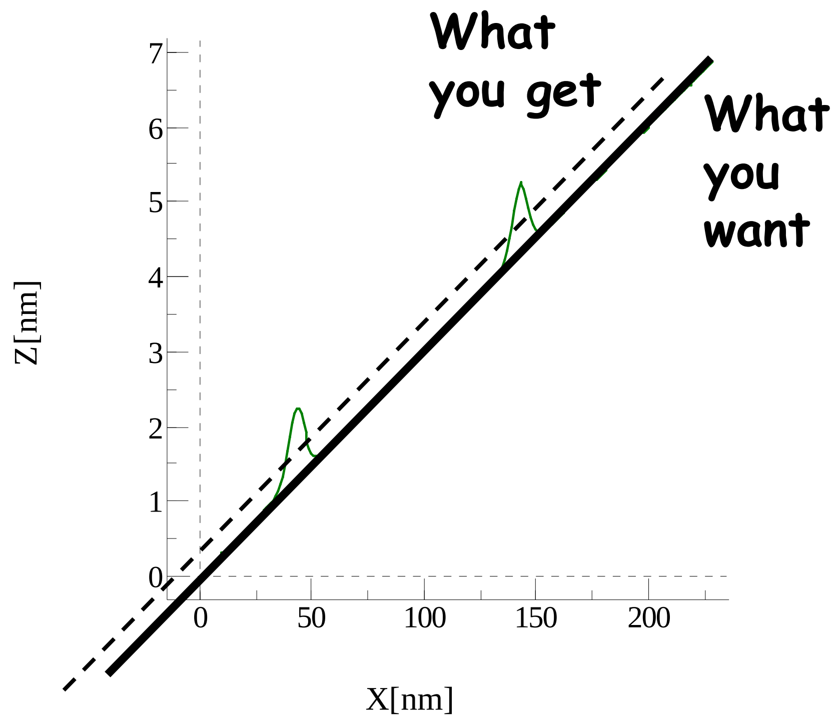
Height profile:



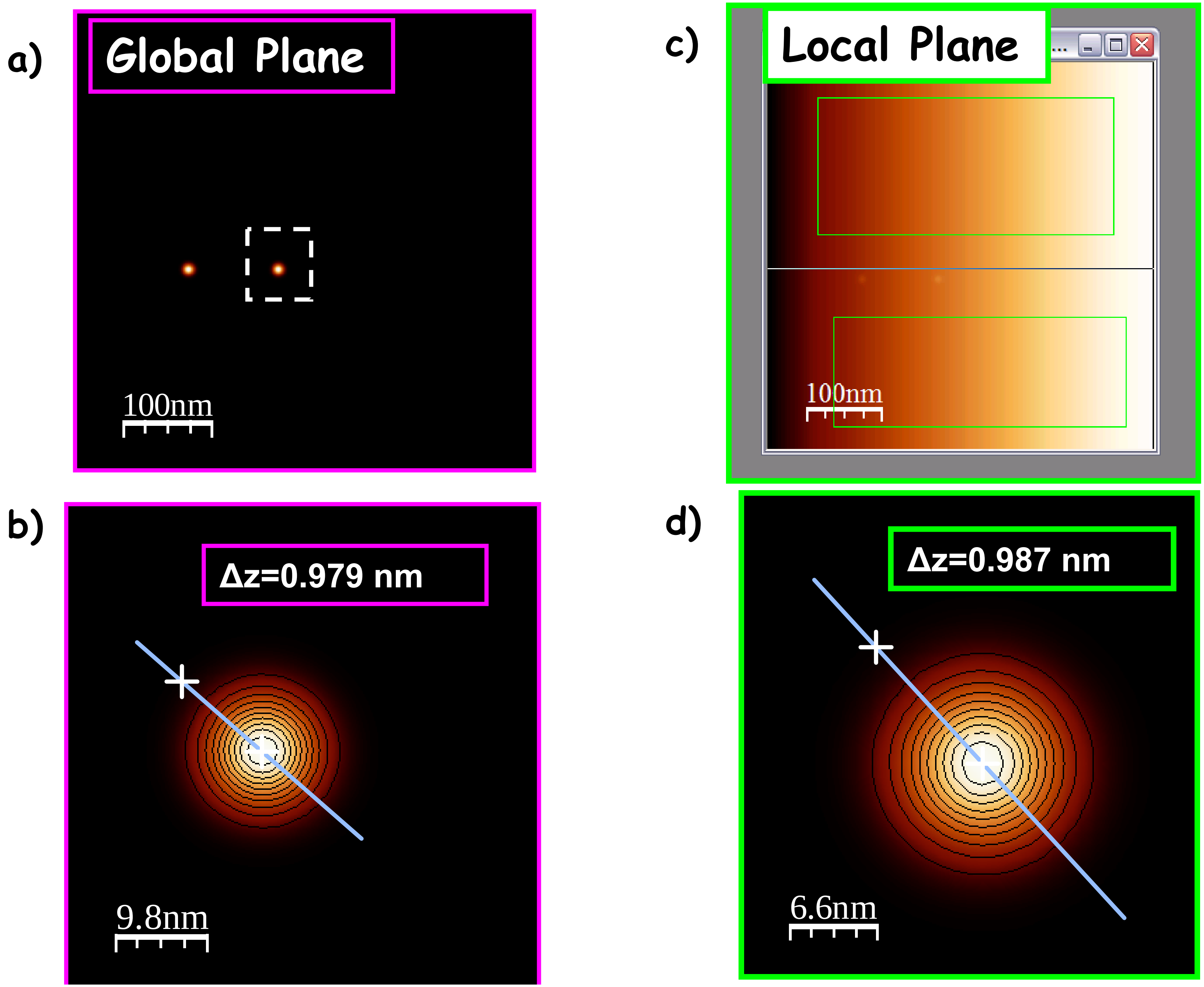
Plane Subtraction

f(x,y)= exp(-0.04*(x-125)^2-0.04*(y-125)^2)
+ exp(-0.04*(x-225)^2-0.04*(y-125)^2)
+ 0.03*(x-225)





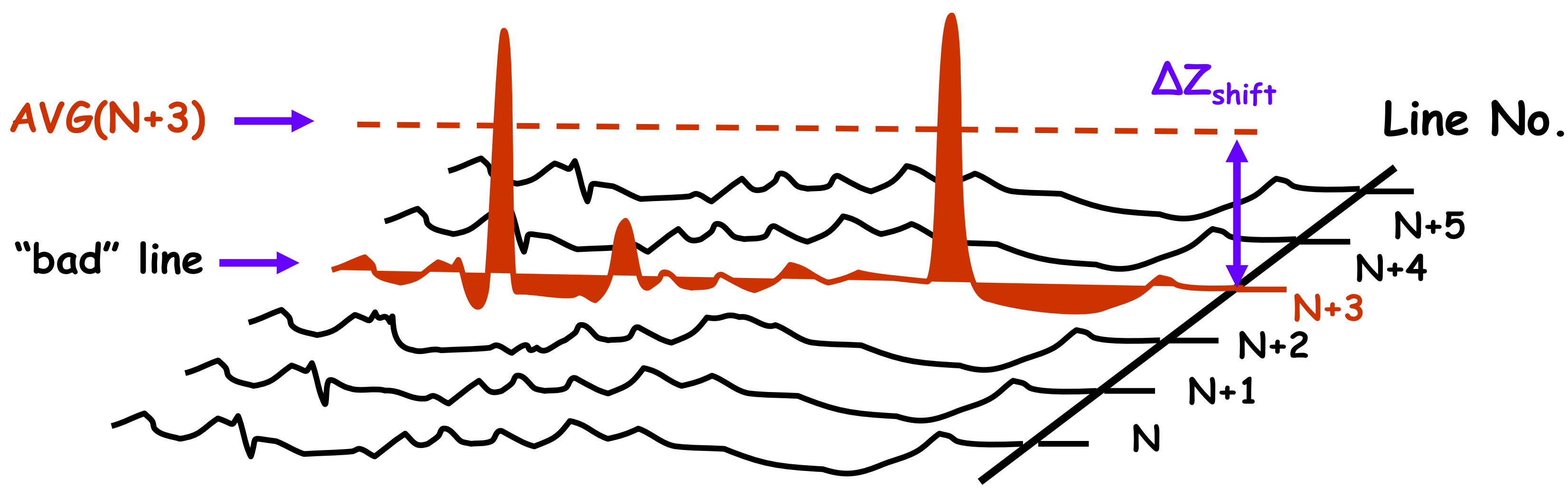
2 clicks


a)
Global Plane
100nm
b)
Δz=0.979 nm
9.8nm
c)
Local Plane
100nm
d)
Δz=0.987 nm
6.6nm

The Flatten/Level Feature

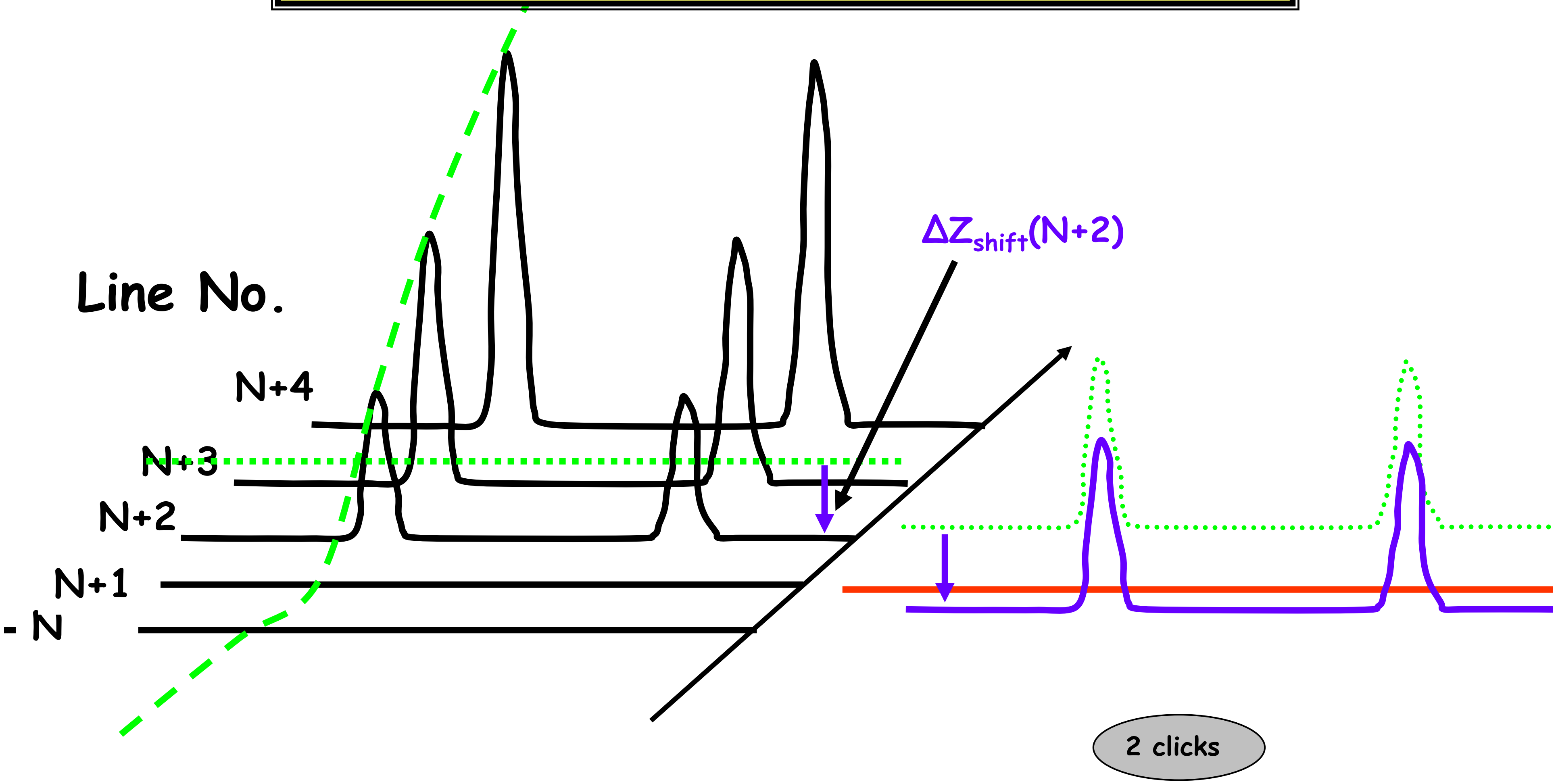
A Simple "Flatten" Algorithm

$\Delta Z_{shift}(N+1) = AVG(N+1) - AVG(N)$
$\Delta Z_{shift}(N+2) = AVG(N+2) - AVG(N+1)$
$\Delta Z_{shift}(N+3) = AVG(N+3) - AVG(N+2)$
etc.

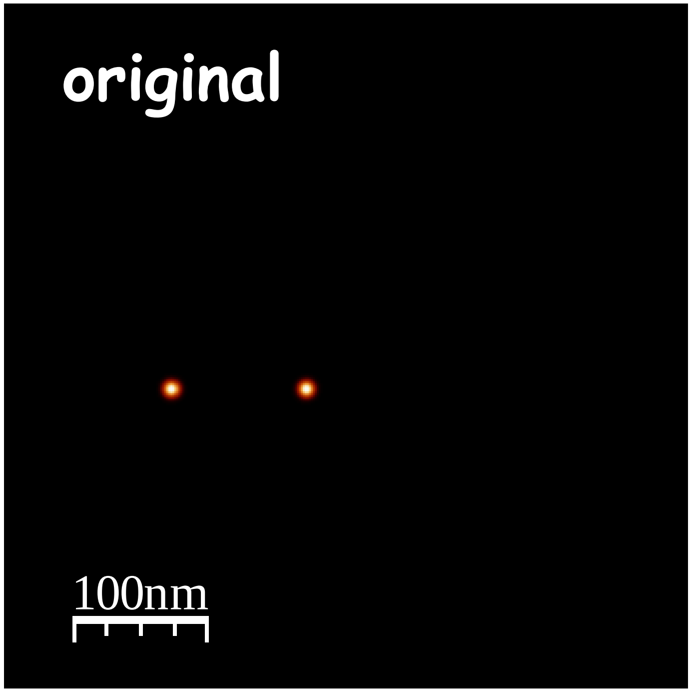


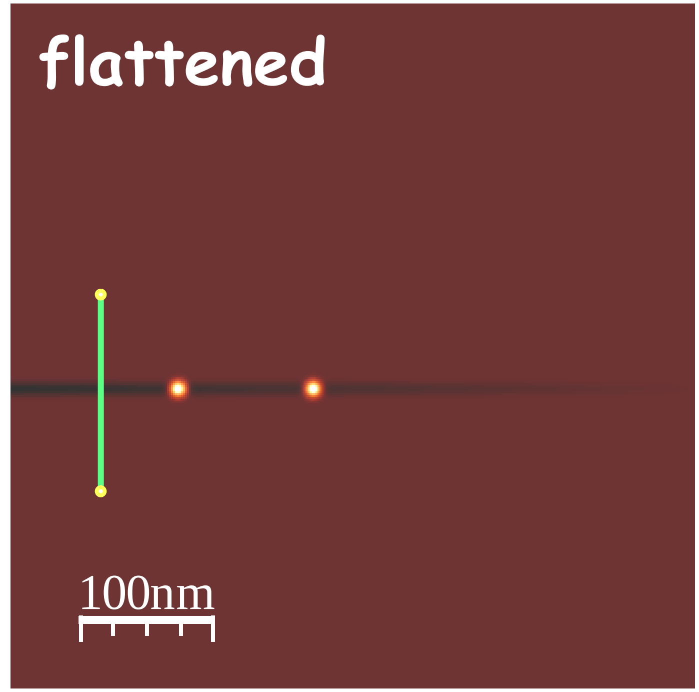
"Flatten" Algorithm Applied to a Localized Feature

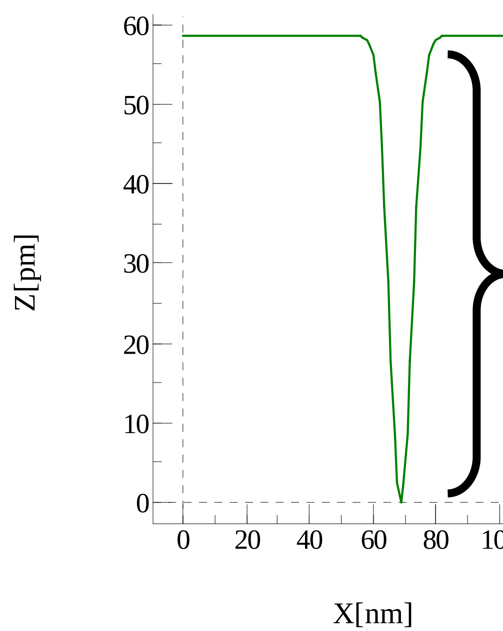
$\Delta Z_{shift}(N+1) = AVG(N+1) - AVG(N)$
$\Delta Z_{shift}(N+2) = AVG(N+2) - AVG(N+1)$
etc.



The Flatten/Level Artifact







This feature is an artifact!

Check: Does your software <u>automatically</u> flatten each image?

Intrinsic Artifacts

Force too large

Abrupt change in properties of substrate

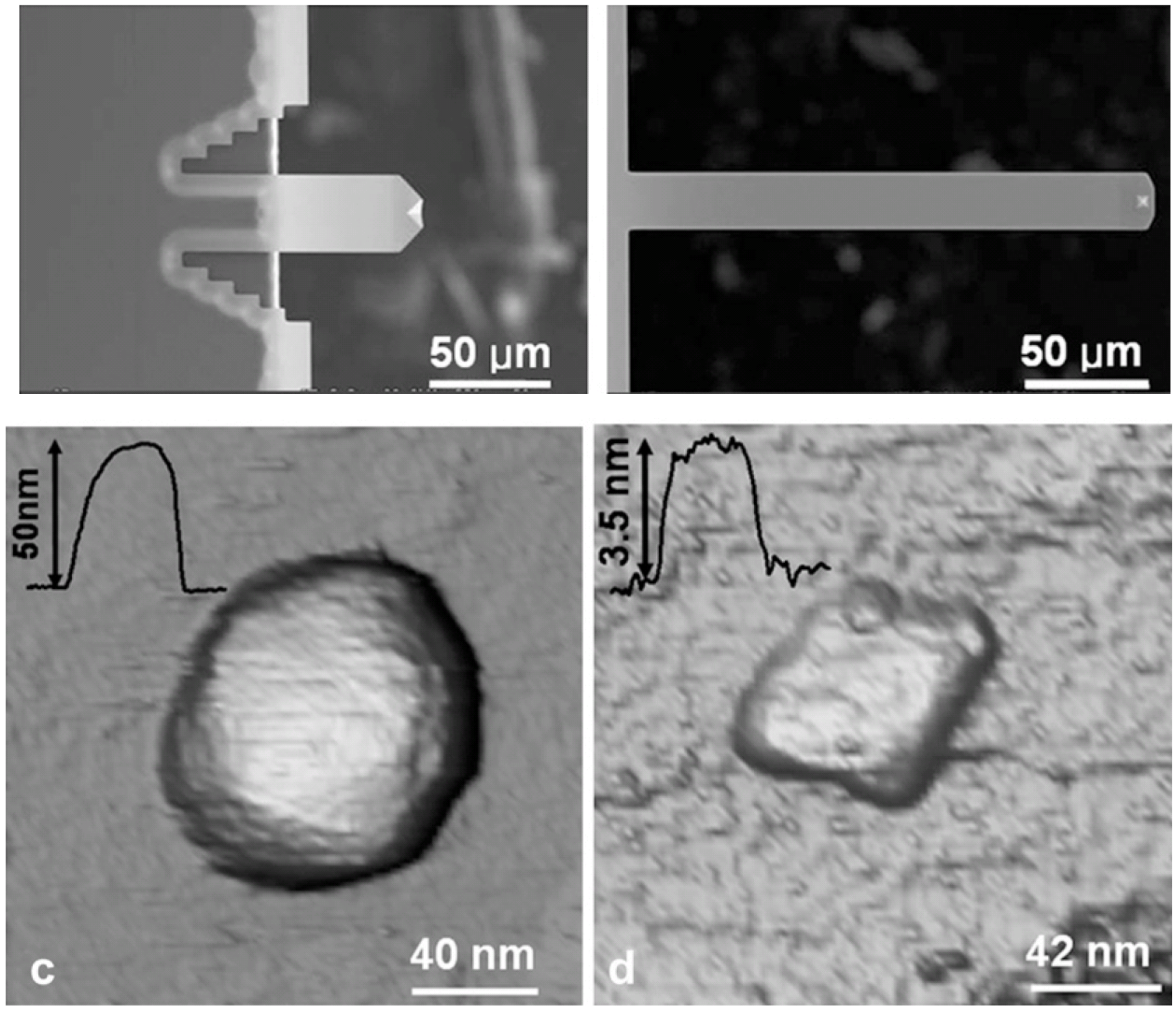
Change in force “volume” due to geometry

Phage Φ29 virial capsids

Crushing force 2-4 nN

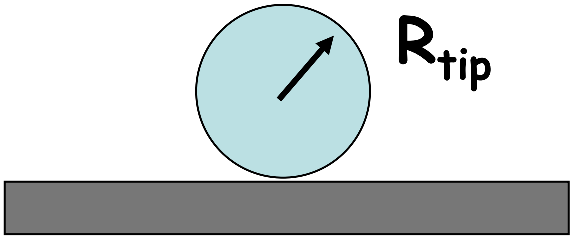
f_o= 8.3 kHz, Q=1.02; k=0.063 N/m f_o= 5.4 kHz, Q=0.47; k=0.072 N/m

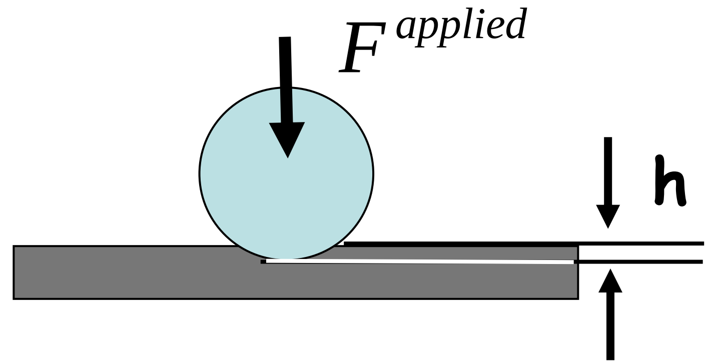
In liquid



Xu et al., Biophys. J. **95**, 2520 (2008).

When is the Force too Big?





Hertz Model:

$$h^3 = \frac{F_{app}^2}{E^{*2} R_{tip}}$$

$$\frac{1}{E^*} = \frac{1-\nu_{tip}^2}{E_{tip}} + \frac{1-\nu_{sub}^2}{E_{sub}}$$

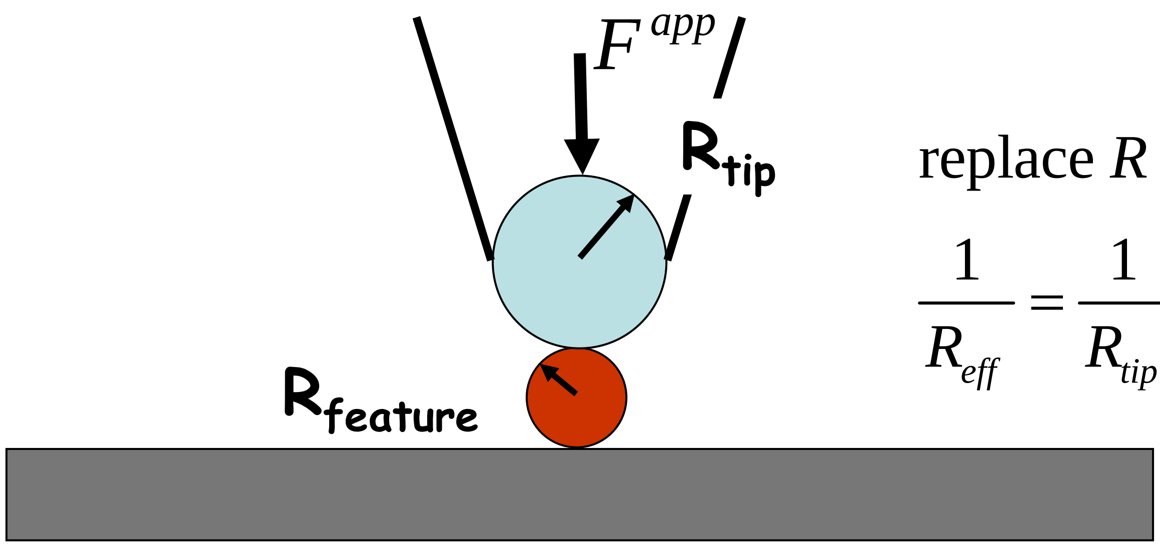
$$let\ h \approx \alpha R_{tip},\ \alpha = 0.01,\ 0.10,\ 0.50$$

$$\alpha^{3/2} R_{tip}^2 \approx \frac{F_{app}^{\max}}{E^*} \Rightarrow F_{app}^{\max} \approx \alpha^{3/2} R_{tip}^2 E^*$$

Look-up Table

α	$\alpha^{1.5}$
0.01	0.001
0.10	0.032
0.50	0.350

Scanning features

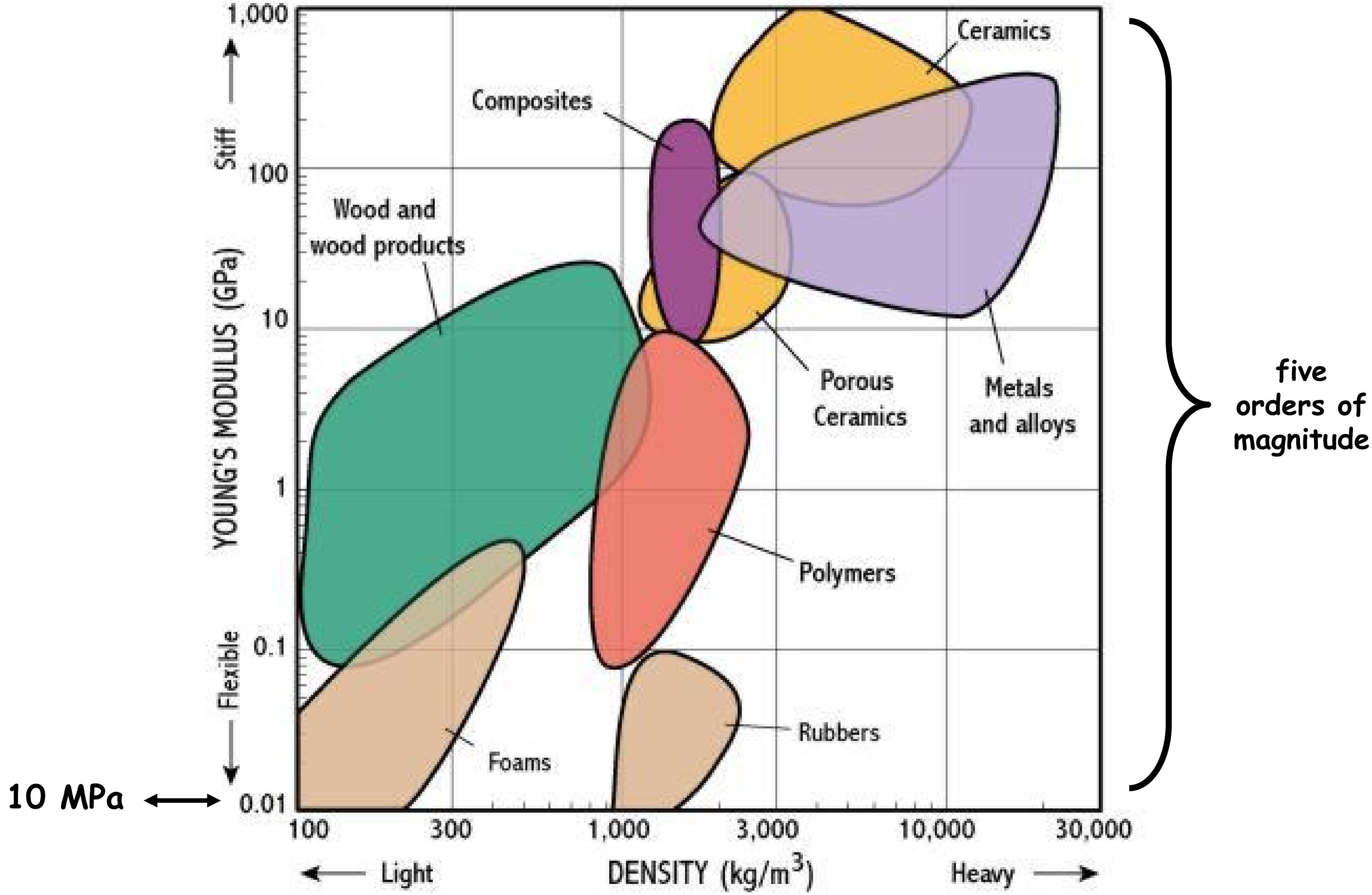


replace R with R_{eff}

$$\frac{1}{R_{eff}} = \frac{1}{R_{tip}} + \frac{1}{R_{feature}}$$

$$\alpha^{3/2} R_{tip}^2 = \alpha^{3/2} R_{eff}^2 \approx \frac{F_{app}^{\max}}{E^*} \Rightarrow \boxed{F_{app}^{\max} \approx \alpha^{3/2} R_{eff}^2 E^*}$$

Young's Modulus – Different Materials



http://www-materials.eng.cam.ac.uk/mpsite/interactive_charts/stiffness-density/NS6Chart.html

Young's Modulus - Biological Materials

REVIEW www.rsc.org/softmatter | Soft Matter

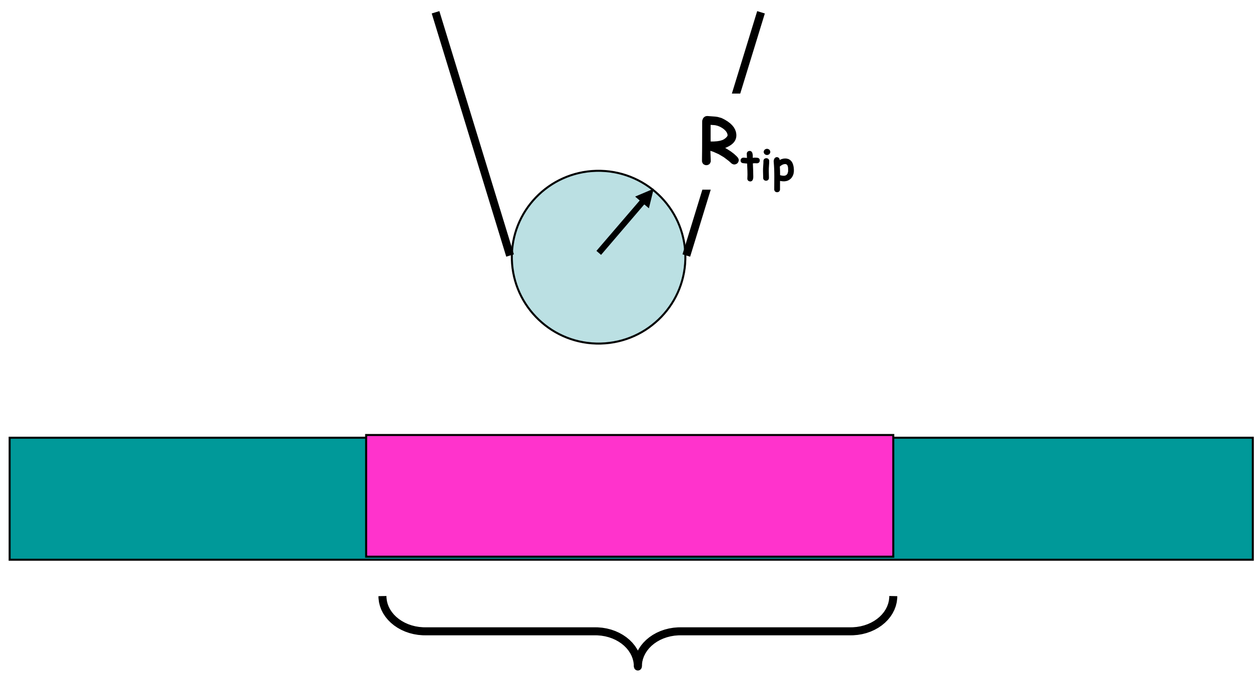
Soft biological materials and their impact on cell function

Ilya Levental,[a] Penelope C. Georges[a] and Paul A. Janmey*[ab]

Tissue type	Animal	Testing method	Elastic modulus	Ref
Achilles' tendon	Rat	Tension	310 Mpa	15
Articular cartilage	Bovine	Compression	950 kPa	86
Skeletal muscle	Rat	Tension	100 kPa	87
Carotid artery	Mouse	Perfusion	90 kPa	88
Spinal cord	Human	Tension	89 kPa	89
Thyroid cancer[a]	Human	Compression	45 kPa	16
Spinal cord	Rat	Tension	27 kPa	90
Cardiac muscle	Mouse	Tension	20–150 kPa	91
Skeletal muscle	Mouse	AFM	12 kPa	13
Thyroid	Human	Compression	9 kPa	16
Lung	Guinea pig	Tension	5–6 kPa	5
Breast tumor	Human	Compression	4 kPa	7
Kidney	Swine	Rheology	2.5 kPa	92
Premalignant breast[b]	Human	Indentation	2.2 kPa	14
Fibrotic liver	Human	Compression	1.6 kPa	93
Liver	Human	Compression	640 Pa	93
Lymph containing metastases	Human	Vibrational resonance	330 Pa	17
Brain	Swine	Indentation	260–490 Pa	94
Lymph Nnode	Human	Vibrational resonance	120 Pa	17
Mammary gland	Human	Compression	160 Pa	7
Fat	Human	Indentation	17 Pa	14

[a] Thyroid papillary adenocarcinoma. [b] Mammary ductal carcinoma *in situ*.

Abrupt change in substrate properties



Change in modulus, adhesion, hydrophobicity, etc.

Abrupt change in material properties

VEDA INPUTS:

Set point 60%

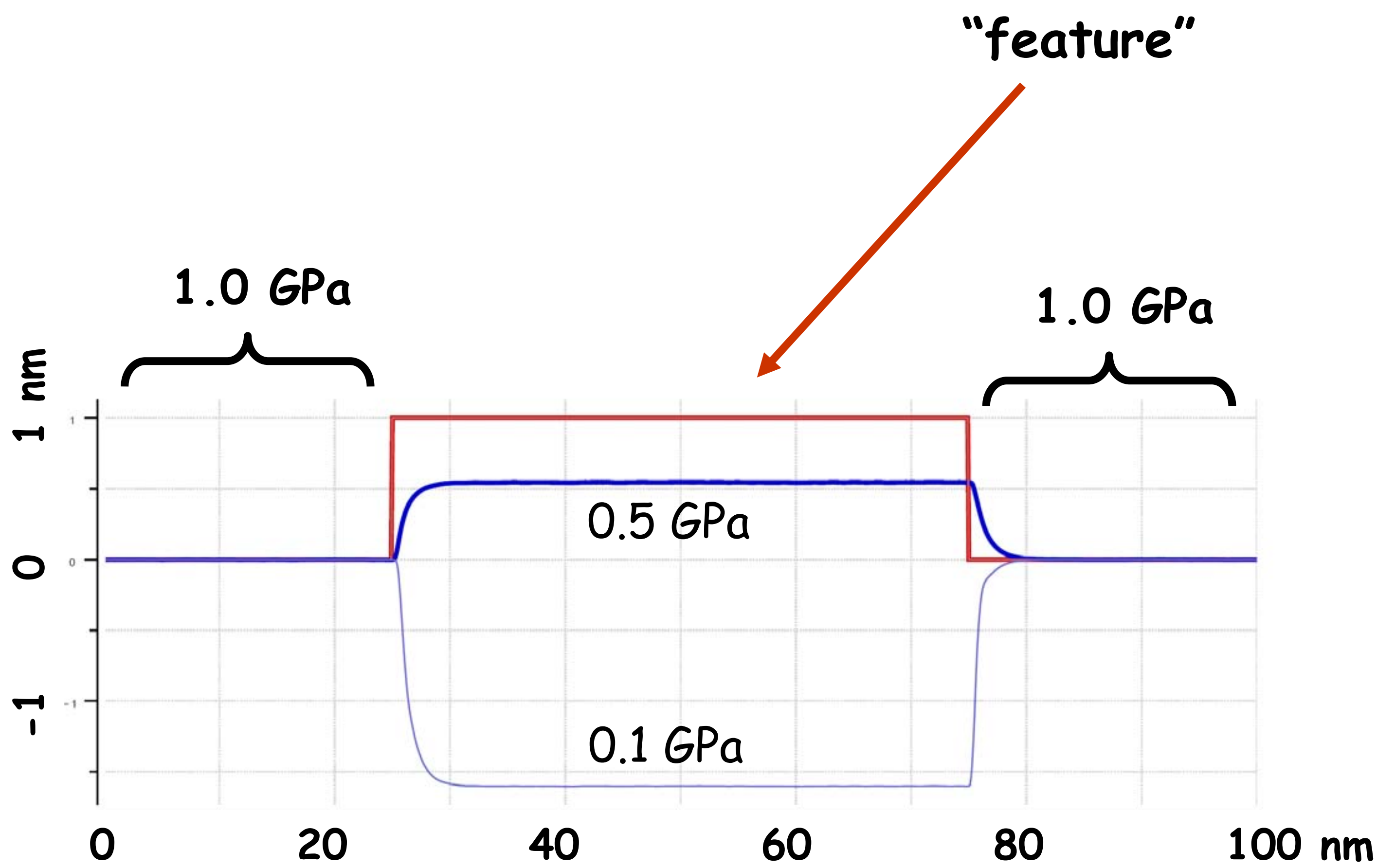
k= 2 N/m

Amplitude = 10 nm

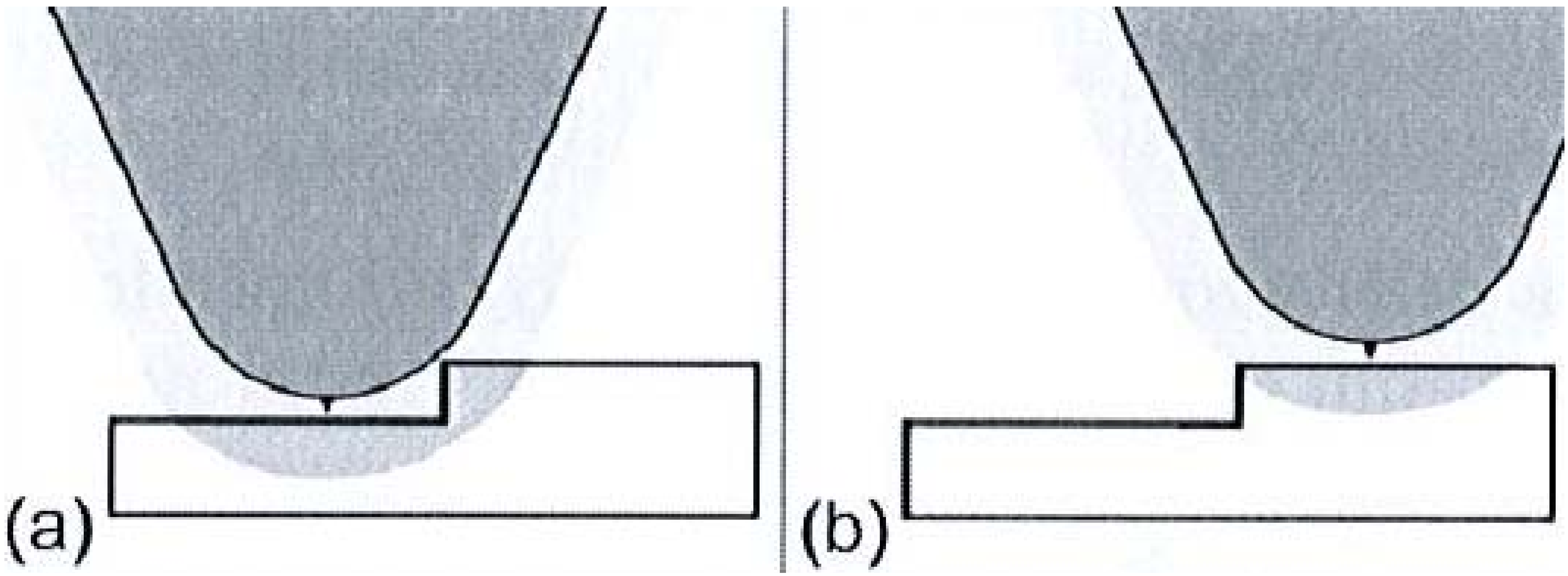
f_o = 30 kHz

R_{tip} = 10 nm

DMT model



Geometrical change in force "volume"



Most important for long range forces, like electrostatics

E. Meyer, H.J. Hug and R. Bennewitz, *Scanning Probe Microscopy*, Springer (2003).

A Few Words on Cleaning SPM Tips and Cantilevers

Helpful Hints are Everywhere!

(24) Piranha-cleaned tips were utilized to exclude deposition of contaminants accumulated on the tip. However, treating tips in piranha makes silicon probes hydrophilic which results in a decrease in the resolution of the LFM images. Formation of the meniscus was also confirmed with tips modified with ODT and dodecylamine. Notably, the use of tips coated with 1-dodecylamine provides significantly better LFM images compared to those taken with bare tips (Piner, R. D.; Hong, S.; Mirkin, C. A. *Langmuir* **1999**, *15*, 5457).

We obtained Au-coated cantilevers from Olympus (OMCL-RC800PB-). Our group developed a two-step pre-cleaning procedure for chemical modification of the Au-coated tips.[36)] First, the tips were oxidized to remove organic contaminants with ozone treatment. Second, the oxidized tips were immersed in hot ethanol (65°C) to reduce the oxidized Au surface. After sputtering 10 nm of Au on the clean tips, the tips were immediately immersed in 1 mM ethanol solution of decanethiol to modify the tip surfaces chemically. After thorough rinsing with pure solvent, the tip was mounted on the scanner head.

Commercial silicon nitride and silicon cantilevers tend to be hydrophilic in nature although their exact hydrophilic/hydrophobic behaviour is unforeseeable because they can be more or less contaminated with hydrocarbons. Cleaning the tips through irradiation with UV light or a glow discharge in an air or oxygen plasma will render the tips very hydrophilic.[9] Both procedures break down the hydrocarbons into smaller compounds, which will react with the ozone created by the UV light or the oxygen radicals in the air or oxygen plasma to form volatile compounds. Surface charges

DPN experiments were performed with ultrasharp silicon nitride microcantilevers with a spring constant of 0.05 N/m, purchased from Vecco Inc. The tips were thoroughly cleaned using ethanol and deionized water and radiated with UV light for 30 min prior to use. The cleaning procedure makes the tip surface hydrophilic which is favorable for the water condensation. The

<u>Conjecture regarding Murphy's 2nd Law</u>:
Any proposed cleaning technique, no matter how well conceived, usually makes matters worse.

A Selected Literature Survey

T. Arai and M. Tomitori, "Scanning Auger Electron Microscopy and Compositional Control of Cantilevers for Ultrahigh Vacuum Atomic force Microscopy, Jp. J. Appl. Phys. **36,** 3855 (1997).

S. Umemura, at al., "Effect of lubricant coating on tips in atomic force microscopy", J. Vac. Sci. Techn. **B16**, 38 (1998).

R.D. Piner, S. Hong, and C. A. Mirkin, "Improved Imaging of Sot Materials with Modified AFM Tips, Langmuir **15,** 5457 (1999).

H.F. Knapp and A.S. Stemmer, "Preparation, Comparison and Performance of Hydrophobic AFM Tips", Surf. Interface Anal. **27,** 324 (1999).

Y.S. Li, et al. "Organic and Inorganic Contamination on Commercial AFM Cantilevers", Langmuir **15**, 6522 (1999).

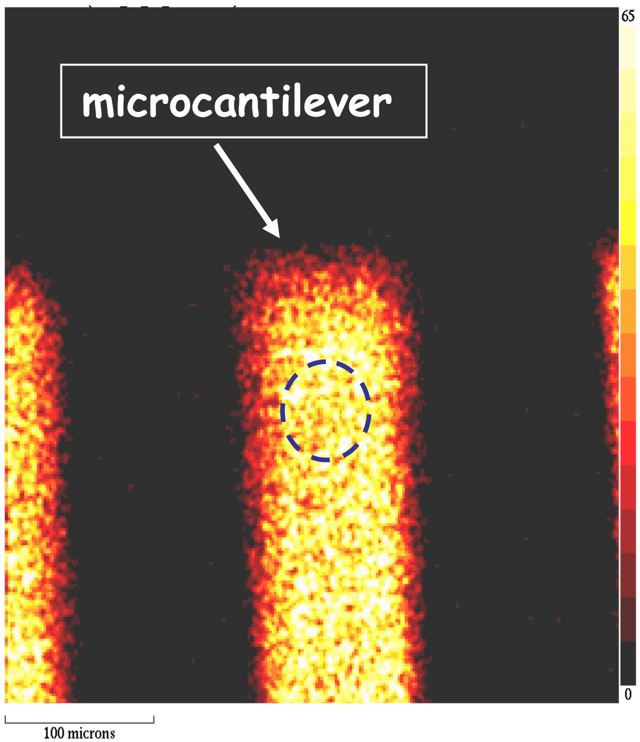
M. Fujihira, et al., "Novel cleaning method of gold-coated atomic force microscope tips for their chemical modification", Ultramicroscopy **82,** 181 (2000).

C. Pereira de Souza, et al., "Implementation of Recycling Routes for Scanning Probe Microscopy Tips", Microsc. Microanal. **8,** 509 (2002).

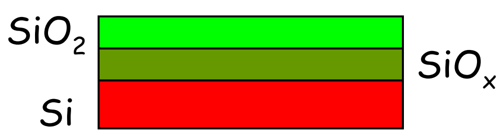
E. Bonaccurso and G. Gillies, "Revealing contamination on AFM cantilevers by Microdrops and Microbubbles", Langmuir **20,** 11824 (2004).

Knowing what's on your tips

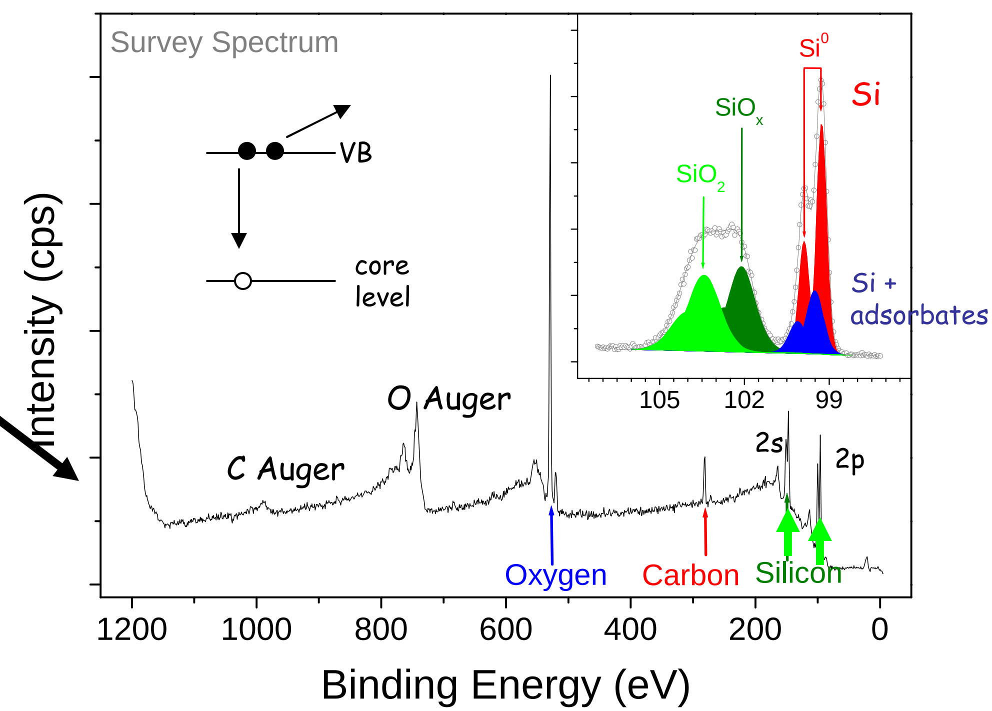
Imaging XPS



O(1s) "image"



Birck Surface Analysis Facility
Purdue University
Dr. Dmitry Zemlyanov



(B.E.=hv-K.E.)

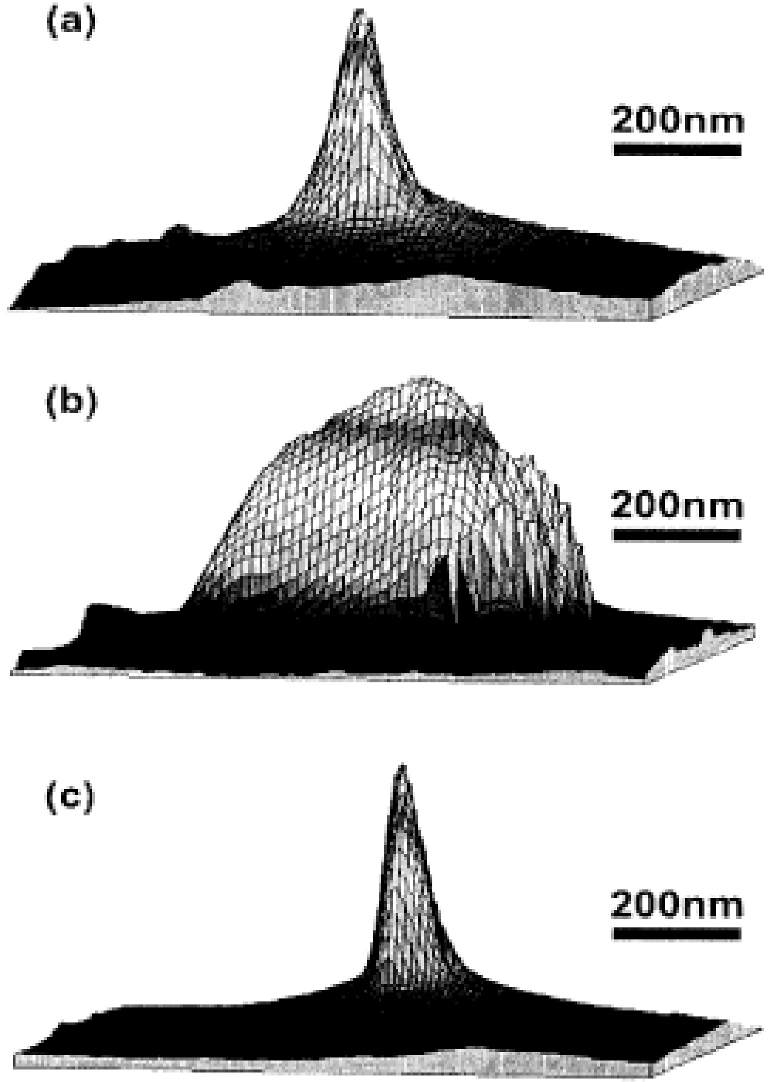
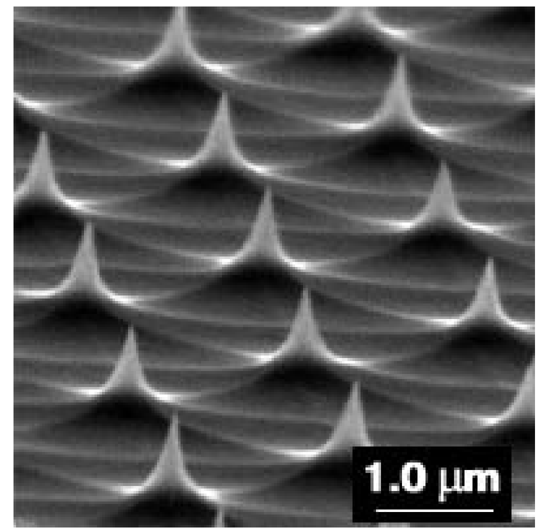


Figure 1. Three-dimensional views of the physical profile of the same SPM tip. **a:** As received by the tip maker. **b:** After intentional contamination with inorganic material. **c:** After cleaning with HF solution. The scale bars indicate the tip dimensions in all, *x*, *y*, and *z*, directions.

Calibration Substrate



Microsc. Microanal. **8**, 509 (2002)

A Procedure for SPM Tip Recovery

Pt-coated Si tips

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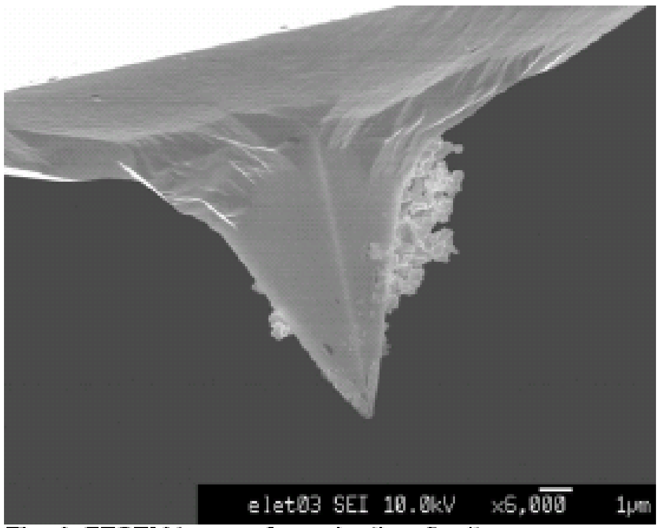

Fig. 1. FESEM image of a probe tip, after its use on a polystyrene latex sample. Note the large number of small particles adhering to the tip sides.

(Removing Particulates)

The tips was thus sonicated within a 50 mL beaker filled with deionized water, partly immersed in a 3L-rectangular water bath, powered by 90 watts of 40 kHz ultrasound, for 5 minutes. Due to its strongly damaging action on many adhesive joints, water is a suitable liquid for cleaning by ultrasonic cavitation, dispensing with the use of any other cleansing agent[5,6], and it is recommended for the removal of particles in the micrometer size range, from solid surfaces.[7]

The fast periodic compression and decompression of a high-surface tension liquid such as water produces a myriad of micro bubbles bursting within the liquid, especially at the existing solid-liquid interfaces. The resulting pressure gradients are sufficient to dislodge the particles, but they can also produce geometrical deformations at the surfaces.

Figure 4 shows a diagram of the set-up used in this work.

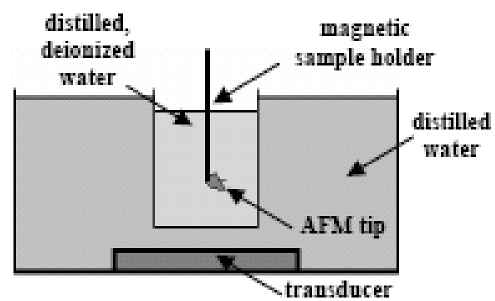

Fig. 4. Schematics of the sample mounting for cleaning.

Simple Method to Check AFM Tip Performance Using a Polymer Film

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Surface Science Western
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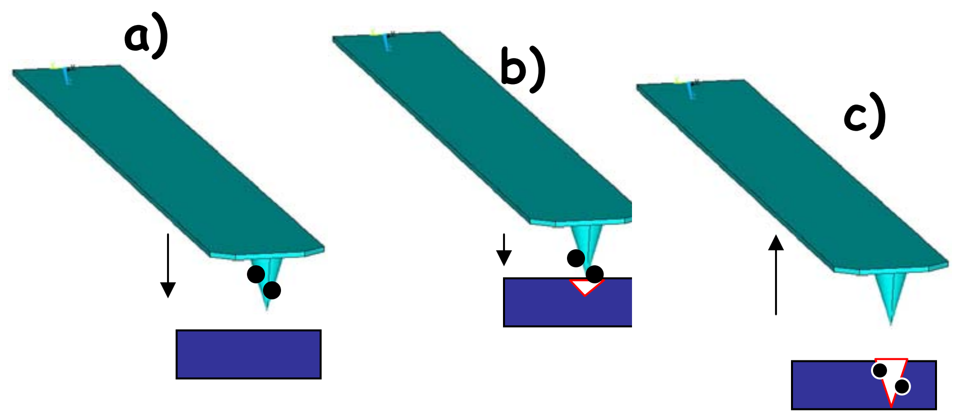
Because the polymer film is soft compared to the silicon tip (Young's modulus for polypropylene is 1-2 GPa, while for silicon it is 132-190 GPa), the polymer will not damage the tip when the tip is pushed into the polymer. This property can be used to clean a contaminated tip, i.e., by pushing the contaminated tip into the polymer, contaminants could be removed from the tip apex.

Another important property of the BOPP is that the polymer film is highly hydrophobic and has a very low surface energy of ~ 30 mJ/m^2 (The surface energy for Si is ~ 1400 mJ/m^2; and the surface tension of water is 72 mJ/m^2). These properties prevent contaminants from accumulating on the surface and hence prevent the contamination of the tip in the evaluation process.

Si tips – particulate contamination



436 *Langmuir, Vol. 17, No. 2, 2001*

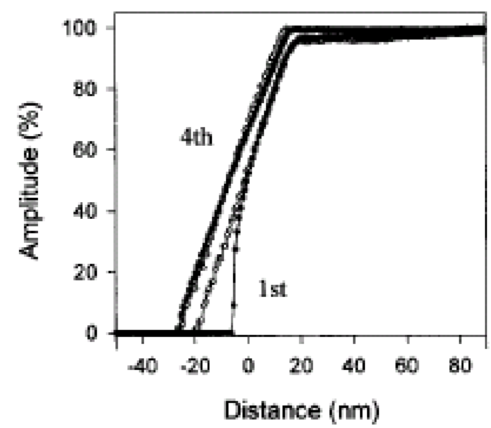


Figure 6. Four amplitude–distance curves were measured after the BOPP film surface shown in Figure 2a was imaged. For clarity only the first and fourth curves are shown, between which are the second and third curves. After the amplitude–distance measurement, the tip was used to obtain the image shown in Figure 2b. The amplitude versus the tip–sample separation when the tip is brought to and retracted from the sample surface is represented by open and filled circles, respectively. The speed of the tip movement was 100 nm/s.

Recommendations

•Don’t store microcantilevers in plastic shipping cases without cleaning microcantilever before use

•Use dedicated teflon or quartz beakers when cleaning (avoids leaching of plasticizers and pyrex).

•Use dedicated tools (tweezers, glass slides, etc.)

•Do not be afraid to clean your tweezers regularly

•Ozone cleaning and Glow Discharge cleaning are relatively easy (no waste or protective equipment required)

•After cleaning, store tips in clean solvent

•Under ambient conditions, hydrophobic tips seem to be better than “as-received” tips

•When in doubt, throw it out!