

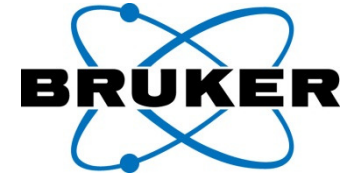
# PeakForce-QNM Advanced Applications Training 2014



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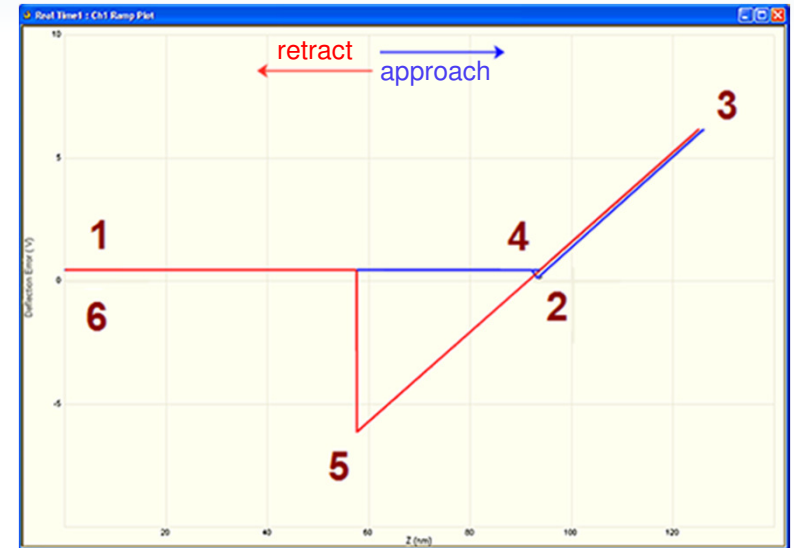
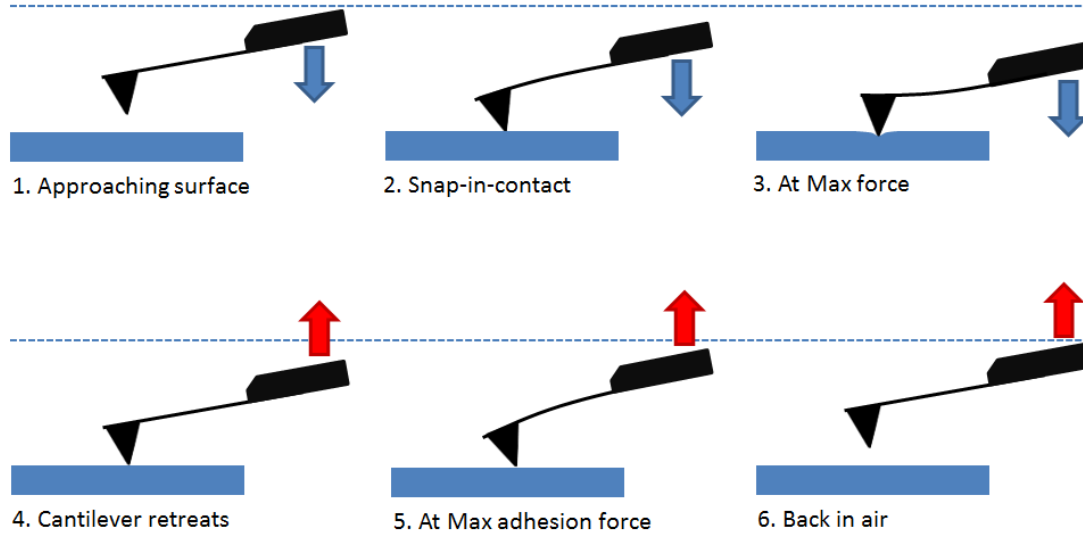
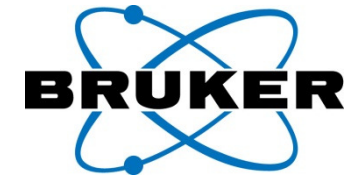


# What is PeakForce QNM?

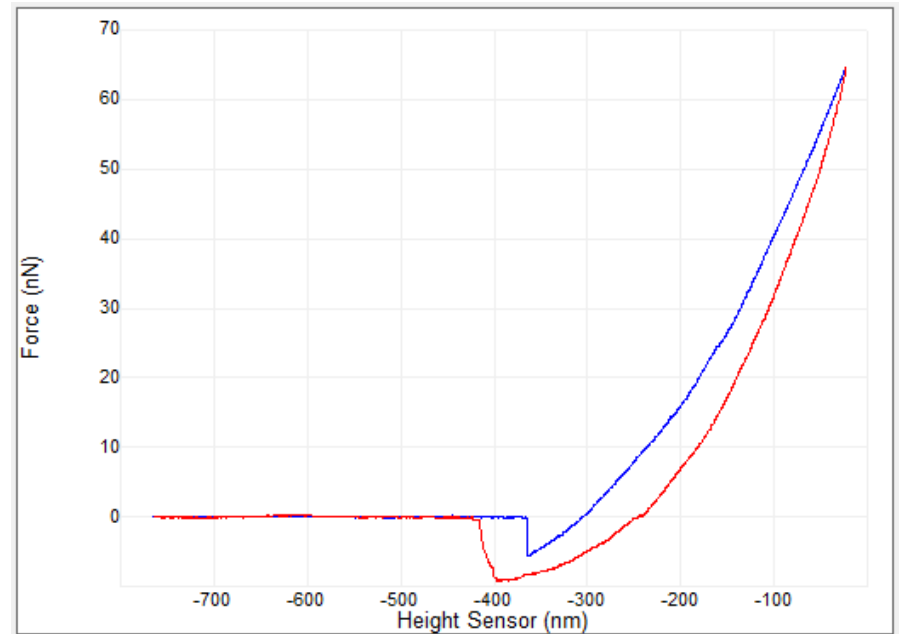
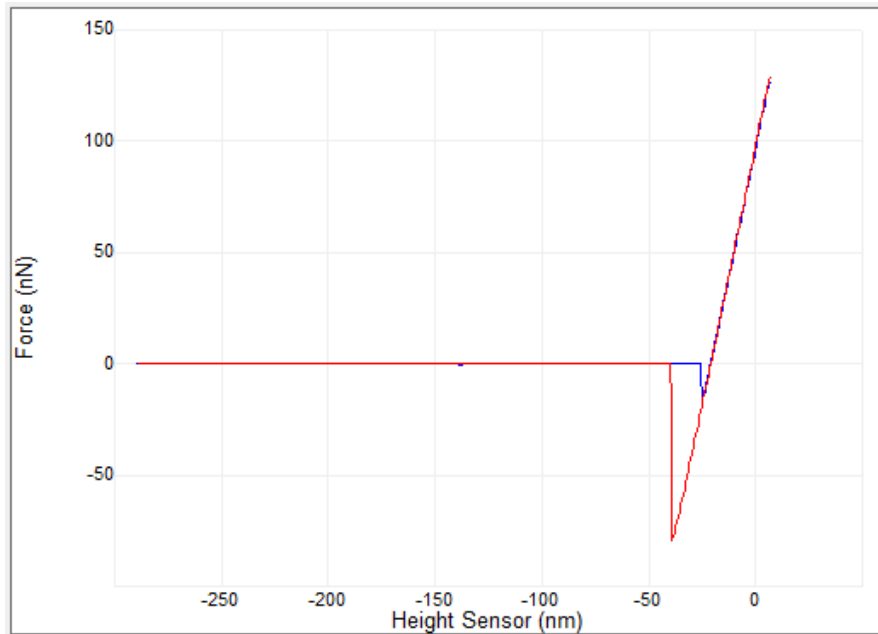
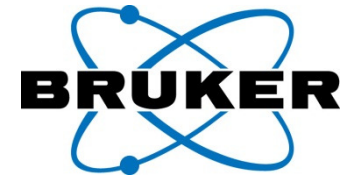


- PeakForce QNM is an imaging mode that produces height images and **Q**uantitative **N**ano-**M**echanical sample property images at the same time.
- Two parts to PeakForce QNM
  - The PeakForce Tapping part
    - PeakForce Tapping is the feedback mode used to track and image the sample surface.
  -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

# Force Distance Curve

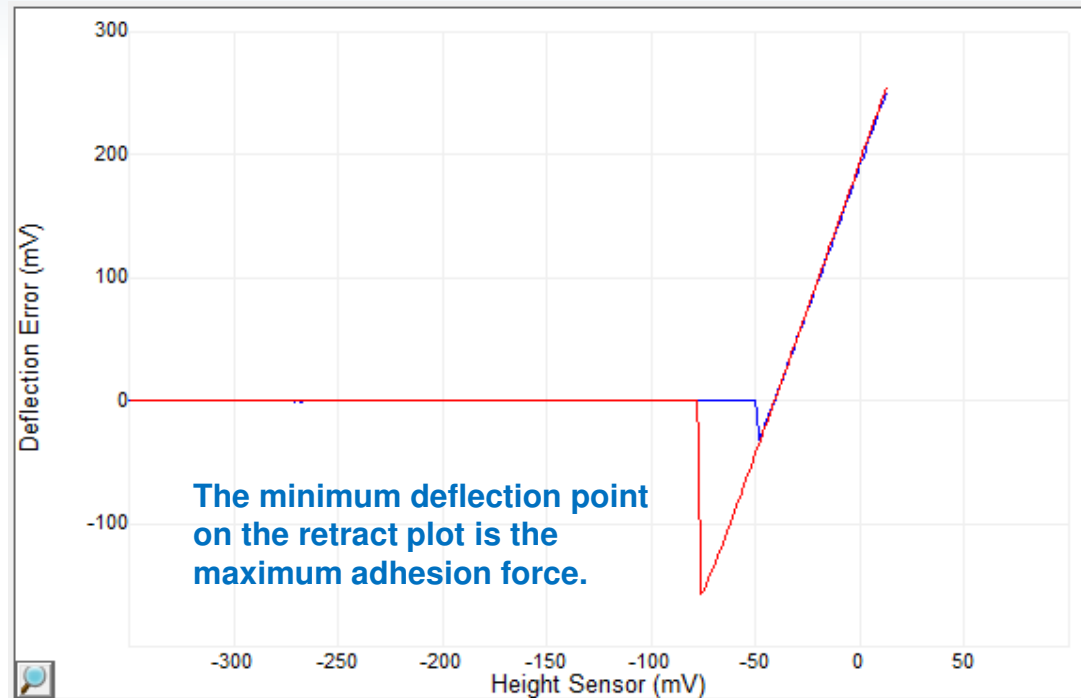


# Force Curve Interpretation



**Question:** What is the different between above two force curves?

# Sample Adhesion Force



- Without calibration, ramp curve give Deflection vs. Z
- Deflection raw unit is in **V**
- How to convert Deflection from **V** to **nm**?
- How to convert Deflection in nm to Force in **nN**?

# Young's Modulus



$$E = \frac{\textit{tensile stress}}{\textit{extensional strain}} = \frac{\sigma}{\varepsilon} = \frac{F/A_0}{\Delta L/L_0} = \frac{FL_0}{A_0\Delta L}$$

Where

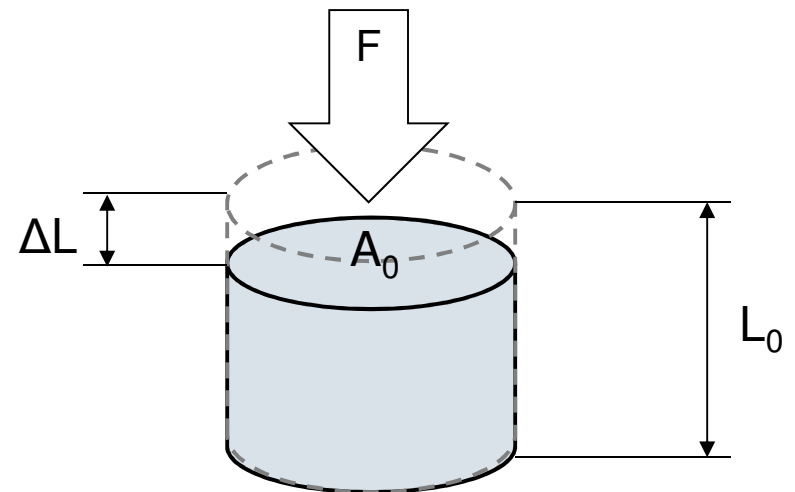
E is the Young's modulus (Modulus of elasticity)

F is the force exerted on an object under tension

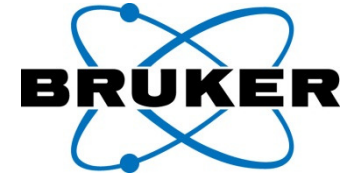
$A_0$  is the area where force is applied

$\Delta L$  is the length change of the object

$L_0$  is the original length of the object



# Sample Modulus: Hertzian Model



$$F = \frac{4}{3} \frac{E}{(1 - \nu^2)} \sqrt{R} \delta^{3/2}$$

or  $(F)^{2/3} = \left( \frac{4}{3} \frac{E}{(1 - \nu^2)} \sqrt{R} \right)^{2/3} \delta$

Linearized Equation

Where

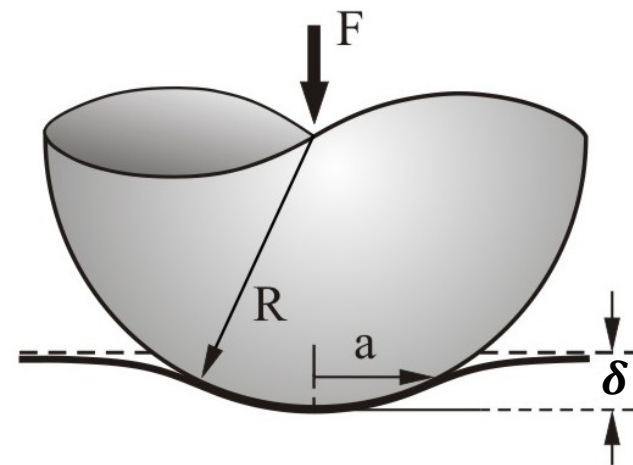
F is the force (from force curve)

E is Young's modulus (fit parameter)

$\nu$  is Poisson's ratio (typically 0.2-0.5)

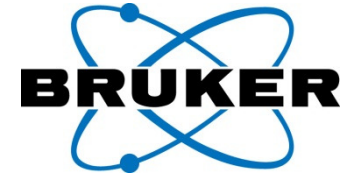
R is the radius of the indenter (tip)

$\delta$  is indentation depth



- Model is valid for  $a \ll R$
- DMT model: Hertzian model with adhesion force
- **Question:** if we use a 5 $\mu$ m radius sphere probe to indent into sample surface 50nm, what is the tip radius **R** for above equation?

# Sample Modulus: Sneddon Model



$$F = \frac{2}{\pi} \frac{E}{(1 - \nu^2)} \tan(\alpha) \delta^2$$

or  $(F)^{1/2} = \left( \frac{2}{\pi} \frac{E}{(1 - \nu^2)} \tan(\alpha) \right)^{1/2} \delta$

Linearized Equation

Where

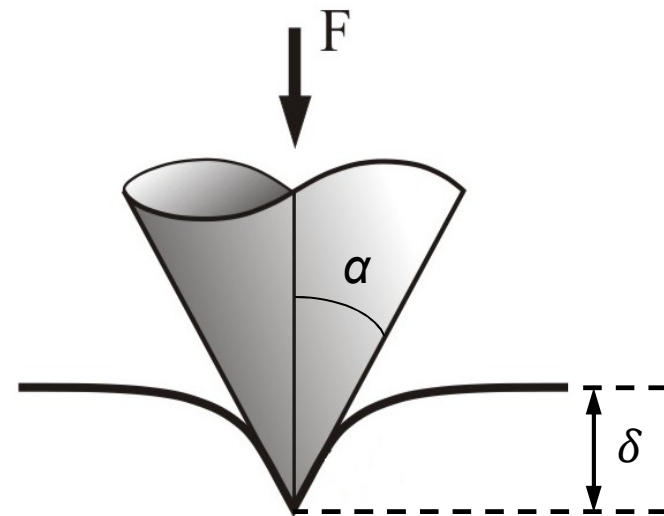
F is the force (from force curve)

E is Young's modulus (fit parameter)

$\nu$  is Poisson's ratio (typically 0.2-0.5)

$\alpha$  is half angle of the indenter

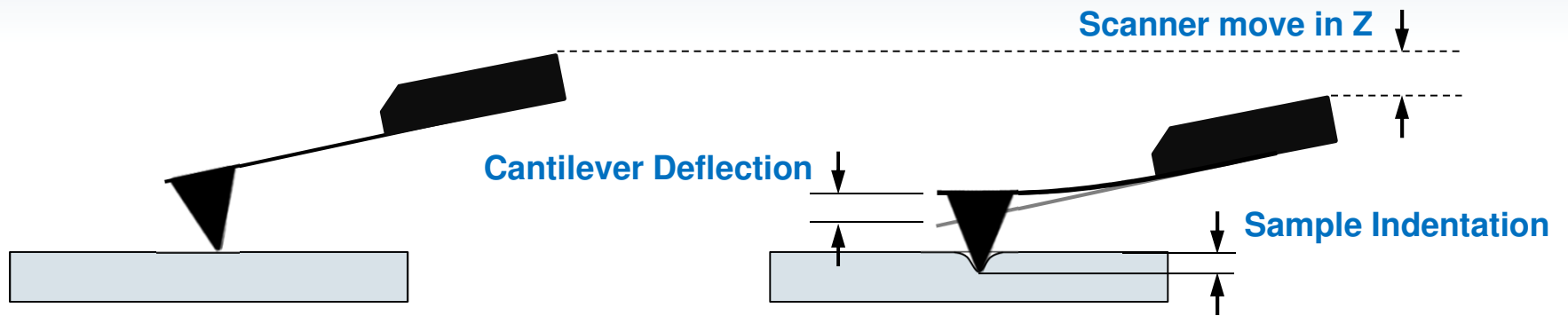
$\delta$  is indentation depth



- Used for large sample indentation
- Uses approach curve, includes plastic deformation
- **Question:** is  $\delta$  same as the Z scanner movement after tip contacts sample surface?

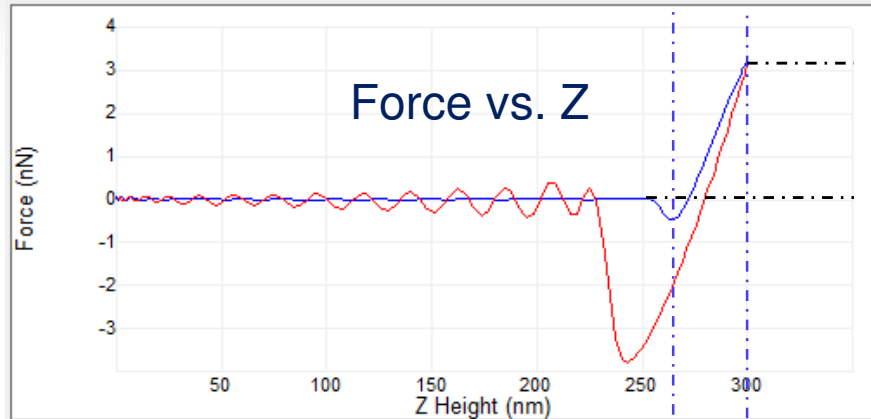
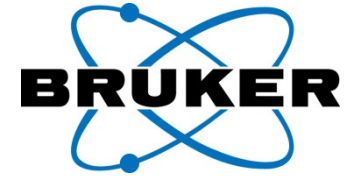


# Indentation Depth



- Scanner movement in Z(↓) direction causes two effects after tip contacts surface:
  - Cantilever deflection(↑)
  - Sample indentation (↓)
- $Z = \text{Sample Indentation} + \text{Cantilever Deflection}$
- Separation: distance between tip and max indentation point
- Indentation depth: distance tip moves after contact position (**indentation depth should be always positive**)

# Force vs. Separation Curve



Channel data

- Image Data
- X Data Type
- Sensitivity
- Plot Units
- Spring Constant
- Display Mode
- Plot Invert
- Phase Offset
- Fit Type
- Active Curve

PeakF Deflection

Ch 2: Z Height

23.00 nm/V

Force

0.3455 N/m

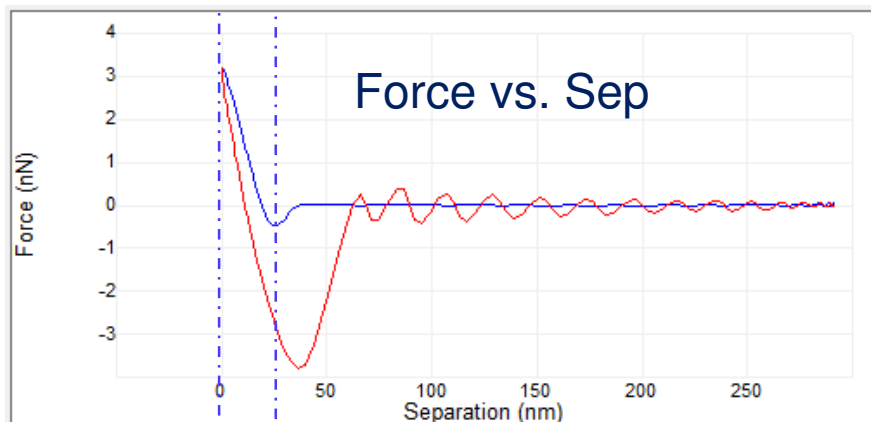
PeakF Deflection vs. Sep

PeakF Deflection vs. Ch 2:

PeakF Deflection vs. Sep

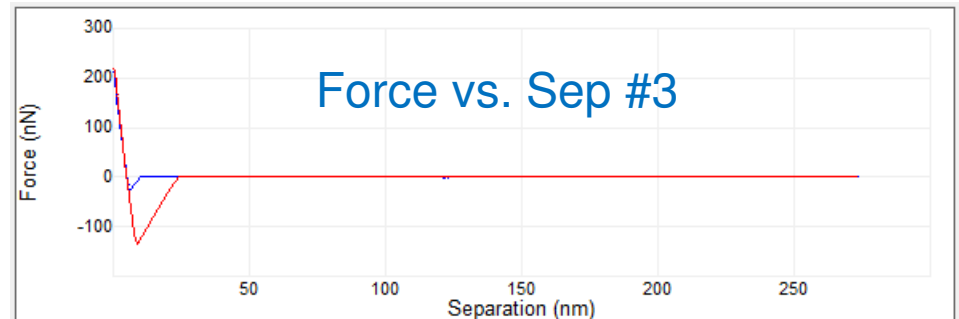
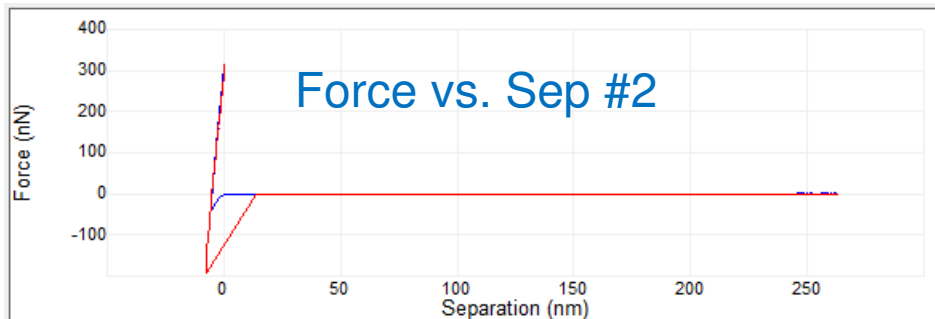
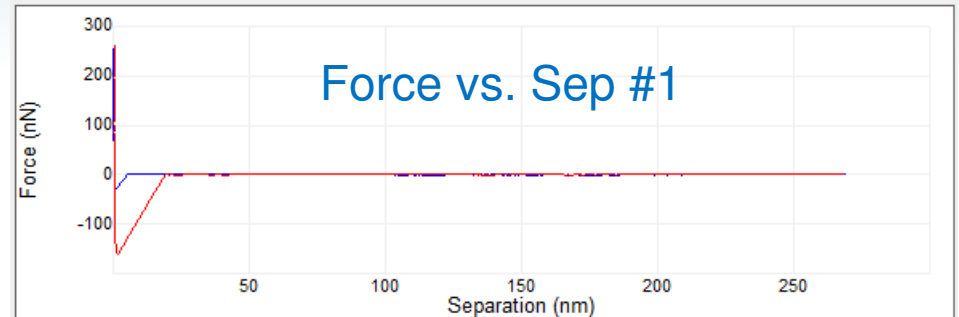
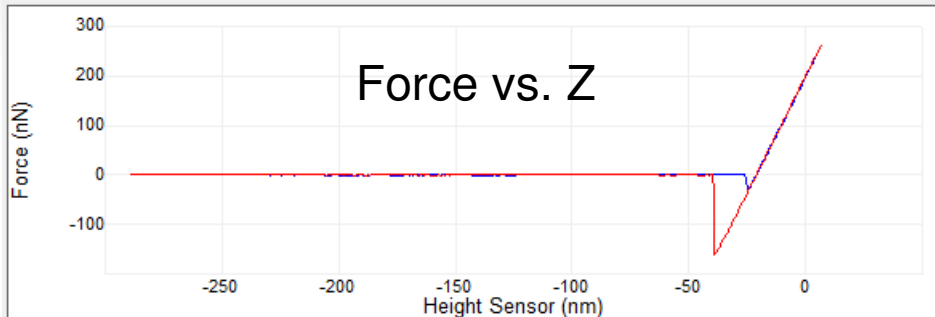
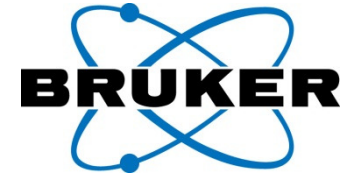
None

Retract



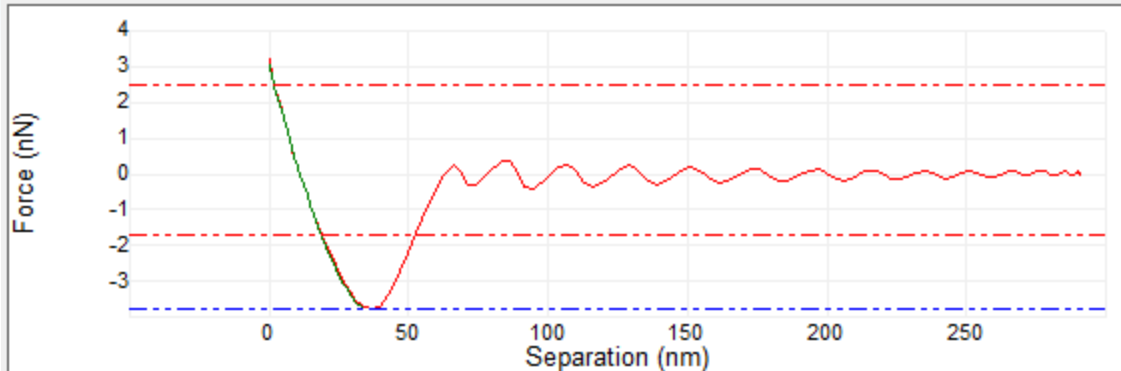
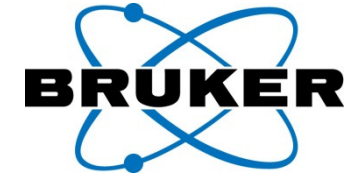
- Force vs. Z curve needs to be converted into Force vs. Sep curve in order to be fitted by the contact models
- This conversion can be easily done in Nanoscope Analysis software

# Example separation curves




- The above **Force vs. Z** curve is obtained by a soft cantilever ramp on a very hard sample, e.g. a ScanAsyst probe on Sapphire sample
- **Questions:** Which of the above **Force vs. Sep** curves is correct?

# How to get modulus from force curve: DMT Model



Inputs	
Active Curve	Retract
Fit Method	Linearized Model
Include Adhesion Force	Yes
Max Force Fit Boundary	90 %
Min Force Fit Boundary	30 %
Fit Model	Hertzian (Spherical)

Results	
R <sup>2</sup>	0.9998
Young's Modulus	3.64 MPa
Reduced Modulus	4.85 MPa

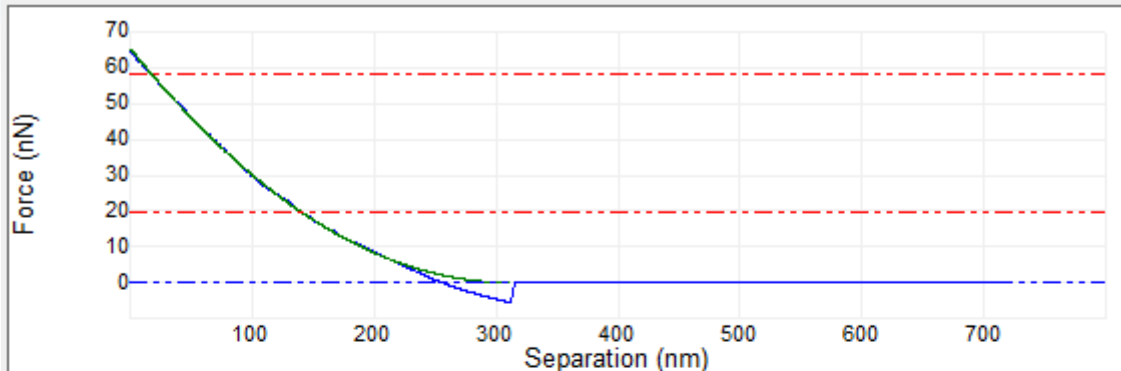
- Click  (Indentation) for force curve analysis
- DMT Model:
  - Use retract curve
  - Fit with linearized model
  - Include Adhesion Force:  
 $F = F_{tip} - F_{adh}$
  - Use Hertzian (Spherical)
  - Set force fit region
- Young's Modulus  $E$  and Reduced Modulus  $E^*$ :

$$E^* = \left[ \frac{1 - \nu_t^2}{E_t} + \frac{1 - \nu_s^2}{E_s} \right]^{-1}$$

If  $E_t \gg E_s$

$$E^* = \frac{E_s}{1 - \nu_s^2}$$

# How to get modulus from FD curve: **Sneddon Model**



- Sneddon Model:
  - Use Extend curve
  - Fit with linearized model
  - Don't include Adhesion Force
  - Use Sneddon (Conical)
  - Set force fit region

Inputs	
Active Curve	Extend
Fit Method	Linearized Model
Include Adhesion Force	No
Max Force Fit Boundary	90 %
Min Force Fit Boundary	30 %
Fit Model	Sneddon (Conical)

Results	
R <sup>2</sup>	0.9996
Young's Modulus	2.98 MPa
Reduced Modulus	3.27 MPa

# Cantilever Selection



## Method\_1

Sample Modulus (E)	Probe	Nominal Spring Constant (k)
1 MPa < E < 20 MPa	<a href="#">ScanAsyst-Air</a>	0.5 N/m
5 MPa < E < 500 MPa	Tap150A, P/N <a href="#">MPP-12120-10</a>	5 N/m
200 MPa < E < 2000 MPa	Tap300A (RTESPA), P/N <a href="#">MPP-11120-10</a>	40 N/m
1 GPa < E < 20 GPa	Tap525A, P/N <a href="#">MPP-13120-10</a>	200 N/m
10 GPa < E < 100 GPa	<a href="#">DNISP-HS</a>	350 N/m

## Method\_2

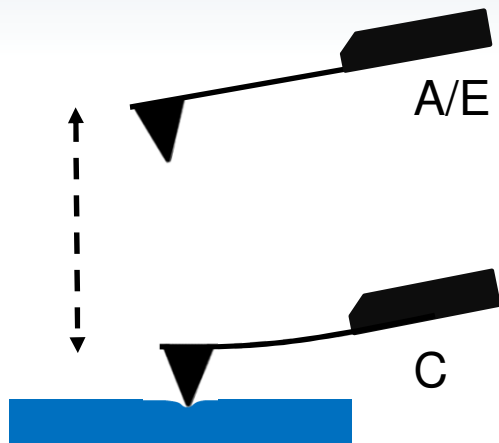
$$F = \frac{4}{3} \frac{E}{(1 - \nu^2)} \sqrt{R} \delta^{3/2}$$

- Cantilever spring constant needs to match with the sample modulus
- For accurate modulus measurement, the amount of sample Indentation(at least 2-5nm) should be comparable to cantilever deflection

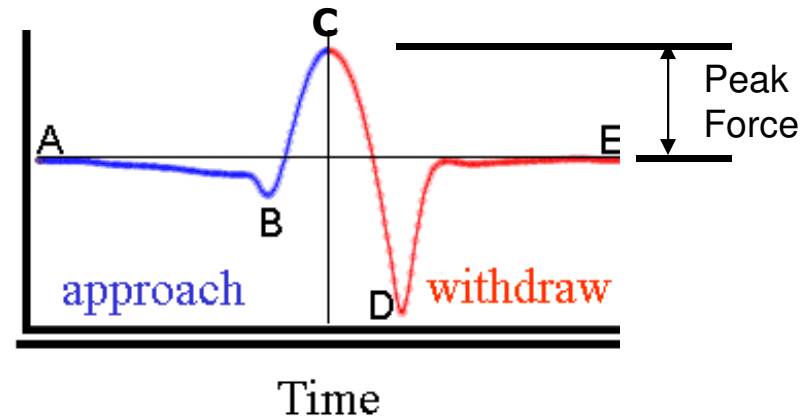
**0.3\*Deflection<Indentation<3\*Deflection**

- **Question\_1:** What spring constant to choose to measure 10MPa modulus with a 5um diameter sphere probe?
- **Question\_2:** How much is the indentation depth when use a diamond probe with 40nm tip radius and 400 N/m spring constant to indent on a Sapphire sample (E=450GPa), and measured cantilever deflection is 10nm?

# Peak Force Tapping

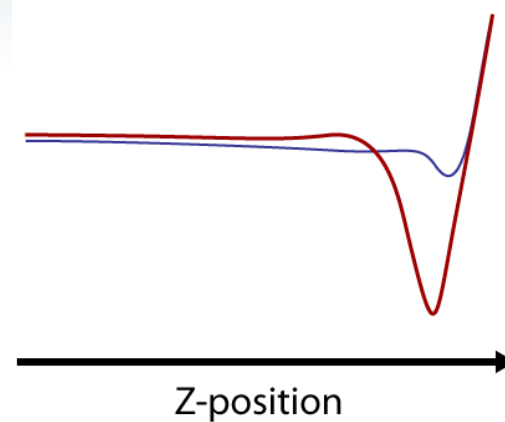
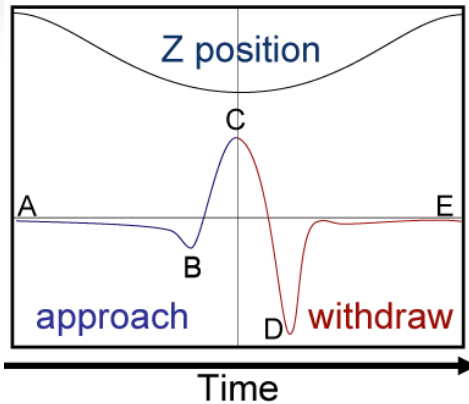
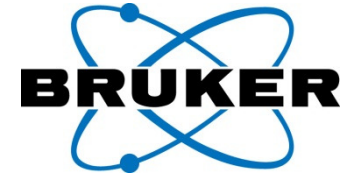


One cycle: photo detector vertical deflection (force) vs time.

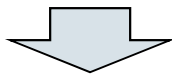


- A - Probe tip 300 nm above sample surface (150nm PFT amplitude). Z piezo pushes probe toward sample.
- B - Tip contacts sample ("snap-to-contact"). Z piezo pushes probe further until PeakForce imaging setpoint is reached.
- C - PeakForce setpoint reached. Probe starts withdrawing from sample.
- D - Probe tip breaks free off sample at maximum adhesion point.
- E - Probe back to starting point 300 nm above sample surface
- **Probe repeat the cycle A->E cycle at fixed frequency, typically 2KHz**

# Difference Between PF-Tapping and FD Curve



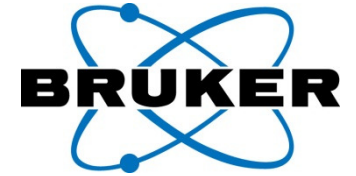
- Sine wave drive signal, 2KHz vs. triangular wave drive signal, 2Hz
- Sync distance + feedback vs. trigger threshold
- Real-time background subtraction vs. offline baseline correction
- Advanced real-time data analysis vs. offline analysis



- Faster speed and higher resolution image
- Accurate force control and gentler tapping
- Real time high resolution mechanical properties mapping
- **Question:** what is the different between PeakForce-Tapping and Tapping?

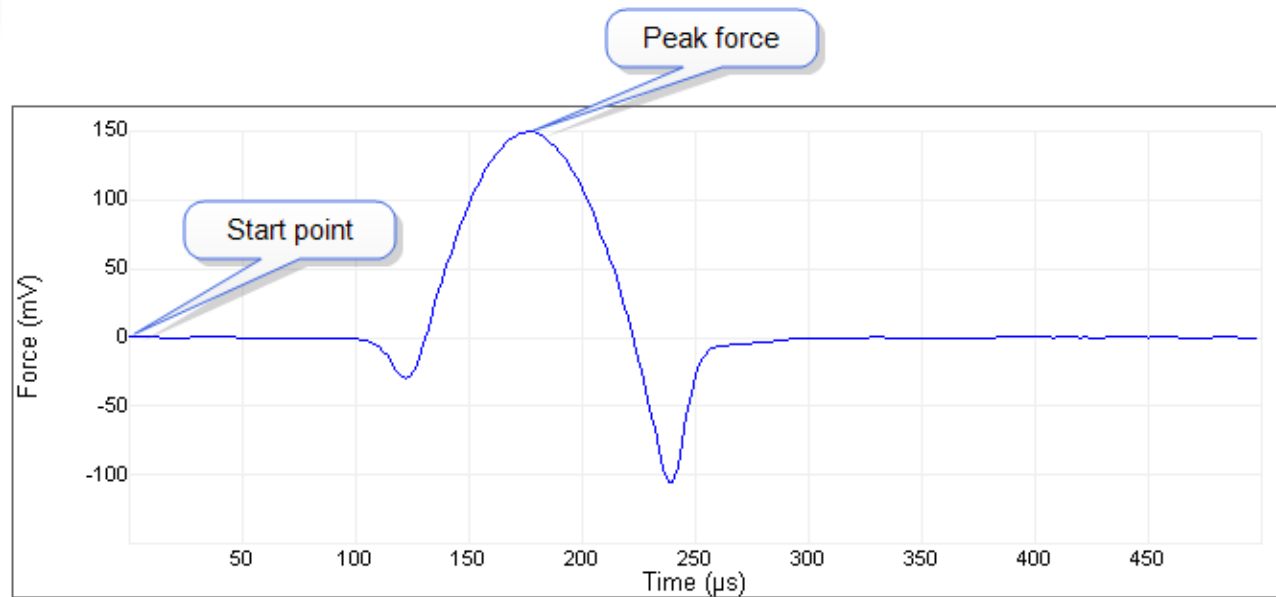
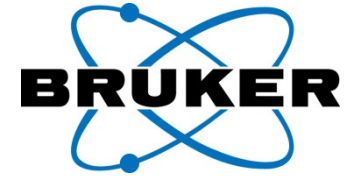


# Difference Between PF-Tapping and Regular Tapping Mode



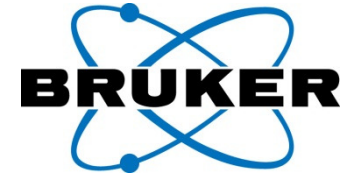
- Z scanner drive vs. Cantilever holder drive
- Off resonance oscillation not require cantilever tune vs. resonance oscillation requires cantilever tune
- Direct force control vs. no direct force control
- Separate modulus and adhesion vs. phase imaging

# What is Synch Distance

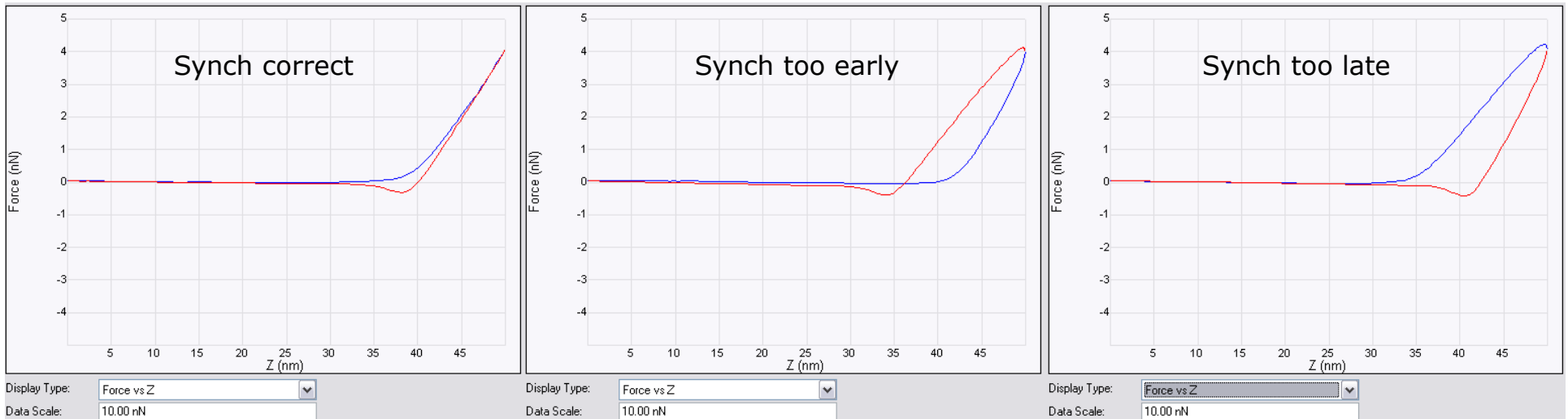


- Sync Distance is the distance (in 2 μsec time steps) between the start point of an extend-retract cycle and the point of peak force.
- On hard sample deflection reaches max when piezo extends to lowest position.
- Sync Distance QNM: used for modulus calculation, should be measured on hard sample, then apply to unknown sample.

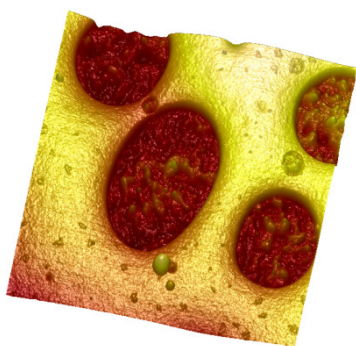
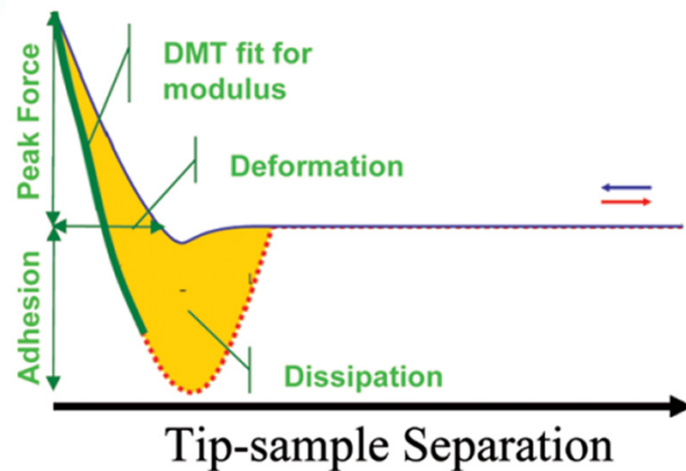
# Synch Distance



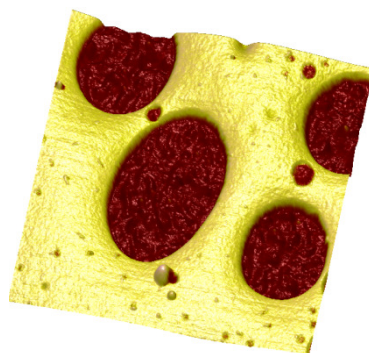
- Two common “synch” problem
  - Late synch- approach curve contains decreasing force before turnaround. Might not be a problem – could be due to sample viscoelasticity
  - Early synch- retract curve contains increasing force at beginning of turnaround.
- Use Auto Config button on hard sample to measure synch distance



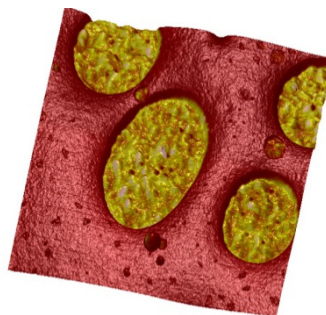
# PeakForce QNM Data Channels



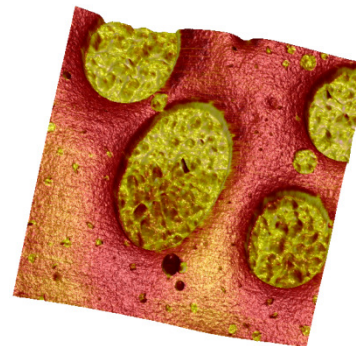
Height



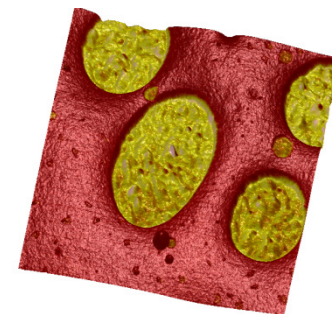
Modulus



Deformation

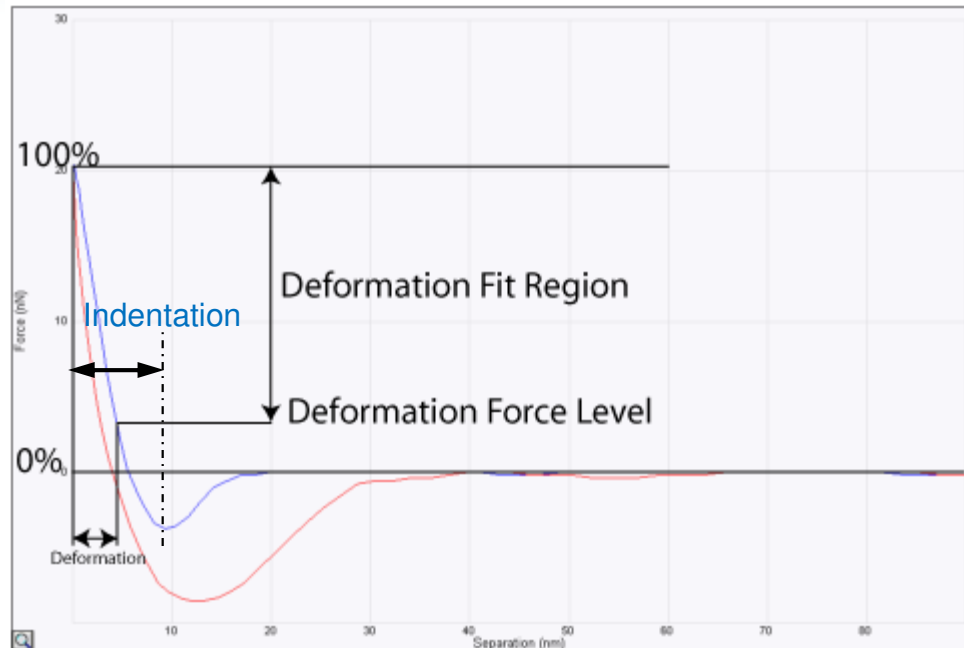


Adhesion



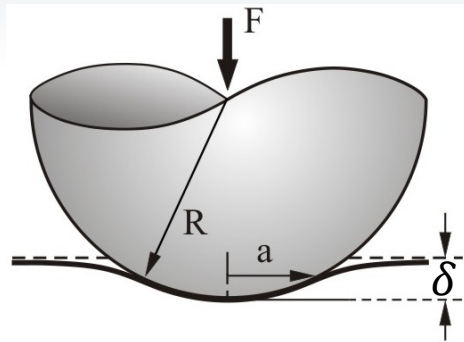
Dissipation

# Deformation

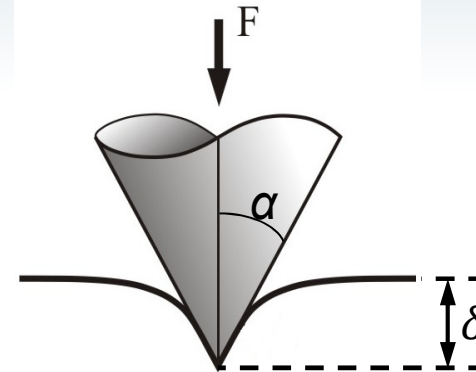


- Softer sample has higher deformation
- Most time deformation is different from sample indentation
- Deformation has close correlation with sample indentation
- Same as indentation, Deformation should not be Negative

# Calibration Parameters



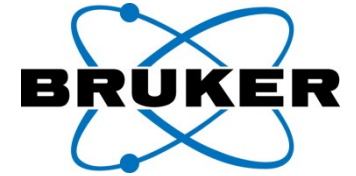
$$F = \frac{4}{3} \frac{E}{(1 - \nu^2)} R^{1/2} \delta^{3/2}$$



$$F = \frac{2}{\pi} \frac{E}{(1 - \nu^2)} \tan(\alpha) \delta^2$$

- **Deflection Sensitivity**
- **Spring Constant**
- **Tip Radius or tip half angle**
- Sample Poisson's Ratio (material property, typically 0.2-0.5)
- **Question:** which of the above parameters is the most important calibration parameter?

# PF-QNM Calibration



- 1. Deflection Sensitivity**
- 2. Spring Constant**
- 3. Tip Radius**



# Def. Sens. : Obtain a F-D curve in RAMP

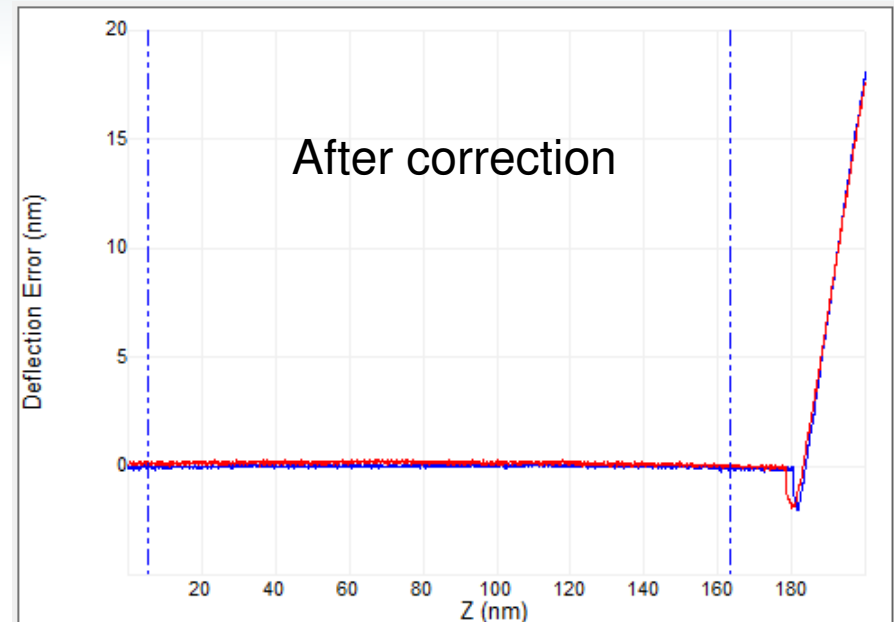
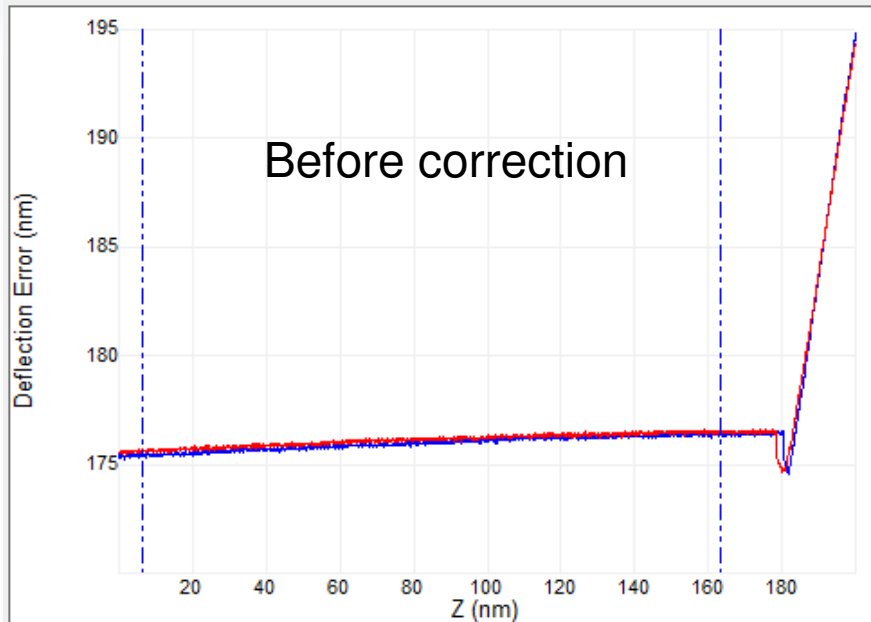
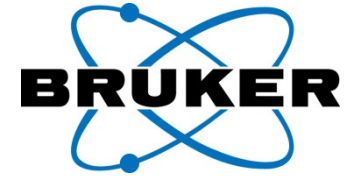



- **Question:** What kind of sample should be used?
- **Question:** Shorter cantilever has higher or lower deflection sensitivity?

- Open PeakForce QNM In Air experiment.
- Load fused silica or sapphire sample.
- Engage
- 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
- Switch to Ramp Mode
- Configure ramp parm's
- Trigger Threshold (Icon & MultiMode)
- ScanAsyst Air or Tap150A - .5V
- RTESPA or Tap525 - .2V

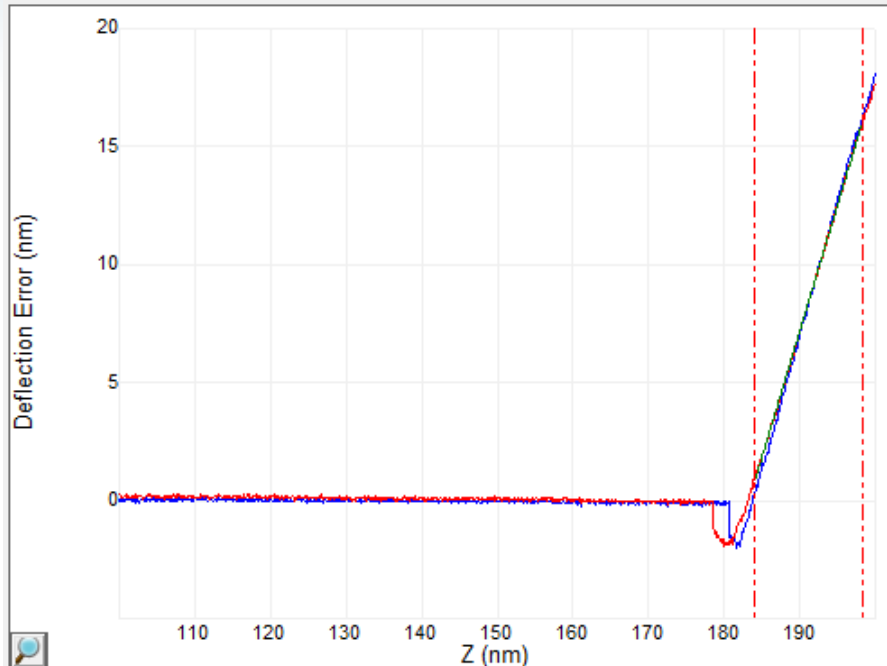
[-] Ramp	
[-] Ramp Output	Z
[-] Ramp size	500.0 nm
[-] Z scan start	7.096 nm
[-] Ramp Rate	1.03 Hz
[-] Forward velocity	1.03 $\mu\text{m/s}$
[-] Reverse velocity	1.03 $\mu\text{m/s}$
[-] X Offset	-160.156 nm
[-] Y Offset	-35.156 nm
[-] Number of samples	512
[-] Spring Constant	0.6000 N/m
[-] Plot Units	Metric
[-] Display Mode	Both
[-] X Rotate	0.00 °
[-] Mode	
[-] Trigger mode	Relative
[-] Data Type	Deflection Error
[-] Trig threshold	0.5000 V
[-] Start mode	Calibrate

# Def. Sens.: Baseline Correction

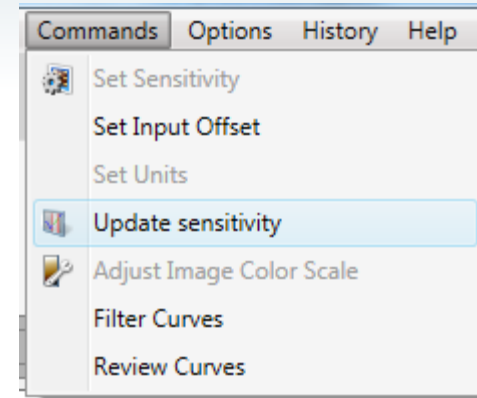


- Because of the optical interference or long range force (magnetic or electric static force), the baseline of the force curve could be not flat.
- Save the ramp curve, and open it in Nanoscope Analysis
- Click on  , define baseline fit region, then correct the baseline

# Def. Sens.: Update Sensitivity with Line Fit

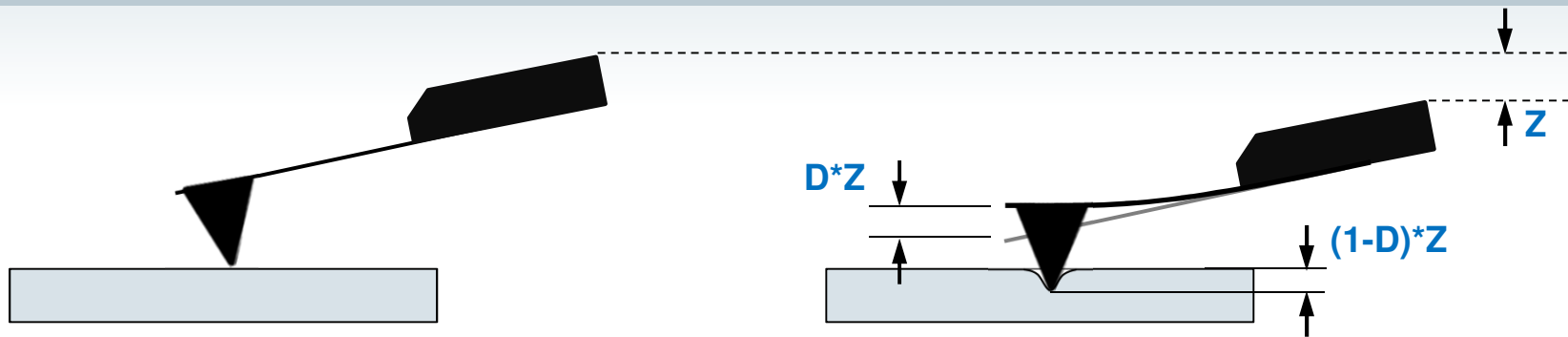


Channel data	
Image Data	Deflection Error
X Data Type	Z
Sens. DeflSens	60.00 nm/V
Plot Units	Metric
Spring Constant	0.4000 N/m
Display Mode	Deflection Error vs. Z
Plot Invert	Normal
Phase Offset	0.00 ms
Fit Type	Line
Active Curve	Retract

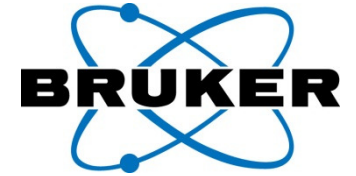


- Acquire at least 3 ramp curves, and calculate the average deflection sensitivity, then type manually in Calibration
- Confirm deflection sensitivity:
  - If it is a standard probe, compare with factory value
  - Deformation on hard sample should be close to zero
  - If not, click "autoconfig"

# Deflection Sensitivity Error Transfers into Indentation Depth Error

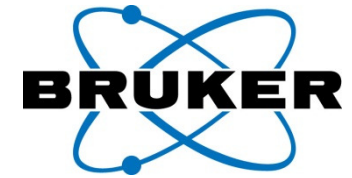


- Z scanner moves:  $Z$
- Cantilever Deflection:  $D*Z$  ( $0 < D < 1$ )
- Indentation depth:  $(1-D)*Z$
- Deflection calibration error:  $e$
- Measured Deflection:  $(1+e)D*Z$
- Calculated Indentation:  $[1-(1+e)D]*Z$
- Force measurement error:  $e$
- Indentation depth error:  $[D/(1-D)]*e$
- The Indentation Depth error introduced by Deflection Sensitivity error could be amplified by a factor of  $D/(1-D)$
- **Question:** what can cause the calculated indentation depth to be **Negative**?

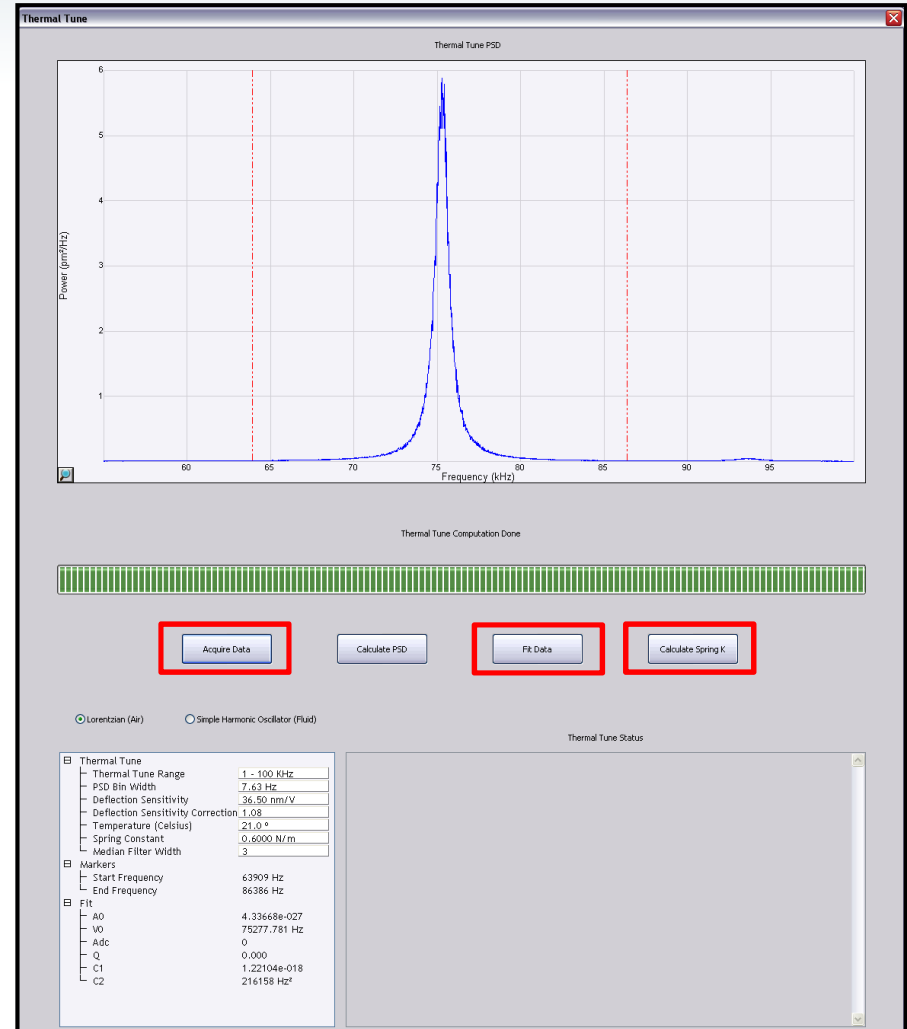


1. Deflection Sensitivity
2. Spring Constant
3. Tip Radius

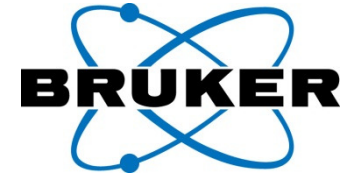
# Spring constant: Thermal Tune, $K < 1 \text{ N/m}$



- **First make sure deflection sensitivity and thermal tune gains are calibrated corrected.**
- Withdraw the probe to prepare for thermal tune.
- Click the Thermal Tune Icon
- Click the "Acquire Data button". When the ramp is finished a plot will appear.
- Drag in cursors for the data fit. The cursors should intersect the 0 baseline on both sides of the peak.
- Click the "Fit Data" button.
- Click the "Calculate Spring k" button.
- Click Yes to accept the k value.
- Close the Thermal Tune window.

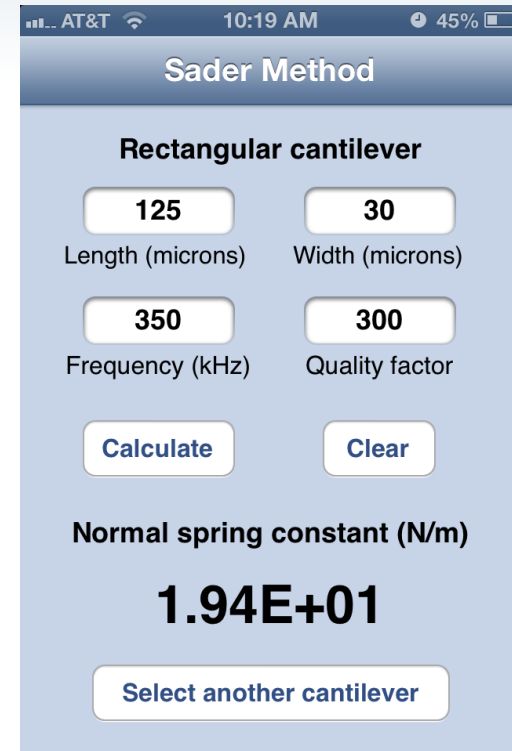


# Spring Constant: Sader Method



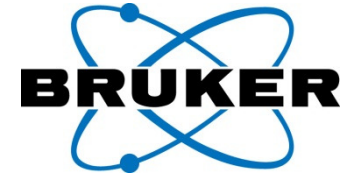
$$k = 7.5246\rho_f w^2 L Q f_0^2 \Gamma_i(Re)$$

$$Re = \frac{2\pi\rho_f f_0 w^2}{4\eta_f}$$



- Sader Method: rectangular shape ( [free iPhone App "Sader Method"](#) )
- Length and Width can be optically measured with good accuracy
- Resonance frequency and Q factor can be obtained in tuning curve (by thermal tune or mechanical tune)

# Spring constant: other methods



- Dimensional Models
  - Dimensional model Simple Beam or Frequency scaling: Rectangular shape

$$k = \frac{Ewt^3}{4L^3}$$

*Simple Beam Method*

$$k \approx \frac{2\pi^3 w (f_0 L \sqrt{\rho})^3}{\sqrt{E}}$$

*Frequency Scaling Method*

- Static Deflection Measurements
  - Reference cantilever method:  $0.3K_{ref} < K < 3K_{ref}$ , all shape
  - Bruker supplies calibrated reference probe, model: CLFC-NOBO, which consists three cantilevers with nominal spring constant at: 0.16N/m, 1.3N/m, and 10.4N/m, respectively

*Application Notes\_094*

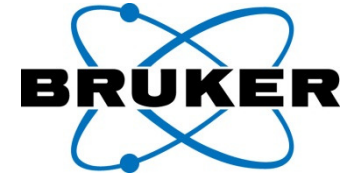


# Uncertainty of Spring Constant Calibration



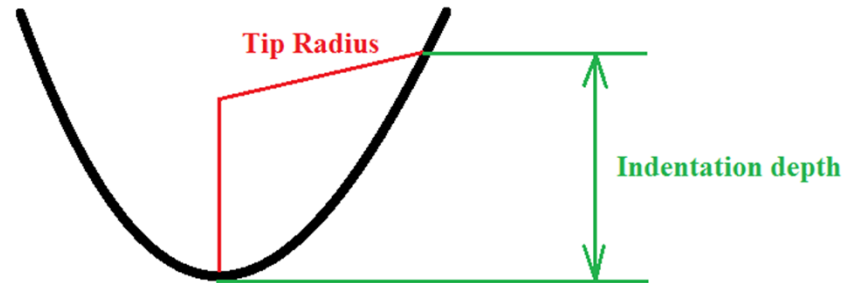
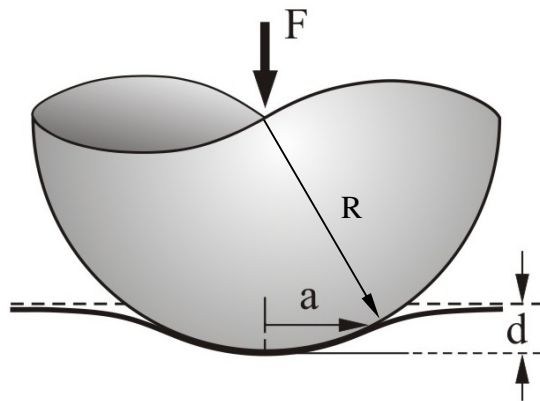
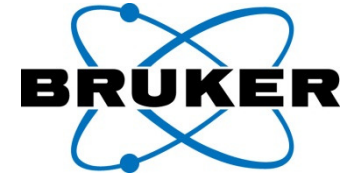
Table 2: Overall uncertainty in spring constants

Method	Uncertainty	Main source of error
Simple beam, Eqn. (1)	~16%	Cantilever thickness
PBA, Eqn. (2)	~26%	Elastic modulus of SiN
Freq. scaling, Eqn. (4)	~9%	Si density
Reference cantilever, Eqn. (5) and (6)	~9%	Deflection sensitivity
Added mass, Eqn. (9c) and (10)	15-30%	Particle diameter
Sader, Eqn. (11)	~4%	Cantilever width
Thermal tune, Eqn. (15a)	~8%	Deflection sensitivity



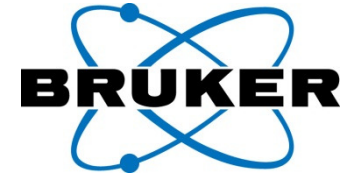
1. Deflection Sensitivity
2. Spring Constant
3. **Tip Radius**

# Tip Radius for DMT Model

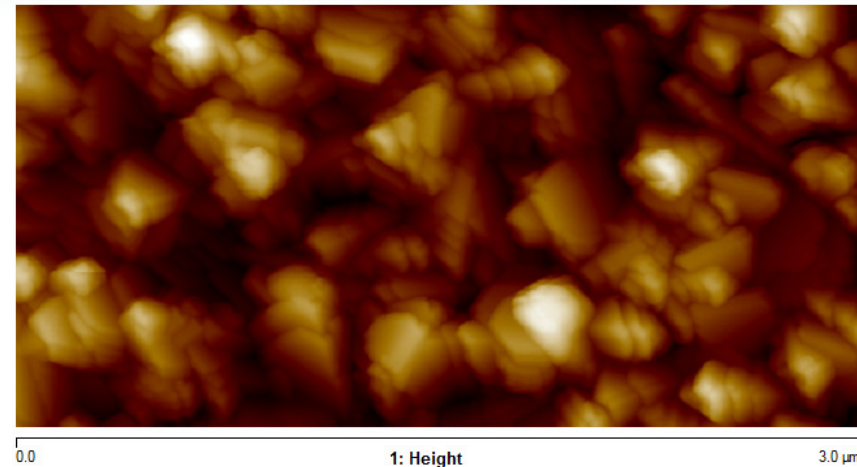


- For spherical probe, the tip radius is independent to the indentation depth
- For conical probe that tip end is not a part of a sphere, the effective tip radius is a function of indentation depth

# Determining Tip Radius with Tip Qual



Scan	
Scan Size	3.00 $\mu\text{m}$
Aspect Ratio	2.00
X Offset	0.000 nm
Y Offset	0.000 nm
Scan Angle	0.00 $^\circ$
Scan Rate	0.501 Hz
Tip Velocity	3.00 $\mu\text{m/s}$
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



- Acquire an Tip Check sample image with parameters as shown
- Open the captured image with NanoScope Analysis
- Set Plane Fit Mode to 1<sup>st</sup> order XY plan fit
- **Question:** How to use DNISP probe to image the tip check sample?

# Determining Tip Radius with Tip Qual



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

136.0 nm

88.8 nm

-82.7 nm

0.0 Tip 93.8 nm

0.0 1: Height 3.0  $\mu$ m

-106.4 nm

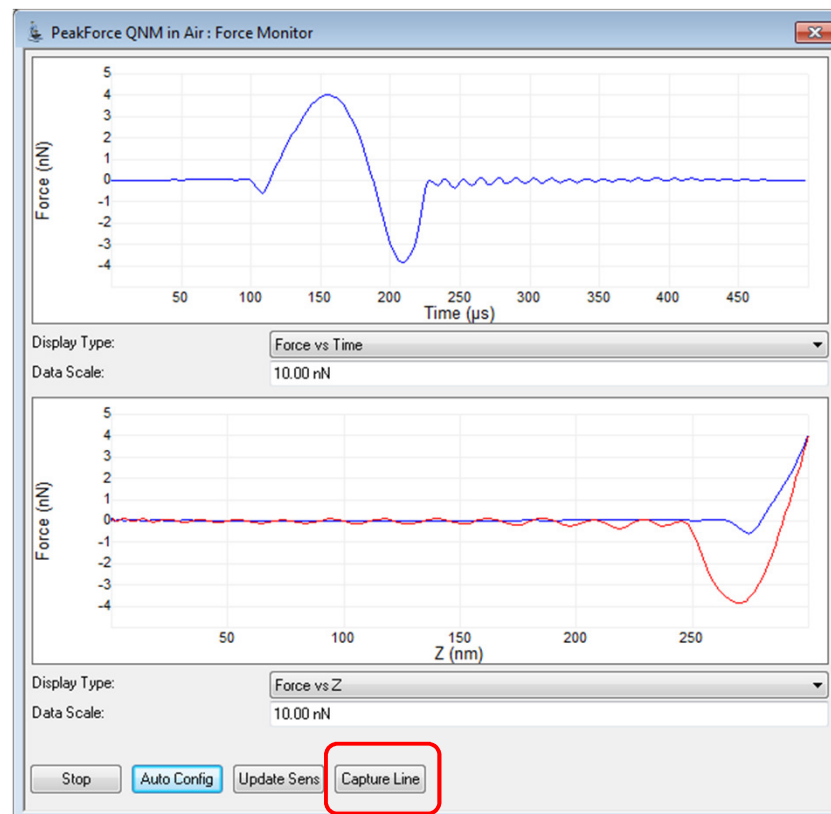
Tip Image Size	100.0 nm
LPF for Max Select	Yes
Low Pass Filter Iterations	Off
Sigma Mult for Spike Rejection	3.500
Sigma Mult for Discontinuity Rejection	3.500
Cross Section 1	
Height 1 from Apex	10.00 nm
Min ETD1 Size	5.000 nm
Good/Worn ETD1 Threshold	15.00 nm
Worn/Bad ETD1 Threshold	20.00 nm
Min y/x Aspect Ratio 1	0.500
Max y/x Aspect Ratio 1	2.000
Cross Section 2	
Height 2 from Apex	20.00 nm

Estimate Tip    Qualify Tip    Save Tip

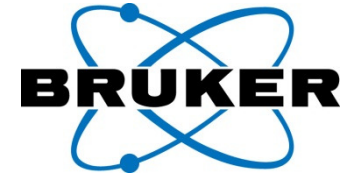
# PFQNM – How To – HSDC & PFQNM Offline (how to determine true indentation depth)



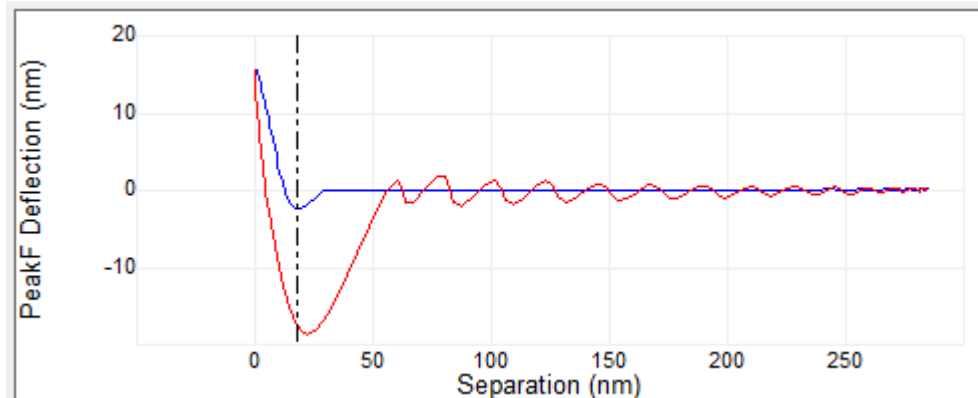
- Capture an image.
- While the image capture is in progress press the Capture Line button on the Force Monitor to capture lines of raw data.
- After the image has finished capturing press the Upload Data button to save the raw HSDC data to a file.



# Indentation depth : Deflection vs. Separation plot of HSDC

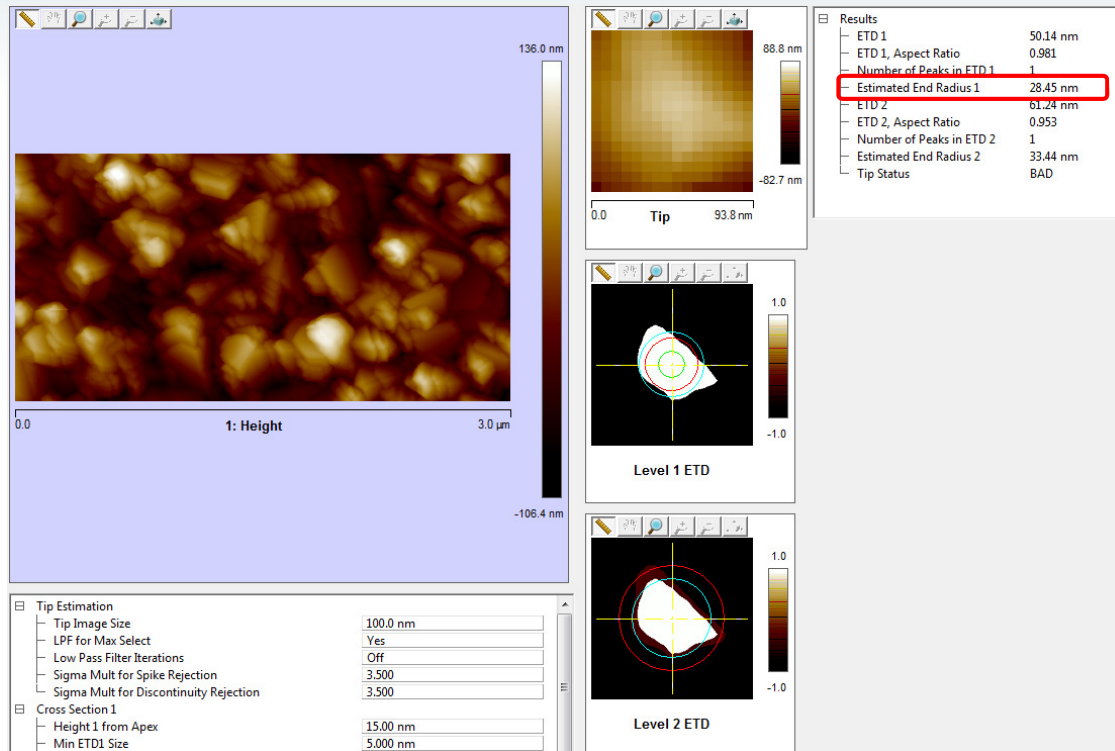
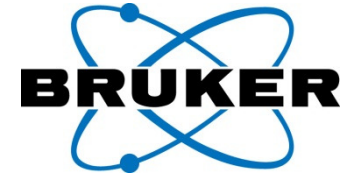


- The distance on a Deflection vs. **Separation** plot's load (extend) curve starting at the "snap-to-contact" point and ending at z fully extended.
  - Use HSDC and QNM HSDC-ForceCurve analysis to export force curve(s) and then reload to display separation plot
  - The force monitor Force vs. Z is a good approximation only if the cantilever deflection is small compared to the deformation



Indentation from deflection vs. separation plot load curve

# Determining Tip Radius with Tip Qual



- Enter the average indentation depth into Height 1 or Height 2 From Apex
  - Assume indentation depth of 15 nm for this exercise
- Click Qualify Tip
- Read the corresponding Estimated End Radius (not ETD or ETD/2)
- Enter Tip Radius into "Cantilever Parameters / Tip Radius" in real time software



# 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, so poisson's ratio of tip doesn't matter.
- Ranges between .2 - .5 Leads to 4-25% difference between the sample's modulus and it's reduced modulus.

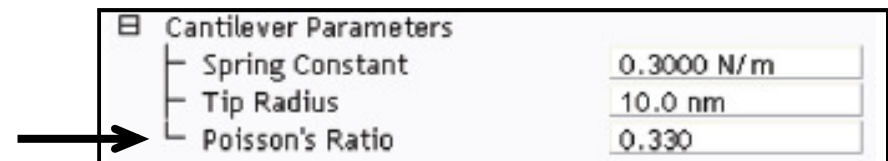
## Relationship between Reduced Young's Modulus ( $E^*$ ) and Poisson's ratio.

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

$\nu_s$  is Poisson's ratio of sample  
 $E_s$  is Young's Modulus of sample  
 $\nu_{tip}$  is Poisson's ratio of tip  
 $E_{tip}$  is Young's Modulus of tip

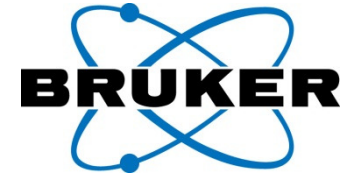
## Estimating Poisson's ratio

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



**Absolute Method  
vs.  
Relative Method**

# 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, but modulus is only weakly dependent on  $R$
  - 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.
- **Question:** how to make sure the indentation depth is close to that on reference sample?

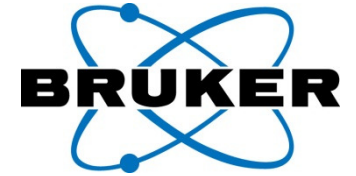
# Relative calibration method



## 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.
  - Can partially correct other error sources
- 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

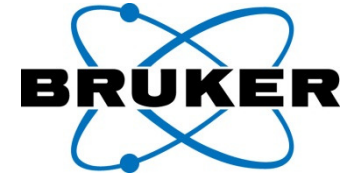
- Advantages:
  - Standard sample with known modulus not required (uncertainties in standard sample modulus and aging eliminated).
- Disadvantages:
  - Susceptible to accumulated errors
  - Takes longer (must measure tip radius and spring constant)

# Which method to use???



- 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?
  - K can't be measured by thermal tune for probes over  $\sim 10-15$  N/m.
- Does the "relative" method work on soft samples like PDMS1 and PDMS2?
  - Yes

# PF-QNM Procedure



## Absolute Method

1. Calibrate Deflection Sensitivity
2. Calibration Spring Constant
3. Image on the tip check sample, if tip radius needs to be calibrated
4. Switch to the unknown sample
5. Manually adjust the PeakForce setpoint to get 5-10nm deformation
6. Capture HSDC data on unknown sample
7. Measure Indentation Depth
8. Open tip check image in Nanoscope Analysis, and use Indentation depth to get the tip radius
9. Type in the tip radius in Nanoscope software and get the sample modulus

## Relative Method

1. Calibrate Deflection Sensitivity
2. Load a reference sample with known modulus
3. Manually adjust the PeakForce setpoint to get 5-10nm deformation
4. Type in an estimated spring constant (i.e. nominal value)
5. Adjust the tip radius so that the measured modulus is close to expect value
6. Record the average deformation
7. Switch to unknown sample
8. Only adjust the PeakForce setpoint so that the average deformation is same as on reference sample
9. The measured modulus is the unknown sample modulus

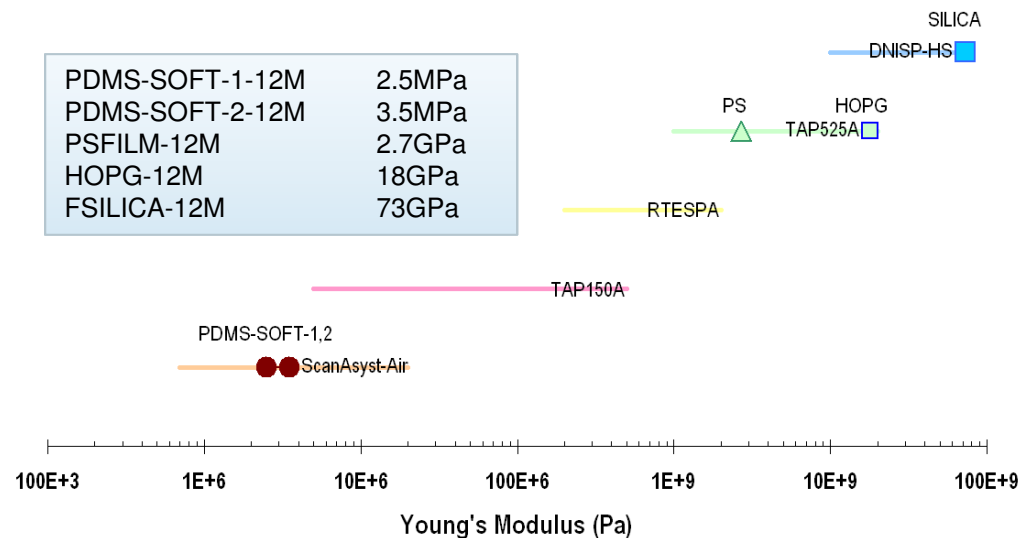
# How to choose probe and reference sample



- If sample modulus is unknown, try probes until one is found that has deformation  $\geq 3 \times$  deflection, with deformation  $\ll R$  and no plastic deformation.
  - Use a reference sample that matches the probe
- If the approximate modulus of the sample is known, use the chart below as a starting point:

Table 2.3a Recommended Probes

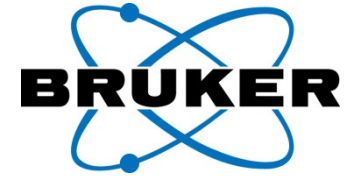
Sample Modulus (E)	Probe	Nominal Spring Constant (k)
0.7 MPa < E < 20 MPa	SNL-A	0.5 N/m
5 MPa < E < 500 MPa	Tap150A	5 N/m
200 MPa < E < 2000 MPa	RTESPA	40 N/m
1 GPa < E < 20 GPa	Tap525A	200 N/m
10 GPa < E < 100 GPa	DNISP-HS	350 N/m





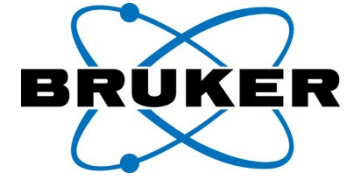
# PF-QNM Parametes

# Engage Force



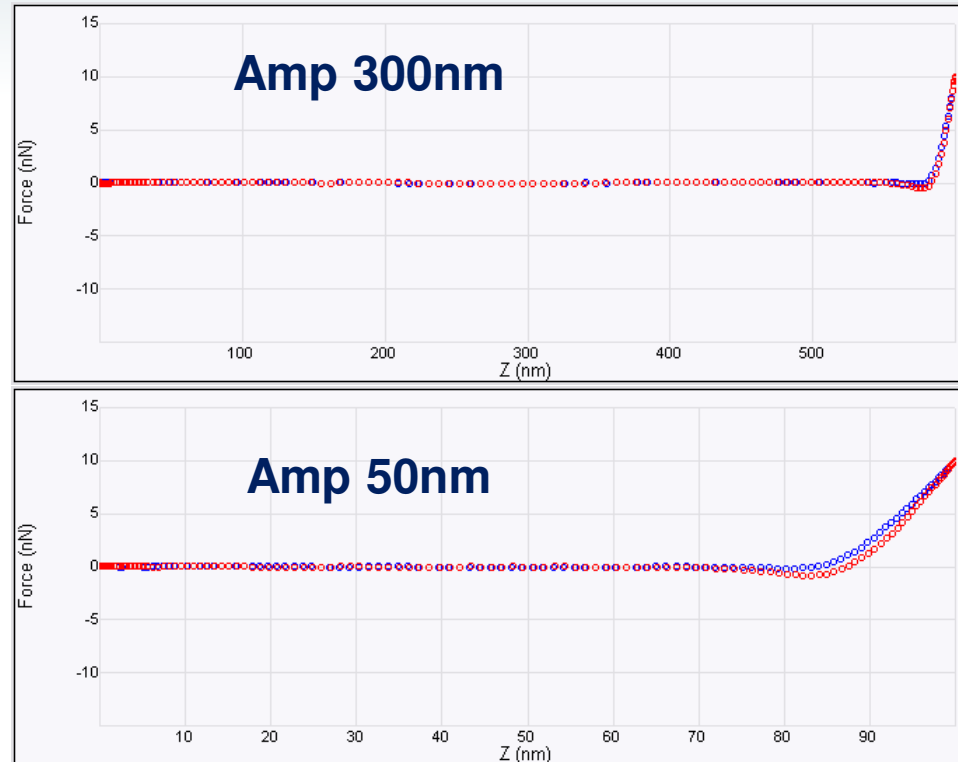
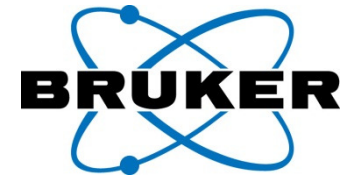
- Engage ignores Peak Force Setpoint
  - Doesn't matter if ScanAsyst Auto Setpoint is On or Off.
- For engaging the Peak Force Engage Setpoint (in other controls) is used.
  - Default .15 V
    - OK for ScanAsyst Air
    - WAY TOO HIGH for stiff probes like RTESPA or TAP 525. Sample and/or probe damage will occur.
    - Try .05 V for stiff probes
- After engage occurs at the engage setpoint the software will
  - Remove background 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

# Setpoint



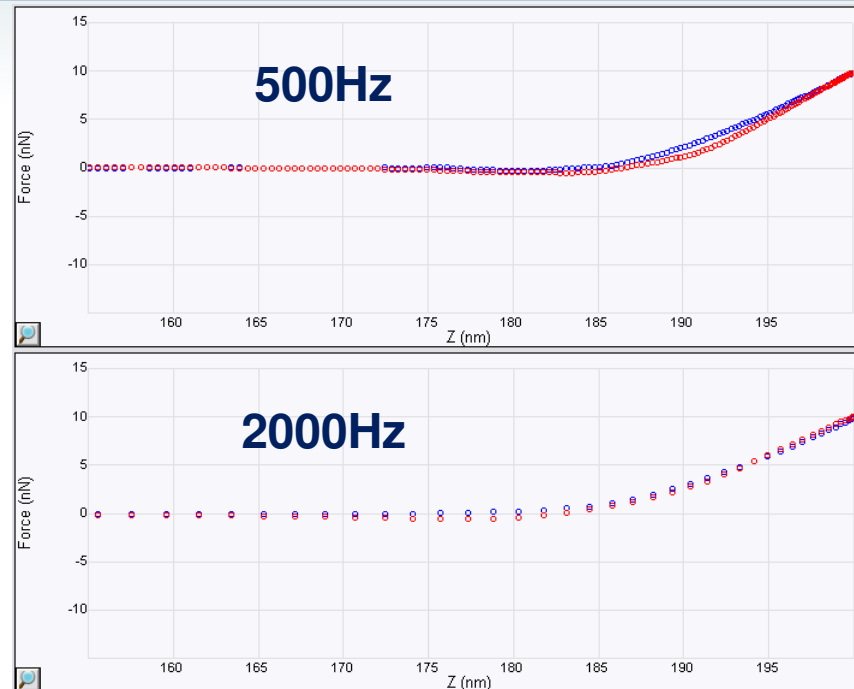
- Set ScanAsyst control to Individual
- Engage with all (setpoint, gain, scan rate, z limit) *On*
- After engaged and stable turn auto setpoint *Off*
  - Changing setpoint while imaging will cause imaging forces to change
  - Indentation depth can be controlled with setpoint
  - If imaging forces change the probe contact area will change
  - If probe contact area changes the tip radius is no longer valid
  - Modulus & adhesion data will shift
- Adjust the setpoint to have at least 2-5nm and less than 10nm deformation for DMT model. For Sneddon model, deformation should be larger, i.e. >30nm

# Amplitude



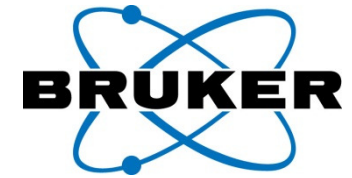
- Amplitude combined with setpoint determines the contact time ratio
- Higher contact time ratio means more data point in contact region
- Higher amplitude sometime is needed to overcome large adhesion force

# Frequency



- Peak Force tapping operates at off cantilever resonance frequency
- Default for Icon and MM is 2KHz, MM HR and Fast Scan is 8KHz
- Higher frequency can scan faster and/or higher resolution for topography
- Lower frequency is preferred for fluid imaging
- DSP force curve analysis speed at 1KHz
- **Question:** why don't use 8KHz on Icon scanner?

# QNM sync Distance

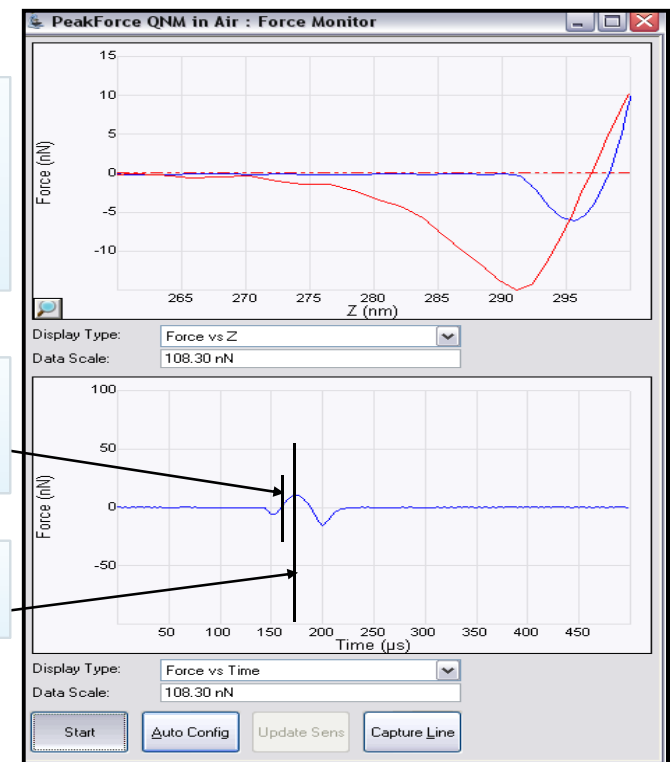


- Use synch distance from hard/elastic material
  - Software changes during autoconfig
  - Can now manually adjust
- Zoom in on interaction region of force monitor.
  - Verify “heart beat” is clean with minimal distortion
  - Watch out for synchronization problems
  - Use auto-config if necessary
- It 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

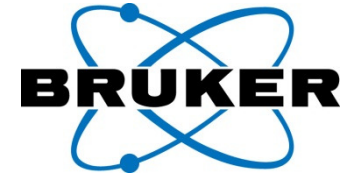
Peak force occurs after synchronization time. withdraw curve should not be to the left of the approach curve

Software thinks that peak force is occurring at this time...

Instead of the actual time peak force occurs



# 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

# Peak Force Capture



The screenshot displays the Bruker software interface for Peak Force Capture. The top toolbar contains several icons, with two red boxes highlighting the 'Peak Force Capture' icon (a camera) and the 'Save' icon (a floppy disk). The main window shows a 'Force Volume' plot on the left, a 'Deflection Error (nm)' vs 'Z (nm)' plot on the right, and a 'Save Curves' button highlighted with a red box. The 'Offline Parameters' section is also visible.

Offline Parameters	
Z display	100.00 nm
Z direction	Retract
Display Mode	Normal
Center plot	Off
Plot Units	Metric
Max Curves Plotted	50
Modulus Fit Model	Hertzian (Spherical)
Deflection Sensitivity	25.000 nm/V
Spring Constant	5.0000 N/m

- One force curve at each image pixel can be captured with PeakForce Capture
- Recommend reduce pixel resolution to 128x128 or less for PeakForce Capture

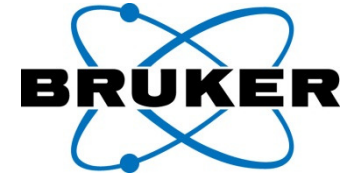


# Artifact



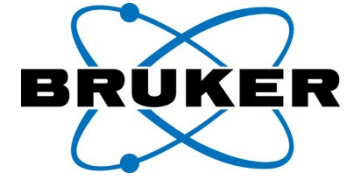
- Sources of artifact
  - Optical, fluid
  - Always use probes with reflective coating
  - Carefully align laser and maximize sum
- Artifact on the force curve base line will appear in data channels.
  - *Ideally*, force curve base line should be
    - On 0 V line
    - Flat (no superimposed, periodic 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

# Sources of error

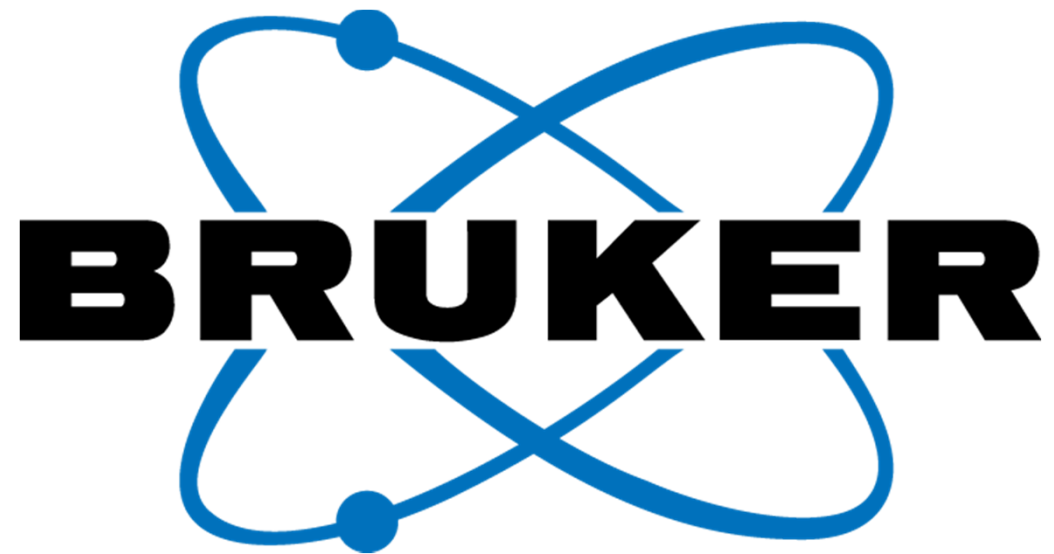


- Thermal Tune
  - When used properly (right probes) thermal tune should be within 10-30% of the real k value
  - $E \sim k/\sqrt{R}$
- Deflection Sensitivity
  - Drift (especially in the first 10 minutes with soft probes)
  - Error in calibration
  - Done on contaminated area of sample (will be too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

# Summary



- Cantilever spring constant need to match the sample modulus
- Deflection Sensitivity is the most important parameter in QNM calibration
- Make sure sync distance is correct, use Auto Config when needed
- Measure sync distance QNM on hard elastic sample, then apply to unknown sample
- Use fixed setpoint for QNM
- Need to have enough Deformation ( $>5\text{nm}$ )
- Relative method is preferred for hard material ( $>1\text{GPa}$ )



[www.bruker.com](http://www.bruker.com)