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 Other Imaging Modalities

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: $I \sim Ve^{-cd}$

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

 $\Delta I_z = (\Delta I_1 + \Delta I_2) - (\Delta I_3 + \Delta I_4) \quad \Delta I_L = (\Delta I_1 + \Delta I_4) - (\Delta I_2 + \Delta I_3)$ $Sum = AI_1 + AI_2 + AI_3 + AI_4$

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.

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

$$
u_{xx} = d_{\parallel} E_x \qquad u_{rr} = d_{\perp} E_x
$$

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.

Piezoceramics Nonlinearity

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:

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

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 http://www.brukerafmprobes.com

Cantilever Parameters

Spring Constant:

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

Resonance Frequency (without tip mass):

 w – width of cantilever

 H – tip height

 p – cantilever mass per unit-length

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

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

- t thickness of cantilever
- f_o resonance frequency
of cantilever (in Hz)

 ρ - density of cantilever $(silicon) = 2.33gm/cm³ =$ 2330 kg/m³

 L – length of cantilever

 E – elastic modulus of

(in the ≤ 110 direction)

cantilever = $1.39x10^{11}N/m^2$

 P – mass of tip

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

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

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

Probe Selection Guide — Material

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

The Lennard-Jones Potential

Bruker Confidential Information

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

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

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 SThM

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 Mon-Magnetic STM

Fluid SCM 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 $1.5 - 2.5$ μ 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

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

Field Support

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

Web Support

- http://nanoscaleworld.bruker-axs.com, http://www.brukersupport.com
- \sqrt{T} Training Webinars

THANK YOU FOR YOUR ATTENTION!

Small Tip Big Science

