Scanning Probe Microscope Training Wenhui Pang



Atomic Force Microscopy 3D Optical Microscopy Tribology Automated AFM Stylus Profilometry Mechanical Testing, Nano Indentation

Bruker Nano Surfaces Division

Innovation with Integrity

Background - Comparison of AFM with BRUKER Other Imaging Modalities

		Optical Microscopy	SEM	TEM	AFM	
Desclutter	XY	200 nm	2 nm	0.1 nm	1 nm	
Kesolution	Z	500 nm	N/A	N/A	0.1 nm	
Sample Preparation		Simple	Moderate Skilled		Simple	
Sample Types		Living or non living	Non living	Non living	Living or non living	
Ambient Atmosphere		Air, Fluid, Vacuum	Vacuum	Vacuum	Air, Fluid, Vacuum	
Field of View		Large	Large enough	Limited	Moderate	
Functionality		Simple	Moderate	Moderate	Advanced	
Speed		Fast	Moderate	Slow	Slow	
Skill Required		Low	Moderate	Advanced	Advanced	
Data Interpretation		Easy	Moderate	Moderate	Complex	

The First SPM in the World





1986 Nobel Prize

G. Binning and H. Rohrer invented the first Scanning Tunneling Microscope in 1981.

STM is the first instrument that can reflect information of material surface in atomic scale.



Data processing

and display

Scanning Tunneling Microscope



was used by Van Vleck in his last publication, the Julian E. Mack Lecture at his Alma Mater, the University of Wisconsin, in 1979, (After B. Bleaney, Contemp. Phys. 25 (1984) 320.)

STM is based on the fact that the tunneling current between a conductive tip and sample is exponentially dependent on their separation.

This can be represented by the equation: I ~ Ve^{-cd}

This technique is typically limited to conductive and semiconducting surfaces

AFM System





Position-Sensitive Photodetectors



The optical system is aligned so that the beam emitted by a diode-laser is focused on the cantilever, and the reflected beam hits the center of a photodetector. Four-section split photodiodes are used as position-sensitive photodetectors (PSPD).



Sum, Vertical and Horizontal





The ΔI_Z value is used as an input parameter in a feedback loop of the atomic force microscope.

 $\Delta I_z = (\Delta I_1 + \Delta I_2) - (\Delta I_3 + \Delta I_4) \quad \Delta I_L = (\Delta I_1 + \Delta I_4) - (\Delta I_2 + \Delta I_3)$ Sum= $\Delta I_1 + \Delta I_2 + \Delta I_3 + \Delta I_4$

Piezoceramic Plate in an External Electric Field



The probe microscope scanners are made of **piezoelectric materials**. Piezoelectric materials change their sizes in an external electric field.

$$u_{ij} = d_{ijk} E_k$$

$$u_{xx} = d_{\parallel} E_{x} \qquad u_{m} = d_{\perp} E_{x}$$

The piezoceramics is polarized polycrystalline material obtained by **powder sintering from crystal ferroelectrics**.





Tubular Piezo-scanner





Z Direction: Change of the internal electrode potential with respect to all external sections results in **lengthening or reduction** of the tube along Z axis.

X, Y Directions: When differential-mode voltage is applied on opposite sections of the external electrode (with respect to the internal electrode) part of the tube reduces in length and increases (where field and polarization directions are opposite). This leads to a **bend** of the tube.



Generally (especially at large control fields) the piezoceramics are characterized by **nonlinear dependence** of the deformation on the field. Thus, deformation of piezoceramics is a complex function of the applied electric field:

$$u_{ij} = u_{ij}(\vec{E})$$

For small control fields the given dependence can be represented in the following way:

$$u_{ij} = d_{ijk}E_k + \alpha_{ijkl}E_kE_l + \dots$$

Typical values of fields E^* , at which nonlinear effects cannot be neglected, are about **100 V/mm**.



Piezoceramics Creep



Piezoceramics creep is a **delay** in the response to **sudden change** of the control electric field value.



The creep results in **appearance of geometrical distortions** in SPM images. Specifically strong influence of the creep occurs, **on initial stages of the scanning process**, or **after a large displacement of the starting point of the scanned area**.

Piezoceramics Hysteresis



Piezoceramics hysteresis is that the piezoceramic deformation depends on the sign of previously applied electric field.



Voltage

To avoid distortions in the SPM images caused by piezoceramics hysteresis, information is stored, in a sample scanning, only while **tracing one of the loop** branches $\Delta Z = f(V)$.



Piezoceramics Hysteresis



Piezoceramics Aging



The sensitivity of piezoelectric materials **decreases exponentially with operation time**. This causes most of the change in the sensitivity to occur **at the beginning of a scanner's life**. Scanners are run approximately 48 hours before they are shipped from the factory to get the scanner past the point where the sensitivity changes dramatically over short periods of time. As the scanner ages, the sensitivity will change less with time, and will eventually get to the point where it very seldom needs recalibrating.



Protection against Acoustic Noise





Acoustic waves directly affect elements of SPM heads, resulting in oscillations of the tip with respect to the sample surface. Various protective enclosures, allowing a sensible reduction of the level of acoustic noise are used to protect the SPM. The most effective protection against acoustic noise is to place the measuring head into a vacuum chamber.

AFM Probes





- An AFM probe has three components: tip, cantilever, and substrate
- There are two different shapes of cantilever: rectangular and triangular
- Key probe parameters: spring constant (k), resonance frequency (f_0) , and tip radius (R)
- For more information, please visit <u>http://www.brukerafmprobes.com</u>

Cantilever Parameters





Spring Constant :

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

Resonance Frequency (without tip mass):

w-width of cantilever

H-tip height

p – cantilever mass per unit-length

$$\rho_{air} = 1.18 kg / m^3$$

$$\eta_{air} = 1.86 \times 10^{-5} \, kg \, / \, m \cdot s$$

- t thickness of cantilever
- f_{g} resonance frequency of cantilever (in Hz)
- ρ density of cantilever (silicon) = 2.33gm/cm³ = 2330kg/m³
- L length of cantilever

E - elastic modulus of

(in the <110> direction)

cantilever = 1.39x10¹¹N/m²

P - mass of tip

 $f_{\theta} = 0.162 \cdot \sqrt{\frac{E}{\rho}} \cdot \frac{t}{L^2} \approx \frac{1}{2\pi} \sqrt{\frac{E}{\rho}} \cdot \frac{t}{L^2}$

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m}}$$

Nitride Probes





A wafer of silicon nitride probes for contact and fluid tapping mode imaging and force measurement. DNP, DNP-S, SNL, DNP-O, ScanAsyst-Air, ScanAsyst-Fluid, ScanAsyst-Fluid+

Silicon Probes





(R)TESP(A), (O)TESPA, OLTESPA, (R)FESP(A) SCM-PIT, SCM-PIC, MESP, DDESP

Factors to Be Considered







Probe Selection Guide — Life Science

Sample		Imaging Environment Probe Family /		Nominal Specifications			Coating s				
	Type Liquid Air Mor		Model	Force Constant (N/m)	Resonant Frequency (kHz)	Radius of Curvature (nm)	Backside	Tip Side	-		
		-	1	c	TESPA	42	320	8	Al	None	
		-	1	lico	RTESPA	40	300	8	A	None	
		-	1	Ś	NCHV-A	42	320	10	Al	None	
	ipids,	~	~		DNP-S	0,06-0,58	18-57	10	Au	None	
	ules teins, I s, etc.)	~	1		MSCT	0.01-0.5	7-120	10	Au	None	
	moleci ds, pro ydrate	~	~	tride	SNL	0.06-0.58	18-57	2	Au	None)
	Bio acia carboh	~	~	con Ni	MSNL	0,01-0,5	7-120	2	Au	None	
	(nucle	~	~	Sili	ScanAsyst- Air	0.2-0.8	45-95	2	AI	None	
	×		-		ScanAsyst- Fluid	0.35-1.4	100-200	20	Au	None	
		~	-		ScanAsyst- Fluid+	0.35-1.4	100-200	2	Au	None	
Cē	<u>0</u>	~	-	con ride	DNP	0.06-0.58	18-57	20	Au	None	7
ien	ů	1	-	Sili Rite	MLCT	0.01-0.5	7-120	20	Au	None	
š		-	~	u	TESPA	42	320	8	Al	None	
Life		-	~	Silico	RTESPA	40	300	8	Al	None	
		-	 ✓ 	0	NCHV-A	42	320	10	Al	None	
		~	-		DNP	0.06-0.58	18-57	20	Au	None	
		× -		DNP-S	0,06-0,58	18-57	10	Au	None		
		1	-		MLCT	0.01-0.5	7-120	20	Au	None	7
	sues	~	-	9	MSCT	0.01-0.5	7-120	10	Au	None	
	Tis	~	-	n Nitrid	SNL	0.06-0.58	18-57	2	Au	None	
		1	-	Silicor	MSNL	0,01-0,5	7-120	2	Au	None	
		~	~		ScanAsyst- Air	0.2-0.8	45-95	2	AI	None	
		~	-		ScanAsyst- Fluid	0.35-1.4	100-200	20	Au	None	7
		~	-		ScanAsyst- Fluid+	0.35-1.4	100-200	2	Au	None	

	AFM Mode							
Probe Attributes	Peak Force/ Scan Asyst	Tapping	Contact	Force Curves	Electrical	Magnetic		
Highest Resolution, Asymmetric Tip		1	-	-	-	-		
Highest Resolution, Symmetric Tip		1	-	-	-	-		
High Resolution, Asymmetric Tip		1	-	-	-	-		
High Resolution, Low Force, Symmetric Tip (sharpened)		~	~	~	-	-		
High Resolution, Lowest Force, Symmetric Tip (sharpened)		~	*	~	-	-		
Ultra-High Resolution, Low Force, Symmetric Tip (extremely sharp)		~	~	~	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)		~	~	~	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-		
High Resolution, Lowest Force, Symmetric Tip (sharpened)	1	-	-	-	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	*	-	-	-	-	-		
Low Force, Symmetric Tip		~	×	~	-	-		
Lowest Force, Symmetric Tip		1	×	~	-	-		
Highest Resolution, Asymmetric Tip		1	-	-	-	-		
Highest Resolution, Symmetric Tip		1	-	-	-	-		
High Resolution, Asymmetric Tip		1	-	-	-	-		
Low Force, Symmetric Tip		1	1	~	-	-		
High Resolution, Low Force, Symmetric Tip (sharpened)		~	~	~	-	-		
Lowest Force, Symmetric Tip		1	1	~	-	-		
High Resolution, Lowest Force, Symmetric Tip (sharpened)		~	~	~	-	-		
Ultra-High Resolution, Low Force, Symmetric Tip (extremely sharp)		~	~	~	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)		1	~	~	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-		
High Resolution, Lowest Force, Symmetric Tip (sharpened)	~	-	-	-	-	-		
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-		



Probe Selection Guide — Material

Sample Type		lma Enviro	ging onment	Probe Family /		Nominal Specifications			Coatings		
		Liquid	Air		Model	Force Constant (N/m)	Resonant Frequency (kHz)	Radius of Curvature (nm)	Backside	Tip Side	-
		•	~		FESP	2,8	75	<10	None	None	
		•	~	LOO O	TESPA	42	320	8	AI	None	
	es		~	Sili	LTESP	48	190	<10	None	None	
	dm	•	~		NCHV-A	42	320	10	AI	None	
	ft Sa	~	~		DNP	0.06-0.58	18-57	20	Au	None	
	s / Sof	~	~		SNL	0.06-0.58	18-57	2	Au	None	3
	ner	•	~	tride	MLCT	0.01-0.5	7-120	20	Au	None	
	Poly	~	~	con Ni	ScanAsyst- Air	0.2-0.8	45-95	2	A	None	
		~		Sills	ScanAsyst- Fluid	0,35-1,4	100-200	20	Au	None	
		~			ScanAsyst- Fluid+	0.35-1.4	100-200	2	Au	None	
als		•	~	Ę	TESPA	42	320	8	A	None	
teri		·	✓	Silico	NCHV-A	42	320	10	A	None	
Mat		Ŀ	 ✓ 	0)	RTESPA	40	300	8	AI	None	
		•	~	E	MESP-RC	2.8	75	25	Ca/Cr	Co/Cr	
		-	~	d Silico	SCM-PIC	0,2	13	20	Pt-Ir	P H r	
	ples		κ.	lodified	SCM-PIT	2.8	75	20	Pt-lr	PHr	
	rd San		~	2	DDESP	42	320	35	Doped Diamond	AI	7
	Hai	~	~		DNP	0.06-0.58	18-57	20	Au	None	
		*	~	ep	SNL	0.06-0.58	18-57	2	Au	None	
		~	~	on Nitri	ScanAsyst- Air	0.2-0.8	45 - 95	2	AI	None	
		~		Silico	ScanAsyst- Fluid	0.35-1.4	100-200	20	Au	None	
		~			ScanAsyst- Fluid+	0,35-1,4	100-200	2	Au	None	

	AFM Mode						
Probe Attributes	Peak Force/ Scan Asyst	Tapping	Contact	Force Curves	Electrical	Magnetic	
High Resolution, Lower Force, Asymmetric Tip		~	-	~	-	-	
Highest Resolution, Asymmetric Tip		1	-	1	-	-	
High Resolution, Long-Lever, Asymmetric Tip		×	-	*	-	-	
High Resolution, Asymmetric Tip		~	-	1	-	-	
Low Force, Symmetric Tip		×	*	×	-	-	
Ultra-High Resolution, Low Force, Symmetric Tip (extremely sharp)		~	*	*	-	-	
Lowest Force, Symmetric Tip		-	~	~	-	-	
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-	
High Resolution, Lowest Force, Symmetric Tip (sharpened)	1	-	-	-	-	-	
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-	
Highest Resolution, Asymmetric Tip		1	-	-	-	-	
High Resolution, Asymmetric Tip		1	-	-	-	-	
Highest Resolution, Symmetric Tip		✓	-	-	-	-	
High Performance, Magnetic Characterization, Asymmetric Tip		~	-	-	~	~	
High Performance, Electrical Characterization, Asymmetric Tip		-	*	-	~	-	
High Performance, Electrical Characterization, Asymmetric Tip		~	-	-	~	-	
Conductive, with Increased Wear Resistance		-	1	-	~	-	
Low Force, Symmetric Tip		1	1	-	-	-	
Ultra-High Resolution, Low Force, Symmetric Tip (extremely sharp)		*	*	-	-	-	
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-	
High Resolution, Lowest Force, Symmetric Tip (sharpened)	~	-	-	-	-	-	
Ultra-High Resolution, Lowest Force, Symmetric Tip (extremely sharp)	~	-	-	-	-	-	

Basic Working Principle



If there is a **sharp enough** and **unique (single valued)** dependence P = P(z) of that parameter on the tip-sample distance, then P can be used in the feedback system (FS) that control the distance between the tip and the sample.



SPM Primary Imaging Modes



$\mathbf{P} = \mathbf{P}(\mathbf{z})$	Working Mode
Tunneling Current i	Scanning Tunneling Microscope (STM)
Cantilever Amplitude A	Tapping Mode AFM™
Cantilever Deflection D	Contact Mode AFM
Cantilever TR Amplitude A _t	Torsional Resonance Mode (TRmode) AFM™
Tip-Sample Force F	PeakForce Tapping AFM™

The Lennard-Jones Potential





Simplified Block Diagram of Contact Mode AFM





Tapping Mode AFM





Tapping Cantilever in Free Air.

Tapping cantilever on sample surface. Note deflection of cantilever and return signal.

Advantages and Disadvantages - Contact BRUKER

Advantages

• Compared with tapping mode under same experimental conditions, the contact mode has higher scan speeds (throughput)

- Rough samples with extreme changes in vertical topography can sometimes be scanned more easily
- Some special applications, such as lithography, SCM, scratch, must be done under contact mode
- Compared with tapping mode, contact mode is a "static" mode, no need to deal with dynamics of cantilever (no tuning needed), feedback control is easier

Disadvantages

- Lateral (shear) forces can distort features in the image
- Forces normal to the tip-sample interaction can be high in air due to capillary forces from the adsorbed fluid layer on the sample surface
- Combination of lateral forces and high normal forces can result in reduced spatial resolution and may damage soft samples (i.e., biological samples, polymers, silicon) due to scraping between the tip and sample

Simplified Block Diagram of Tapping Mode AFM





Advantages and Disadvantages- Tapping BRUKER

Advantages

• Compared with contact mode in air, can achieve higher lateral resolution on most samples (1nm to 5nm)

• Compared with contact mode, tapping mode has lower forces and less damage to soft samples imaged in air

• Lateral forces are virtually eliminated, so there is no scratching

Disadvantages

- Slightly slower scan speed than contact mode AFM
- Need to deal with dynamics of cantilever, feedback loop is harder to adjust
- Cannot be easily operated in vacuum environment
- Fluid operation is difficult
- Tip-sample interaction force is not directly controlled

Peak Force Tapping Control





TESP (42 N/m) on Si, MM8

ScanAsyst Advantages



- Automatic image optimization results in faster, more consistent results, regardless of user skill level
- Direct force control at ultra-low forces helps protect delicate samples and tips from damage
- Elimination of cantilever tuning, setpoint adjustment, and gain optimization makes even fluid imaging simple



Secondary Imaging Modes



Derivation of Contact Mode

LFM cAFM TUNA PFM SCM SSRM SSRM

Derivation of TRmode™

TR cAFM TR TUNA **Derivation of Tapping Mode™**

Phase Imaging™ EFM MFM TP-KPFM

Derivation of PeakForce Tapping

ScanAsyst™ PeakForce QNM™ PeakForce TUNATM PeakForce SSRM PeakForce KPFM



Non-Imaging Modes – Force Curve



Surface Modification



• Surface Modification Techniques: Nanolithography, Nanoindentation, Nanoscratching, Nanomanipulation











Basic Components of SPM - Dimension Edge





Nanodrive Controller





Stage System





Dimension Stargate Head





Dimension SPM Probe Holder





Standard



Non-Magnetic



STM



Fluid



SCM



CAFM, TUNA

Resolution Issues



Lateral Resolution

Tip Shape: **The radius of curvature of the end of the tip will determine the highest lateral resolution** obtainable with a specific tip. The sidewall angles of the tip will also determine its ability to probe high aspect ratio features.

Pixelization

Vertical Resolution

Scanner

Z Limit

Noise



Tip-Sample Convolution











Tip Shape Issues



The smaller the radius of curvature, the smaller the feature that can be resolved. A sharper tip will be able to laterally resolve smaller features than a dull tip with a larger radius of curvature. The accumulation of debris on the end of the tip can also dull the tip and result in image distortion.



Probe Limited Resolution

Dirty Probe

Note: A dull or dirty tip **may not affect** the measurement of the **vertical dimensions** of these samples.





Dull or dirty tip



Double or multiple tips

Tip Sidewall Image



Another tip artifact occurs on very tall samples or samples where the slope of the features is greater than the slope of the tip. This causes the tip sidewall to interact with the sample instead of the tip apex. The typical appearance is that of an almost linear ramp-like artifact around the feature, as shown below:



Tip/Sample Contamination



Check is there any dust or debris on the sample top surface or backside. If yes, clean it as needed. If the sample is hydrophilic and exposed in ambient air for long time, if there is any soft or loose residue remain on the sample after sample preparation, which can easily contaminate the tip. If the sample is not in good condition, and hard to recover, get a fresh clean sample.



Contamination from tip or sample surface

Tapping Mode Artifacts: Not Tracking



In tapping mode, if cannot track surface, **decrease the Amplitude setpoint**, **decrease the scan rate or increase the I gain and P gain**.



Insufficient tapping force An excessively fast scan rate Gain values set too low

Tapping Mode Artifacts: High Frequency Operation





In tapping mode, decrease the Drive Frequency.

Optical Interference





Interference between the incident and reflected light from the sample surface can produce a sinusoidal pattern on the image with a period typically ranging between $1.5 - 2.5 \mu m$.

It can usually be reduced or eliminated by adjusting the laser alignment so that more light reflects off the back of the cantilever and less light reflects off the sample surface, or by using a cantilever with a more reflective coating (MESP, TESPA).

Bow





Second order bow: the arch-shaped bow



Third order bow: the arch-shaped bow

Technical and Application Support



Customer Care Center Support

- ✓ 400 Telephone Support: 400-890-5666
- ✓ Team Viewer Remote Control
- ✓ Email Support: support.bns.cn@bruker.com
- ✓ Free Test/Repair service for warranty/service contract customers
- ✓ Free Training service for warranty/service contract customers
- ✓ 6 big basic training classes every year (2 in Beijing, 3 in Shanghai, 1 in Guangzhou)
- \checkmark 6 big advanced training classes every year in Beijing

Field Support

- ✓ Free Field Service for warranty/service contract customers
- ✓ Billable Support for out of warranty customers
- ✓Return Visit

> Web Support

✓ <u>http://nanoscaleworld.bruker-axs.com</u>, <u>http://www.brukersupport.com</u>







THANK YOU FOR YOUR ATTENTION!







Small Tip Big Science

