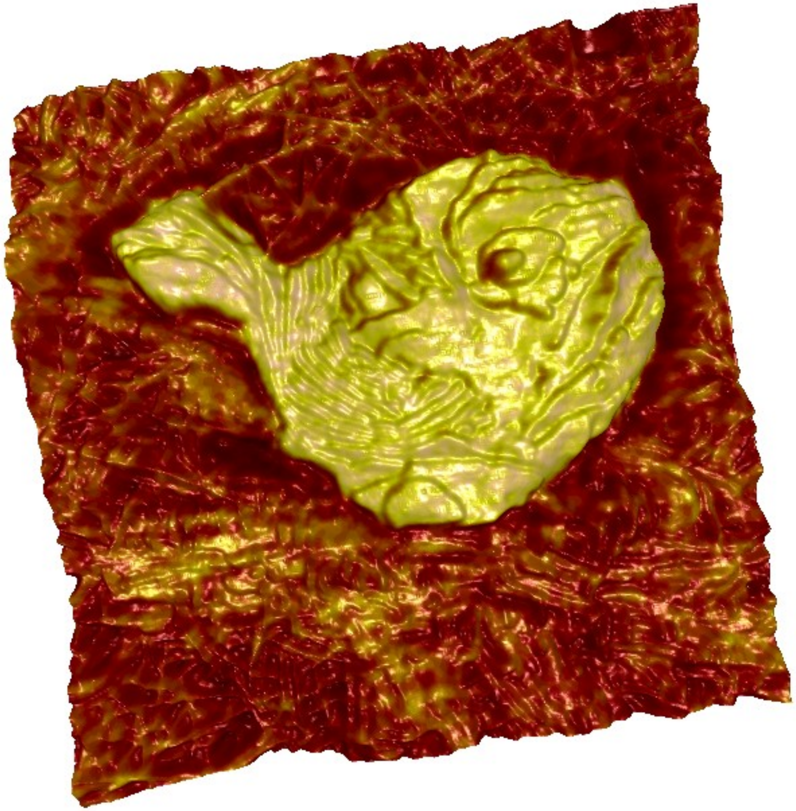
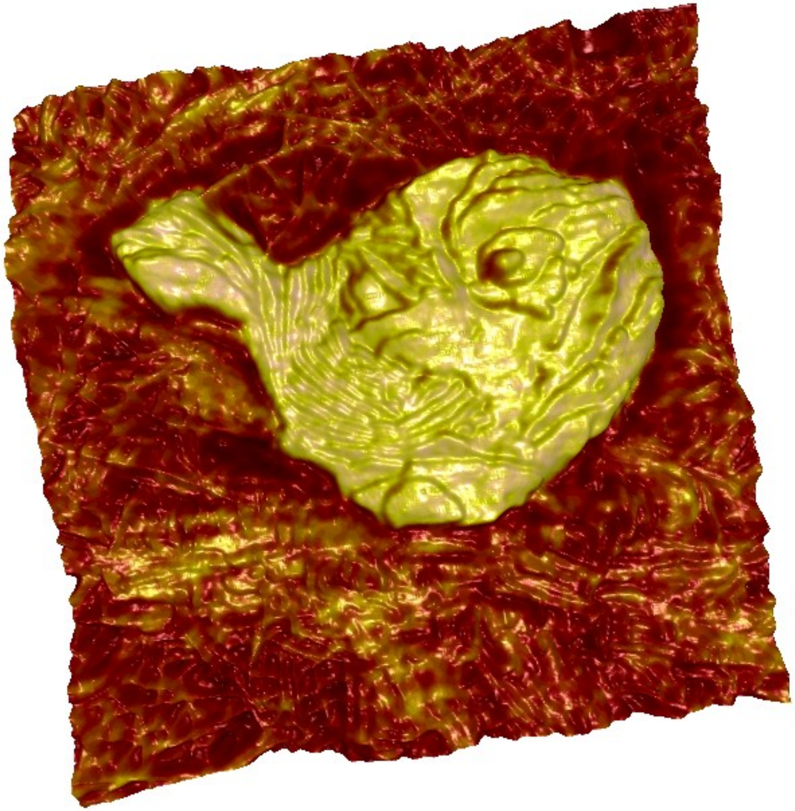
PeakForce QNM

Mike Maybrun, November, 2010



PEO/SPP – Adhesion overlay on height

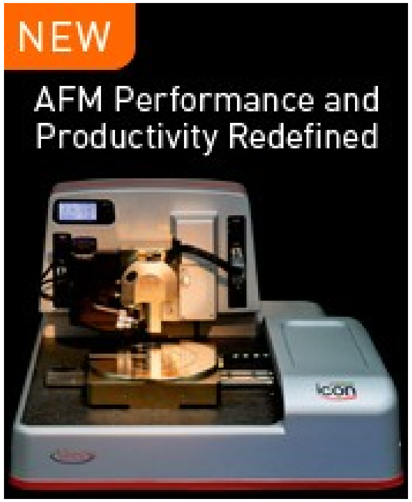
Overview of PFQNM



PEO/SPP – Adhesion overlay on height

PeakForce QNM is available on all NanoScope V8.10 Platforms

Dimension icon



MultiMode 8

BioScope Catalyst

What is PeakForce QNM?

- PeakForce QNM is an imaging mode that produces height images and **Q**uantitative **N**ano-**M**echanical sample property images at the same time.
- Two parts to PeakForce QNM
  - The PeakForce Tapping part
    - PeakForce Tapping is the feedback mode used track and image the sample surface.
  - The QNM part
    - PeakForce Tapping mode produces force curves
    - The force curves are used to extract quantitative material properties data.

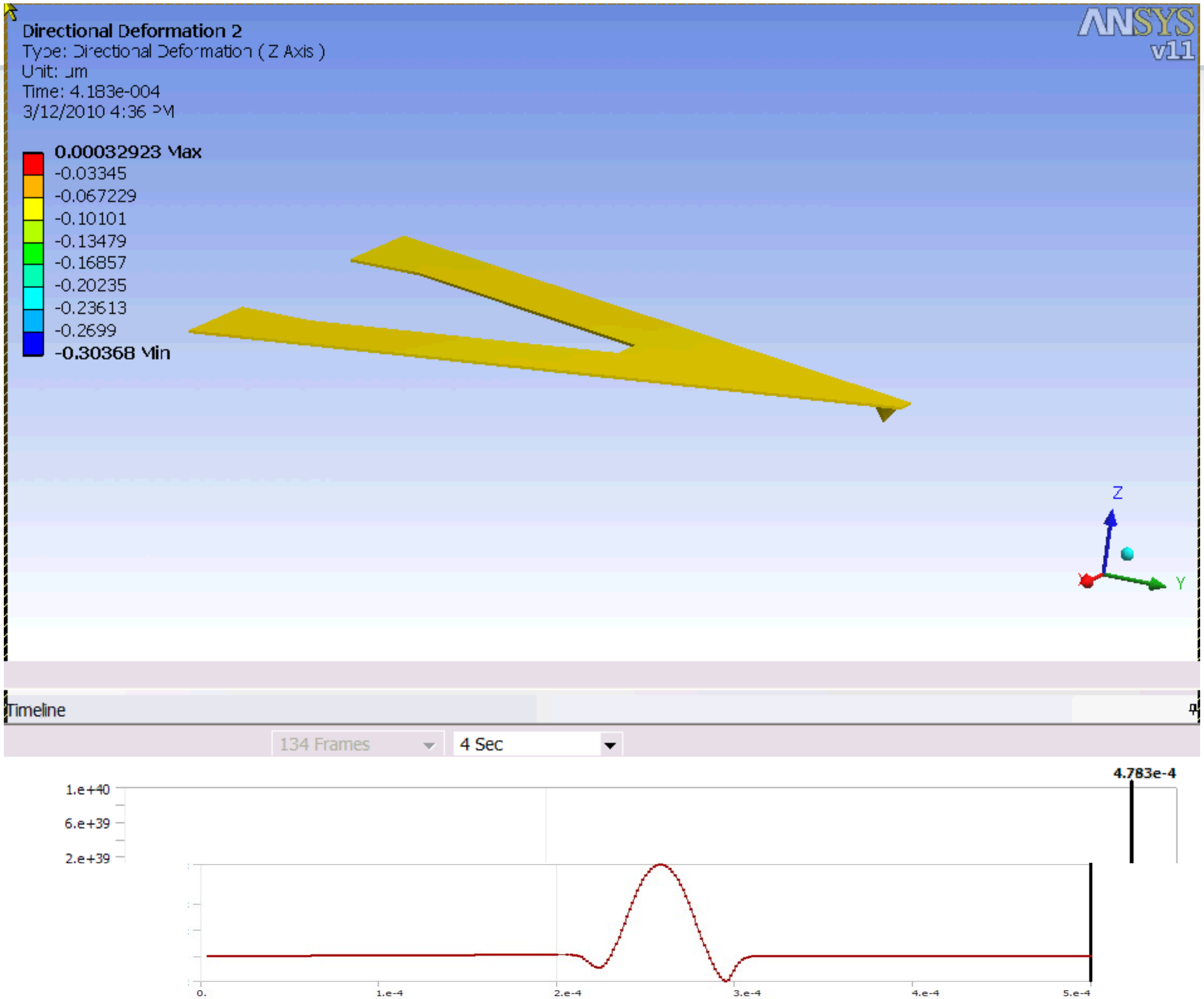
The PeakForce part
PeakForce Tapping Mode - 1

- In PeakForce Tapping mode, the Z scanner is typically modulated at a few kHz. The peak interaction force is used as control feedback for imaging the sample. This technique tracks the surface, producing force curves at a high rate (2000 per second) while the scanner moves in a raster pattern in the XY plane.

- With the *direct peak force control* as low as tens of pN in Peak Force Tapping mode, sample deformation depths and lateral force can be very small. There is minimal damage to the probe or sample. PeakForce QNM can provide compositional mapping of a complex composite material at resolution similar to Tapping Mode.

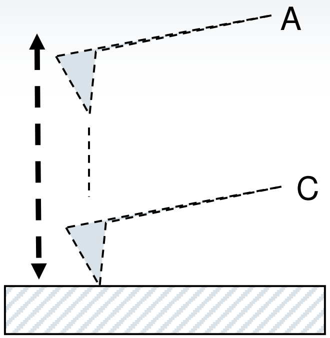
- No need to tune the cantilever, *easy and stable* operation in liquid environments and under low or high temperatures.

The PeakForce part
PeakForce Tapping Mode - 2

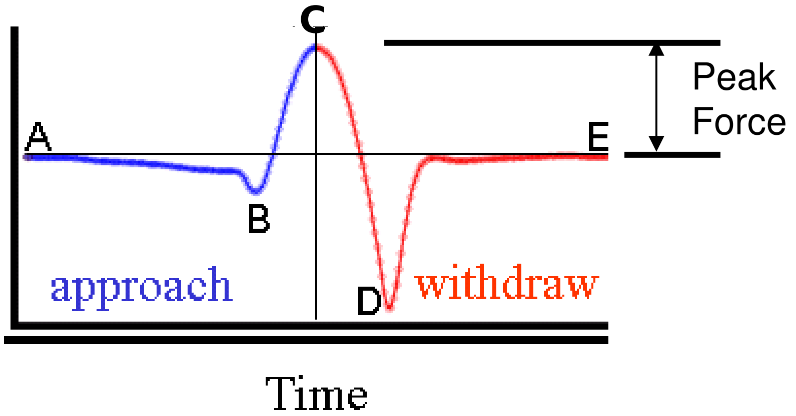


- Animation for Peak Force Tapping mode operation Finite Element Analysis
- With Z scanner modulated at 2 kHz, the cantilever is performing fast force curves and the peak interaction force is used for direct force feedback

The PeakForce part
Peak Force Tapping Mode - 3



One cycle: photo detector vertical deflection (force) vs time.

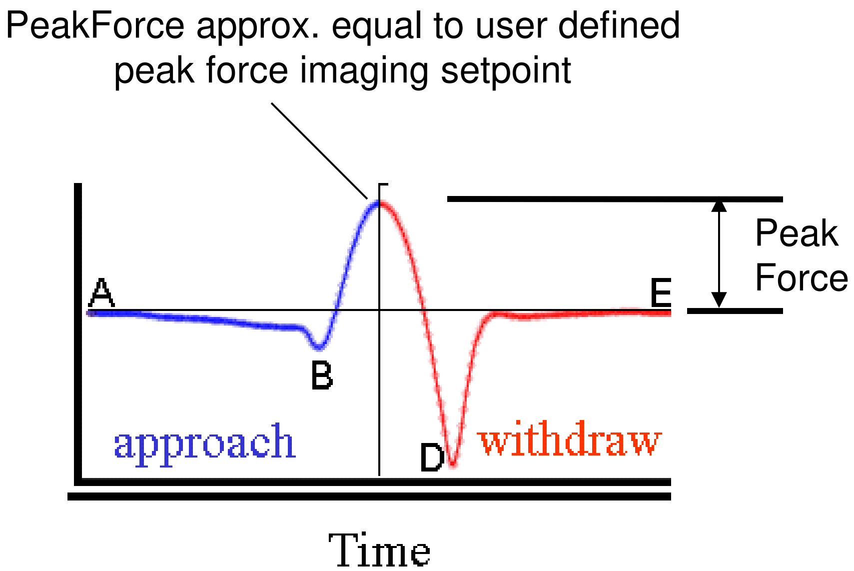


The graph shows a single approach/retract cycle. This happens at every XY pixel location in the image at a rate of ~2000/s.

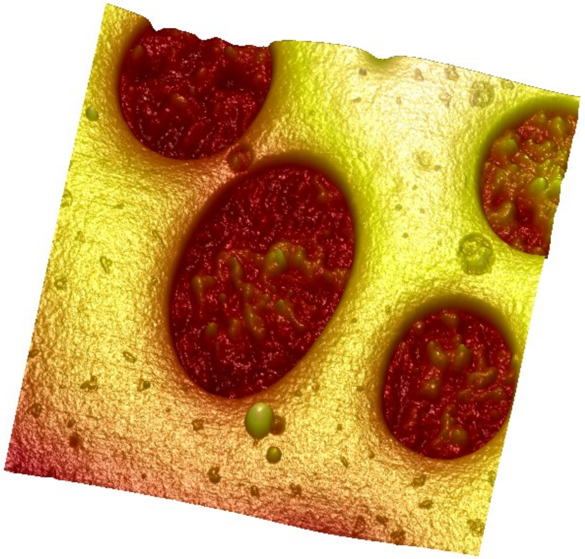
- A - Probe tip 300 nm above sample surface. Z piezo pushes probe toward sample.
- B - Tip contacts sample (“snap-to-contact”). Z piezo pushes probe further until PeakForce imaging setpoint is reached.
- C – PeakForce setpoint reached. Probe starts withdrawing from sample.
- D – Probe tip breaks free of sample at maximum adhesion point.
- E – Probe back to starting point 300 nm above sample surface

The PeakForce part
Peak Force Tapping Mode - 4

- Height image is rendered by plotting the Z piezo voltage required to keep the probe tracking the surface topography at the user specified peak force setpoint.
  - Difference between peak force setpoint and peak force at a given pixel is peak force error.



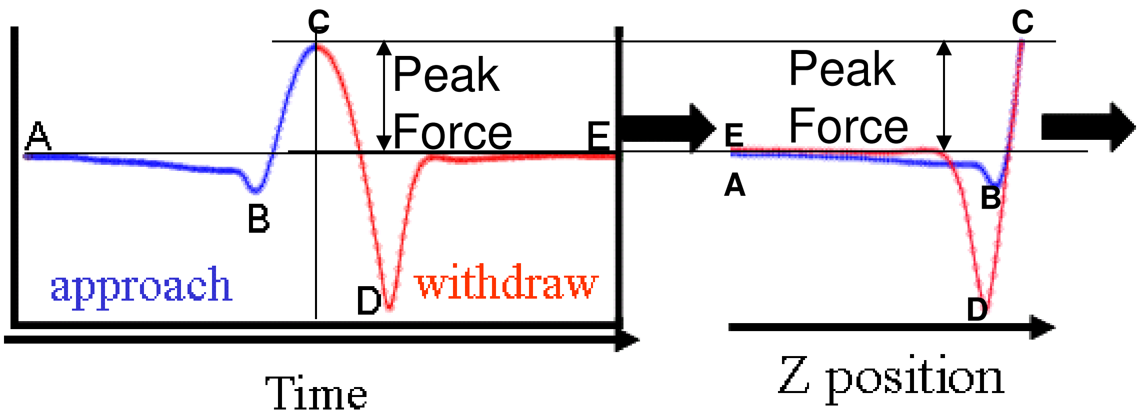
One cycle: photo detector vertical deflection (force) vs time.



Height

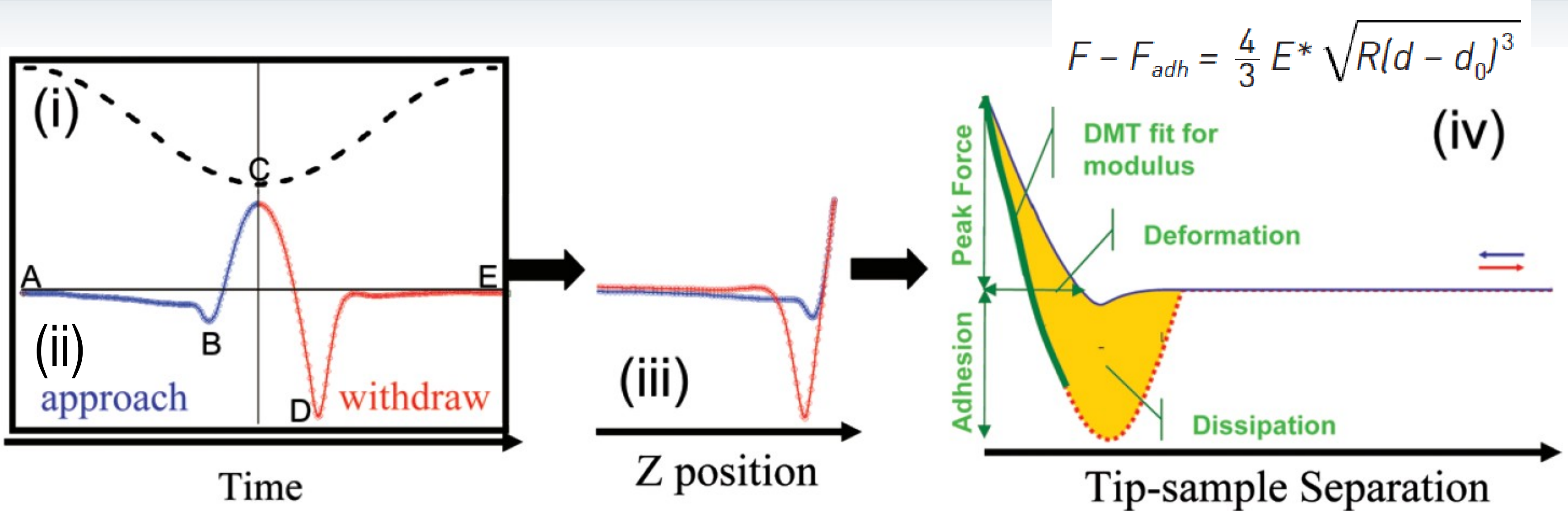
The QNM Part
Force curves…at every xy position

- The horizontal axis (time) of the left plot can easily be converted to Z probe position.



- Recall from a previous section that typical force curves collected at single XY sample points can reveal material properties.
- Similarly, the force curves produced as a result of PeakForce Tapping mode imaging can be used to extract material properties data along with the topography (height) and at imaging speeds.

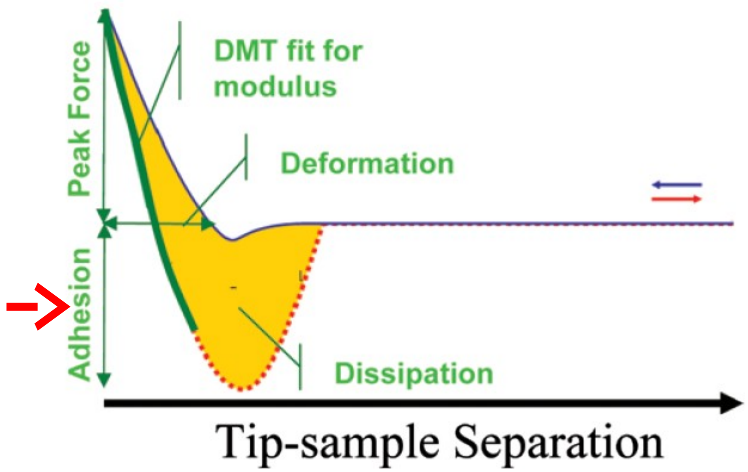
The DMT model

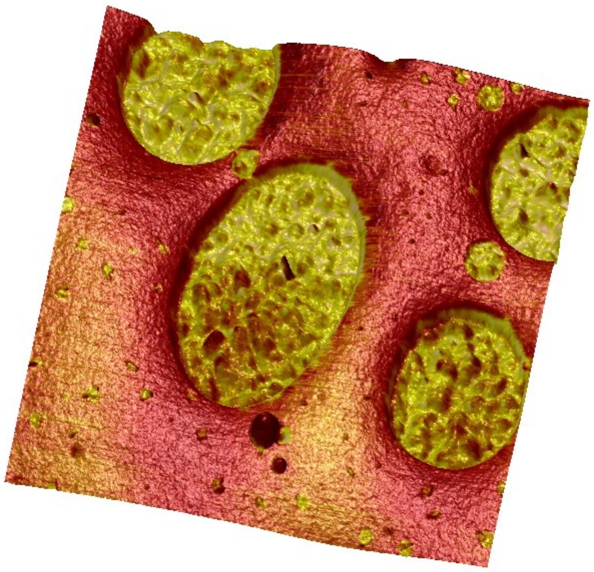


- Force vs Time → Force vs. Z → Force vs. Separation
  - Quantitative data comes from Force vs. Separation plots

The QNM part
Adhesion…at every XY position

- Adhesion – indication of sample "stickiness"
  - In the image brighter (yellow) areas are higher adhesion (stickier)
  - Higher adhesion corresponds to lower minimum on the probe withdraw (red) plot.

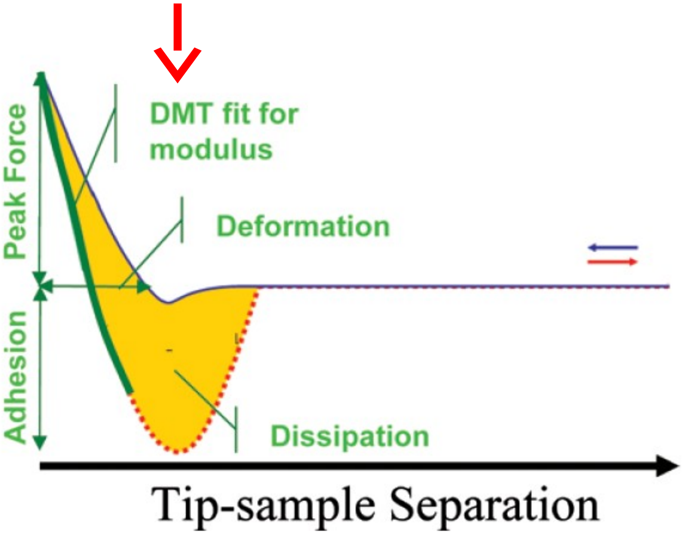


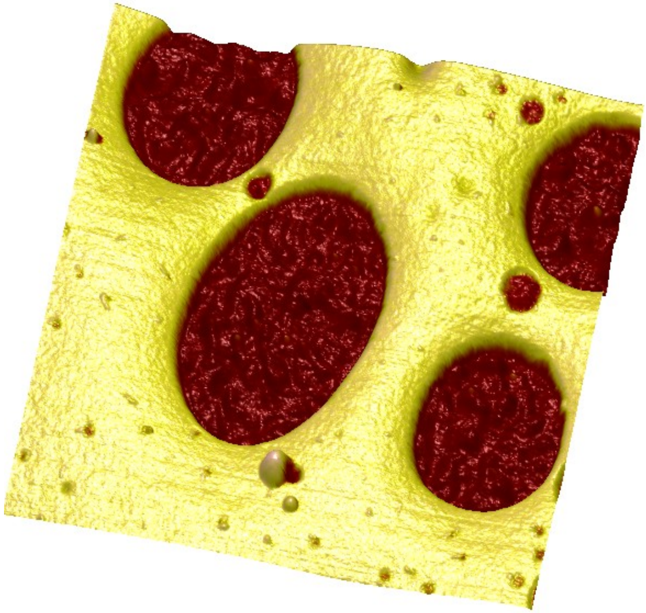


Adhesion Image

The QNM part
DMT Modulus…at every XY position

- DMT Modulus – sample “stiffness”
  - in the image below dark (brown) areas are softer.

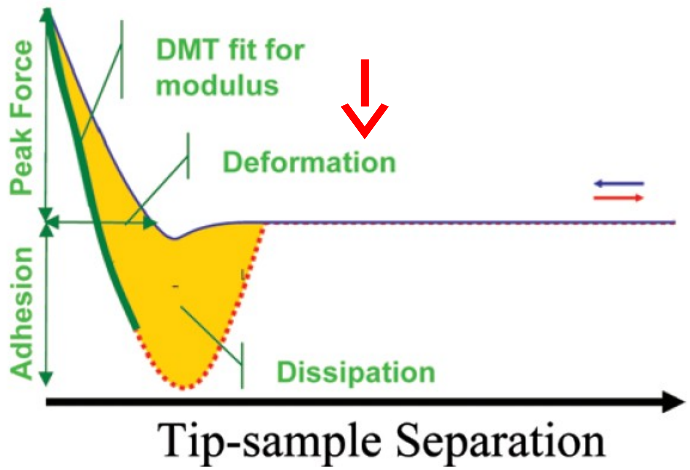


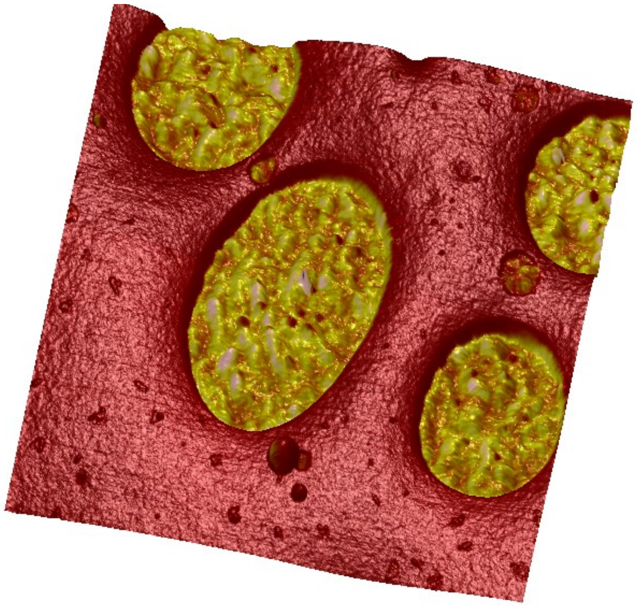


Modulus Image

The QNM part
Deformation…at every XY position

- Deformation – approximation of indentation depth
  - In the image below yellow areas indicate more sample deformation under the probe (softer material).

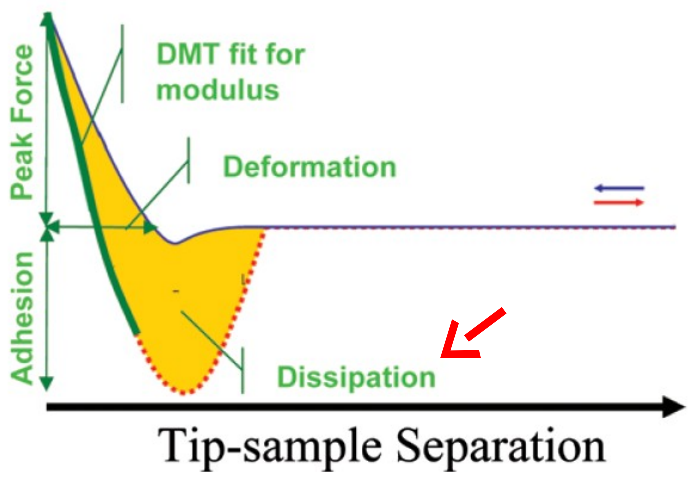


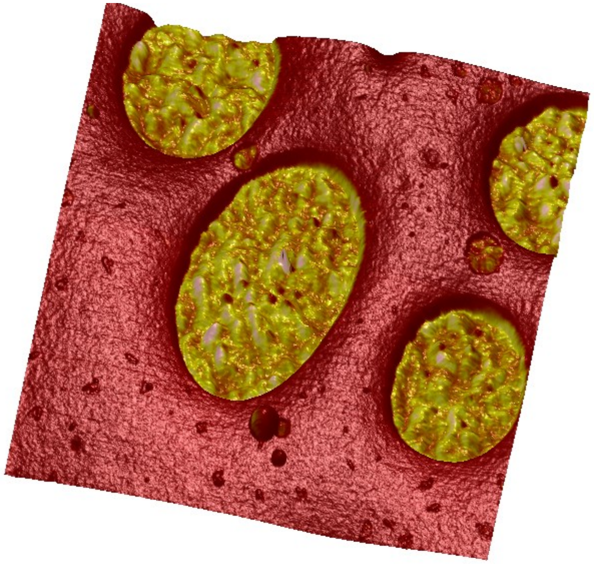


Deformation Image

The QNM part
Dissipation…at every XY position

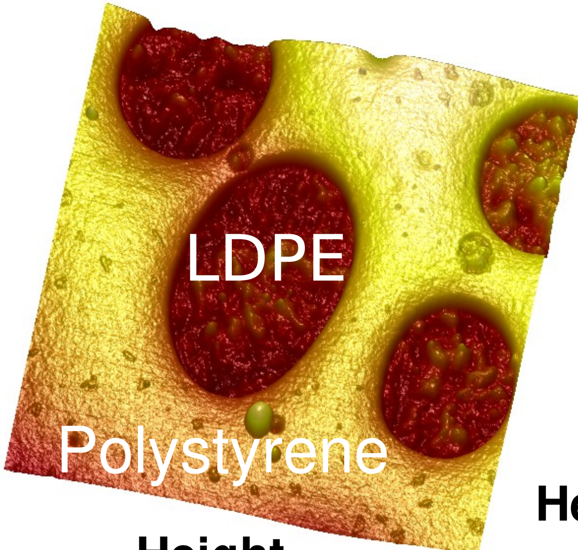
- Dissipation – energy lost by probe during interaction with the sample
  - in the image below yellow areas indicate higher dissipation



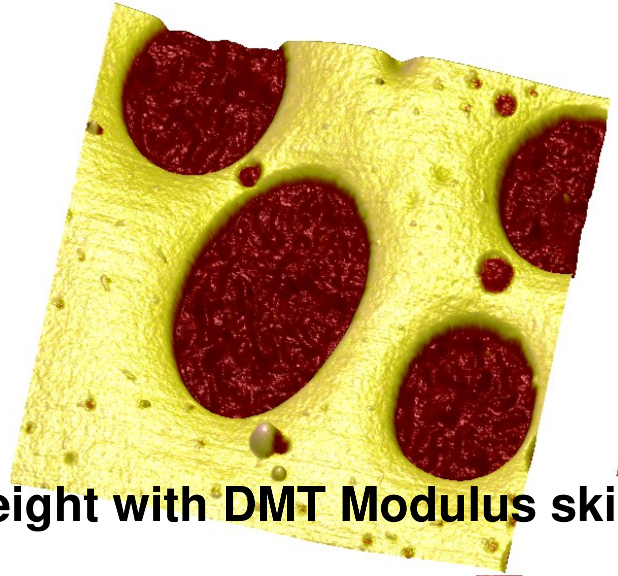


Dissipation Image

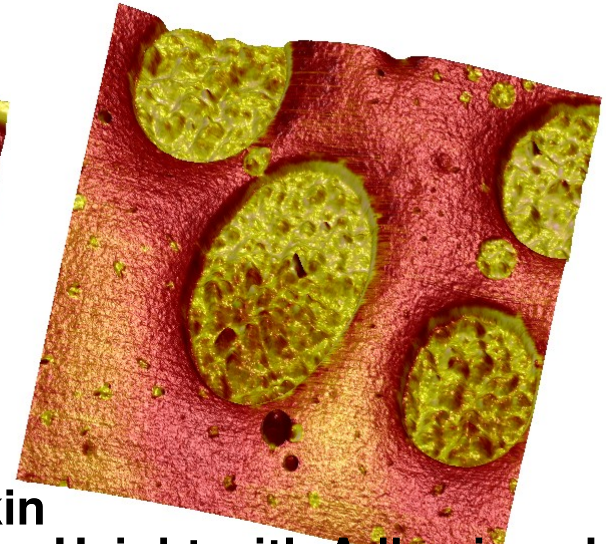
PeakForce QNM – The complete picture
Example: Polystyrene-LDPE



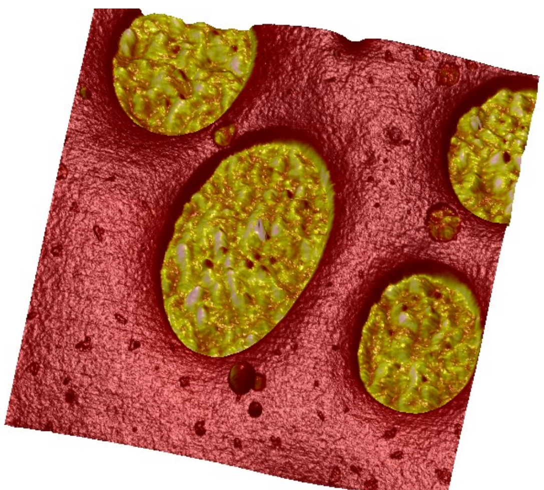
Height



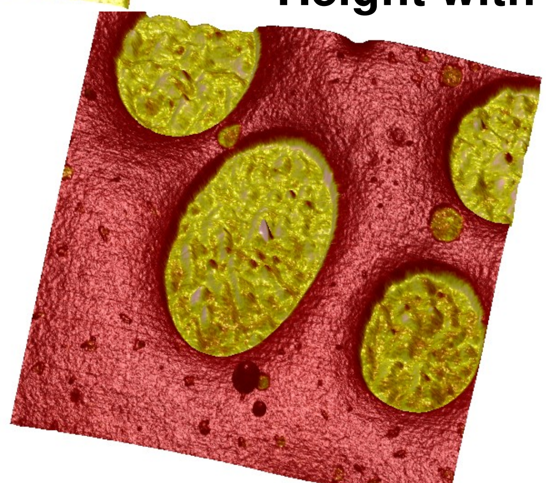
Height with DMT Modulus skin



Height with Adhesion skin



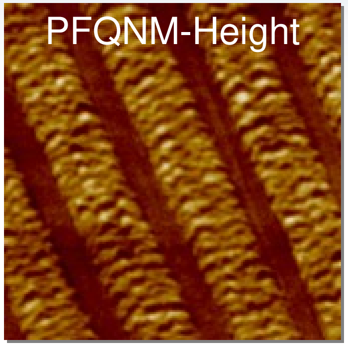
Height with Deformation skin

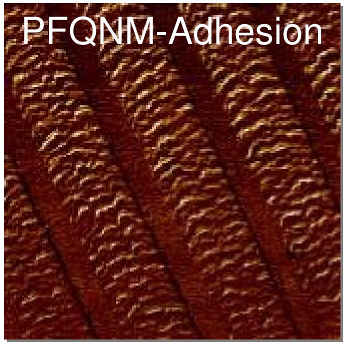


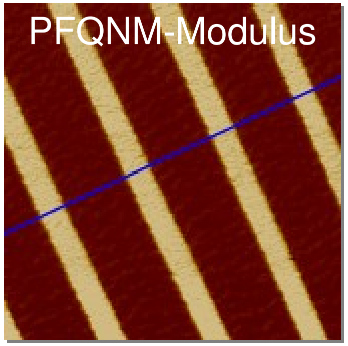
Height with Dissipation skin

- Low-density polyethylene (LDPE) rubber material is softer, stickier, with larger deformation and dissipation compared to Polystyrene.

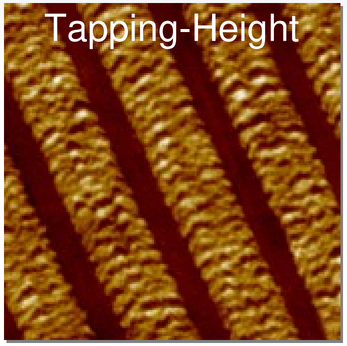
Peak Force QNM: Unambiguous material property mapping

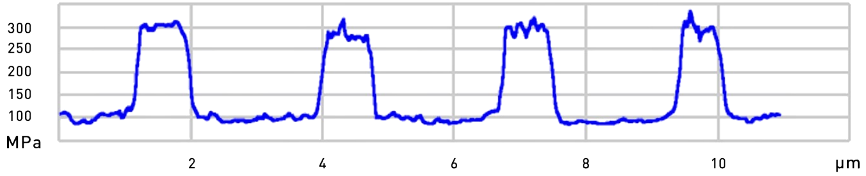




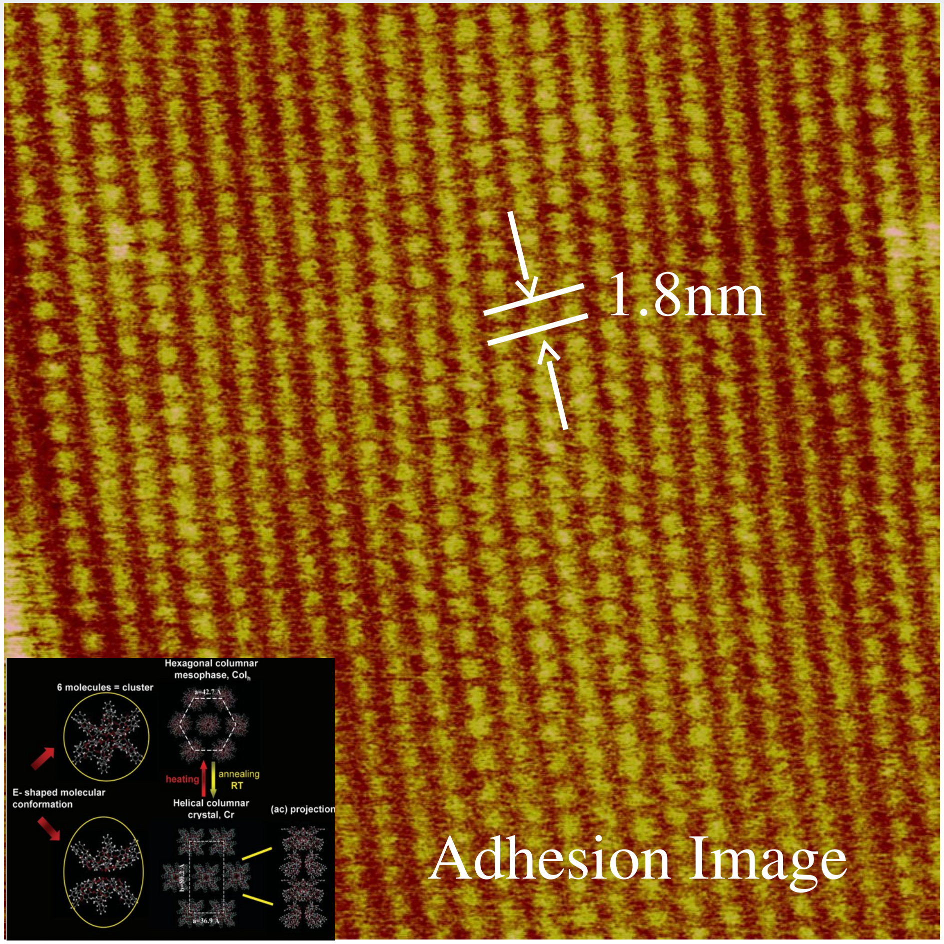


- Multilayered polymer film, 10 µm scans
  - Left: PeakForce QNM images
  - Right: TappingMode images
- Section plot illustrates the ability to measure the modulus various across the polymer layers
- Comparison of the Adhesion and Phase images clearly shows that the phase contrast is primarily due to adhesion. Modulus measurement decouples from other data (such as phase, adhesion which have signatures in the height signals), giving distinctive layered properties quantitatively.
- This example highlights the value of being able to separately, unambiguously, and quantitatively measure nanoscale adhesion and modulus variations with PeakForce QNM.





PeakForce QNM: Direct force control results in high resolution QNM mapping

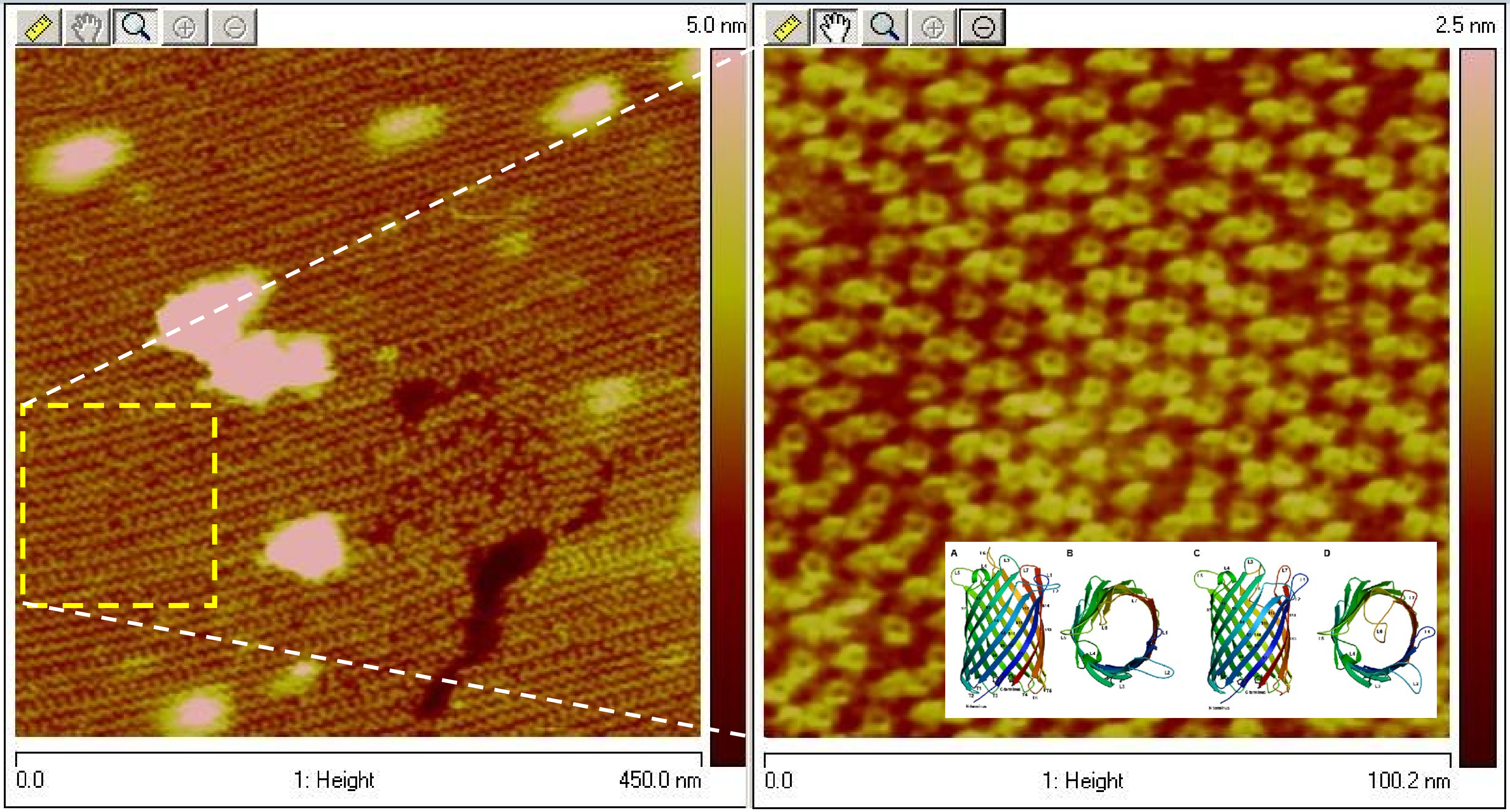


Molecular resolution material property mapping for semi flexible star-shaped mesogen (liquid crystal)

Domitri Ivanov, Institut de Chimie des Surfaces et Interfaces (ICSI)

- Peak force can be as low as ***tens of pN***.
- Ultra low force and direct force control enable the high resolution QNM imaging of liquid crystal which has ***1.8 nm*** periodicity along the molecular chains and 4nm between molecular chains.

PeakForce QNM: Direct force control results in stable and easy imaging in liquids (Peak force around 50 pN)



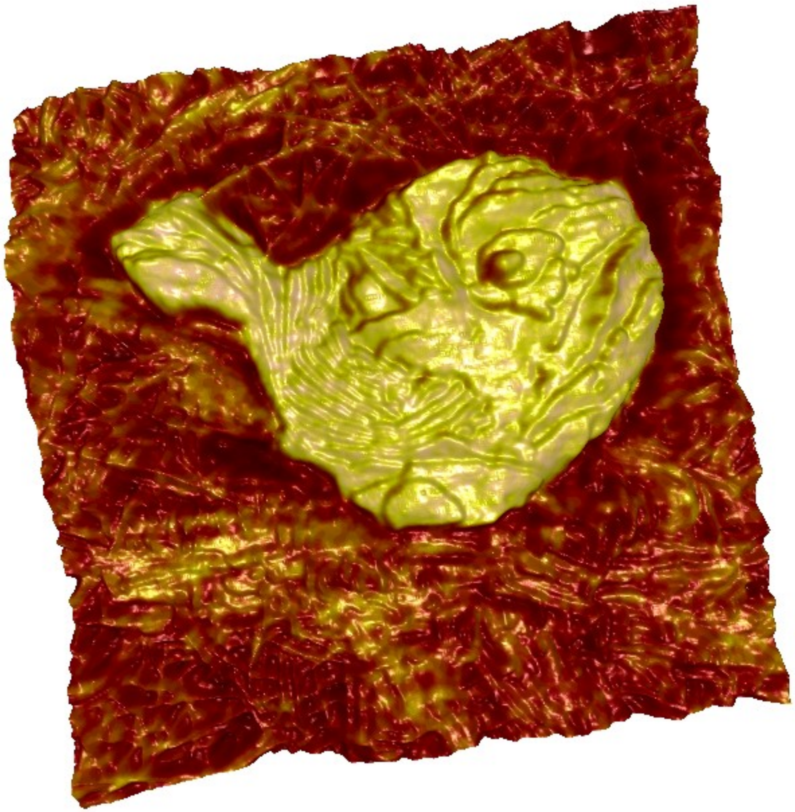
OmpG Membrane Pore (data courtesy of C. Bippes and D. Muller, Dresden Univ.)

- ***Direct force control*** as low as 50pN results in high resolution images in liquid
- No need to tune the cantilever; operating Peak Force Tapping in liquid is as ***easy and stable*** as in air.

Techniques for Mechanical Mapping

	PeakForce QNM	HarmoniX	TappingMode Phase Imaging	Single Harmonic	Dual AC	Pulsed Force Mode	Force Volume
Young's modulus and adhesion mapping	Yes	Yes	Mixed & parameter dependent	Mixed & parameter dependent	Mixed & parameter dependent	Qualitative	Possible offline
Deformation depth mapped?	Yes	Yes	No	No	No	No	Possible offline
Quantitative Modulus range	0.7 MPa - 70 GPa	10 MPa - 10 GPa	--	--	--	--	<1 MPa - ~100 GPa
Adhesion noise level	<10 pN	200 pN	--	--	--	<1 nN	<10 pN
Feedback on Peak Force?	Yes	No	No	No	No	Yes	Yes
Peak force	<100 pN	<5 nN	<3 nN	<10 nN	<5 nN	<20 nN	<50 nN
Lateral resolution	<5 nm	<10 nm	<5 nm	<10 nm	<10 nm	<50 nm	<100 nm
Simultaneous high resolution imaging	Yes	Yes	Yes	Yes	Yes	Moderate	No
Mapping Time	4 minutes	4 minutes	4 minutes	4 minutes	4 minutes	4 minutes	1-8 hours
Easy to use?	Yes	No	Yes	No	No	No	No

Calibration Methods & Best Practices



PEO/SPP – Adhesion overlay on height

Summary

- Probes must be a good “match” to sample
  - Need both indentation & probe deflection
  - Soft samples (PDMS; 2.5-3.5 MPa) require soft probe (ScanAsyst Air; k ~ .5 N/m)
  - Hard samples (PS-Film; 2.7 GPa) require stiff probe (Tap300A k = 40 N/m)

- Two methods to calibrate probes for QNM
  - Absolute
    - Use for soft materials (PDMS with ScanAsyst Air probe)
  - Relative
    - Must use for hard materials (PS-Film with Tap525)
    - Can also use for soft materials

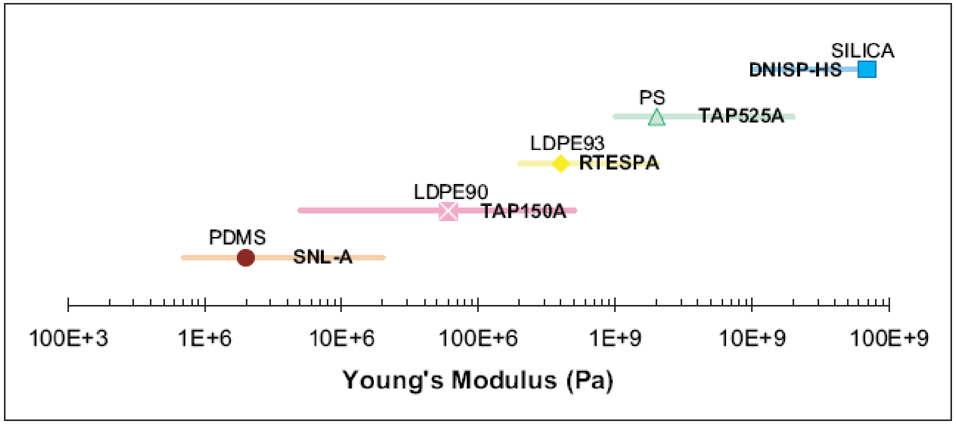
Probe choice

- While the final DMT Modulus value of the unknown sample is not known, typically the range it falls into will be. It is important to choose a probe that produces sufficient sample Deformation while still retaining high force sensitivity. Here are Bruker's recommendations:

Table 2.3a Recommended Probes

Sample Modulus (E)	Probe	Nominal Spring Constant (k)
0.7 MPa < E < 20 MPa	SNL-A	0.5 N/m
5 MPa < E < 500 MPa	Tap150A	5 N/m
200 MPa < E < 2000 MPa	RTESPA	40 N/m
1 GPa < E < 20 GPa	Tap525A	200 N/m
10 GPa < E < 100 GPa	DNISP-HS	350 N/m

Figure 4.2a Modulus ranges covered by various probes. The modulus of the reference sample for each range is indicated as well



Relative vs. Absolute

- $E \sim k/\sqrt{R}$ (E=modulus, k=spring constant, R=tip radius)
- Main differences between “relative” and “absolute” methods.
  - Absolute method requires direct measurement of k and R
    - R must be evaluated at indentation depth
    - If indentation depth changes R can be re-evaluated
  - Relative method uses reference sample to force the *ratio* of $k/\sqrt{R}$ to be correct at a given indentation depth.
    - To get modulus of unknown sample adjust setpoint until the indentation depth is close to that used to establish $k/\sqrt{R}$ on the reference sample.

Relative calibration method

- Relative Method
  - Advantages:
    - Avoids accumulated errors
    - Faster calibration (does not require direct tip radius measurement)
    - Cantilever spring constant measurement not required for quantitative DMT Modulus Data.
  - Disadvantages
    - Requires reference sample with known modulus, close to the value of the sample of unknown modulus that will be measured.

Absolute calibration method

- Absolute Method
  - Advantages:
    - Standard sample with known modulus not required (uncertainties in standard sample modulus and aging eliminated).
  - Disadvantages:
    - Susceptible to accumulated errors
    - Takes longer (must measure tip radius and spring constant)

About the absolute method

- When to use the absolute method
  - Soft samples like PDMS1 and PDMS2 in the reference sample kit
- Why does the absolute method work well with soft samples / soft probes?
  - Thermal tune for measuring k is accurate
  - Tip Qual with the tip check works well and doesn't damage soft probes
  - The probe tip itself is not deformed by the sample
- Why does the absolute method not work well with hard samples / stiff probes?
  - Thermal tune for determining k is inaccurate on high frequency probes
  - The tip can be deformed by the sample (DMT model falls apart)

About the relative method

- When to use the “relative” method
  - If a reference sample of known modulus whose value is close to that of the sample to be measured is available.
  - Hard samples like PS-Film in the standard sample kit
  - Soft samples if preferred over absolute method
- Why is the “relative” preferred for hard samples?
  - It’s not really a choice. k can’t be measured by thermal tune for probes over ~10-15 N/m.
- Does the “relative” method work on soft samples like PDMS1 and PDMS2?
  - Yes

Calibration steps for absolute method

- Choose proper probe (use guideline chart)
- Enter estimated sample Poisson's ratio (use guideline chart)
- Calibrate Deflection Sensitivity on glass or sapphire
  - Glass ok for TAP 150A and ScanAsyst Air
- Thermal Tune for k
- Image Tip Check. Open NanoScope Analysis. Perform 1st Order XY Plane Fit. Open Tip Qual (do not evaluate tip R yet but leave open for later use).
- Engage with auto setpoint, gain, scan rate, and z limit on.
- Make sure synch is ok. Auto config if necessary. (more details on following slides)
- Turn auto setpoint and z limit off
- Set proper Noise Threshold (.2 nm for PDMS1 or PDMS 2)
- Determine indentation
  - Use force monitor to estimate
- Return to Tip Qual and evaluate Tip Radius at estimated indentation depth. Enter into Tip Radius parameter.
- Capture image
- Determine DMT Modulus from Roughness/Image Raw Mean

Calibration steps for relative method

- Choose proper probe (use guideline chart).
- Enter estimated sample Poisson's ratio (use guideline chart).
- Calibrate Deflection Sensitivity on glass or sapphire.
  - Glass is ok for ScanAsyst and Tap150 probes. Use sapphire for RTESPA and stiffer.
- Thermal Tune for k (soft probes only; for stiff probes enter the nominal value).
- Set Engage Setpoint to .05 V for probes stiffer than ScanAsyst Air.
- Engage with auto setpoint, gain, scan rate, and z limit on.
- Make sure synch is ok. Auto config if necessary.
- Turn auto setpoint and z limit off.
- Set proper Noise Threshold (.2 nm for samples in sample kit).
- Image reference sample.
- Note indentation depth.
  - From force monitor for soft, adhesive samples; from deformation channel for hard
- Adjust Tip Radius and/or Cantilever Spring Constant until DMT Modulus data matches known value of reference.
- Image unknown sample and adjust setpoint until indentation depth is close to where it was when the reference sample was measured.
- Evaluate DMT Modulus using Roughness/Image Raw Mean.

Sample Poisson's Ratio

- Even though this is in "Cantilever Parameters" this parameter refers to Poisson's ratio for the sample.
- Use supplied table if unknown…or set to 0 for "Reduced Young's Modulus" in DMT Modulus channel.
- E_{tip} assumed to be infinite.
- Ranges between .2 - .5 Leads to 4-25% difference between the sample's modulus and it's reduced modulus.

Relationship between Reduced Young's Modulus (E*) and Poisson's ratio.

$$E^* = \left[\frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_{tip}^2}{E_{tip}} \right]^{-1}$$

ν_s is Poisson's ratio of sample

E_s is Young's Modulus of sample

ν_{tip} is Poisson's ratio of tip

E_{tip} is Young's Modulus of tip

Guidelines for estimating Poisson's ratio

E_s	ν_s
$E_s < 100$ MPa	0.5
$0.1 < E_s < 1$ GPa	0.4
1 GPa $< E_s < 10$ GPa	0.3

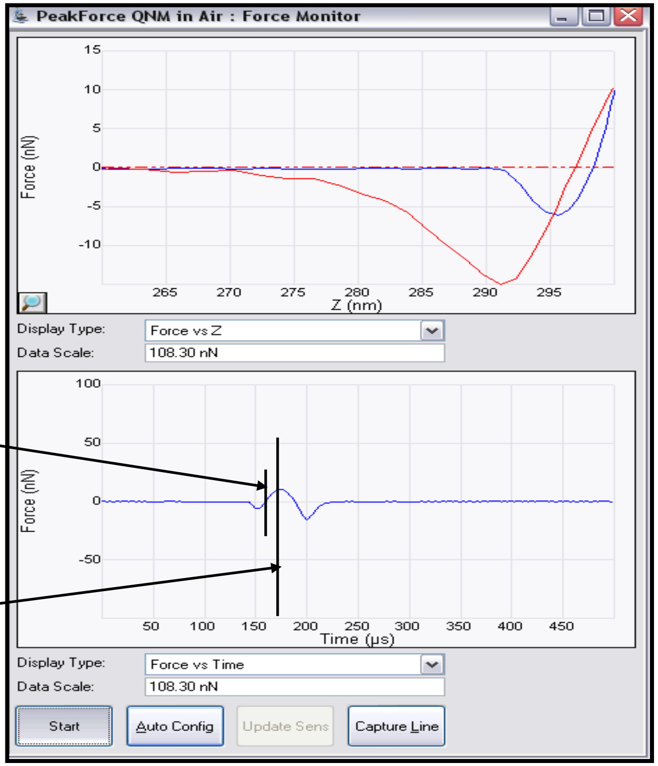
Synchronization Timing

- Zoom in on interaction region of force monitor.
  - Verify "heart beat" is clean with no distortion.
  - Watch out for synchronization problems.
  - Use auto-config if necessary.
- It is important that Peak Force Tapping is properly synchronized for QNM data
  - "good synch" means software looks for peak force at the right cycle time
  - Approach is fully retracted to synch distance (time)
  - Retract is synch distance (time) to fully extended

Peak force occurs after synchronization time. withdraw curve should not be to the left of the approach curve

Software things that peak force is occurring at this time…

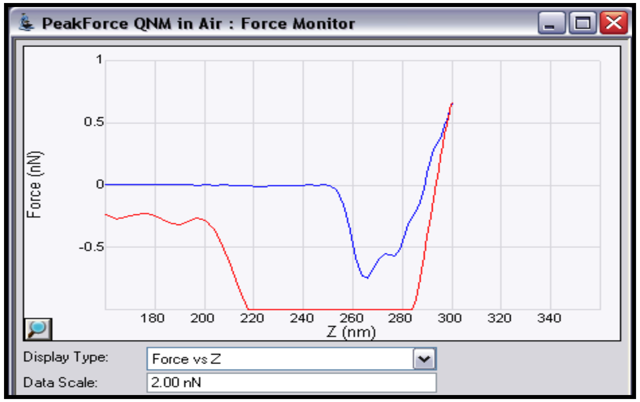
Instead of the actual time peak force occurs



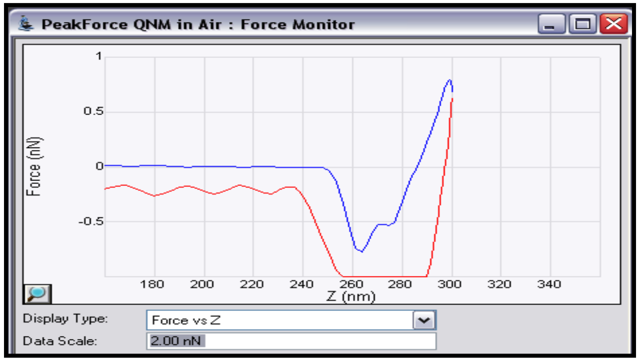
Synch problem examples

- Undock Force Monitor
- Zoom in on contact region of Force vs. Z plot
- Check for synchronization problem
  - Two common "synch" problem
    - Late synch- approach curve contains decreasing force before turnaround.
    - Early synch- retract curve contains increasing force at beginning of turnaround.
  - Use Auto Config button to correct synch problems

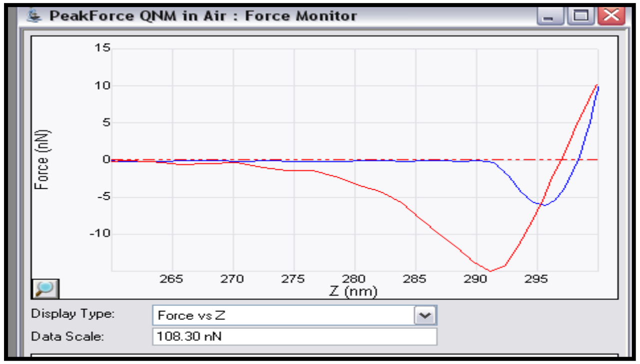
Synchronization correct



Synchronization too late



Synchronization too early



Artifact

- Sources of artifact
  - Optical, fluid
  - Always use probes with reflective coating
  - Carefully align laser and maximize sum
- Artifact on the force curve base line will appear in data channels.
  - *Ideally*, force curve base line should be
    - On 0 V line
    - Flat (no superimposed, periodic artifact)
    - Not moving up and down periodically
- Auto Config button on Force Monitor can often be used to correct
  - Background artifact is corrected upon engaging in air (every scan frame in fluid)
  - If problems develop after engaging try the Auto Config button on the Force Monitor

Setpoint

- Set ScanAsyst control to Individual
- Engage with all (setpoint, gain, scan rate, z limit) *On*
- After engaged and stable turn auto setpoint *Off*
  - Changing setpoint while imaging will cause imaging forces to change
  - Indentation depth can be controlled with setpoint
  - If imaging forces change the probe contact area will change
  - If probe contact area changes the tip radius is no longer valid
  - Modulus & adhesion data will shift

Auto Z Limit & Noise Threshold

- Auto Gain control needs to know how much feedback oscillation or noise is acceptable
- Noise Threshold sets allowable feedback background “noise”
- If Auto Z Limit is On the Noise Threshold is controlled automatically but takes 1 ½ scan frames after engaging to adjust (too long)
  - For example on really flat surfaces Z Limit and Noise Threshold will be lowered on really flat surfaces
- If feedback oscillations are too high, and you don’t want to wait 1 ½ scan frames, turn off Auto Z Limit and set Noise Threshold appropriately
  - Use common sense
    - Noise Threshold of 1 nm would not work well for really flat surface.
    - Noise Threshold of 1 nm would be ok for surface like Tip Check

Engage Force

- Engage ignores Peak Force Setpoint
  - Doesn’t matter if ScanAsyst Auto Setpoint is On or Off.
- For engaging the Peak Force Engage Setpoint (in other controls) is used.
  - Default .15 V
    - OK for ScanAsyst Air
    - WAY TOO HIGH for stiff probes like RTESPA or TAP 525. Sample and/or probe damage will occur.
    - Try .05 V for stiff probes
- After engage occurs at the engage setpoint the software will
  - Remove back ground artifact from force curve base line (scan size changes to 10 nm for about 5 seconds.
  - Adjust setpoint automatically if ScanAsyst Auto Setpoint in On
  - Adjust setpoint to Peak Force Setpoint if ScanAsyst Auto Setpoint is Off

Other Models

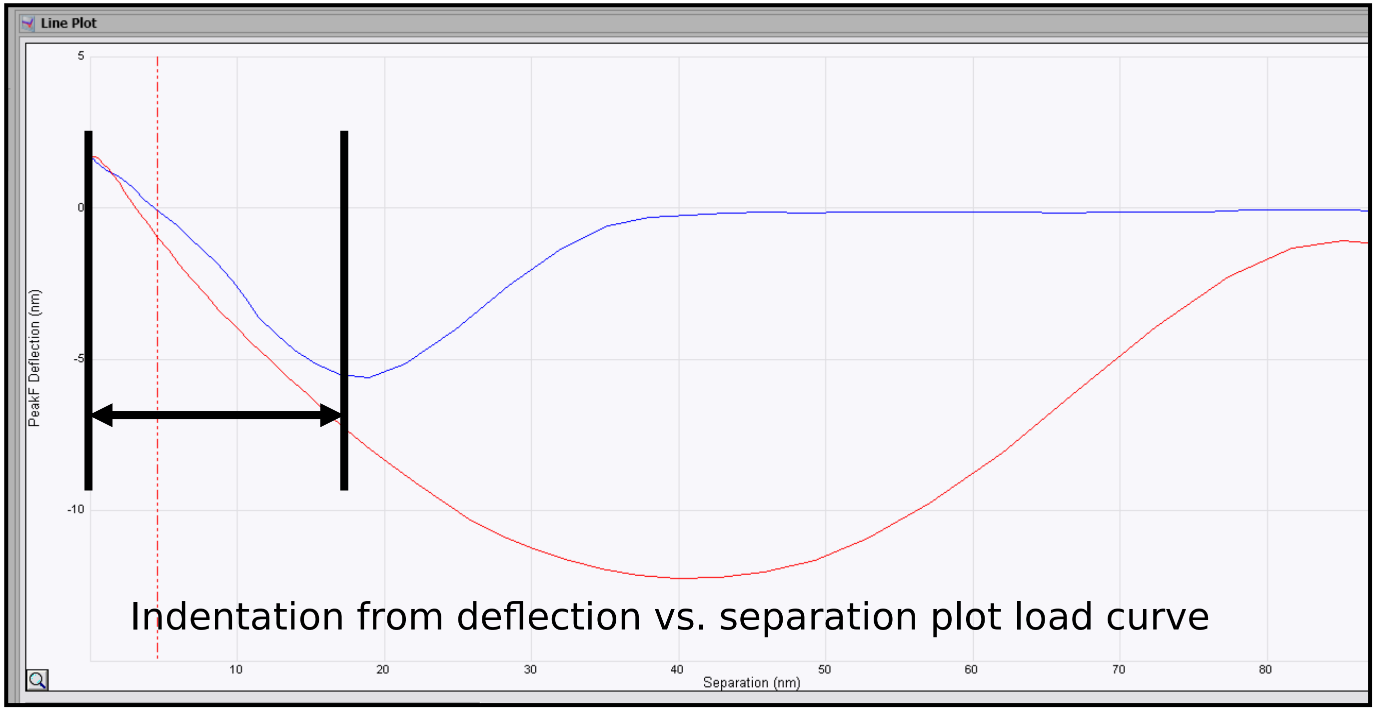
- Our implementation uses the “DMT” model for modulus data
  - Not always the best model
- What are the options?
  - None for the real-time modulus data channel
  - Several scan lines of raw data can be captured using HSDC (capture line button on Force Monitor)
  - Force curves can be exported with PFQNM Offline in NanoScope Analysis and fit to different models outside of NanoScope

Deformation data (caution)

- Deformation (data channel) is an estimate of indentation.
- Deformation is a data channel.
  - Force vs. Separation curves are being computed in the back ground while imaging.
  - Deformation data is calculated (crunched)
- Deformation channel is a reasonable estimation (sometimes) of indentation on hard sample / stiff probe combinations
- Deformation is a bad estimation of indentation on soft sample / probe combinations (like PDMS and ScanAsyst Air) when there is high adhesion

Indentation on sample like PDMS (dmt model)

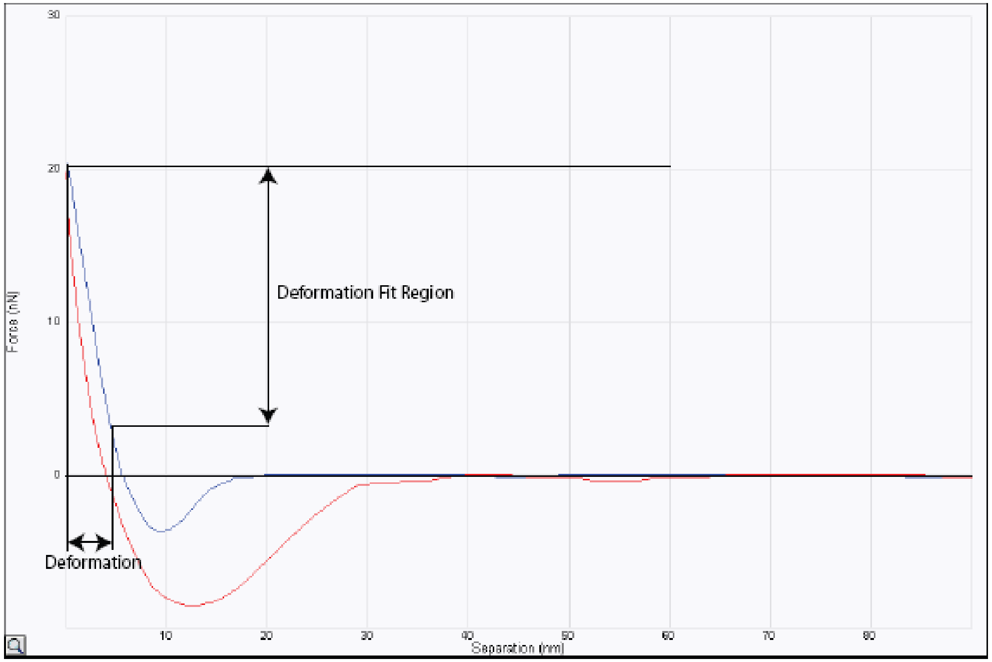
- The distance on a Deflection vs. **Separation** plot's load (extend) curve starting at the "snap-to-contact" point and ending at z fully extended.
  - The force monitor Force vs. Z is a good approximation



Indentation from deflection vs. separation plot load curve

Deformation

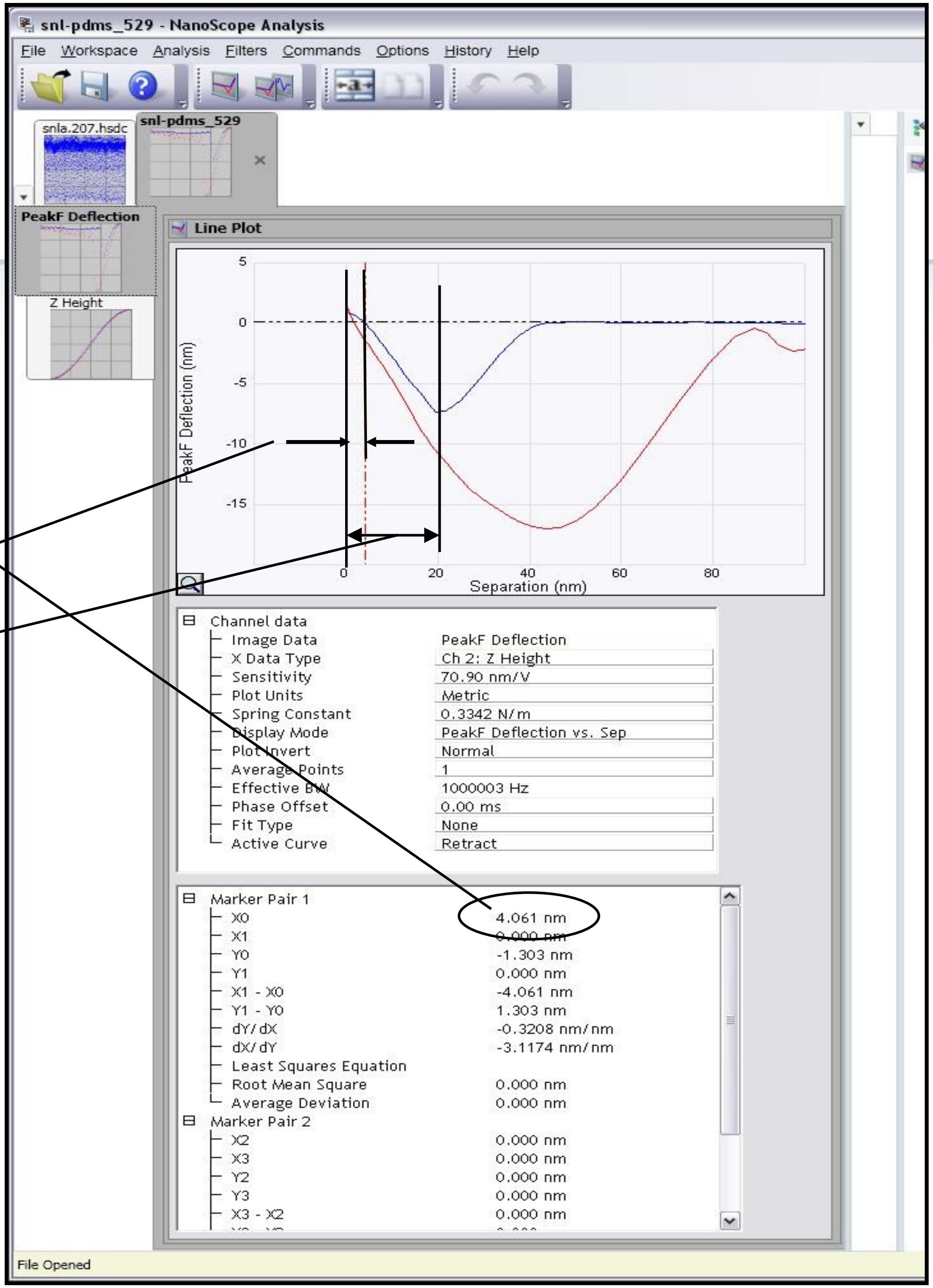
- How does NanoScope compute deformation data for the real-time deformation map?
  - NanoScope uses the region beginning approximately at the intersection of the load curve (not snap-to-contact) with the 0 baseline and ending at the maximum z extension.
  - The “deformation fit region” parameter then reduces another 15% by default



Deformation on PDMS

NanoScope will assign a pixel value of 4 nm to the deformation map based on this force vs separation plot.

It’s more like ~20 nm

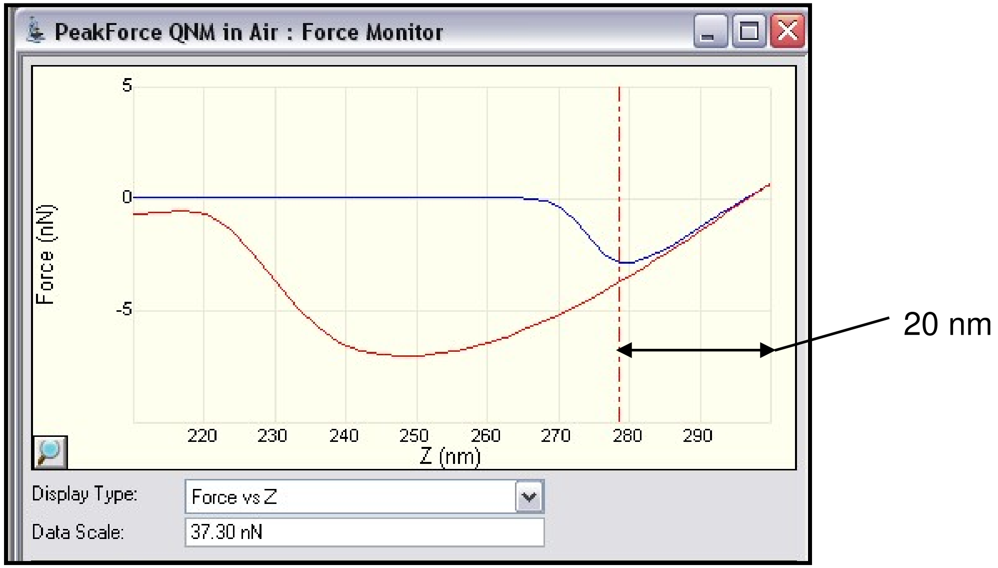


- This matters because when absolute is used the effective tip radius is much lower at a “Height From Apex” value of 4 nm than at 20 nm.

- Since E ~ k/√R the modulus calculation would be too high if the tip radius was calculated at 4 nm above the tip apex.

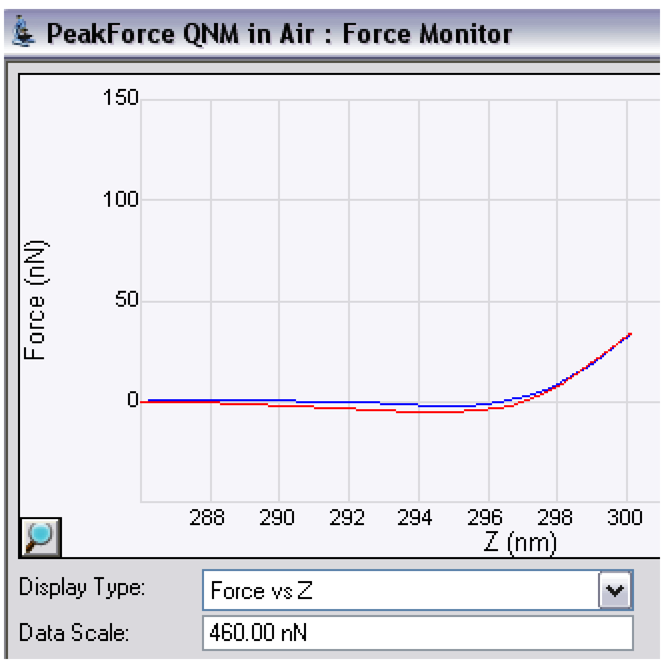
Estimating indentation from the force monitor

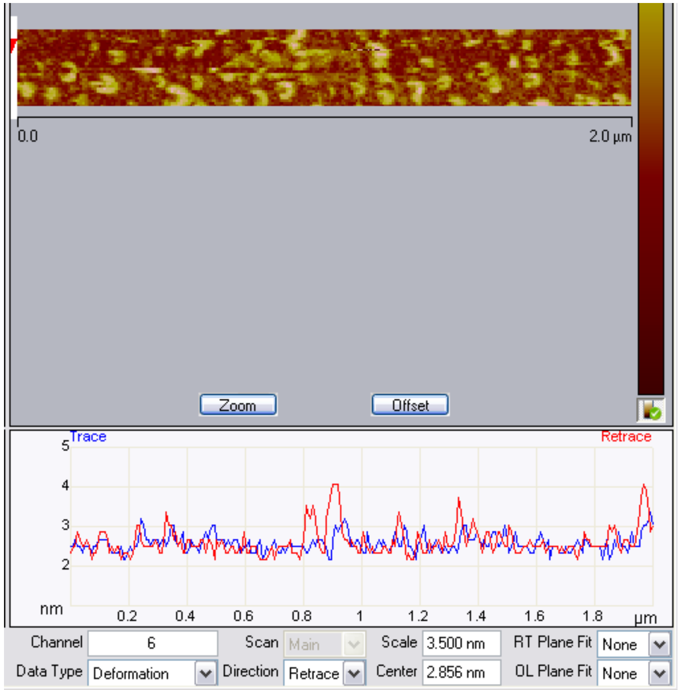
- To estimate indentation of soft samples like PDMS with a ScanAsyst Air probe the force monitor can be used
  - Distance on Z axis from snap-to-contact to end of approach plot



Deformation as approximation of indentation on hard samples

- Deformation is a good approximation of indentation on hard sample / stiff probe
  - On the approach plot there is not significant snap-to-contact (on z or separation axis) because the probe is stiff
  - If you are instructed to estimate indentation from the deformation data channel use the average value from the line trace.
  - If there is no substantial snap to contact on approach the "deformation fit region can be set to 100%





Summary

- Use the Force Monitor to estimate indentation for PDMS1 and PDMS2 (soft, adhesive samples).
- Use the Force Monitor or the deformation data channel to estimate indentation for harder samples like PS-Film if they are in close agreement.
    - If the deformation data still doesn't match well just use the Force Monitor
- If in question use HSDC and PFQNM Offline in NanoScope Analysis to estimate indentation.

Sources of error

- Thermal Tune
  - When used properly (right probes) thermal tune should be within 10-30% of the real k value
  - $E \sim k/\sqrt{R}$
- Deflection Sensitivity
  - Drift (especially in the first 10 minutes with soft probes)
  - Error in calibration
  - Done on contaminated area of sample (will be too high)
  - Modulus data depends on (deflection sensitivity)^3
- Sample doesn’t work well for DMT model
- Probe shape not ideal

Tip Shape of new tips

- QNM relies on contact mechanics (DMT model) that assume simple tip geometry
  - Hemi-sphere
  - Flat punch
  - Cone
- Tip shapes are not simple or predictable even when new

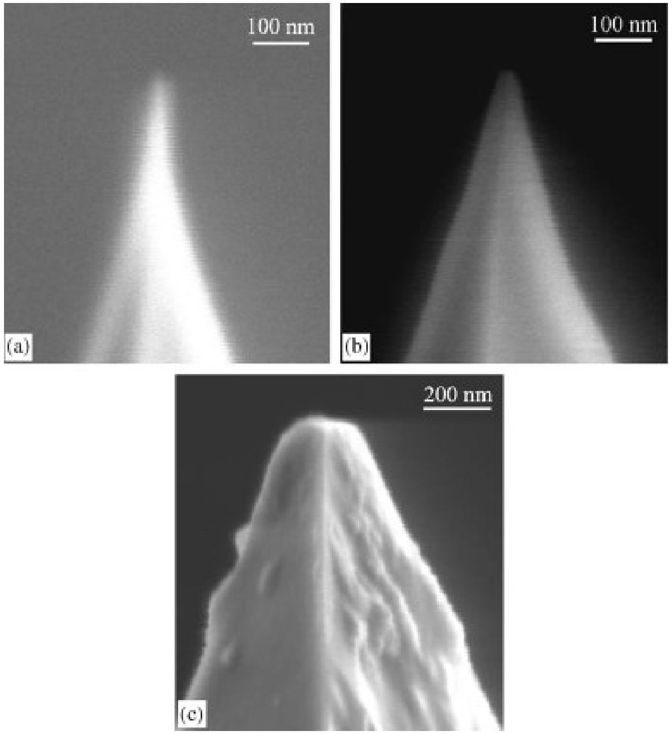


Fig. 2. SEM images of brand new tips revealing the wide differences in possible new-tip geometry. The scale is the same for all three images. (a) Sharp tip with radius of curvature $R < 10$ nm.(b) Tip with $R \sim 20$ nm. (c) Very blunt tip with possible surface contamination not seen in other tips.

Kopycinska-Mueller et. al. (2006) Ultramicro Volume 106: 466-474

Tip shape changes with use

- Tips dull
- Tips pick up debris
- Tips fracture

- How often do you re-characterize the tip with prolonged use?
  - Typically it is done once before the experiment begins

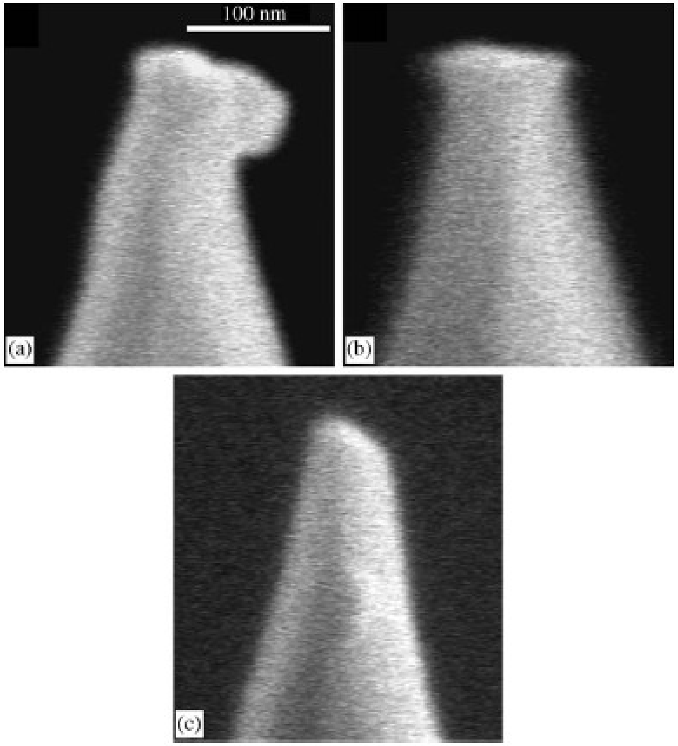
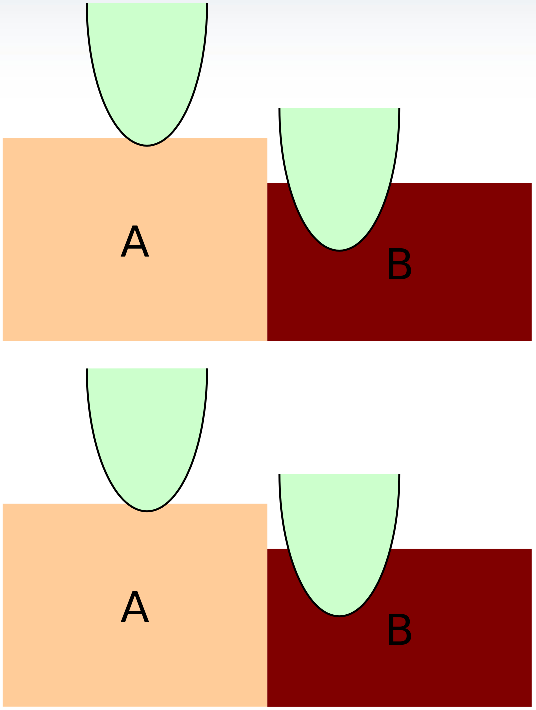


Fig. 3. SEM images of three tips affected by fracture in the AFAM measurements. The images were acquired after the (a) third, (b) fifth, and (c) first AFAM tests. The scale is the same for all three images.

Kopycinska-Mueller et. al. (2006) Ultramicro Volume 106: 466-474

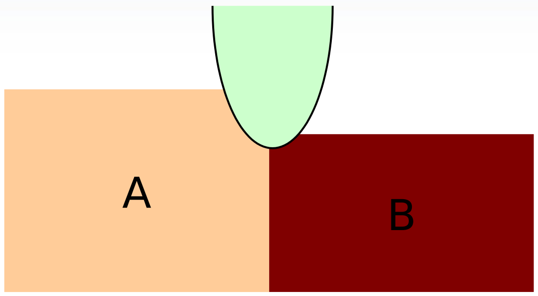
Sample heterogeneity

- Heterogeneity
  - E ~ k/√R
  - R evaluated at indentation depth
  - If indentation depth changes significantly then R changes significantly
  - If R changes then E changes
  - Conclusion: samples with components of much different modulus values will pose a challenge.
    - Example: PS-LDPE (2 GPa – 50 MPa)
    - Probe (Tap 525) that deforms PS by about 5 nm could deform LDPE about 50 nm



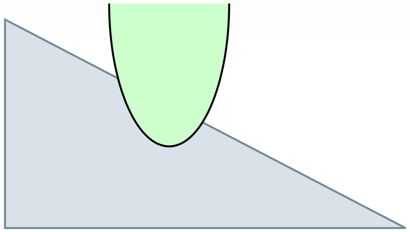
Edge artifact

- Feature transitions
  - High modulus changes at material interface (edges) is usually pretty but artifact



Sample flatness

- Sample “flatness”
  - The flatter the better
  - Angles cause problems with indentation contact area



PeakForce QNM

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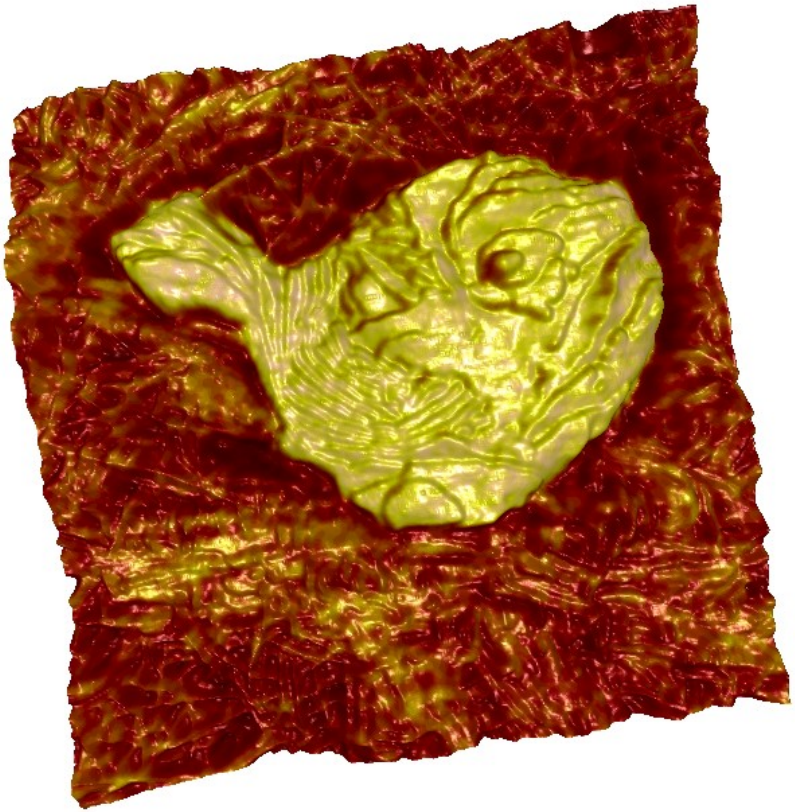
Exercise 1

Deflection Sensitivity calibration
Determining k with Thermal Tune
Determine Tip Radius with Tip Qual



PEO/SPP – Adhesion overlay on height

Exercise 1 summary

- Deflection Sensitivity calibration.
  - Deflection sensitivity calibration is very important for qnm work. We will calculate deflection sensitivity from 3 separate ramps and make sure the values are in close agreement (better than 5%).
- Thermal Tune
  - Use Thermal Tune to determine cantilever spring constant (k)
- Tip Radius
  - For absolute method tip radius must be determined at average indentation depth.
  - Tip check sample is imaged first. Later, when sample of interest is imaged and indentation depth is known, the tip radius is evaluated using Tip Qual
  - For this exercise we will assume the indentation depth is 10 nm.

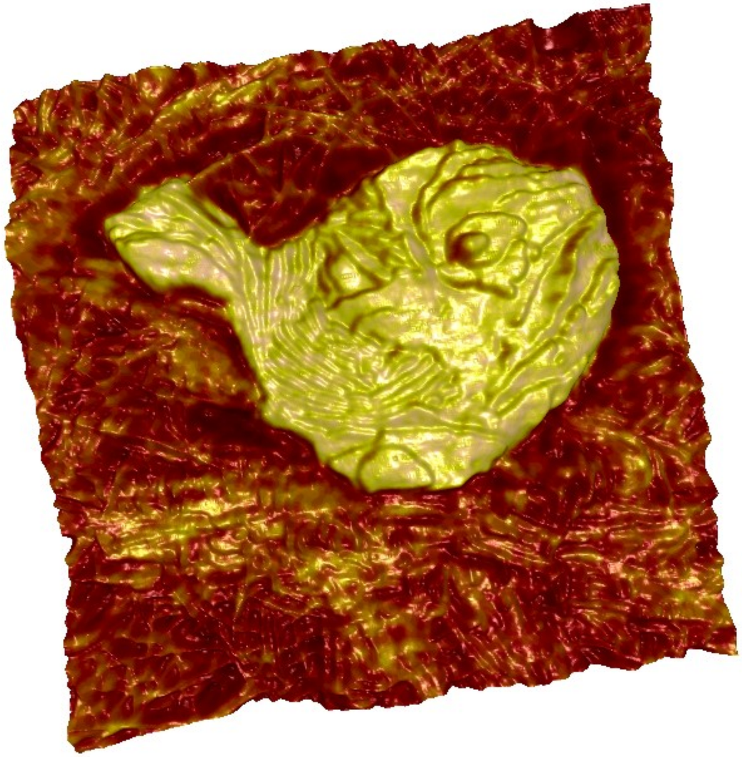
Exercise 1 instructions

- Use ScanAsyst Air Probe
- Calibrate Deflection Sensitivity and k with Thermal Tune
  - Use “PFQNM – How To – Def. Sens. and thermal Tune”
  - Record value of deflection sensitivity
  - Record value of k
- Image Tip Check and use Tip Qual to determine Tip Radius
  - Assume indentation depth of 10 nm for “Height from Apex 1”
  - Use “PFQNM – How To – Determine Tip Radius Using Tip Qual”
  - Record value
- Leave probe on system and workspace open for next exercise

Exercise 1 Discussion

- What was deflection sensitivity?
- What was k?
- What was tip radius?

PFQNM – How To - Def. Sens. and thermal Tune



PEO/SPP – Adhesion overlay on height

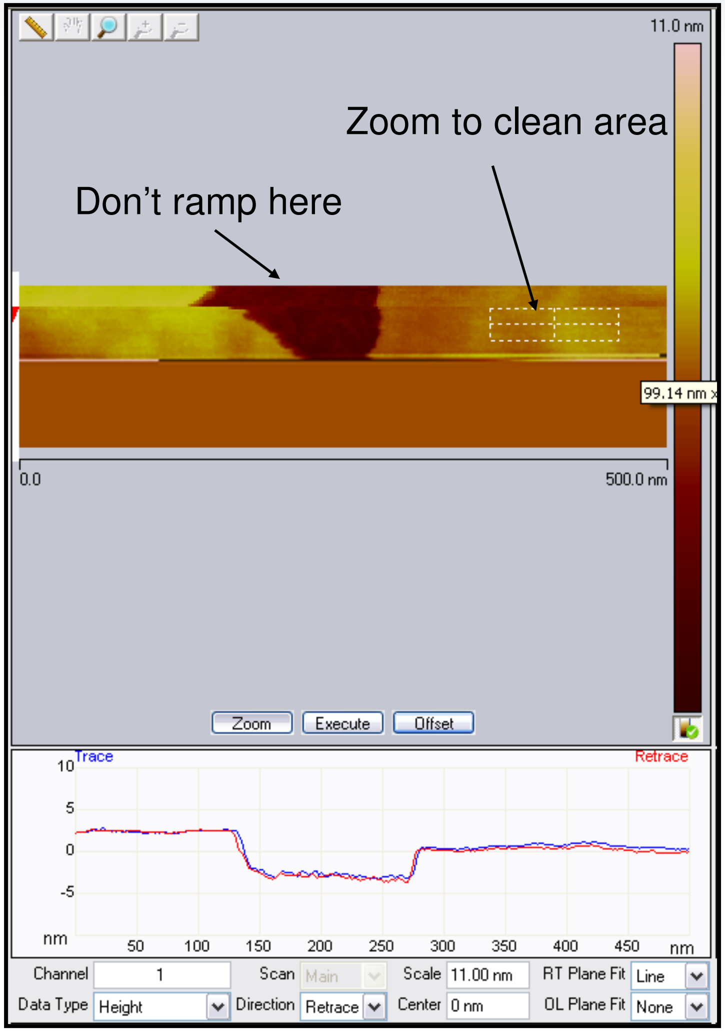
Def. Sens. and thermal Tune 1

- Open PeakForce QNM In Air experiment.
- Load ScanAsyst Air probe.
- Load fused silica or sapphire sample.
- Configure settings to engage.
- Engage

Scan	
Scan Size	500 nm
Aspect Ratio	4.00
X Offset	0.000 nm
Y Offset	0.000 nm
Scan Angle	0.00 °
Scan Rate	0.501 Hz
Tip Velocity	0.501 µm/s
Samples/Line	256
Lines	64
Slow Scan Axis	Enabled
Feedback	
Peak Force Setpoint	0.01238 V
Feedback Gain	30.00
LP Deflection BW	40.00 kHz
ScanAsyst Noise Threshold	0.500 nm
ScanAsyst Auto Control	Individual
ScanAsyst Auto Gain	On
ScanAsyst Auto Setpoint	On
ScanAsyst Auto Scan Rate	On
ScanAsyst Auto Z Limit	On
PeakForce QNM Control	
Peak Force Amplitude	150 nm
Lift Height	44.9 nm
Cantilever Parameters	
Spring Constant	0.6000 N/m
Tip Radius	10.0 nm
Sample Poisson's Ratio	0.500
PeakForce QNM Limits	
Force Limit	1.237 V
Dissipation Limit	4096 Arb
DMTModulus Limit	4096 Arb
LogDMTModulus Limit	32.00 log(Arb)
Limits	
Z Limit	6.044 um
Deflection Limit	24.58 V
Other	
LP Deflection	Enabled
Tip Bias Control	Ground
Sample Bias Control	Ground
Units	Metric
Minimum Engage Gain	10.0
Peak Force Engage Setpoint	0.1500 V
Bidirectional Scan	Disabled
Tip Serial Number	
Output 1 Data Type	Off
Output 2 Data Type	Off

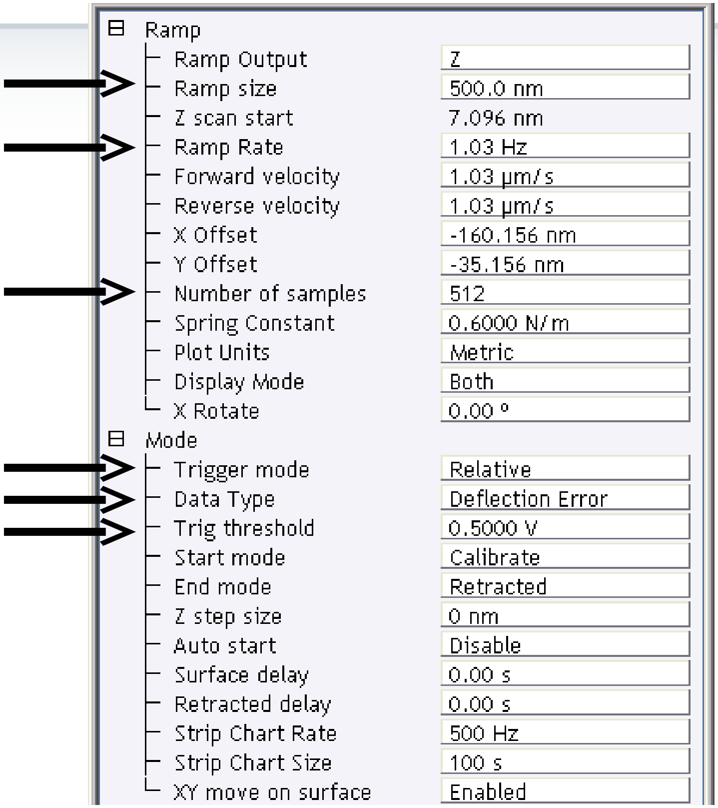
Def. Sens. and thermal Tune 2

- Make sure the center of the image area where the ramp will be performed is clean and flat. If it's not move or zoom to a clean area.



Def. Sens. and thermal Tune 3

- Switch to Ramp Mode
- Configure ramp parm's
- Trigger Threshold (Icon & MultiMode)
    - ScanAsyst Air or Tap150A - .5V
    - RTESPA or Tap525 - .2V



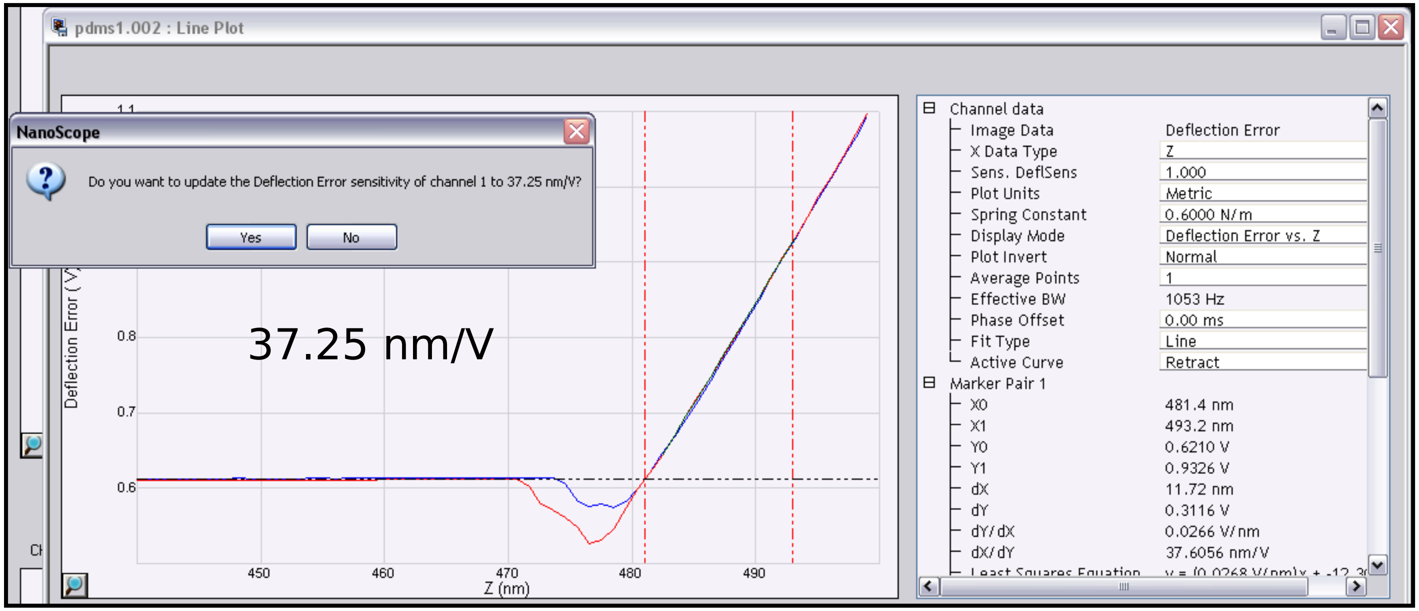
Def. Sens. and thermal Tune 4

- Tip: Calibrate deflection sens. from captured ramps because:
  - Doing so from real-time ramp only uses 2 points for the line fit.
  - Doing so offline uses multiple points for the line fit.
- Click ramp single then click capture to capture the ramp.
- Repeat two more time so that 3 ramps are captured.

Def. Sens. and thermal Tune 5

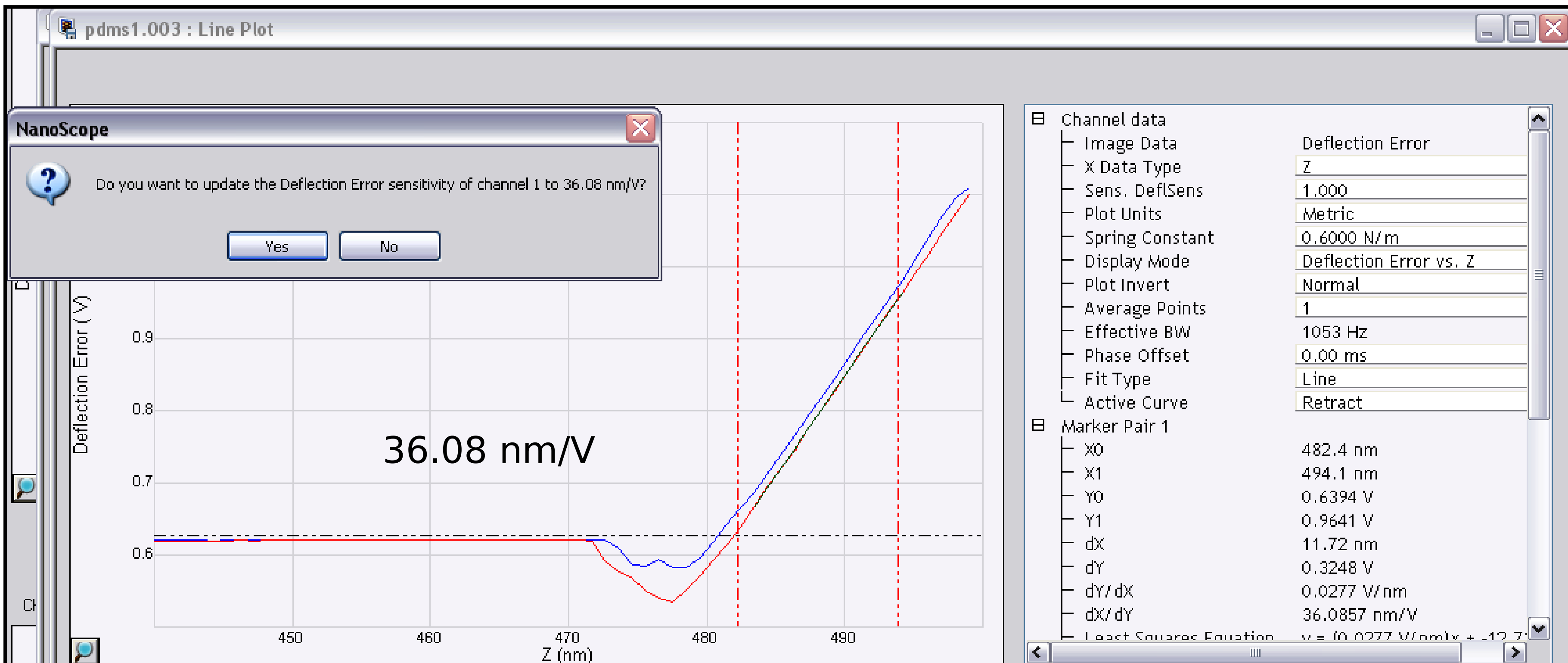
- Open the first captured ramp
- Zoom into the contact region
- Set Active Curve to Retract
- Set Fit Type to Line (this will help to visually verify the fit)
- Drag in cursors for the fit
- Click the Update Sensitivity Icon and note the value.
- Click Yes to update the deflection sens. in this file

Tip: Updating def. sens. in captured ramp files only saves the value in that file. The final value will need to be manually entered into the detector calibration window.



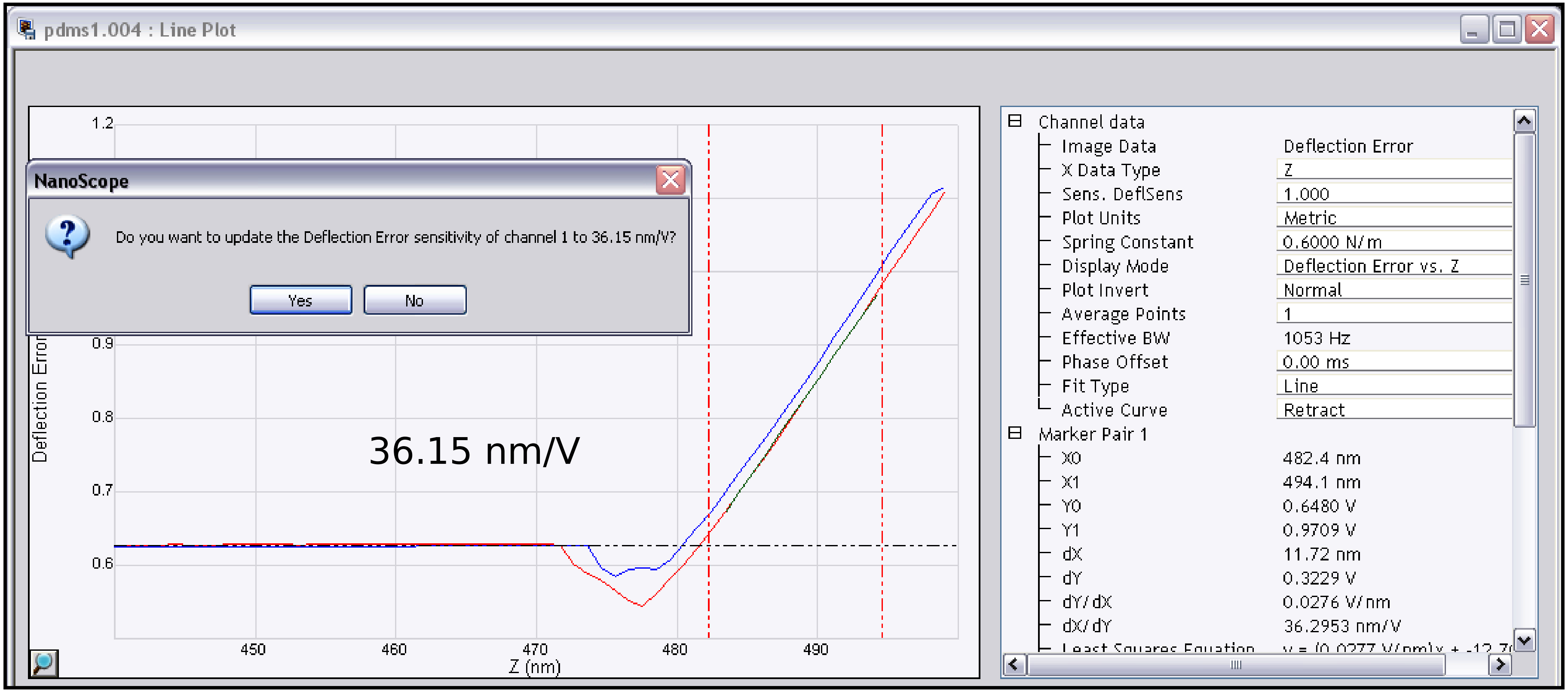
Def. Sens. and thermal Tune 6

- Deflection sensitivity cont'd
- Repeat deflection sensitivity on second ramp.



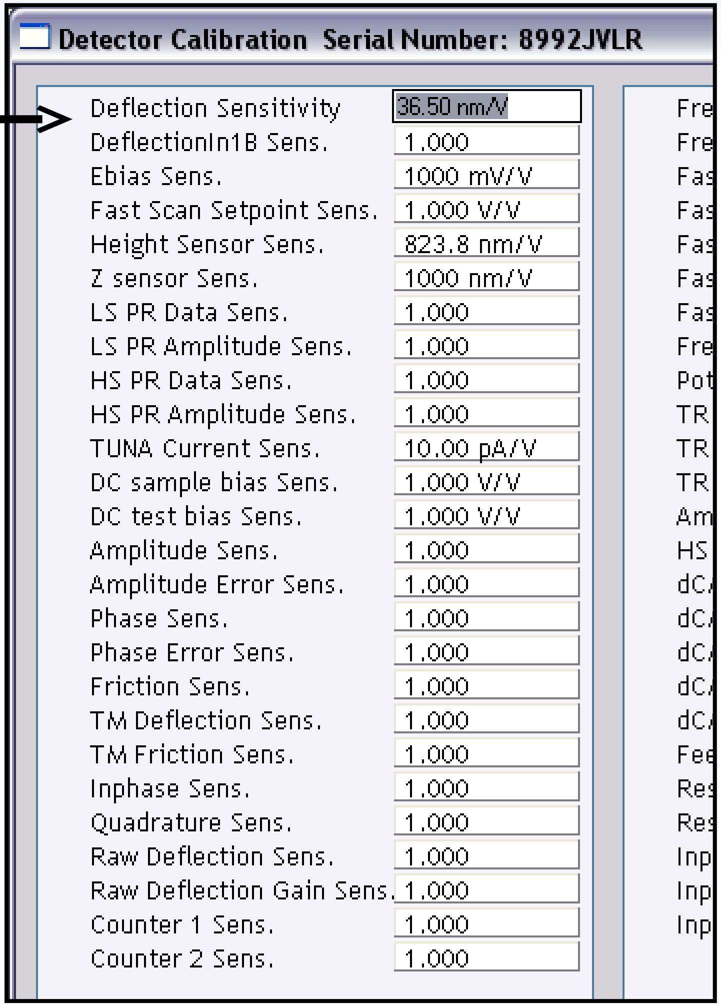
Def. Sens. and thermal Tune 7

- Deflection sensitivity cont'd
- Repeat deflection sensitivity on third ramp.



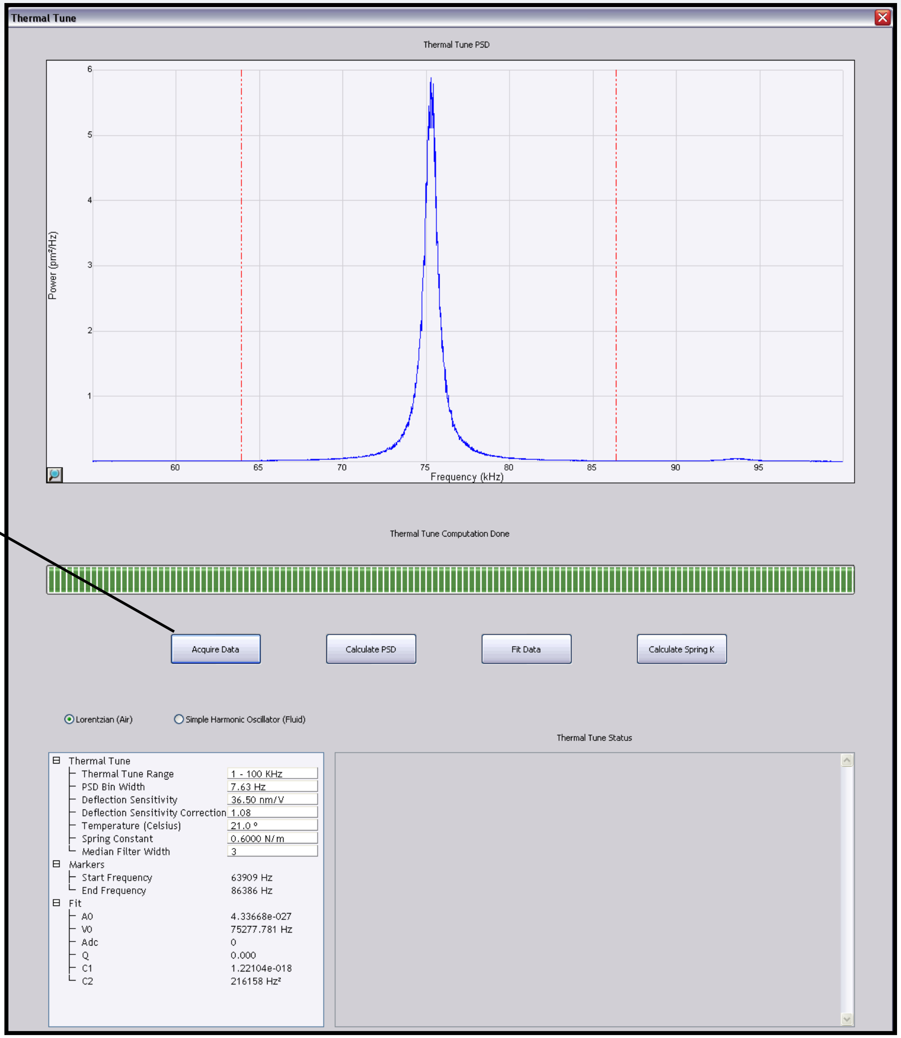
Def. Sens. and thermal Tune 8

- Deflection sensitivity cont’d
- Make sure the three def. sens. values are close in value.
  - The values should not differ by more than ~5%
  - If one of them is substantially different do not use the value.
- Calculate the average def. sens. from the three ramps.
- Enter the value, including units, into Calibrate/Detector/Deflection Sens.



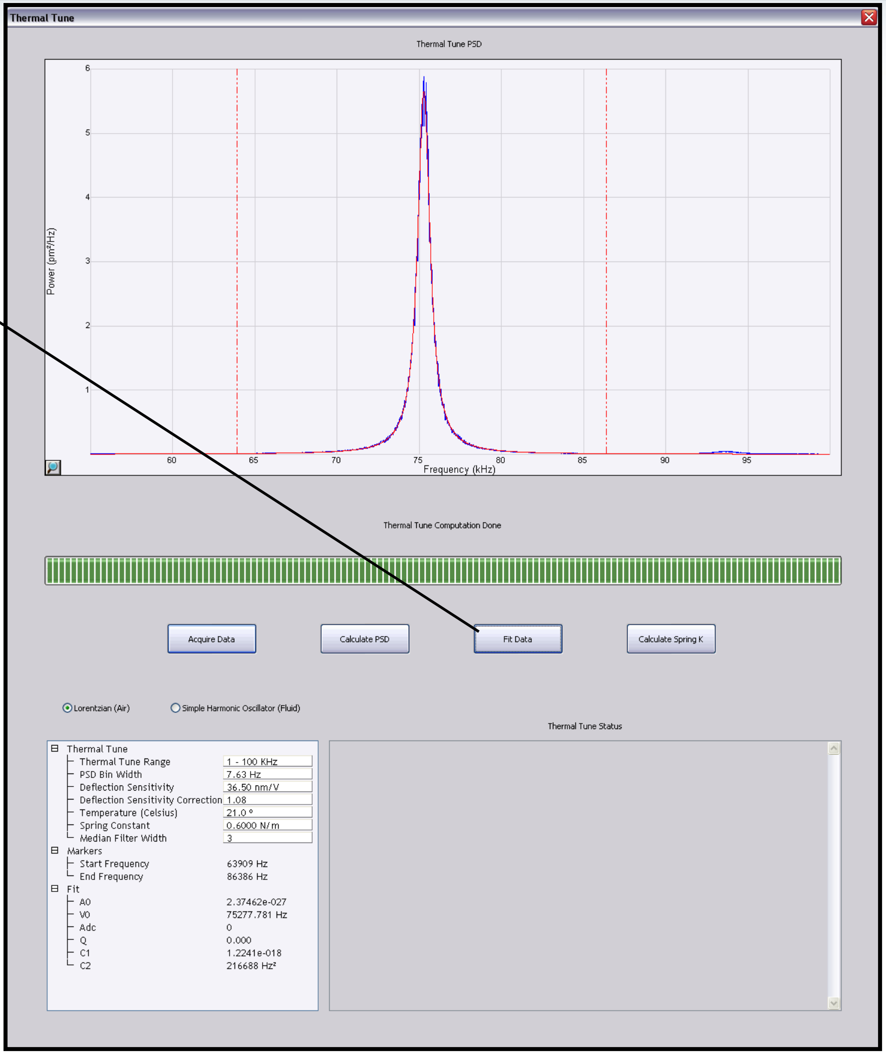
Def. Sens. and thermal Tune 9

- Withdraw the probe to prepare for thermal tune.
  - (if using a multimode withdraw 5x so the tip is 100um over surface)
- Click the Thermal Tune Icon
- Click the Acquire Data button. When the ramp is finished a plot will appear.
- Zoom in on the peak. Make sure 0 baseline is present on both sides of the peak.
- Drag in cursors for the data fit. The cursors should intersect the 0 baseline on both sides of the peak.



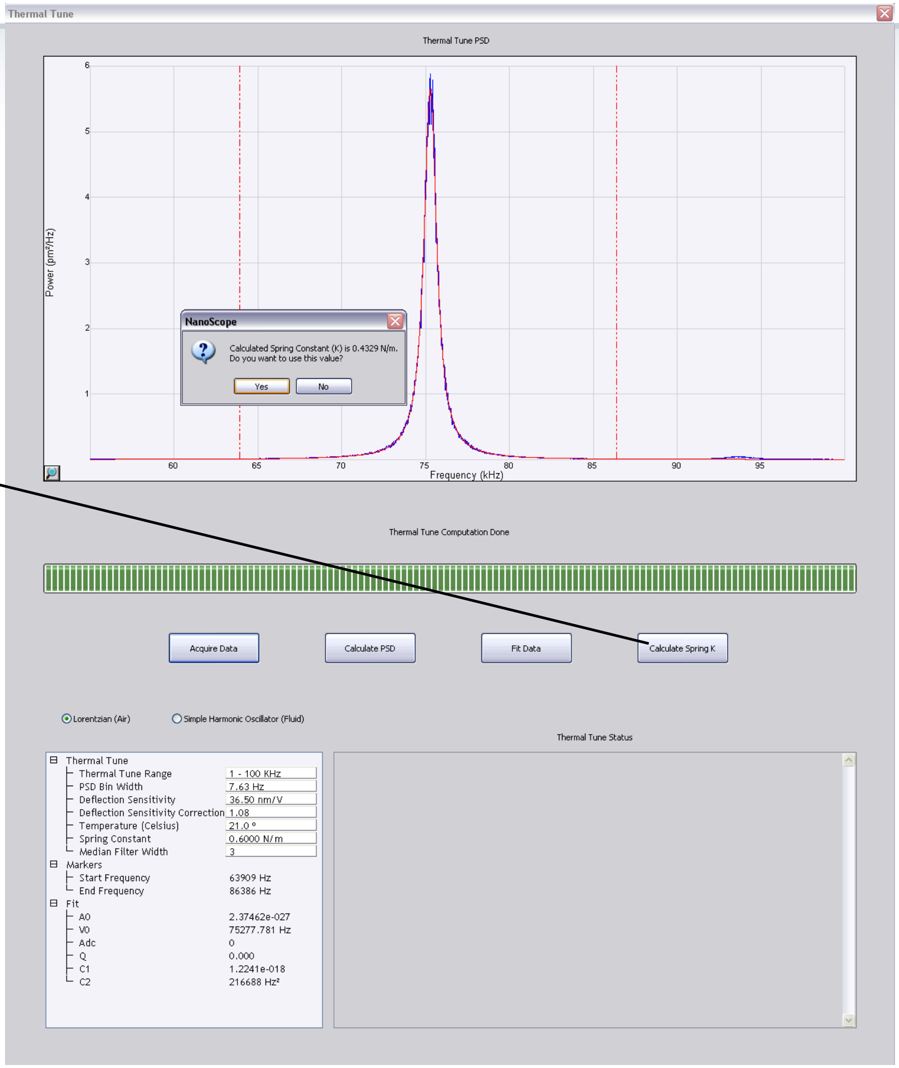
Def. Sens. and thermal Tune 10

- Click the Fit Data button.



Def. Sens. and thermal Tune 11

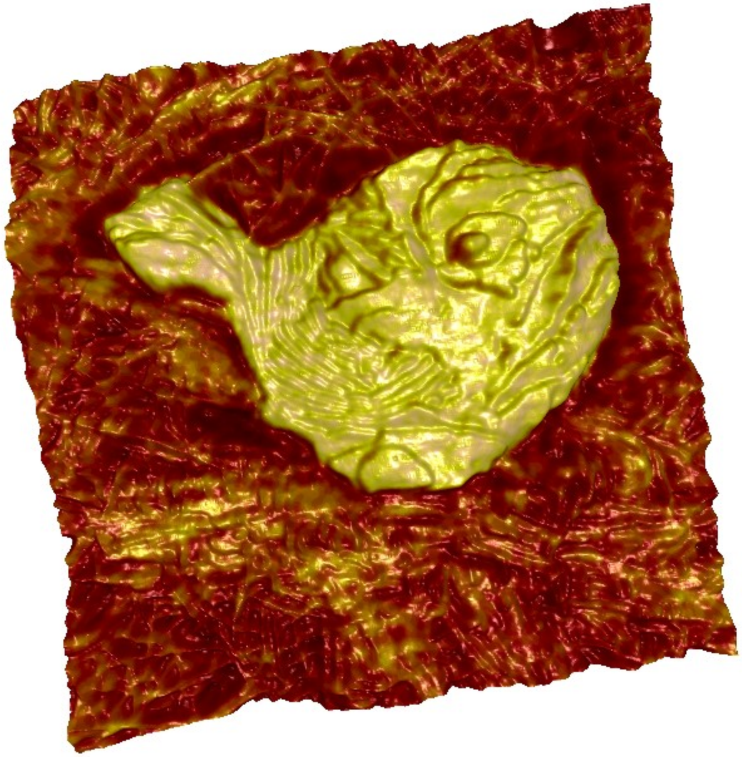
- Click the Calculate Spring k button.
- Click Yes to accept the k value.
- Close the Thermal Tune window.



Tip: The usual value for ScanAsyst Air probes is 0.4-0.8 N/m.

PFQNM – How To –

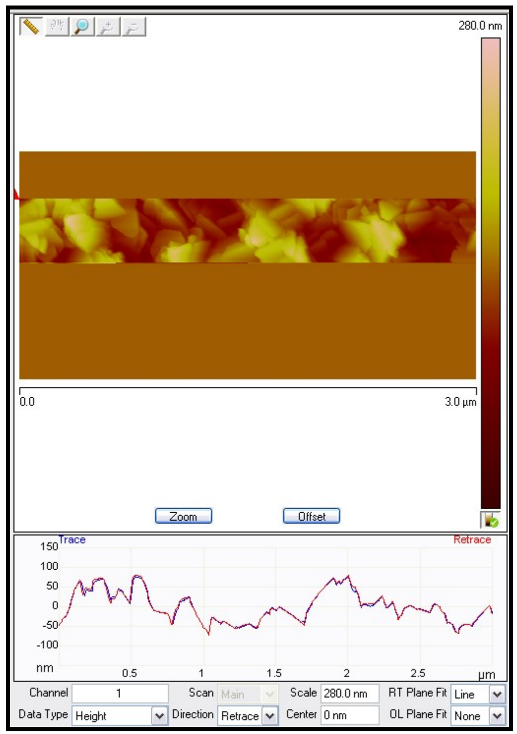
Determining Tip Radius with Tip Qual



PEO/SPP – Adhesion overlay on height

Determining Tip Radius with Tip Qual 1

- Load the Tip Check sample.
- Configure parameters as shown.
- Engage
- Capture Image

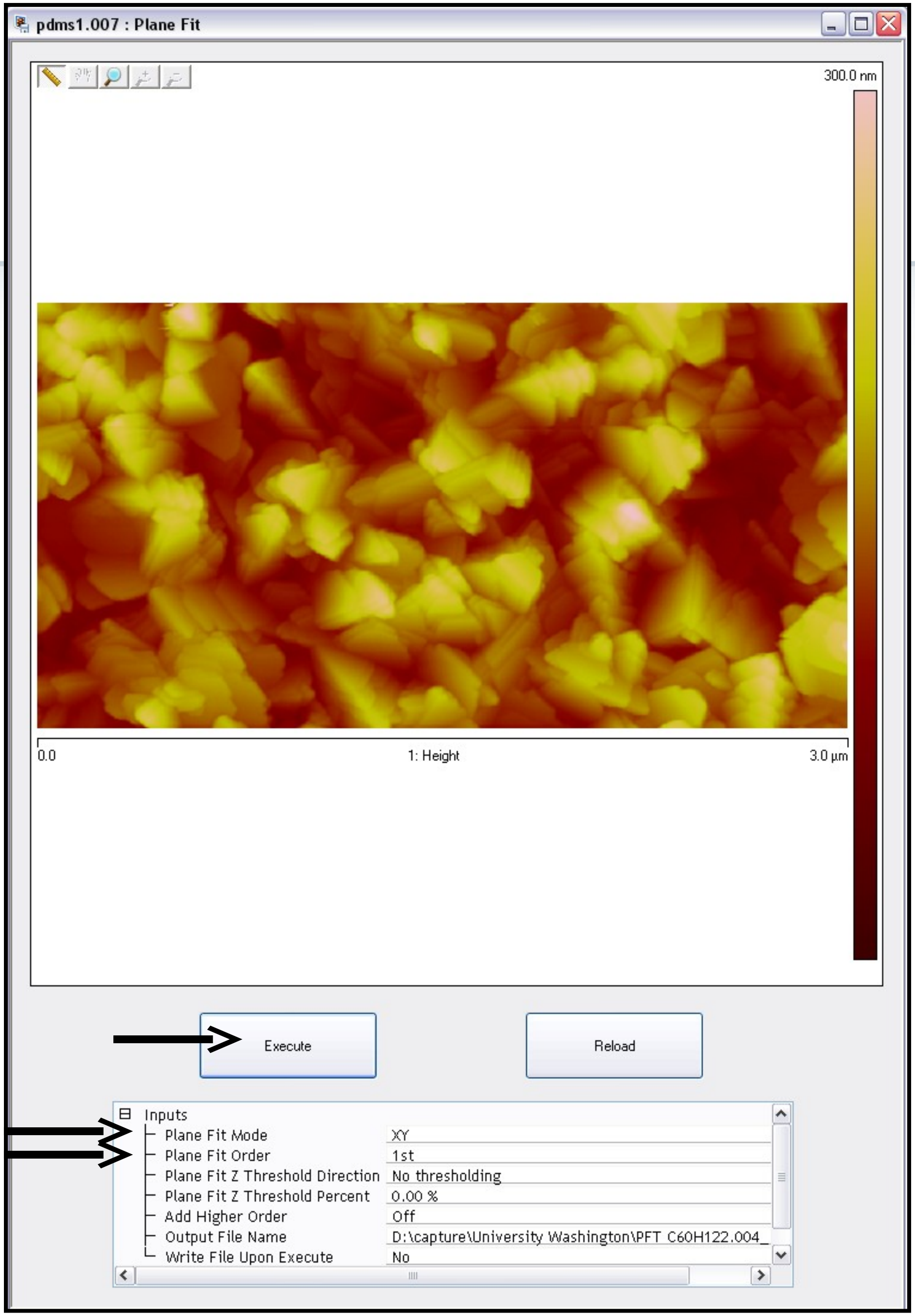


Scan	
Scan Size	3.00 µm
Aspect Ratio	2.00
X Offset	0.000 nm
Y Offset	0.000 nm
Scan Angle	0.00 º
Scan Rate	0.501 Hz
Tip Velocity	3.00 µm/s
Samples/Line	512
Lines	256
Slow Scan Axis	Enabled
Feedback	
Peak Force Setpoint	1.890 nN
Feedback Gain	14.92
LP Deflection BW	40.00 kHz
ScanAsyst Noise Threshold	1.00 nm
ScanAsyst Auto Control	Individual
ScanAsyst Auto Gain	On
ScanAsyst Auto Setpoint	On
ScanAsyst Auto Scan Rate	Off
ScanAsyst Auto Z Limit	Off
PeakForce QNM Control	
Peak Force Amplitude	150 nm
Lift Height	23.2 nm
Cantilever Parameters	
Spring Constant	0.4329 N/m
Tip Radius	10.0 nm
Sample Poisson's Ratio	0.500
PeakForce QNM Limits	
Force Limit	10.00 nN
Dissipation Limit	1.000 MeV
DMTModulus Limit	2.000 GPa
LogDMTModulus Limit	32.00 log(Pa)
Limits	
Z Limit	6.044 um
Deflection Limit	24.58 V
Other	
LP Deflection	Enabled
Tip Bias Control	Ground
Sample Bias Control	Ground
Units	Metric
Minimum Engage Gain	10.0
Peak Force Engage Setpoint	0.1500 V
Bidirectional Scan	Disabled
Tip Serial Number	
Output 1 Data Type	Off
Output 2 Data Type	Off

Determining Tip Radius with Tip Qual 2

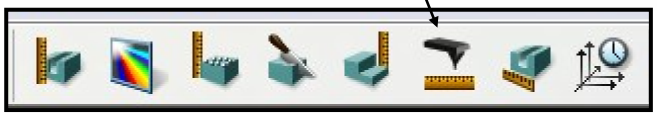
- Open the captured image with NanoScope Analysis
- Set Plane Fit Mode to XY
- Set Plane Fit Order to 1st
- Click Execute

Plane Fit Icon

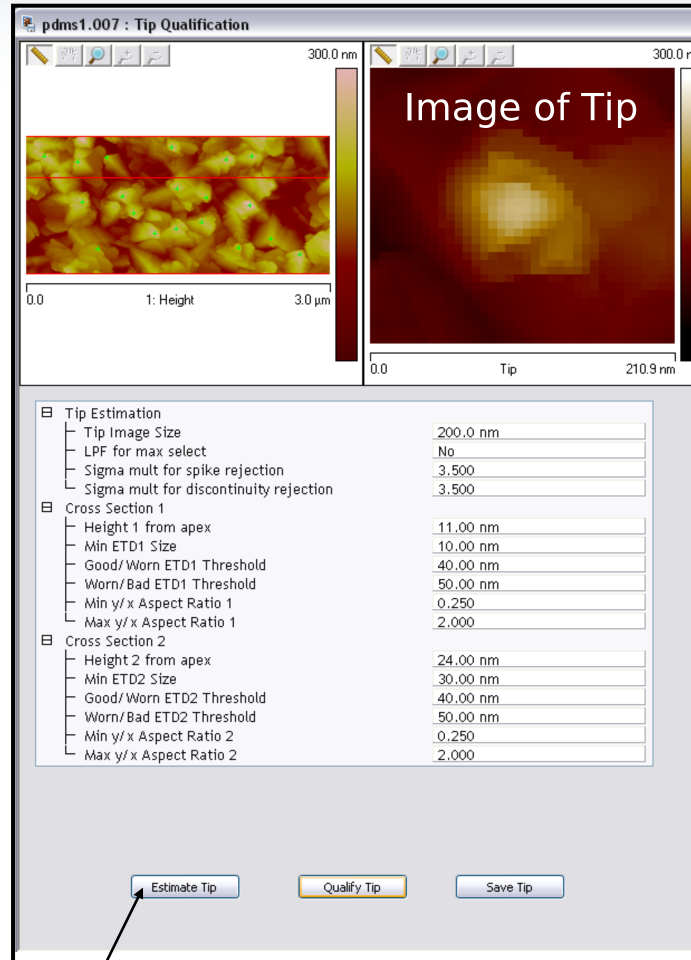


Determining Tip Radius with Tip Qual 3

Tip Qual Icon



- Click the Tip Qual Icon
- Set Tip Image Size to 100 nm
- LPF for max select On
- Click on Estimate Tip
  - An image of the Tip now appears in the image window



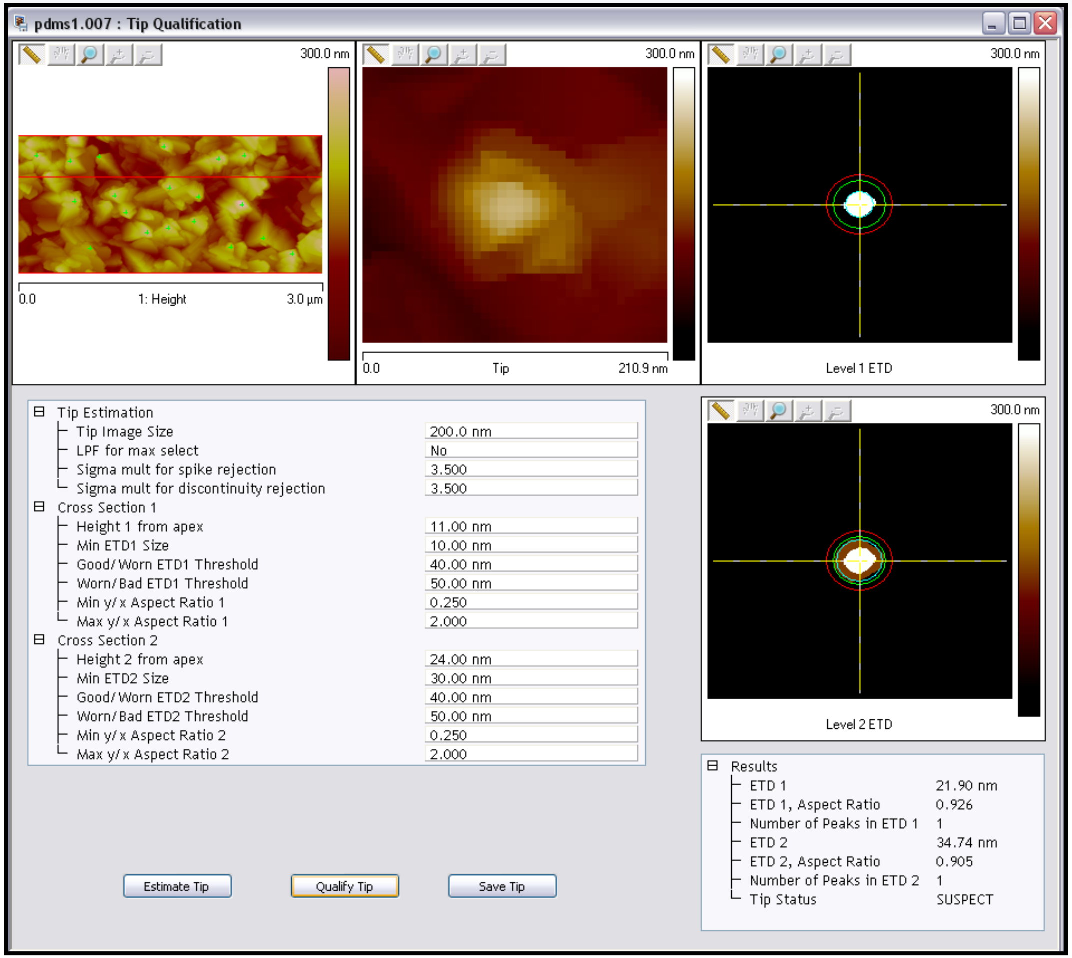
Estimate Tip

Determining Tip Radius with Tip Qual 4

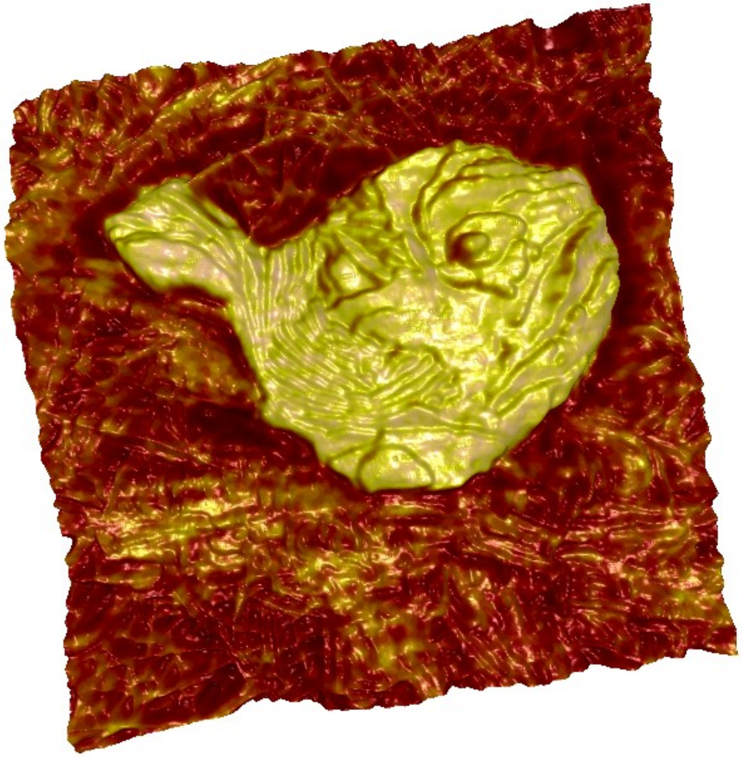
- The next step requires evaluating the tip radius at a specified indentation depth called “Height from Apex 1”
- The indentation depth can come from
  - An estimate from the force monitor (soft samples like PDMS)
  - Captured HSDC data converted to Force vs. Separation plots (more accurate)
  - The deformation data channel (harder sample / probe combinations like PS Film)

Determining Tip Radius with Tip Qual 5

- Enter the average indentation depth into Height 1 From Apex
  - Assume indentation depth of 10 nm for this exercise
- Click Qualify Tip
- Read estimated tip diameter from ETD 1
  - ETD 1 is ~ 22 nm
  - Tip R is ~ 11 nm
- Enter Tip Radius into “Cantilever Parameters / Tip Radius” in real time software .



Exercise 2
Absolute method on PDMS 1



PEO/SPP – Adhesion overlay on height

Exercise 2 Overview

- Use the absolute method to determine the DMT Modulus of PDMS 1 from the sample kit.
- The first steps of the calibration procedure were done in Exercise 1
  - Deflection sensitivity
  - Thermal tune for k
  - Image of Tip Check captured
- Assuming the same cantilever is still on the system and the workspace has not been changed there is no need to repeat those steps.
- We will use the Force Monitor in this exercise to estimate indentation depth (rather than the deformation data channel)

What is acceptable value for PDMS 1?

- Nominal value is 2.5 MPa
- 2.5-5 MPa is “normal” and acceptable for this class
  - Usually measured results are too high rather than too low

Class Exercise 2 – Absolute method on PDMS 1

- Load ScanAsyst Air probe (try to use from previous exercise)
- Enter estimated sample Poisson's ratio (use guideline chart – sample will be 2.5 MPa)
- Calibrate Deflection Sensitivity on glass or sapphire (may use from previous exercise)
- Thermal Tune for k (may use from previous exercise)
- Image Tip Check. Open NanoScope Analysis. Perform 1st Order XY Plane Fit. Open Tip Qual (do not evaluate tip R yet but leave open for later use). (may use from previous exercise)
- Engage with auto setpoint, gain, scan rate, and z limit on.
- Make sure synch is ok. Auto config if necessary. (more details on following slides)
- Turn auto setpoint and z limit off
- Set proper Noise Threshold (.2 nm for PDMS1 or PDMS 2)
- Set scan size = 2um, aspect ratio = 4, samples / line = 256
- Note the average value of scope trace on deformation data channel (record value for discussion later) – this step is for illustration only
- Estimate indentation from force monitor (record value on note pad)
- Return to Tip Qual (from exercise 1) and evaluate Tip Radius at estimated indentation depth. Enter into Tip Radius parameter (record tip radius on note pad)

Class exercise 2 – cont'd

- Capture image
- While Image Capture is on press Capture Line on force monitor and upload the data for later use.
- Determine DMT Modulus from Roughness / Image Raw Mean with NanoScope Analysis (record the value on note pad)
- Be prepared to report / discuss
  - The measured DMT modulus from PDMS 1
  - The estimated indentation from the force monitor
  - The tip radius at that indentation depth
  - The average indentation depth as estimated from the captured Deformation channel
- Leave the probe on the system and the work space open for next exercise

Exercise 2 Discussion

- Discuss results
  - The measured DMT modulus from PDMS 1
  - The estimated indentation from the force monitor
  - The tip radius at that indentation depth
  - The average indentation depth as estimated from the captured Deformation channel
  - If the deformation channel was used to estimate indentation depth and that value was plugged into “Height from Apex 1” to evaluate tip radius
    - Would Tip radius have been higher or lower?
    - Would that have made the DMT Modulus value higher or lower?

PeakForce QNM

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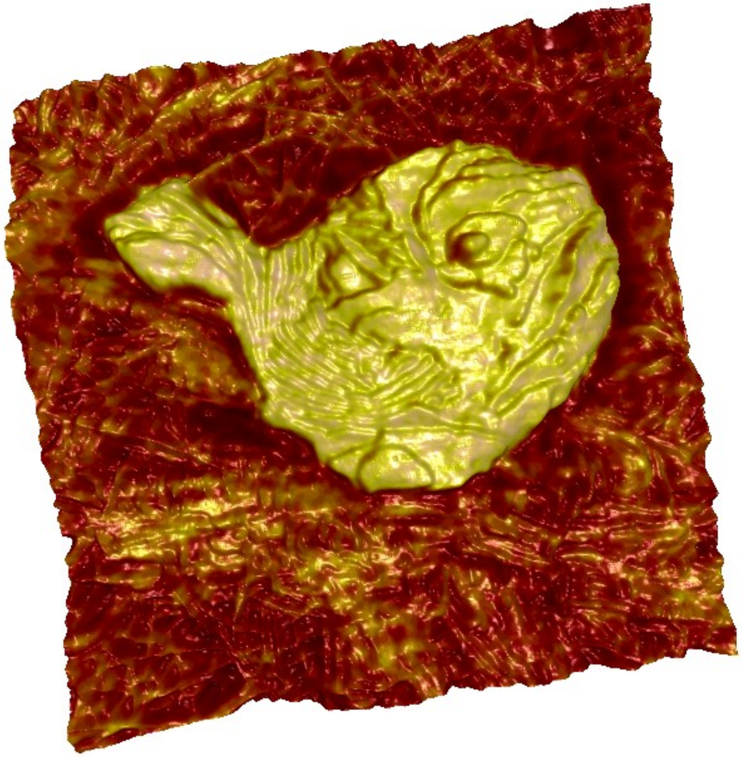
Exercise 3
Absolute method on PDMS 2



PEO/SPP – Adhesion overlay on height

Exercise 3 Overview

- Hopefully there is a calibrated probe on the afm still from exercise 2
- In this exercise we will measure the DMT Modulus of PDMS 2 from the sample kit (nominal value 3.5 MPa)
- Main “take-aways” from this exercise
    - More practice
    - Paying attention to whether Tip Radius needs to be re-evaluated
        - Indentation depth may have changed because this sample is slightly stiffer
    - Hopefully show ~ 1 MPa difference in DMT Modulus from PDMS 1
        - If PDMS 1 was 3.5 MPa then PDMS 2 should be ~ 4.5 MPa

Exercise 3 instructions

- Measure the DMT Modulus of PDMS 2 using the absolute method
  - The probe calibration and tip check images already exist from the previous exercise.
  - Use the force monitor to estimate indentation depth (record the value)
  - Make sure to reevaluate the tip radius if your indentation depth changed from where it was on PDMS 1
  - Record the value of PDMS 2

Exercise 3 Discussion

- Estimated indentation depth
- Tip radius value (if different)
- DMT modulus of PDMS 2

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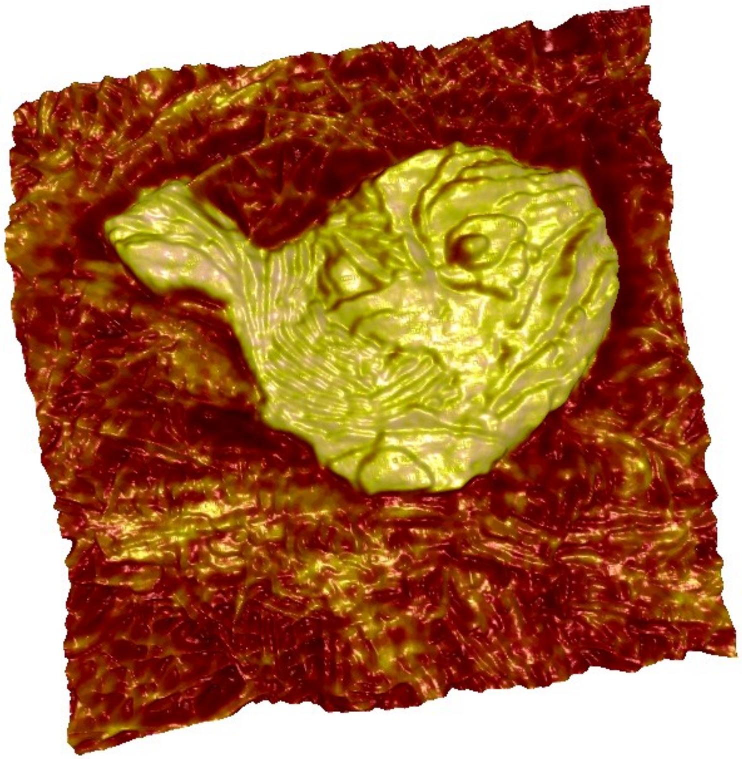
Exercise 4

Capturing raw data using Capture Line
Using PFQNM Offline
Calculate indentation



PEO/SPP – Adhesion overlay on height

Exercise 4 Summary

- Capturing raw data (force curves) while imaging is a powerful capability.
  - Customers may want to analyze with outside program using model other than DMT
- Main “take-aways” for this exercise
  - Learn how to capture raw data using HSDC (Capture Line)
  - How to work with the data (PFQNM Offline)
  - How to use raw data to export force curves and view force vs. separation

Exercise 4 instructions

- In the Exercise 2 you estimated the indentation depth from the force monitor and then used that value to evaluate tip radius. You also captured a line of high speed data.
- Use that line of high speed data to evaluate the real indentation depth
  - See “PFQNM – How to – Calculate Indentation with PFQNM Offline” starting on third slide
- Report / discuss
  - Measured indentation from HSDC offline
  - How does that compare to the indentation estimated from the force monitor?
  - If tip radius had been evaluated at the indentation depth from HSDC offline how would the modulus have changed?

Exercise 4 – Use HSDC to determine true indentation depth

- In the Exercise 2 you estimated the indentation depth from the force monitor and then used that value to evaluate tip radius. You also captured a line of high speed data.
- Use that line of high speed data to evaluate the real indentation depth
  - Use “PFQNM – How to – Calculate Indentation with PFQNM Offline” starting on slide 3

Exercise 4 discussion

- Report / discuss
  - Measured indentation from HSDC offline
  - How does that compare to the indentation estimated from the force monitor?
  - If tip radius had been evaluated at the indentation depth from HSDC offline how would the modulus have changed?

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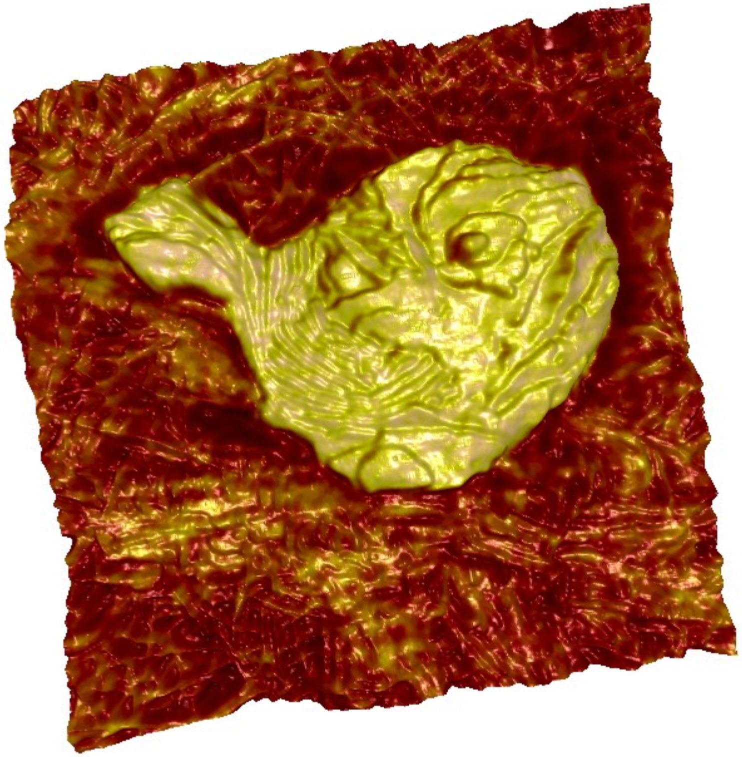
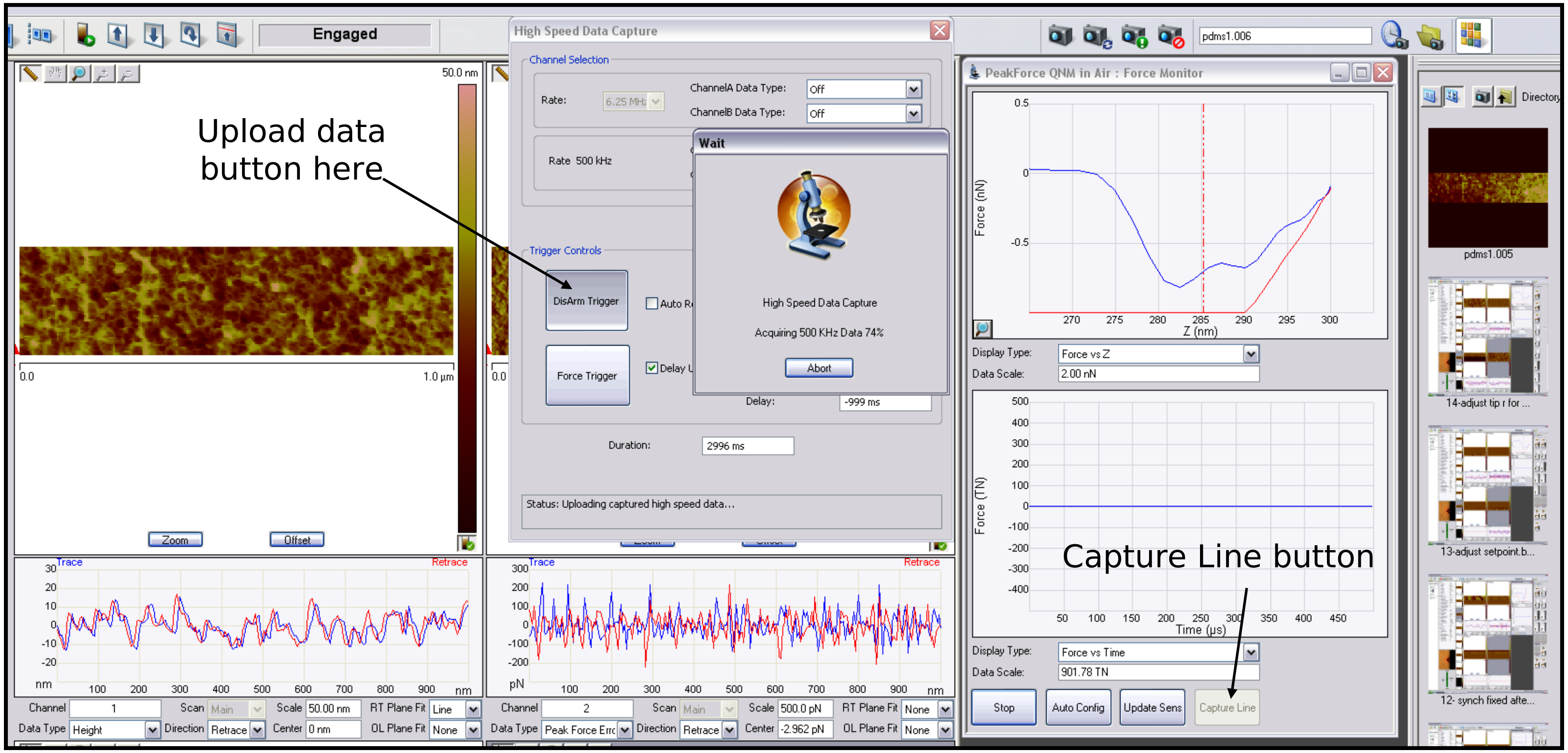
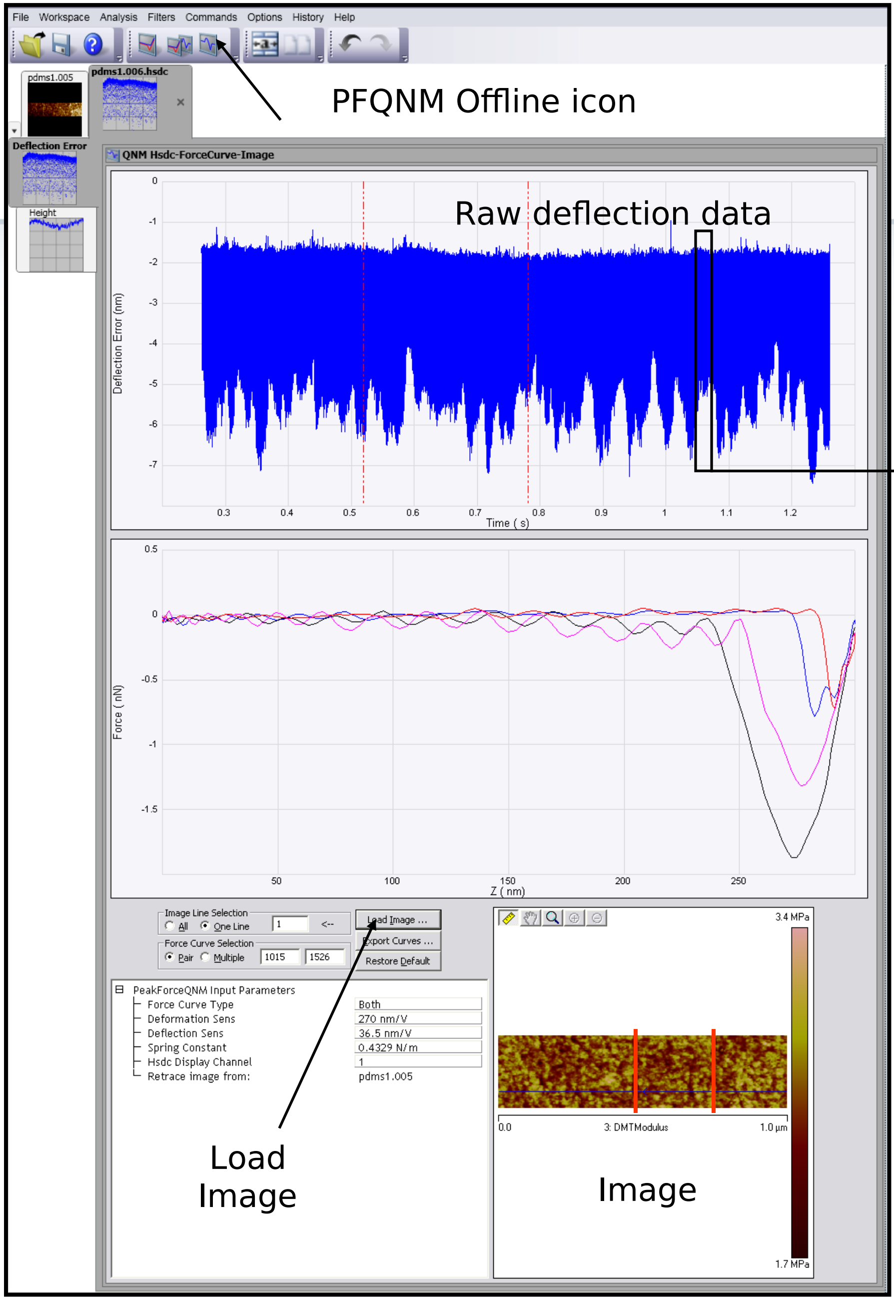
PFQNM – How To –
HSDC & PFQNM Offline
(how to determine true indentation depth)

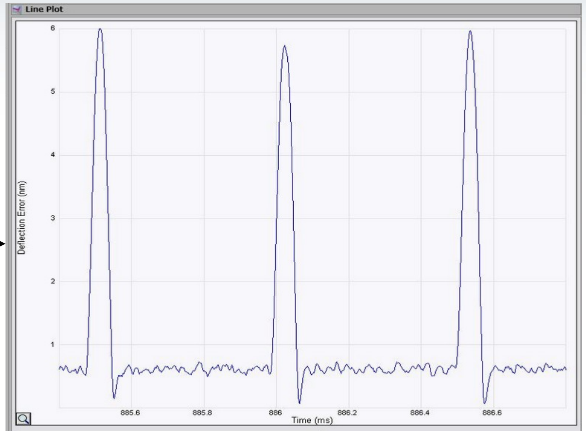


PEO/SPP – Adhesion overlay on height

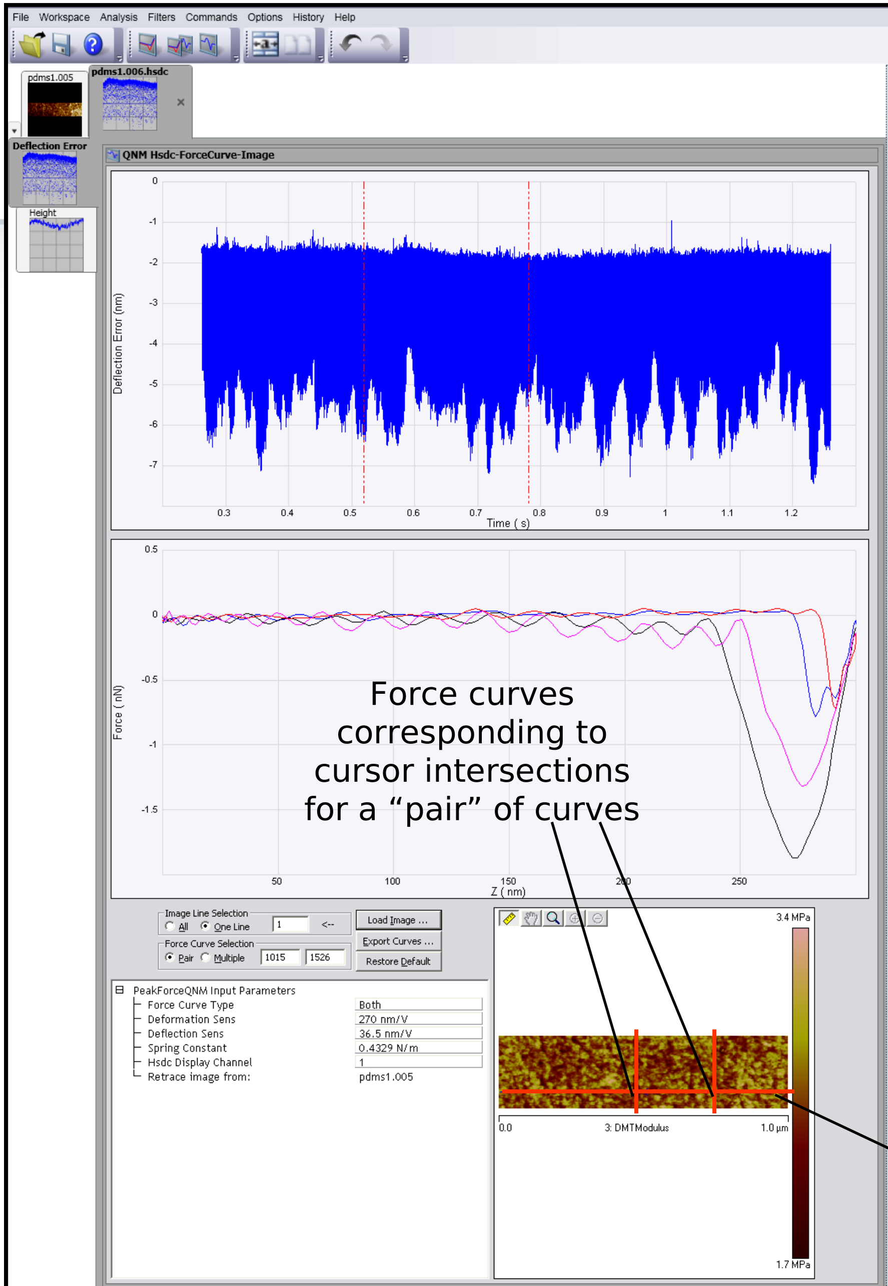
- Capture an image.
- While the image capture is in progress press the Capture Line button on the Force Monitor to capture lines of raw data.
- After the image has finished capturing press the Upload Data button to save the raw HSDC data to a file.







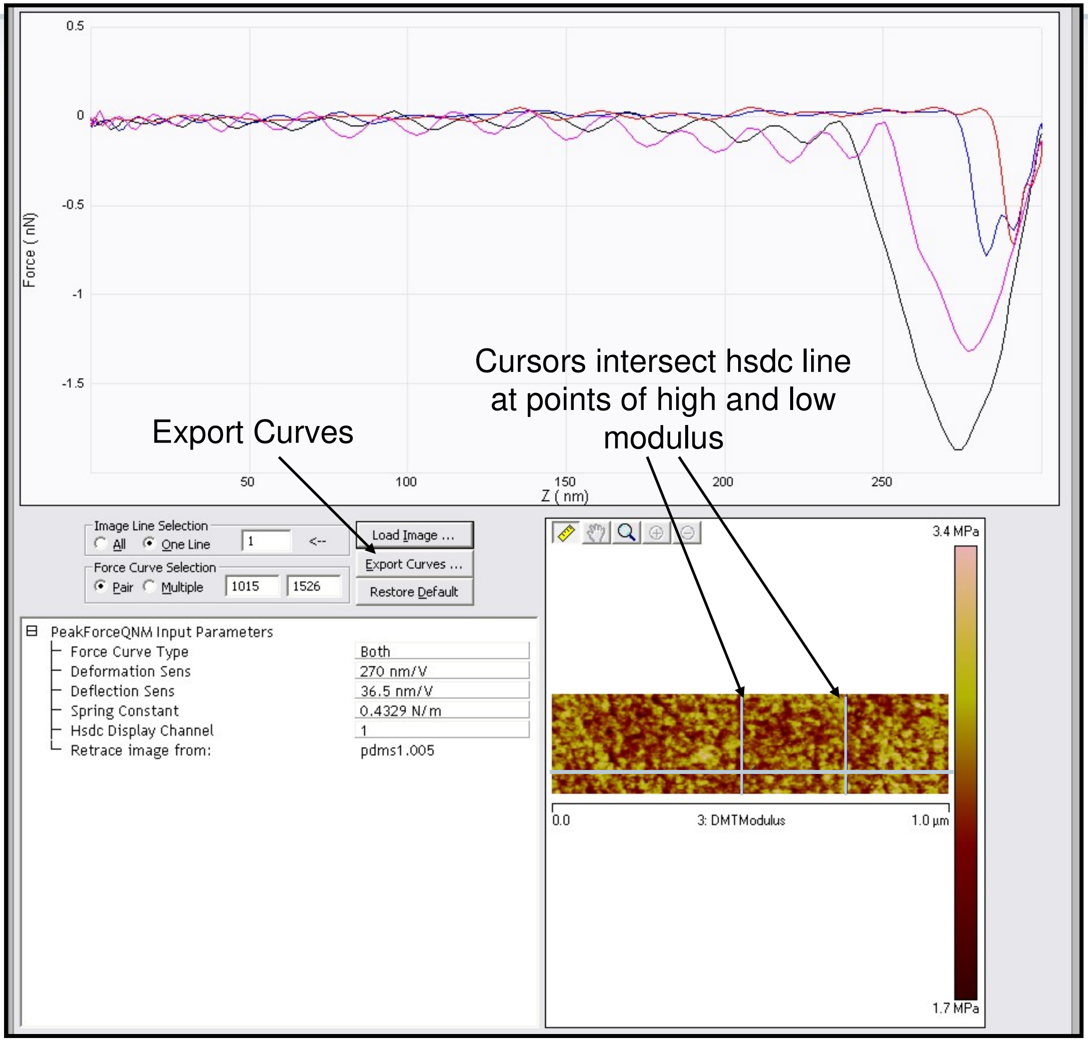
- Open the raw data file (*.hsdc) using NanoScope Analysis.
- Click the PFQNM Offline icon.
- Click the Load Image button and navigate to the captured image file that corresponds to the captured raw data.
- Right-click on the image display and choose the DMT Modulus channel for display.



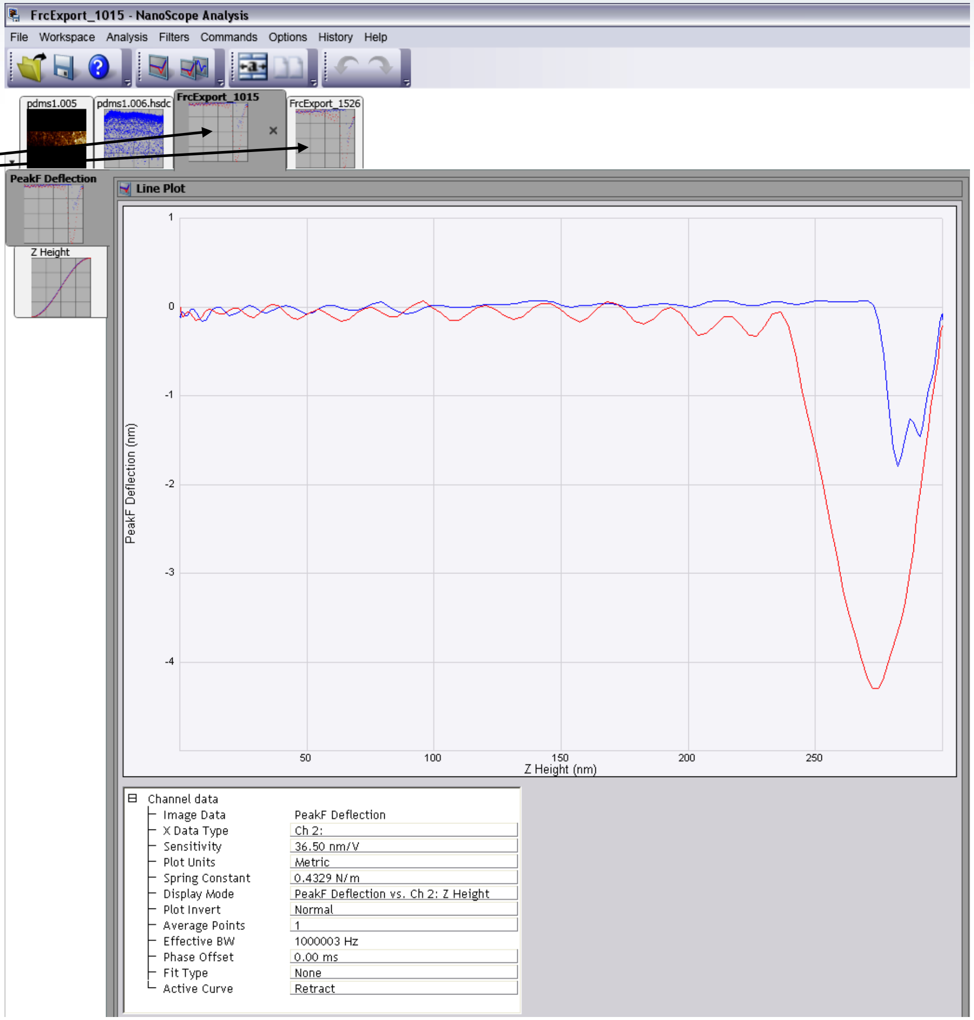
Active scan line

- Often more than one scan line is captured
- Active scan line shown by horizontal cursor in image area
- In "Image Line Selection" area choose display "One Line"
- Control which of the captured lines to display with the number parameter to the left of the Load Image button
- Use the "Force Curve Selection" area to display either a pair of curves (located at the intersections of the vertical and horizontal cursors in the image area) or all of the curves between the vertical cursors.

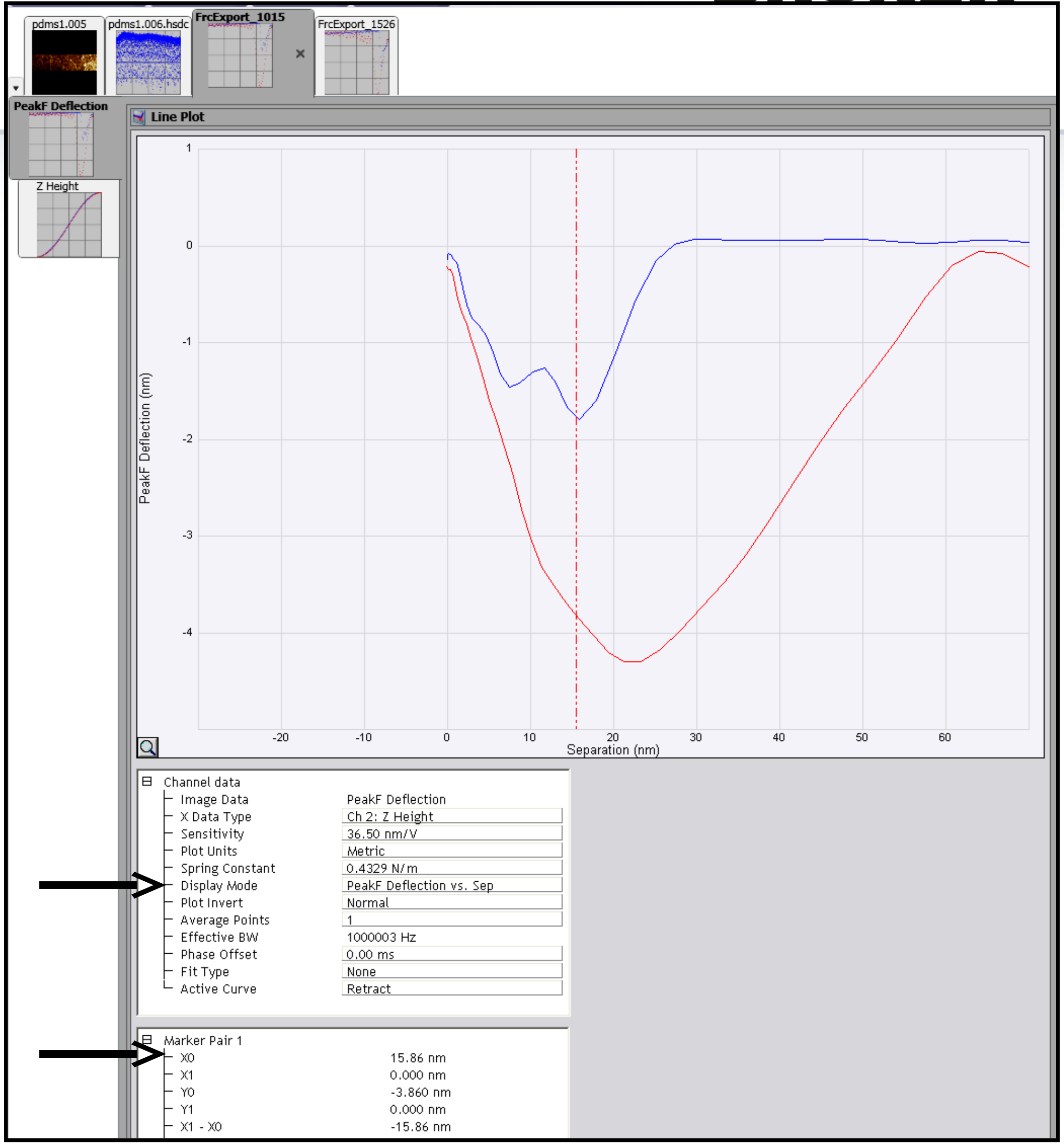
- Place cursors at points of interest
- Click Export Curves and click yes when the dialog appears.
  - A pair of force curves has now been exported from the raw data.



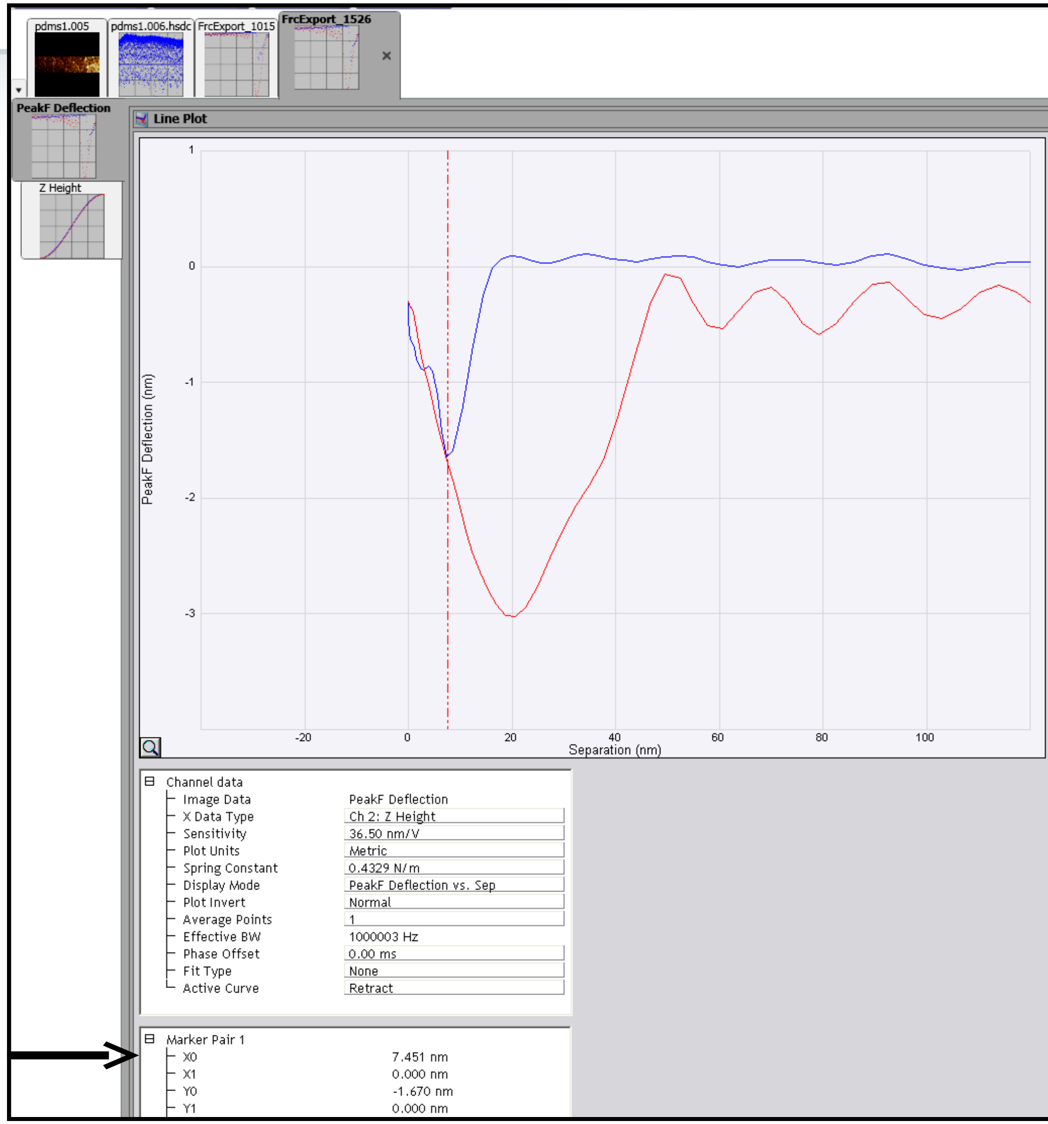
- Open both of the exported force curves.



- Activate the first force curve.
- Set the Display Mode to Peak Deflection vs. Sep.
- Drag in a cursor so that it intersects the minimum force on the load curve.
- Note the X0 value. This is the penetration depth for this force curve.
  - ~16 nm



- Determine the indentation depth on the second force curve and note the value.
    - ~7 nm



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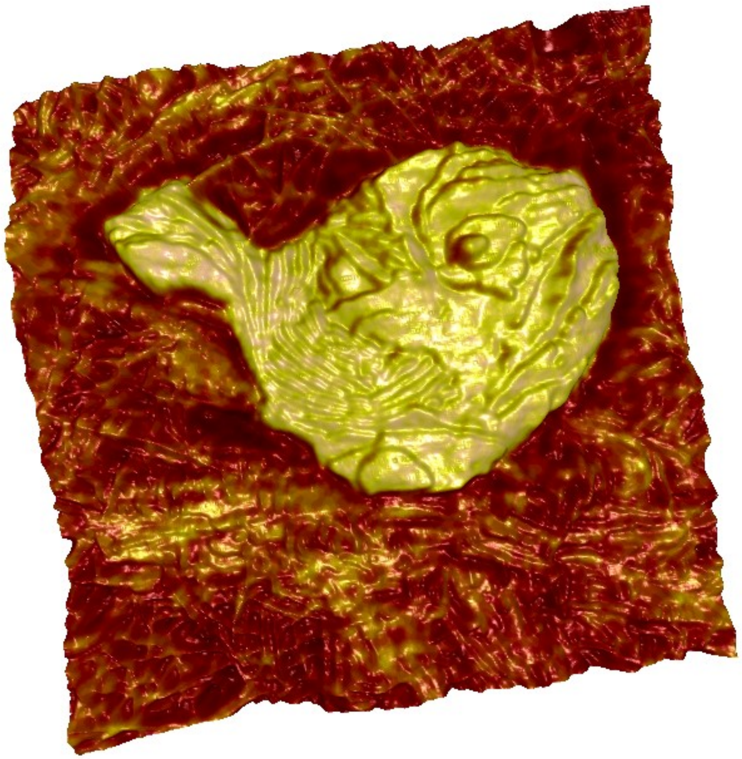
Class Exercise 5

Relative method on PS Film and PS-LDPE



PEO/SPP – Adhesion overlay on height

Exercise 5 Summary

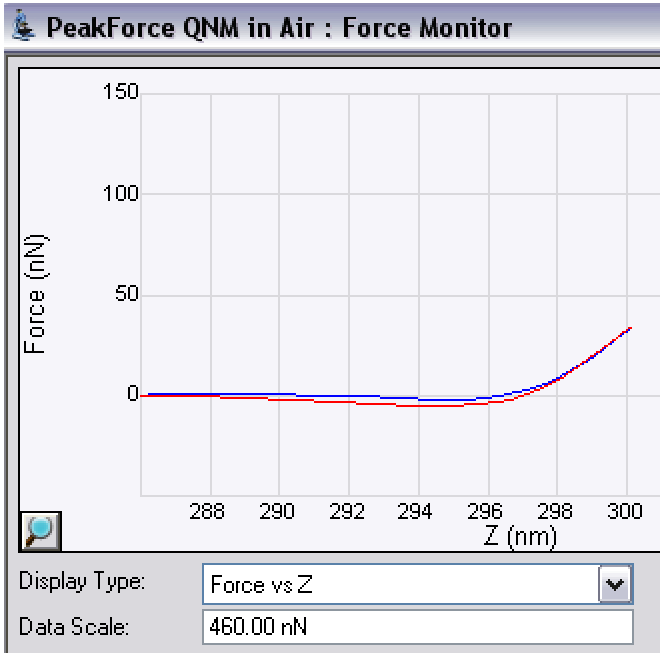
- In this exercise we will use the relative method to determine the DMT modulus of the PS (polystyrene) area of the PS-LDPE sample
- Main "take-aways" from this exercise
  - Being careful with imaging forces with stiffer probes
  - Smaller indentation depths
  - The deformation data channel works better with small attractive region on approach plot
  - Adjusting Limits
  - Setting the ratio of $k/\sqrt{R}$ (instead of measuring each)

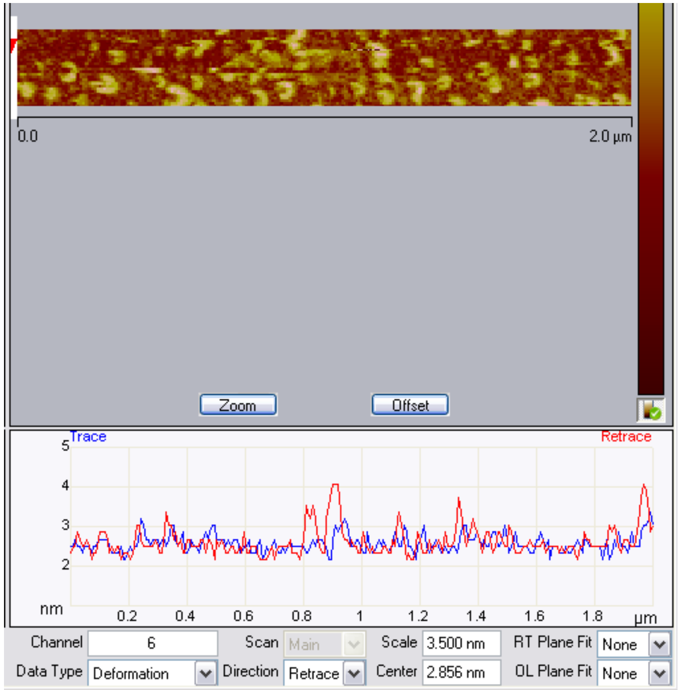
Exercise 5 – relative method on PSFilM and PS-LDPE samples

- Load Tap300A (RTESPA) probe
- Calibrate Deflection Sensitivity on glass sapphire
  - Don’t forget to set Peak Force Engage Setpoint to .05V
  - When ramping set relative trigger to .15V
  - Record value for later discussion
- Load PS Film sample (2.7 GPa)
- Enter 25 N/m for k
- Enter estimated sample Poisson’s ratio (.3 based on guideline chart).
- Set Engage Setpoint to .05 V
- Set ScanAuto to Individual and
  - Auto setpoint, auto scan rate, auto z limit off
- For setpoint type in .01V and then enter (will convert to force)
- Set proper Noise Threshold (.2 nm for samples in sample kit)
- Engage
- Set scan size = 2 um, aspect ratio = 4, samples / line = 256

Class Exercise 5 – cont'd

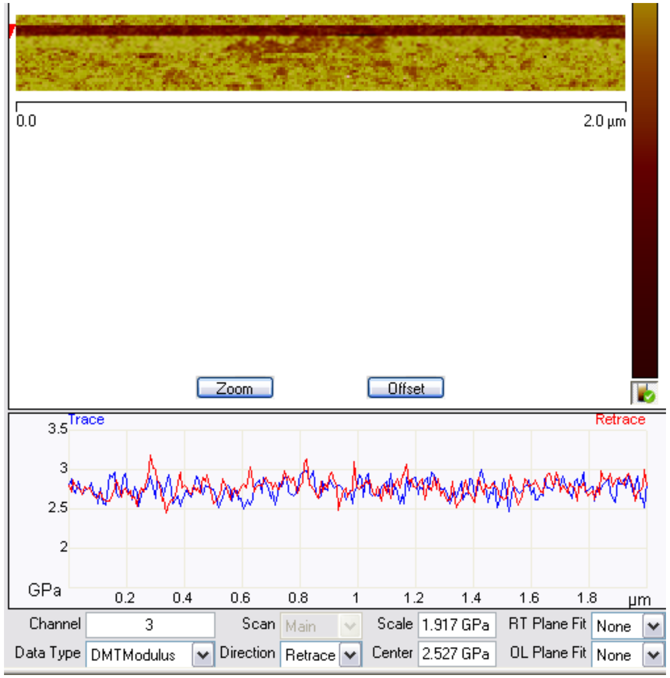
- Adjust setpoint until there is 2-5 nm indentation based on either force monitor or deformation data channel
  - If sample is being damaged or modified try lowering the setpoint but don't make the indentation any less than 2 nm
- Set Peak Force Limit to ~ 20 x the peak force setpoint
- Record estimated indentation depth from scope trace data on the deformation channel





Exercise 5 cont'd

- Adjust DMT Modulus Limit until you can read the data on the scope trace easily (start with ~ 200 GPa)
- Adjust Tip Radius and/or Cantilever Spring Constant until DMT Modulus data matches known value of reference (2.7 GPa).
- Make final adjustment to DMT Modulus Limit (if it needs to be lowered again)
  - 50 GPa should work well



Exercise 5 cont'd

- Capture an image
- Load PS – LDPE
- Set scan size = 3 um, aspect ratio = 4, samples / line = 256
- Engage
- Zoom into an area of PS and auto-config (as many times as necessary) to get good synch
  - On a 2 component sample auto config should be done in stiffer area
- Increase the scan size to 3 um again
  - After increasing the scan size is there sample modification / damage in the previous scan area?
- Adjust setpoint (if necessary) until indentation depth (scope trace on Deformation channel) is close to where it was when the reference sample was measured.
- Capture an image
- Evaluate DMT Modulus (of the PS area) using Roughness/Image Raw Mean
  - Record the value for discussion
- Evaluate the average deformation in an area of LDPE
  - Record the value for discussion

Exercise 5 – discussion

- Report / discuss
  - Deflection sensitivity
  - Indentation depth
  - DMT modulus of PS region of PS – LDPE
  - Why the deformation is “ok” to use to estimate indentation with this sample
  - Deformation in the LDPE area
  - Why can’t the modulus data be trusted in the LDPE area?