Image Quality & PeakForce Tapping Advanced Applications Training



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Outline



- AFM Basics
- Contact Mode
- Tapping Mode
- PeakForce Tapping
- Image Quality
- Practice

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What is Atomic Force Microscope (AFM)





- AFM uses a sharp tip to image the sample surface
- Every AFM has following key components: probe, scanner, controller, and software







- An AFM probe has three components: tip, cantilever, and substrate
- Two most common building materials: Si and Si₃N₄
- Two different shapes of cantilever: rectangular and triangular
- Question: which one of the three components is the least critical for AFM imaging

Cantilever spring constant, resonance frequency, tip radius, and dimensions





 f_0 : Cantilever resonance frequency ρ : Density of the material

<u>50 nm</u>

Probe Selection



Life Sciences

Biomolecules

Cells

	Irobo	Imaging Environme		Imaging Nominal Specifications		tions	Coatings				AFM Mode				
Fami	ly/Model	Liquid	Air	Force Constant (N/m)	Resonant Frequency (kHz)	Radius of Curvature (nm)	Back Side	Tip Side	Probe Attributes	Peak Force/ ScanAsyst	Tapping	Contact	Force Curves	Electrical	Magnetic
	DNP	1	-	0.06-0.58	18–57	20	Au	None	Low force, Symmetric tip	-	~	1	~	-	-
	MLCT	~	-	0.01-0.5	7–120	20	Au	None	Lowest force, Symmetric tip	-	1	~	~	-	-
Silicon Nitride	<u>FastScan-</u> <u>B</u>	~	-	1–3	300-600	8	Ti/Au	None	Highest resolution, symmetric tip, higher optical sensitivity and lower force	~	~	~	~	_	_
	<u>FastScan-</u> <u>C</u>	~	-	0.4–1.2	130–290	8	Ti/Au	None	Highest resolution, symmetric tip, higher optical sensitivity and lower force	~	~	~	~	-	-
	FastScan- Dx	1	-	0.21-0.29	90–130	8	Proprietary	None	Highest speed and force control on soft biological samples	~	~	-	-	-	-

Tissues

Materials

▶ Polymers/Soft Samples

Hard Samples

More Information of Bruker Probes



Ohana	Resonant Freq. kHz			Spring Const. N/m			Length µm			Width µm		
Snape	Nom.	Min.	Max.	Nom.	Min.	Max.	Nom.	Min.	Max.	Nom.	Min.	Max.
Rectangular	300	200	400	40	20	80	125	115	135	35	30	40

Tip Specification

This probe uses a rotated tip to provide a more symmetric representation of features over 200nm.



Geometry:	Rotated (Symmetric)
Tip Height (h):	15 - 20µm
Front Angle (FA):	15 ± 2º
Back Angle (BA):	25 ± 2 °
Side Angle (SA):	17.5 ± 2 °
Tip Radius (Nom):	8 nm
Tip Radius (Max):	12 nm
Tip SetBack (TSB)(Nom):	15 µm
Tip Set Back (TSB)(RNG):	5 - 25 µm

www.brukerafmprobes.com

Tip Schematic

Cantilever Specification

Aluminum reflective coating on the backside of the cantilever. The AI Reflective coating increases the laser signal (A+B) by up to 2.5 times. Although not necessary for general imaging, reflective coating is recommended for thin cantilevers, highly reflective samples, and machine vision applications.



Material:	0.01 - 0.025 Ωcm Antimony (n) doped Si
Geometry:	Rectangular
Cantilevers Number:	1
Cantilever Thickness (Nom):	3.75 μm
Cantilever Thickness (RNG):	3.0 - 4.5 µm
Back Side Coating:	Reflective Aluminum
Top Layer Back:	40 ± 10 nm of Al

Cantilever schematic

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Optical Lever and PSPD





- AFM use an optical lever to generate feedback signal
- When the cantilever bending changes, the laser spot position on the PSPD changes, and generate electrical signal
- SUM = A + B + C + D
- Vertical deflection=[(A+B)-(C+D)]/SUM

Optical Lever





- Angular movement of the cantilever causes deflection change
- Deflection Sensitivity (nm/V) ∝ L; Lower value of Deflection Sensitivity means cantilever is more sensitive (better sensitivity)
- Shorter cantilever has better deflection sensitivity (e.g. PFM application)
- Force Sensitivity (nN/V)=k*(Def. Sens.) ∝ 1/L², where k ∝1/L³; longer cantilever for better force control
- Lower value of Force Sensitivity means cantilever is more sensitive

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Contact Mode





Contact Mode Image and Data Channels







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    Contact Mode
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- Sample: PS-LDPE
- Probe: ScanAsyst-Air
- Scan Angle: 90Deg

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Contact Mode Parameters







- Contact Mode Key Parameters:
 - Scan size
 - Scan angle
 - Scan rate
 - Scan resolution
 - Feedback gains
 - Deflection setpoint
 - Z range

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Contact Mode Summary



- Advantages:
 - Simple and direct force control
 - Provides lateral force signal
 - Tip has solid contact with sample, good for electrical measurement: TUNA/C-AFM, SCM, Piezoresponse
- Disadvantages:
 - Relative high force and shear force cause tip wearing and possible sample damage, even with soft cantilever
 - Sensitive to optical interference and laser signal drift

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TappingMode





- Cantilever oscillates at its resonance frequency
- Cantilever oscillation amplitude decrease with tip interacts with sample surface
- TappingMode uses cantilever oscillation amplitude as feedback signal for imaging. Typical amplitude setpoint is 10-100nm.

Tune Cantilever





- Make sure cantilever is seated well in the probe holder
- The tuning curve should have one signal peak
- Do a surface tune or fast thermal tune if needed

Cantilever Tuning Trouble Shooting





- Reposition the cantilever in the probe holder
- Clean the probe holder slot
- Make sure the spring clip is holding the probe securely

Tapping Mode Surface Tracking





• Use trace & re-trace to avoid tracking artifacts

Tapping Mode Parameters Optimization





- How to improve tracking:
 - Increase feedback gains, may increases noise
 - Reduce amplitude setpoint, but also increases tip-sample force
 - Lower scan speed or reduce scan size, but also increases scanning time

Tapping Phase: Sine Wave





- Example:
 - Detector signal: y₂=1*sin(1000t-90°)
 - Drive signal: y₃=2*sin(1000t+0^o)
 - Phase = $-90^{\circ}-0 = -90^{\circ}$

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Phase Imaging







Patches of Polydiethylsiloxane on Si substrate. The sample topography shows overall height undulations that can obscure the presence of distinct properties.

The contrast observed in the phase image arises from mechanical sample properties and clearly reveals more details within Polydiethylsiloxane.

- Tapping phase signal is the phase difference between the cantilever oscillation signal (measured by PSPD) and the cantilever driving signal
- Phase signal correlates to the sample surface properties (modulus and adhesion)

Cantilever Tuning Curve





- Drive frequency is fixed for Tapping Mode imaging
- **Question:** at what frequency the cantilever oscillates?
- **Question:** what is the cantilever oscillation amplitude in free air?

Cantilever Oscillation



For a simple driven harmonic oscillator:

$$\omega = \sqrt{\frac{k}{m^*}} \qquad \qquad F = -k \times z \\ k = -\frac{\partial F}{\partial z}$$

Force Gradient changes the effective spring constant.

$$k_{eff} = k - \frac{F_{ts}}{\partial z}$$
 $\omega_{eff} = \sqrt{\frac{k - \frac{\partial F_{ts}}}{m^*}}$



Force Between Tip and Sample





- Repulsive force gradient effectively increases the spring constant
- Attractive force gradient effectively decreases the spring constant

Force gradient causes resonance frequency shift





- The force gradient equivalents to an additional spring, $\Delta \mathbf{K} = -\frac{\partial \mathbf{F}}{\partial \mathbf{Z}}$
- Resonance frequency shift due to spring constant change, $\omega = \sqrt{\frac{\kappa}{m}}$

Cantilever Tuning with 5% offset to the left of resonance frequency





- In Tapping mode, cantilever need to be tuned before imaging
- Tapping frequency is set close to cantilever's resonance frequency

5% Peak Offset on Left





- When tip approaches surface, amplitude initially increase because of the attractive force
- Phase decreases in attractive regime, and increase in repulsive regime

Cantilever Tuning at resonance frequency









- Amplitude decreases from the beginning
- The small kink is the transition from attractive regime to repulsive regime

Cantilever Tuning with 5% offset to the right of resonance frequency





5% Peak Offset on Right





- In bi-stable regime, with same amplitude setpoint, the tip could be at two different tip-sample distance
- At same tip-sample distance, the phase could change from very negative to very positive
- Imaging in bi-stable regime generates artifacts

Tapping Artifacts in Bi-Stable Regime





• At sample peak position is tip is tapping in repulsive regime

Attractive Regime vs. Repulsive Regime



8.0 nm

-5.0 nm

50.0°

20.0°



Phase

400.0 nm

400.0 nm

- Feature looks larger in attractive regime
- Tip can not penetrate the water layer on surface
- No Phase contrast in attractive regime

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Height Sensor

Phase

Attractive Regime vs. Repulsive Regime





- Tapping in repulsive regime give more details and sharper image
- Measured surface roughness is higher when tapping in repulsive regime

Attractive Regime vs. Repulsive Regime





- When tip operated in attractive regime:
 - Tip is sensing the attractive force, which is long range interaction
 - Tip-sample distance at peaks is shorter than in valleys, less stable
 - Tip cannot penetrate surface water layer
 - Phase signal does not related to surface properties
- When tip is operated in repulsive regime:
 - Tip is sensing the repulsive force, which is short range interaction
 - Tip-sample distance is same at peaks and valleys, more stable
 - Tip can penetrate surface water layer, and image on solid surface
 - Phase signal relates to surface properties

Tapping Mode Cantilever Response Time





• Cantilever response time constant: $\tau = 2Q/f_0$

How To Scan Fast





FastScan-A probe Typical f_0 =1.4MHz



FastScan Z Scanner

- Two key components to enable fast scan speed:
 - FastScan probe: high resonance frequency with close triangle shape to reduce Q
 - FastScan Z scanner: piezo stack with reduced mass loading to increase Z bandwidth

Tapping Mode Summary



- Advantages:
 - Less tip and sample wear and usually higher resolution
 - Usually less normal tip-sample force than contact mode, no shear force
 - Phase signal relates to sample properties
 - Less sensitive to optical interference and laser signal drift
- Disadvantages:
 - More complicate
 - No direct force control
 - Tip intermittently contact with surface, not compatible with some electrical measurement
 - Requires tuning of modulation to match cantilever resonance

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Force Distance Curve





Peak Force Tapping





- In Peak Force Tapping, the tip performs a very fast force curve at every pixel in the image
- The peak interaction force of each of these force curves is then used as the imaging feedback signal
- Peak Force Tapping operates typically operates at 2 KHz, and it does not depend on the resonance frequency

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PeakForce Tapping Parameters

1.00 µm

0.000 nm

0.000 nm 0.00 °

1.01 Hz

256

256

1

On

5.000

0 V

On Off

1.000 nN

40.00 kHz

Individual

1.00 nm

2.52 µm/s

Enabled

1.00



⊟ Scan

- Scan Size
- Aspect Ratio
- X Offset
- Y Offset
- Scan Angle
- Scan Rate
- Tip Velocity
- Samples/Line
- Lines
- Slow Scan Axis
- Scan Single Frame Number
- XY Closed Loop
- Feedback
 - Feedback Gain
 - Peak Force Setpoint
 - Analog2
 - LP Deflection BW
 - ScanAsyst Noise Threshold
 - ScanAsyst Auto Control
 - ScanAsyst Auto Gain
 - ScanAsyst Auto Setpoint
 - ScanAsyst Auto Scan Rate
 - ScanAsyst Auto Z Limit
- Peak Force Tapping Control
 - Peak Force Amplitude
 - Peak Force Frequency
 - Lift Height

UII .	
On	
On	
150 nm	
2 KHz	
44.9 nm	

- PeakForce Tapping Parameters
 - Scan parameters same as other modes
 - Feedback gain: only one gain
 - PeakForce Setpoint
 - ScanAsyst Auto Control
 - Noise threshold
 - PeakForce Amplitude
 - PeakForce Frequency
 - Engage setpoint

Sew tip

- Peak Force Engage Amplitude
- Peak Force Engage Setpoint
 - Engage int. gain

Yes	
150 nm	
0.1500 V	
10.0	

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[🖯] General Engage

Force Monitor & Auto Config





- Force monitor gives realtime display of the force curve
- Auto Config:
 - Background subtraction
 - Synch distance
- Question: what is the ringing after tip break off from surface?

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Patented background subtraction is unique to PeakForce Tapping





PeakForce Tapping

- Background subtraction removes distortions from the high-speed force curves, enabling the use of lower imaging forces and quantitative measurement of material properties from the curves.
- Background subtraction enables:
 - Higher resolution
 - Gentler imaging
 - Quantitative nanomechanics

Patented synchronous peak force detection is unique to PeakForce Tapping





PeakForce Tapping

- Synchronous peak force detection calibrates the precise position of the peak force in each cycle. By measuring the force at a defined position, it enables lower forces and lower noise.
- Synchronous peak force detection enables:
 - Higher resolution
 - Gentler imaging

Negative PeakForce Setpoint





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Mica in Water: Atomic Resolution Peak Force Tapping





Roughness measurement with PFT







- Same area was continuously scanned for 600 images
- A fresh tip is used
- Peakforce Setpoint: 0.5nN
- 1st image RMS roughness: 0.684nm
- 600th image RMS roughness: 0.667nm

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Roughness measurement with PFT



2.000 500.0 pN

40.00 kHz

0.500 nm

Individual

Off

Off

Off

Off

50.0 nm

1 KHz

146.7 144.9

90 %

30 %

15 %

100 nm

Threshold Crossing

🖯 🛛 General Engage		
 Sew tip 	Yes	
 Peak Force Engage Amplitude 	150 nm	
 Peak Force Engage Setpoint 	0.05000 V	
Engage int. gain	5.00	
H Tapping Engage		
∃ Stage Engage		
 Sample clearance 	1000 µm	
 SPM safety 	100 µm	
 SPM engage step 	0.486 µm	
 Withdraw Z Pos 	Smart Lift	
Load/Unload height	3000 µm	
∃ Smart Engage		
 Engage Mode 	Standard	
 Fast engage velocity 	50.0 %	
 Fast engage threshold 	100 %	
└── Fast engage height	-5000 µm	
Height Engage		

B Actuated Probe Engage

- Engage Setting:
 - PeakForce Engage Setpoint
 - SPM engage step

- Feedback and PFT Control:
 - Feedback Gain

⊟ Feedback

Feedback Gain

Peak Force Setpoint LP Deflection BW

ScanAsyst Noise Threshold

ScanAsyst Auto Control

ScanAsyst Auto Setpoint

ScanAsyst Auto Z Limit

Peak Force Frequency

Sync Distance New

Sync Distance QNM

Adhesion Algorithm Max Force Fit Boundary

Min Force Fit Boundary

Deformation Force Level

Peak Force Tapping Control
 Peak Force Amplitude

Lift Height

ScanAsyst Auto Scan Rate

ScanAsyst Auto Gain

- PeakForce Setpoint
- ScanAsyst Auto Control: Individual
- PeakForce Amplitude

|--|

PeakForce Tapping vs. TappingMode --3 most important differences--



Three important characteristics unique to PeakForce Tapping:

- #1: Direct control of the tip-sample force at known low values
 - Benefits: Gentler operation and higher resolution
- *#*2: Direct measurement of the force-distance interactions
 - Benefits: Quantitative nanomechanics
- #3: Operates far below the cantilever resonance
 - Benefits: Simplicity and consistency

Direct control of the tip-sample force



PeakForce Tapping

- Measuring the complete force-distance interaction at every pixel enables PeakForce Tapping to directly control the image force at low, precisely known levels.
- Direct force control enables:
 - Easier to achieve high quality image



- Tapping mode does not directly control the tip-sample force. It can only control the tapping oscillation, which is just one factor in the tip-sample force. It's extremely difficult to estimate the actual force and the force can vary even if the amplitude is held constant.
- Without direct force control:
 - May cause more tip wear or sample damage if not operated correctly



You know the amplitude difference, but what force is it applying to the sample?

Defection (μs)

Tip-sample force is unambiguous and directly controlled, even down <50 pN

Direct force-distance measurement



PeakForce Tapping

- PeakForce Tapping directly obtains the complete force-distance interaction at every pixel. By simply applying standard indentation models we can easily obtain quantitative modulus measurements. It's easy to understand and completely accessible to verify.
- Direct force measurement enables:
 - Quantitative nanomechanics



Modulus is measured using standard, well accepted indentation models

Tapping Mode

- Tapping mode can't directly measure the force-distance interaction. Various attempts have been made to model the tapping interaction and reconstruct a complete force interaction, but the complexity of these approaches has prevented any consensus or standard.
- Without direct force measurement:
 - Extremely difficult to obtain quantitative modulus estimates

Loss tangent imaging? DART? AM-FM Viscoelastic Mapping? Contact Resonance? Band Excitation? Dual AC Imaging?

Too many options. Too many limitations and complications. No one agrees where or when or if various approaches are correct.

Operates below the cantilever resonance



PeakForce Tapping

- Operating at a fixed frequency below the cantilever resonance greatly simplifies PeakForce Tapping operation. First, there's no need to tune the cantilever resonance. Second, and less obvious, is that feedback is simpler and more stable when operating off resonance.
- Sub-resonance operation enables:
 - Simplicity and consistency
 - ScanAsyst



Off-resonance operation makes possible the automatic optimization of imaging parameters in ScanAsyst.

Tapping Mode

- Tapping mode operates at the cantilever resonance frequency. This requires tuning the tapping frequency for every probe and adjusting it when it shifts. Adjusting gains is also more sensitive.
- Operating on resonance means:
 - More complexity



A typical cantilever tune in liquid. Which peak is the best peak?

ScanAsyst Advantage



• Advantage:

- The tip touches the surface very briefly in each tapping cycle. The shear force between the tip and sample is minimized
- Peak Force Tapping has direct control of the force between the tip and sample, very small peak force can be easily achieved
- In Peak Force Tapping mode, AFM does a F-D curve at every pixel, which includes a lot of useful information about the sample surface (details see PF-QNM)
- Peak Force Tapping does not rely on cantilever resonance frequency, no need to tune the cantilever. This is a great advantage for imaging in liquid
- ScanAsyst can automatically optimize the key scanning parameters to obtain high quality images
- Disadvantage:
 - Scan speed is limited by the peak force tapping frequency, but similar to regular Tapping Mode

Brief summary of AFM imaging modes



	Contact Mode	TappingMode	PeakForce Tapping
First introduced	1986 (original mode)	1992	2009
Tip-sample interaction	Tip scans in constant contact with the sample	Cantilever is oscillated at its resonance, so the tip intermittently contacts or "taps" the sample	Whole probe is ramped sinusoidally, so the tip intermittently contacts or "taps" the sample
Tip oscillation	Not applicable	At cantilever resonance (typ. 10-1000's kHz) with typ. amplitude of 1-10's nm	Below cantilever resonance (typ. <10 kHz) with typical amplitude of 10-100's nm
lmaging feedback	Constant force (cantilever deflection)	Constant tapping amplitude	Constant peak force
Sketch			
Off surface	F ₀	A _{free}	F_0 F_0
On surface	F _{SP}	A _{SP}	F ₀ F _{peak}

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Image Quality (Ishikawa Diagram)





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Image Quality: Probe





- Probe
 - **Tip:** bad tip, tip shape, tip radius
 - **Cantilever:** coating, tuning, stiffness
 - Substrate: mounting, contamination

Image Quality: Probe, Examples





• AFM image is a convolution of sample topography and tip shape

Image Quality: Sample





- Sample:
 - Sample preparation: immobilization, mounting
 - Sample condition: charging, contamination, degradation

Image Quality: Hardware





- Hardware:
 - Limitations: scanner, controller, system
 - **Others:** optical interference, piezo hysteresis, drift

Image Quality: Software





- Z resolution = (Z range, nm)/65536
- If Z range is 10um, Z resolution = 10,000nm/65536= 0.15nm
- If Z range is 1um, Z resolution = 1,000nm/65536= 0.015nm

Software:

- Scanning parameters:
 - feedback gains
 - setpoint
 - scan resolution
- Engage settings:
 - engage setpoint
 - engage mode
- Calibrations:
 - XYZ
 - detector
 - coupling
- Image processing:
 - flattening
 - filtering

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Image Quality: Environment





- Environment:
 - Noise: acoustic, electrical
 - Vibration: ground vibration, building vibration

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Practice#1: PFT mode imaging





- In PFT mode, optimize scanning parameters to get high quality image on a LDPE sphere in PS-LDPE sample
- Image the same sample with Tapping mode to compare

Practice#2: Contact mode imaging





• In contact mode, try different probes and parameters to get optimal image quality on PF-TUNA sample

Practice#3: Tapping mode imaging





• In Tapping mode, try different frequency and amplitude settings to image on the flat area of calibration grid, and compare the images



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