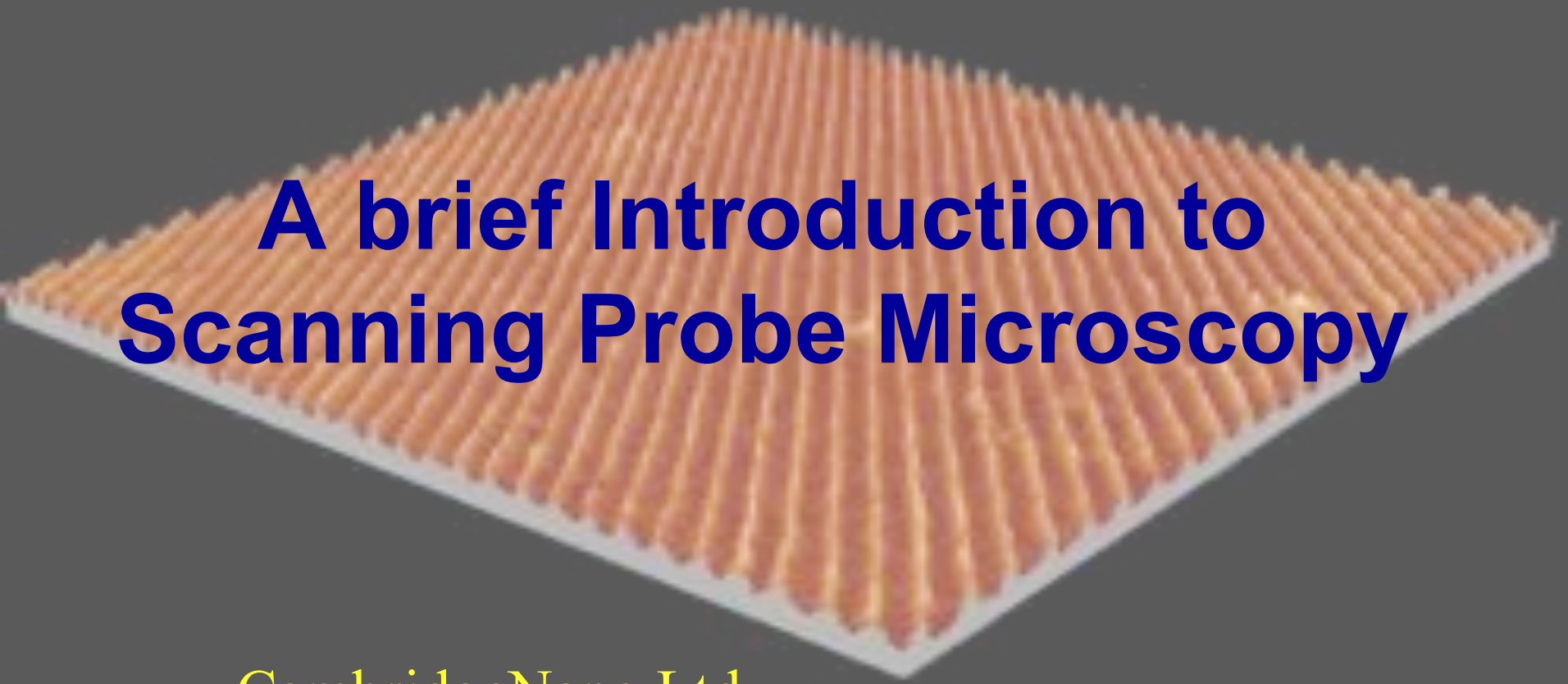
CambridgeNano

Showing You the World

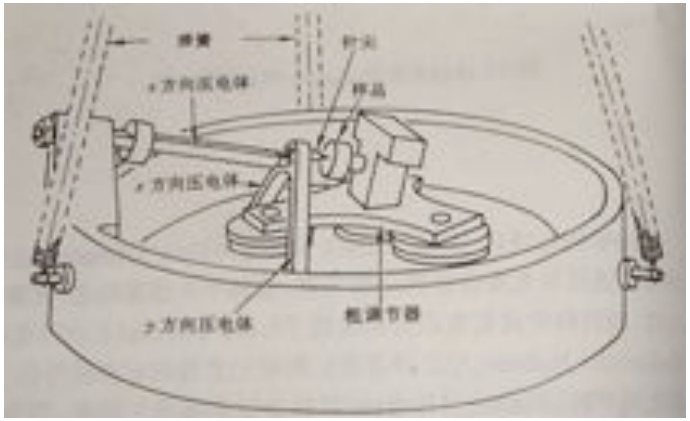


A brief Introduction to Scanning Probe Microscopy

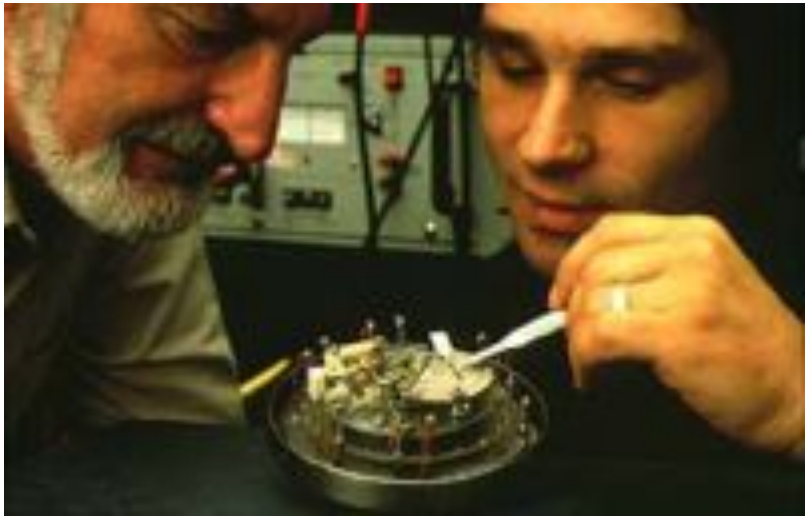
CambridgeNano Ltd.
www.cambridgenano.co.uk

A brief history of Scanning Probe Microscopy

- The first Scanning Tunnelling Microscope (STM) was invented by Binning and Rohrer of IBM in 1981, Zurich, Switzerland.
- On 1986, Binning and Rohrer were awarded the Nobel Prize in Physics.
- The Atomic Force Microscope (AFM) was invented in 1986.



Design of the first STM



Binnig (R) and Rohrer (L) with the first STM

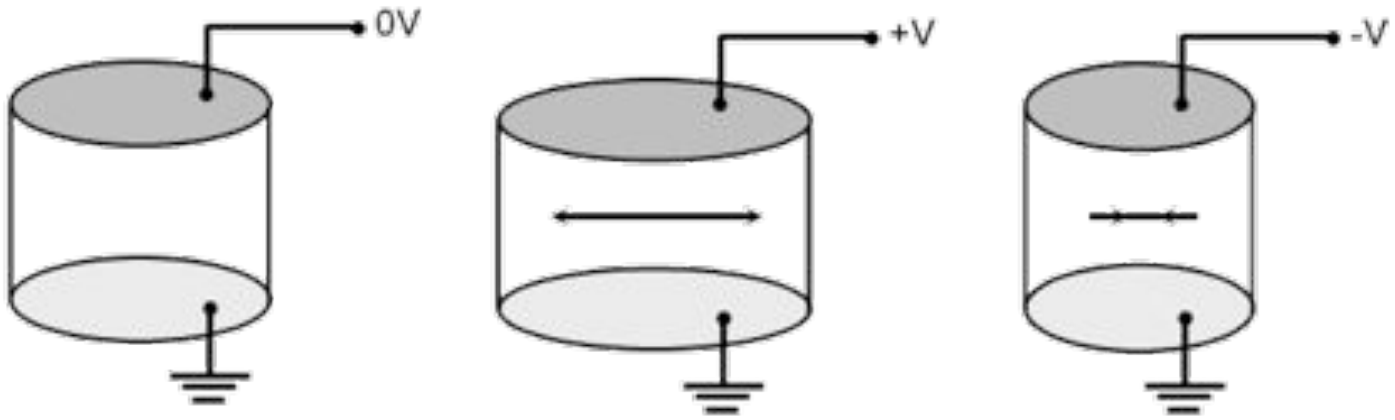
The first AFM

The Scanning Probe Microscope family

SPM: forms images of surfaces using a physical probe that scans the specimen. An image of the surface is obtained by mechanically moving the probe in a raster scan of the specimen, line by line, and recording the probe-surface interaction as a function of position.

- Scanning Tunneling Microscope (STM)
- Atomic Force Microscope (AFM)
  - Contact Mode AFM
  - Dynamic (intermittent-contact) Mode AFM
  - Phase Imaging
  - Lift Mode
- Lateral Force Microscope (LFM)
- Magnetic Force Microscope (MFM)
- Electric Force Microscope (EFM)
- ……

Piezoelectric effect and Scanners

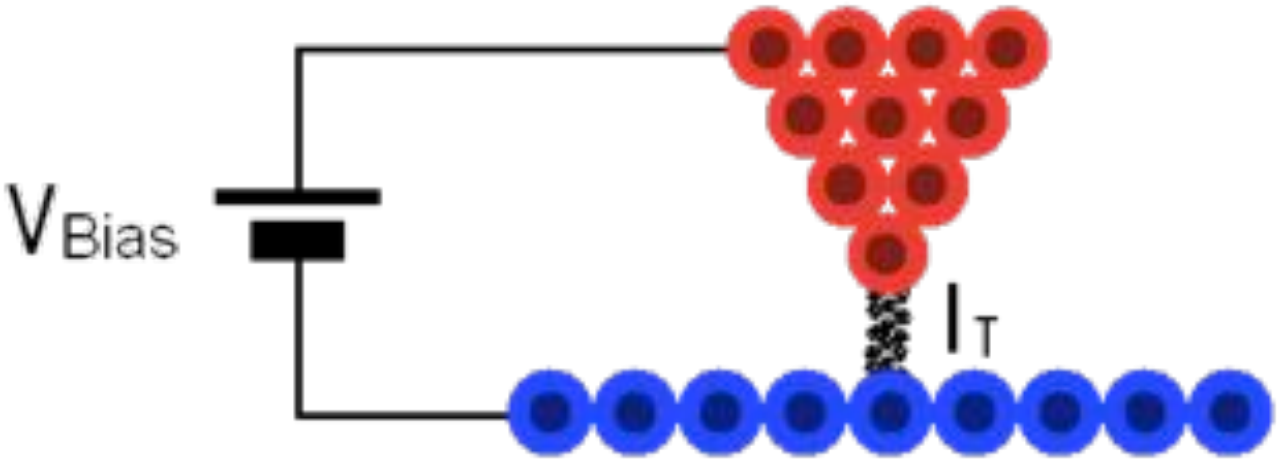


Length changes according to the applied voltage

Scanners are made of piezo materials:

- X and Y voltage: scanner moves horizontally. (for scanning)
- Y voltage: scanner moves vertically. (for topography)

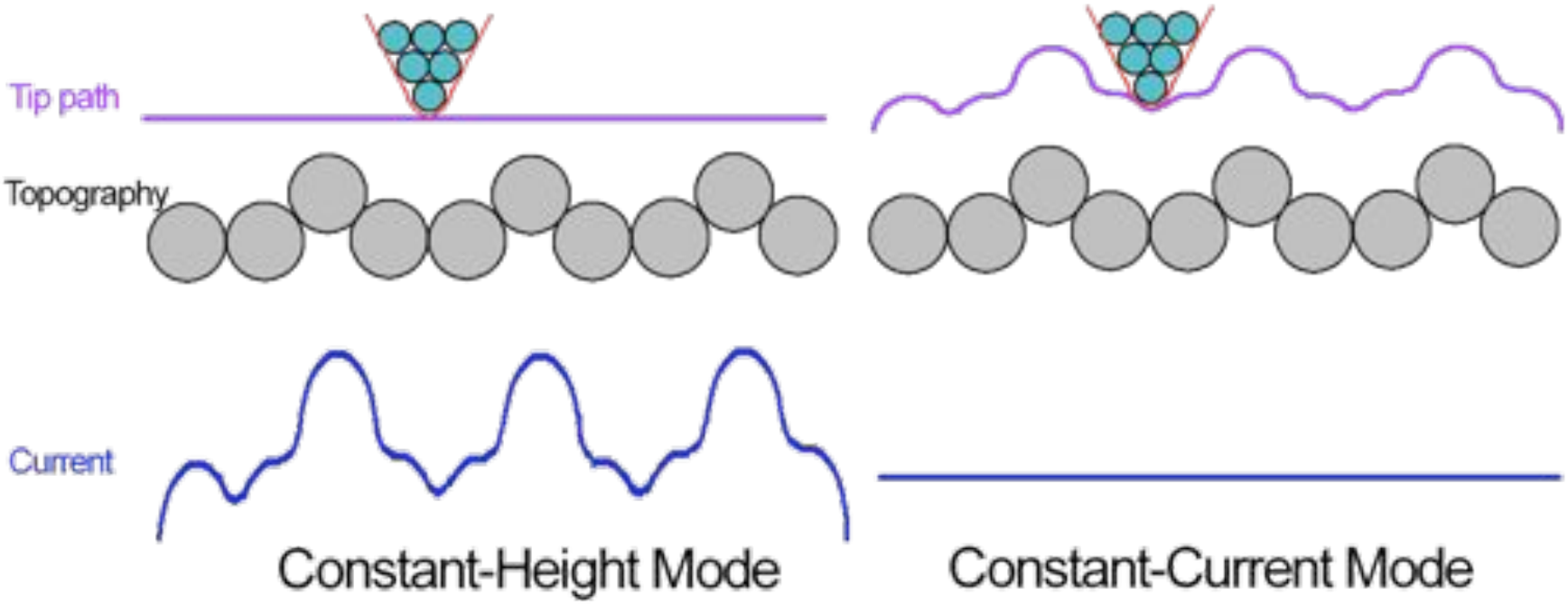
Quantum tunneling and STM



Tunneling Effect: $I_T \sim V_{Bias} e^{-c \cdot S}$

- IT: Tunneling Current
- VBias: Constant Bias voltage applied to tip and sample
- C: Constant
- S: Distance between tip and sample

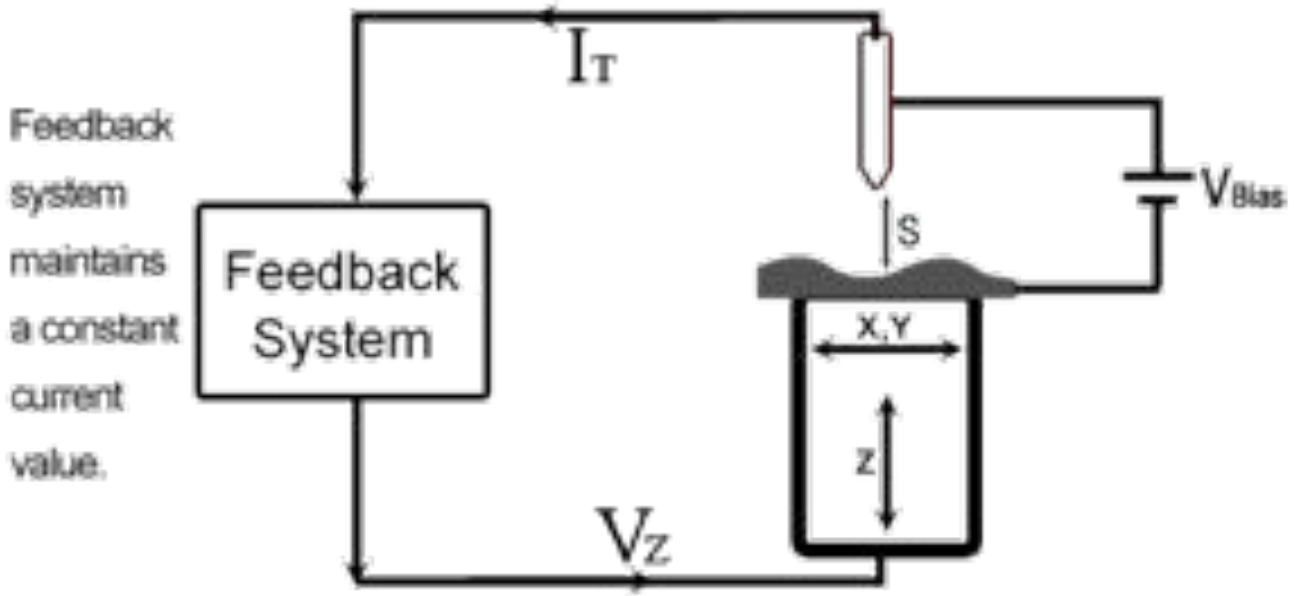
Operation modes of STM



Constant-Height mode: No feed-back control.

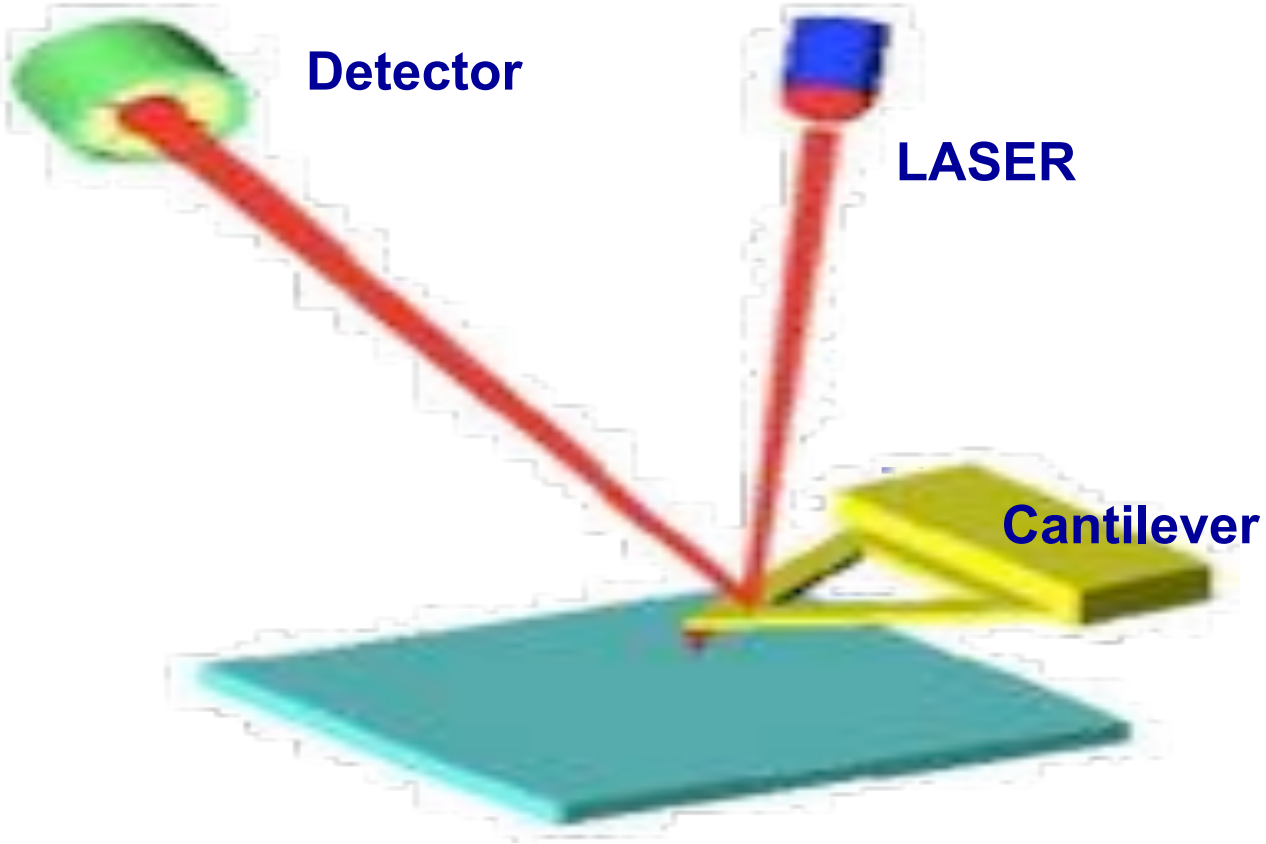
Constant-Current mode: Feed-back control activated. Tip path equals the sample topography.

STM System



- Tip approaches to sample, current occurs.
- Vz is controlled by feedback system to maintain a constant current which is called Setpoint.
- V(x,y) of each scanning point (x,y) is recorded.
- Sample topography T(x,y) can be calculated by V(x,y).

Atomic Force Microscope



- There is a force between the tip and the sample.
- This causes the cantilever to deflect.
- The deflection is measured by a Laser and detector (photodiode).

AFM- Contact Mode

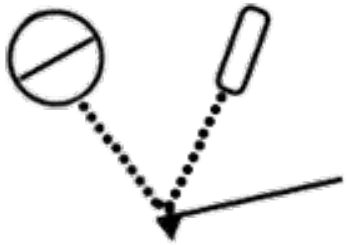
No force between tip and sample, no cantilever deflection.

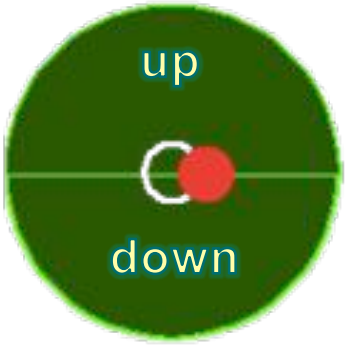
Repulsion between tip and sample, cantilever deflects upwards.

Repulsion between tip and sample, cantilever deflects downwards.

- X: Deflection of cantilever;
- k: Force constant of cantilever;
- F=kx

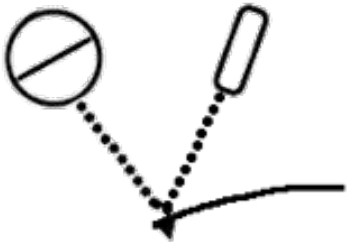
Cantilever Deflection on Detector by Laser





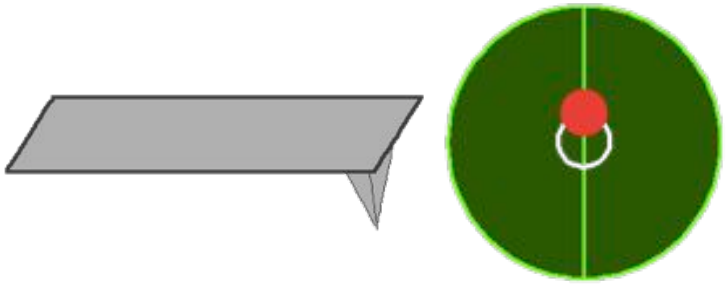
No deflection: Up-Down=0

Upwards: Up-Down>0

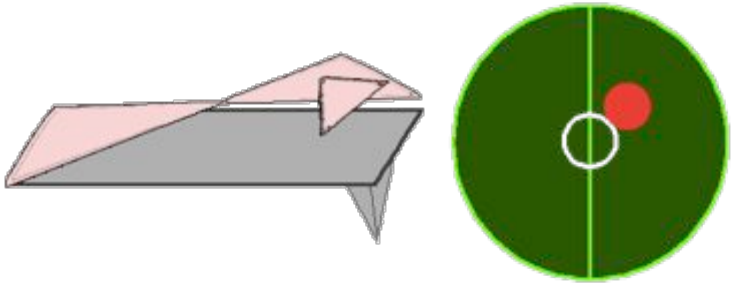


Downwards: Up-Down<0

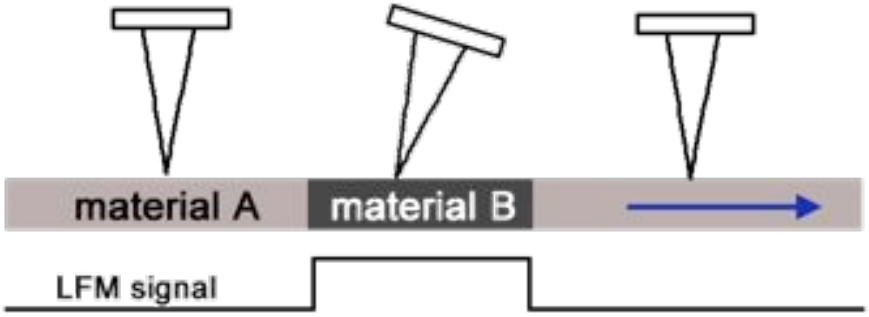
Lateral deflection of cantilever

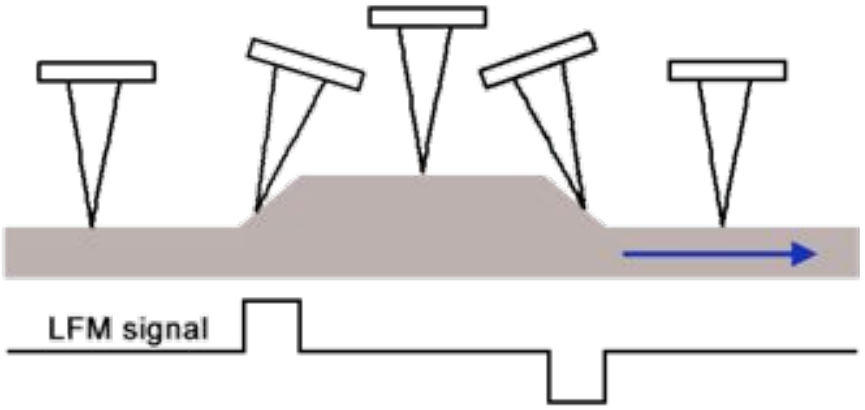


No friction: Left-Right=0

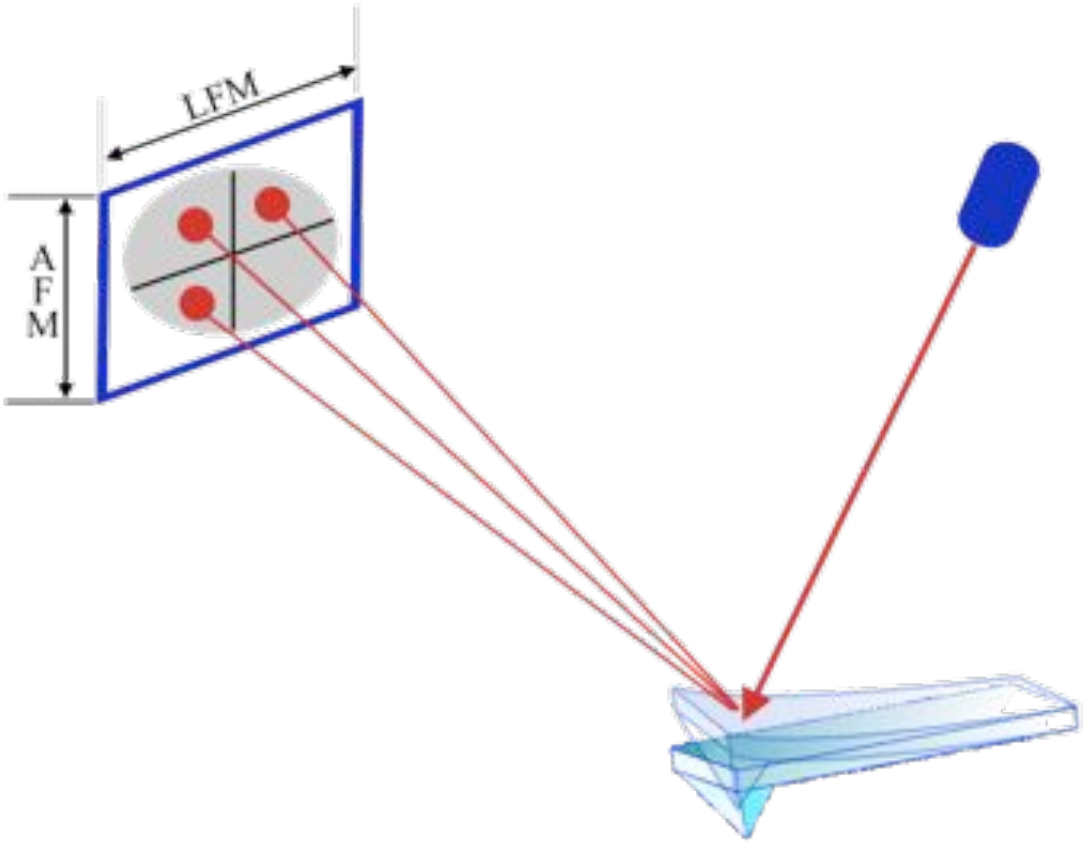


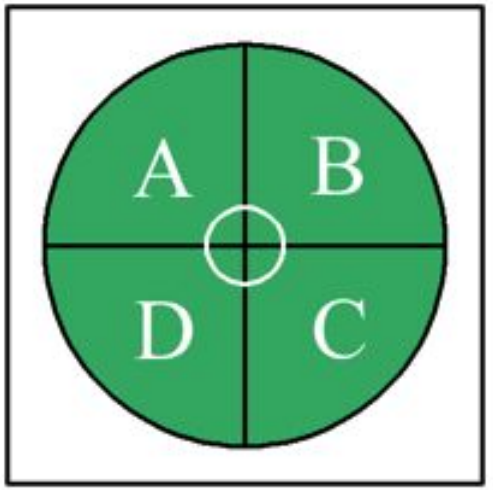
Friction: Left-Right≠0





AFM and LFM

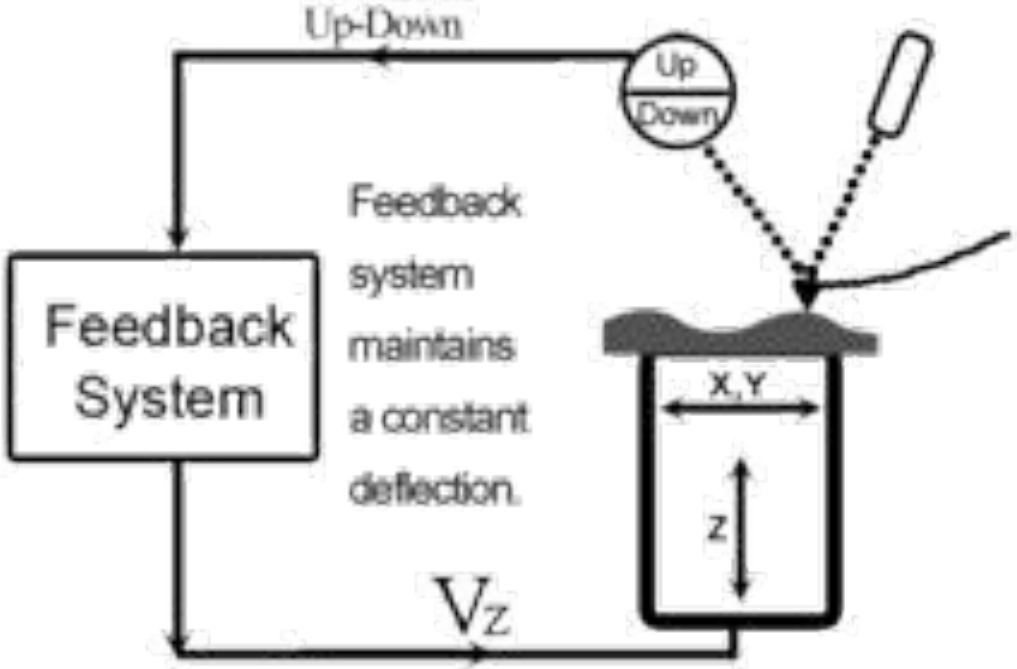




(A+B)-(C+D)：AFM signal

(A+D)-(B+C)：LFM signal

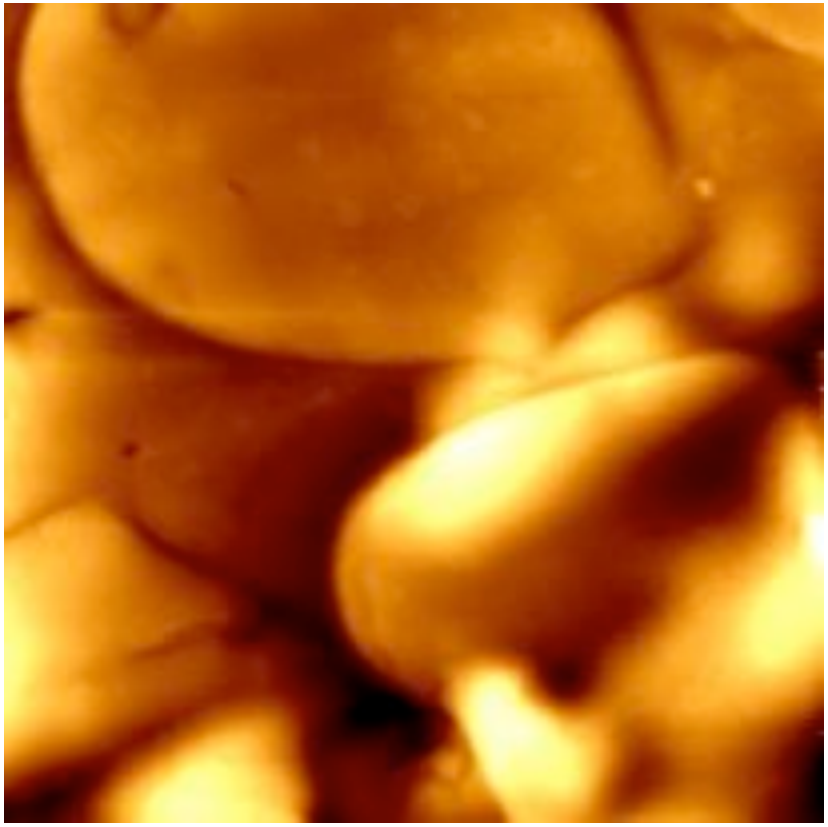
Contact Mode AFM

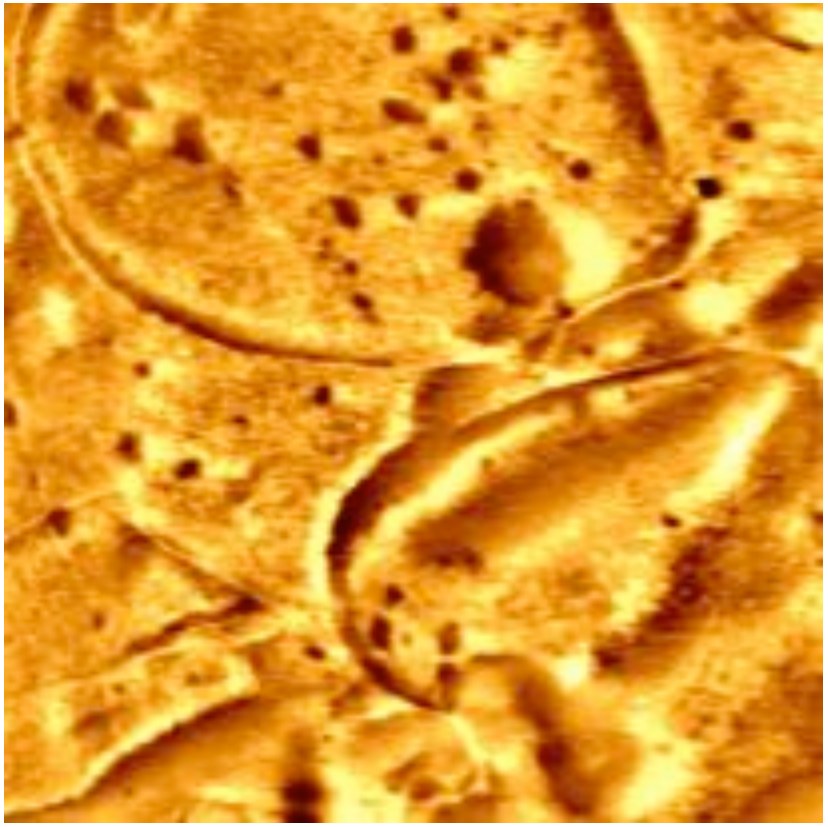


- The tip comes into contact with the sample, and deflects upwards.
- Up-Down Signal of the detector changes.
- Vz is controlled by the feedback system to maintain a constant Up-Down signal, which is called the “Setpoint”.
- V(x,y) of each scanning point (x,y) is recorded.
- Sample topography T(x,y) can be calculated by V(x,y).

Samples of AFM and LFM

Blood Cells (12μm)

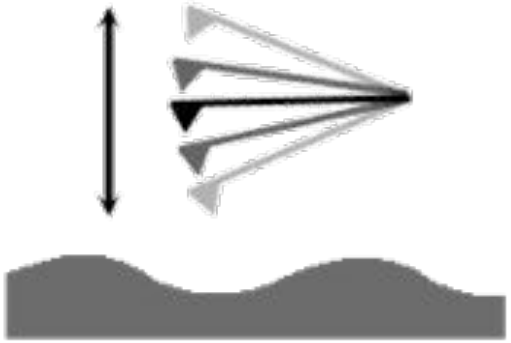


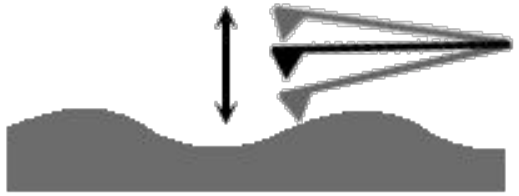


Topography **LFM**

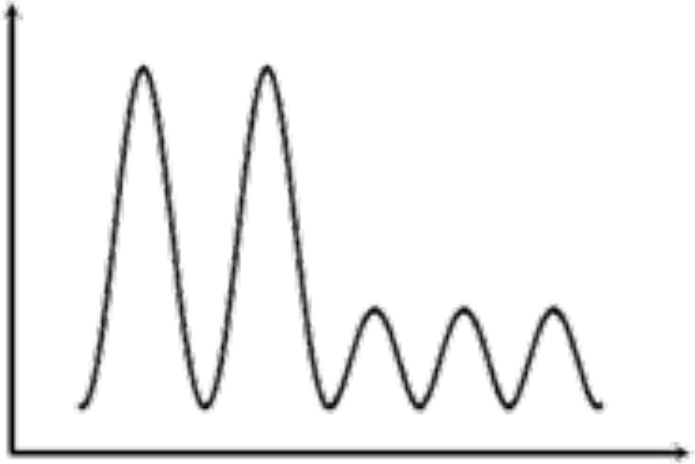
Low friction parts can be observed on LFM image.

AFM- dynamic Mode





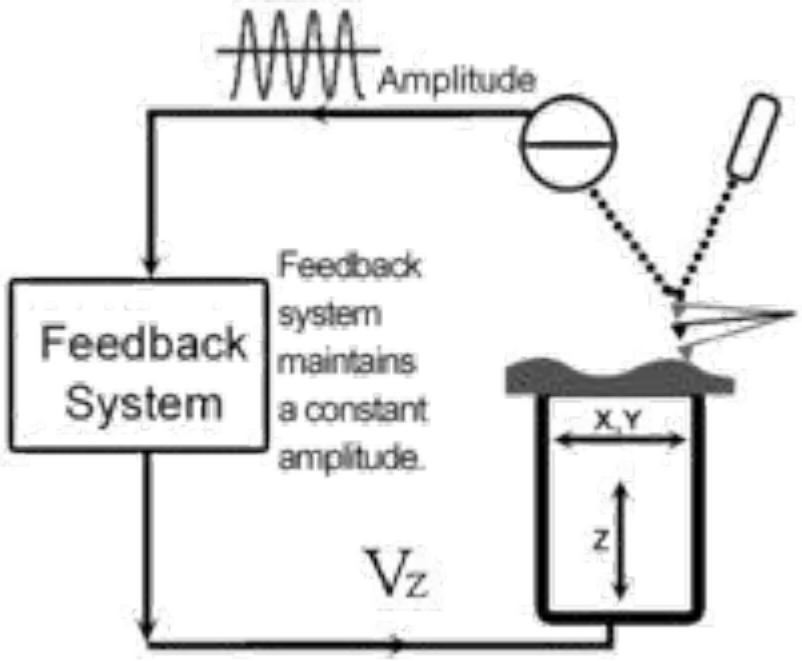
Cantilever is vibrating normal to the surface.



Tip moves close to sample, amplitude is reduced and phase shifts.

Changes in topography cause changes in Amplitude and phase. The feedback loop alters the tip-sample distance to maintain a constant amplitude.

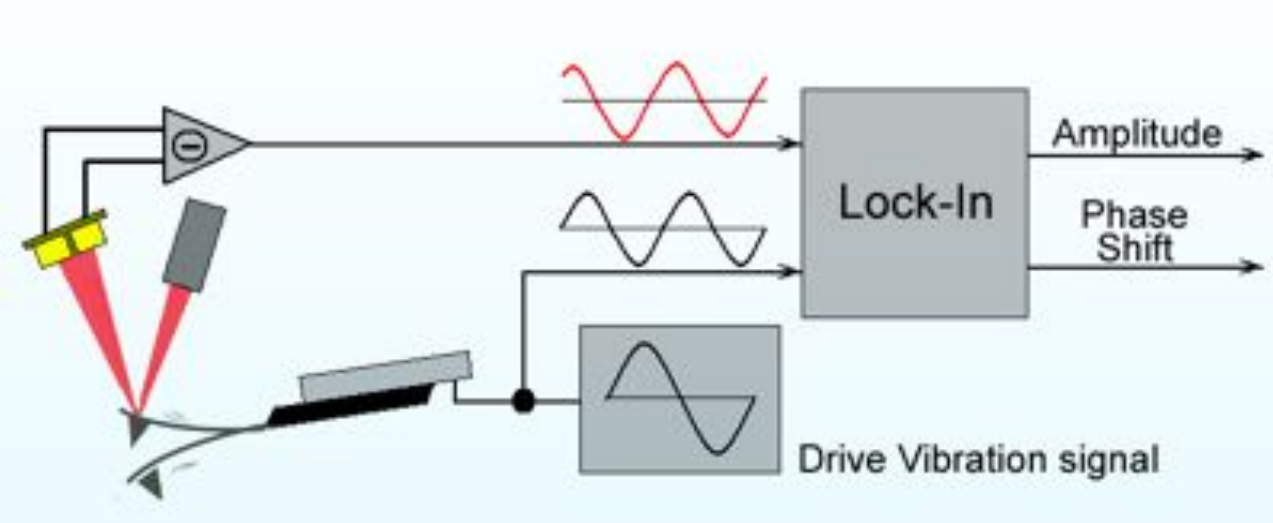
Dynamic Mode in more detail

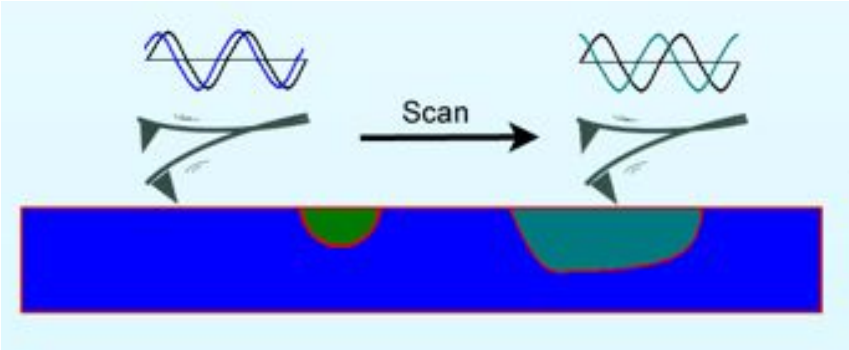


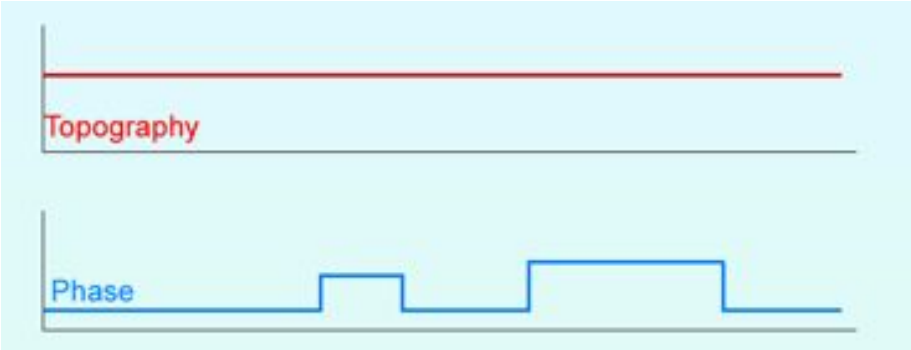
- An internal oscillator drives the cantilever via a small piezo plate.
- The oscillation amplitude is reduced when tip taps the sample.
- Vz is controlled by the feedback system to maintain a constant Up-Down signal which is called the “Setpoint”.
- V(x,y) of each scanning point (x,y) is recorded.
- Sample topography T(x,y) can be calculated by V(x,y).

Phase Imaging

- As the tip scans over the sample surface, changes in the force gradient experienced by the tip (which have many possible origins, including stray electric & magnetic fields, and differences in the mechanical properties of the sample) cause the frequency and phase of the oscillating cantilever to shift. The phase image is often collected as it has higher resolution than the topography image, but it rarely leads to quantitative information, apart from a few select cases.



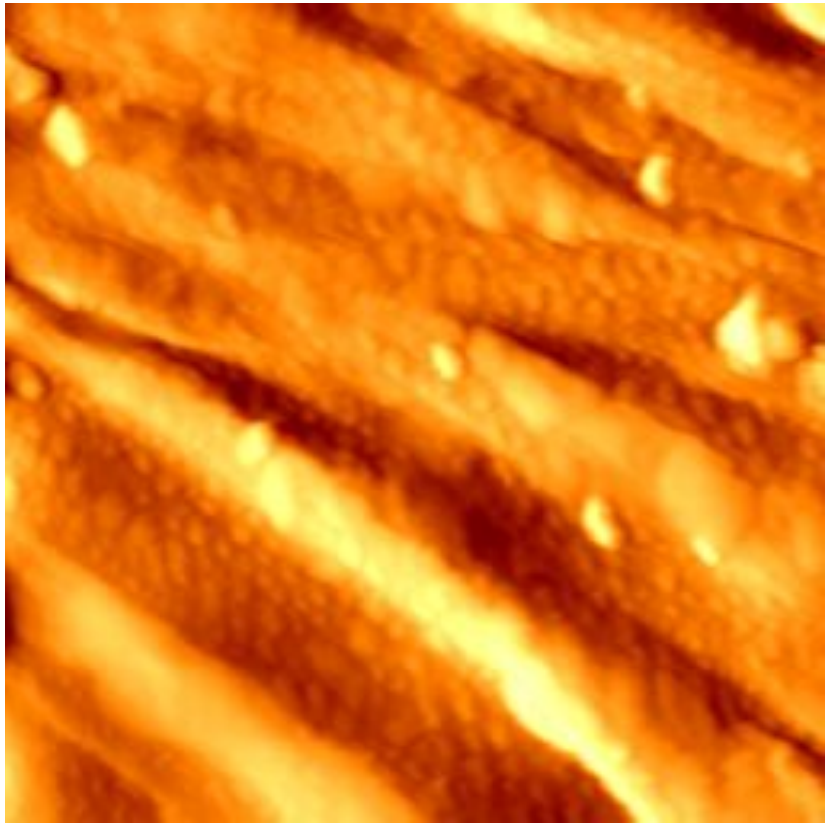




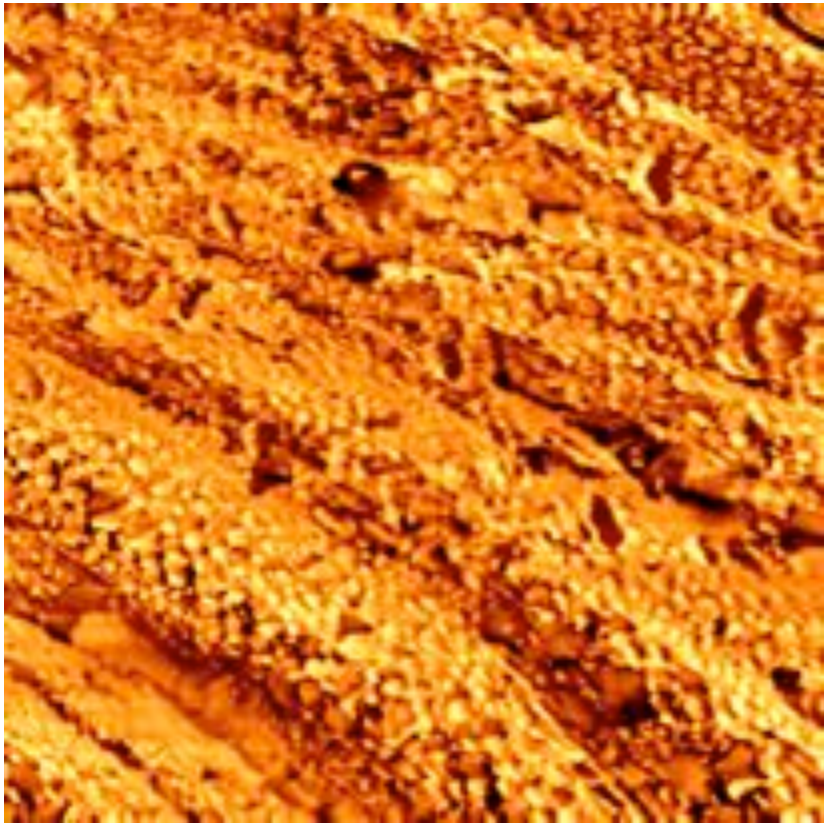
The relative phase of the oscillator and the cantilever depends on local sample properties

Example of dynamic Mode

Organic Film (9.38μm area)



Topography

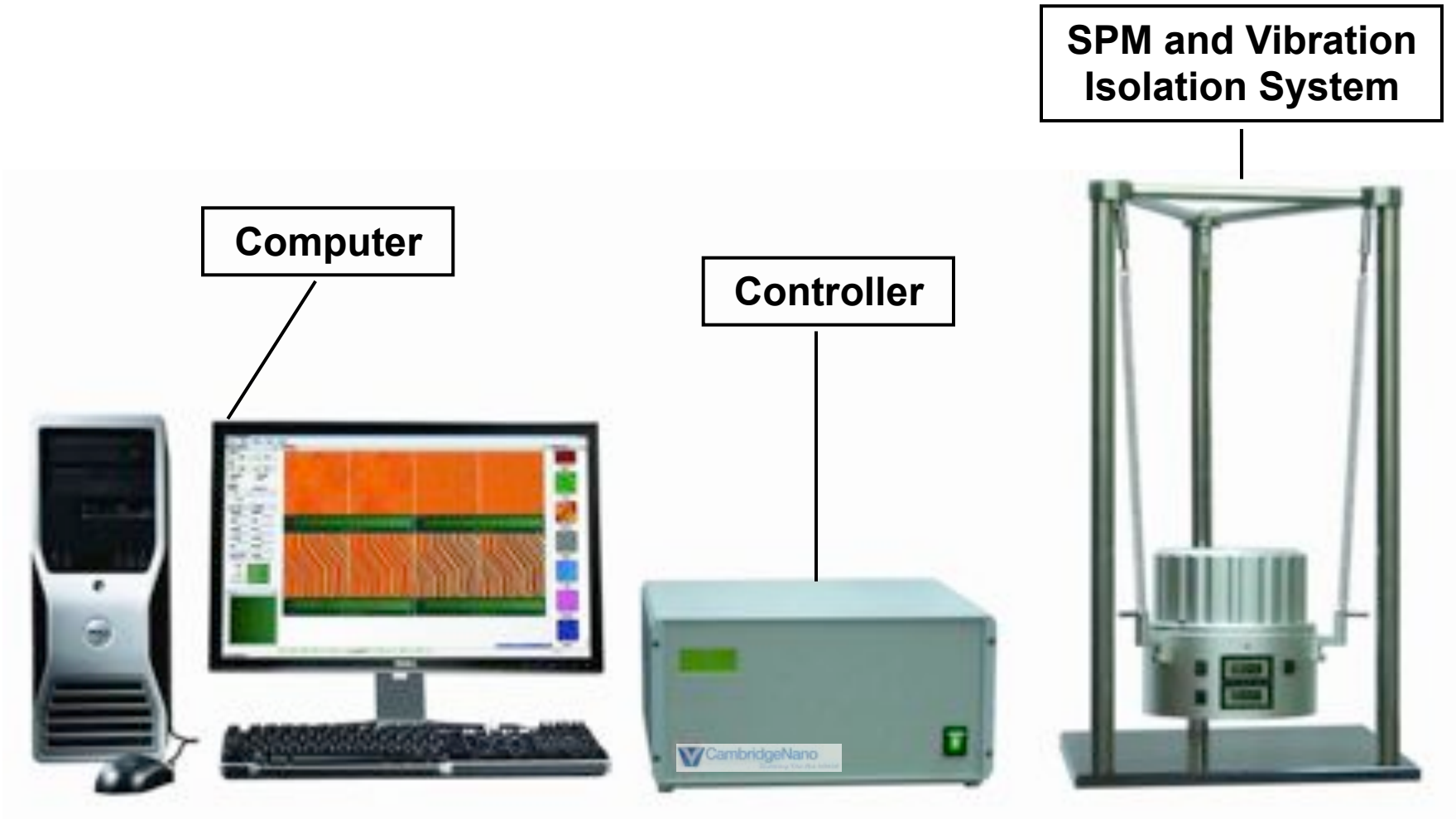


Phase Image

How to choose between Contact and dynamic Mode

	Contact Mode	Tapping Mode
Scan Speed	Higher	Lower
Lateral Forces	Yes	No
Soft Sample	Unsuited	√
Unstable Sample	Unsuited	√

The CN6000 SPM System



CN6000 parts

SPM Head

SPM Base

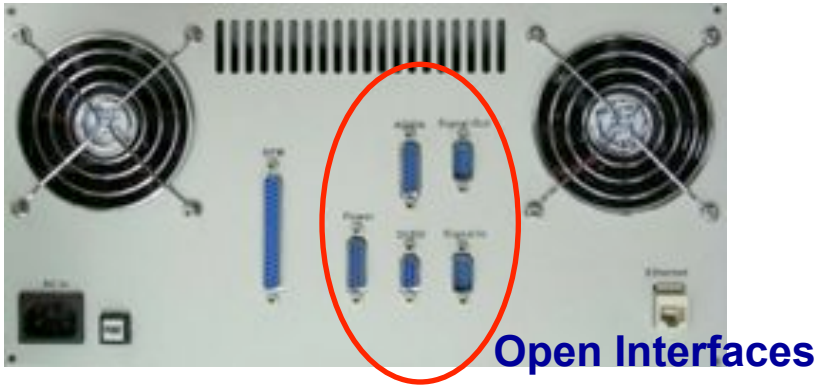
Interchangeable Scanners

STM Tipholder

AFM Tipholder

The Controller

Front panel



Rear panel

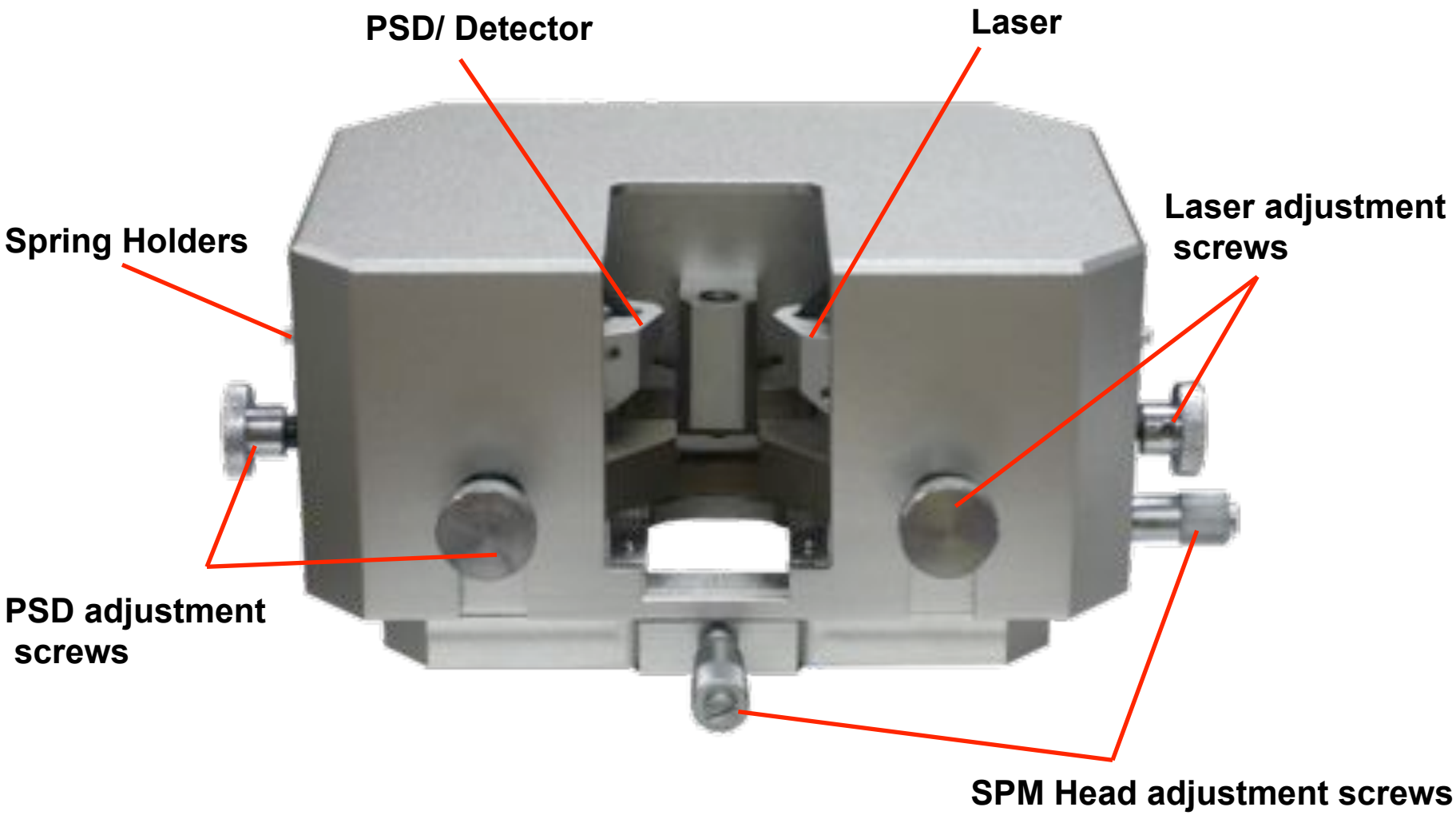
Line 1: Serial Number

Line 2: Controller software version and MAC address

Line 3: Data incoming and outgoing

Line 4: System status

SPM Head


PSD/ Detector
Laser
Spring Holders
Laser adjustment screws
PSD adjustment screws
SPM Head adjustment screws

SPM Base

Scanners

Warning

- **Storage**

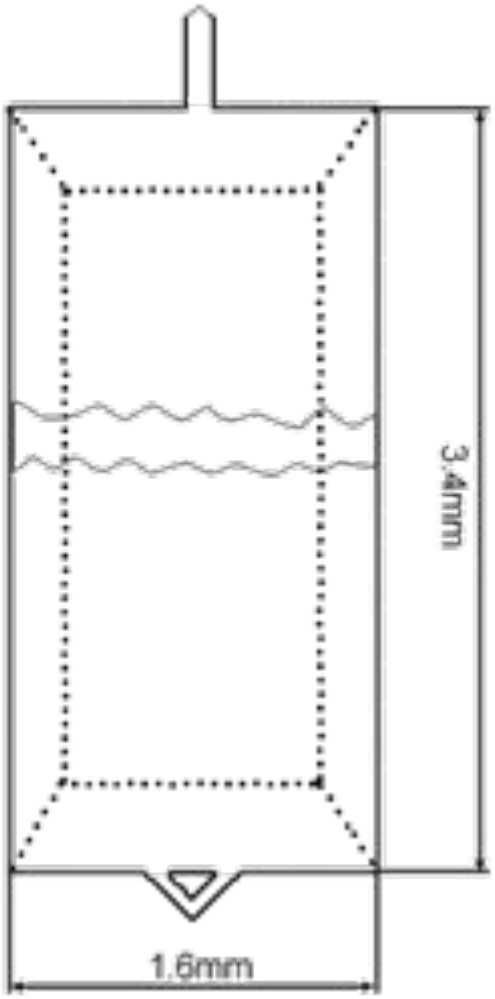
  Scanners must be kept in a sealed box with desiccant.

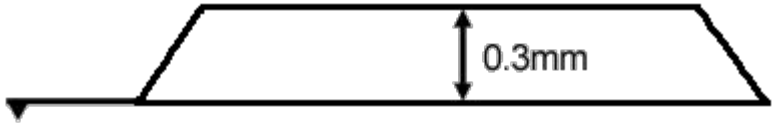
  Be very careful when installing or removing the scanner.

- **Use**

Cover must be removed before use.

Probes for AFM





Slope on probe's edge

Material: Silicon

Shape: Triangle or rectangle

Coating: Al on the backside, gold or Pt

Geometrical Parameters:
Length, Width and Thickness

Force Constant and Frequency

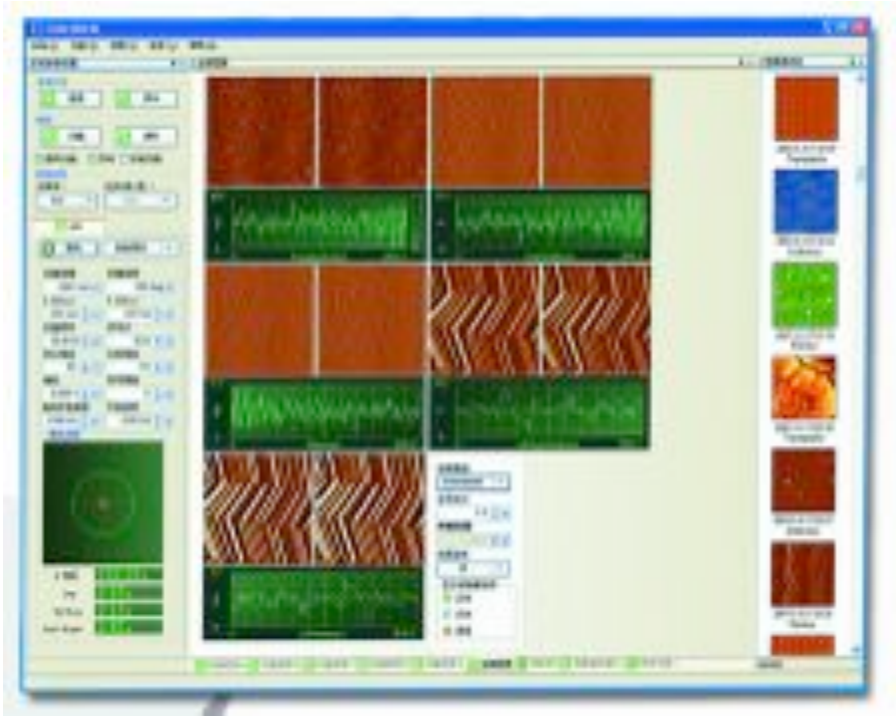
Tip: tip geometries, tip height…

Difference between probes for contact and dynamic mode

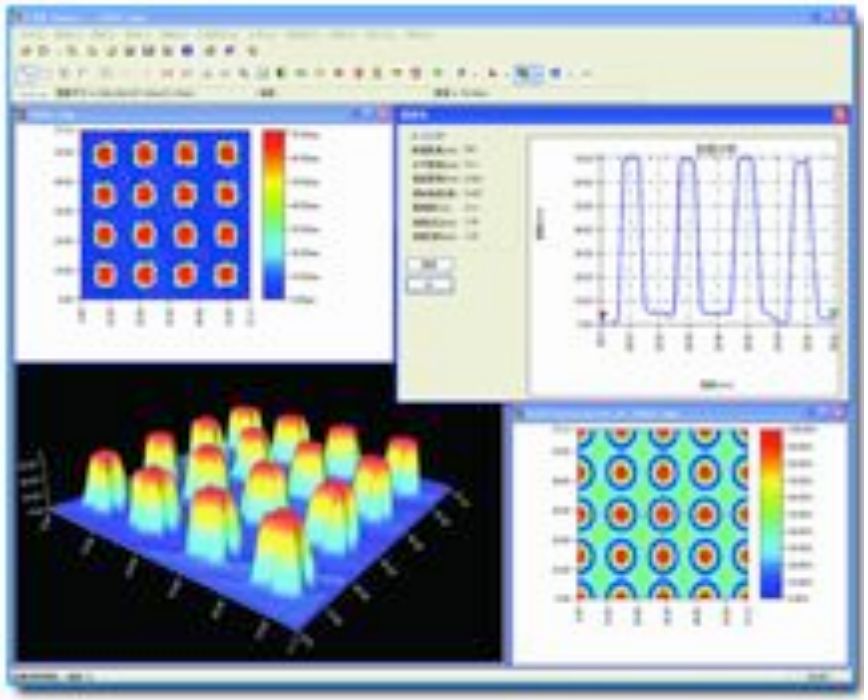
	Contact Mode	Dynamic Mode
Length	450μm	125μm
Width	50μm	30μm
Thickness	2μm	4μm
Force Constant	0.2N/m	40N/m
Frequency	13kHz	300kHz

Software

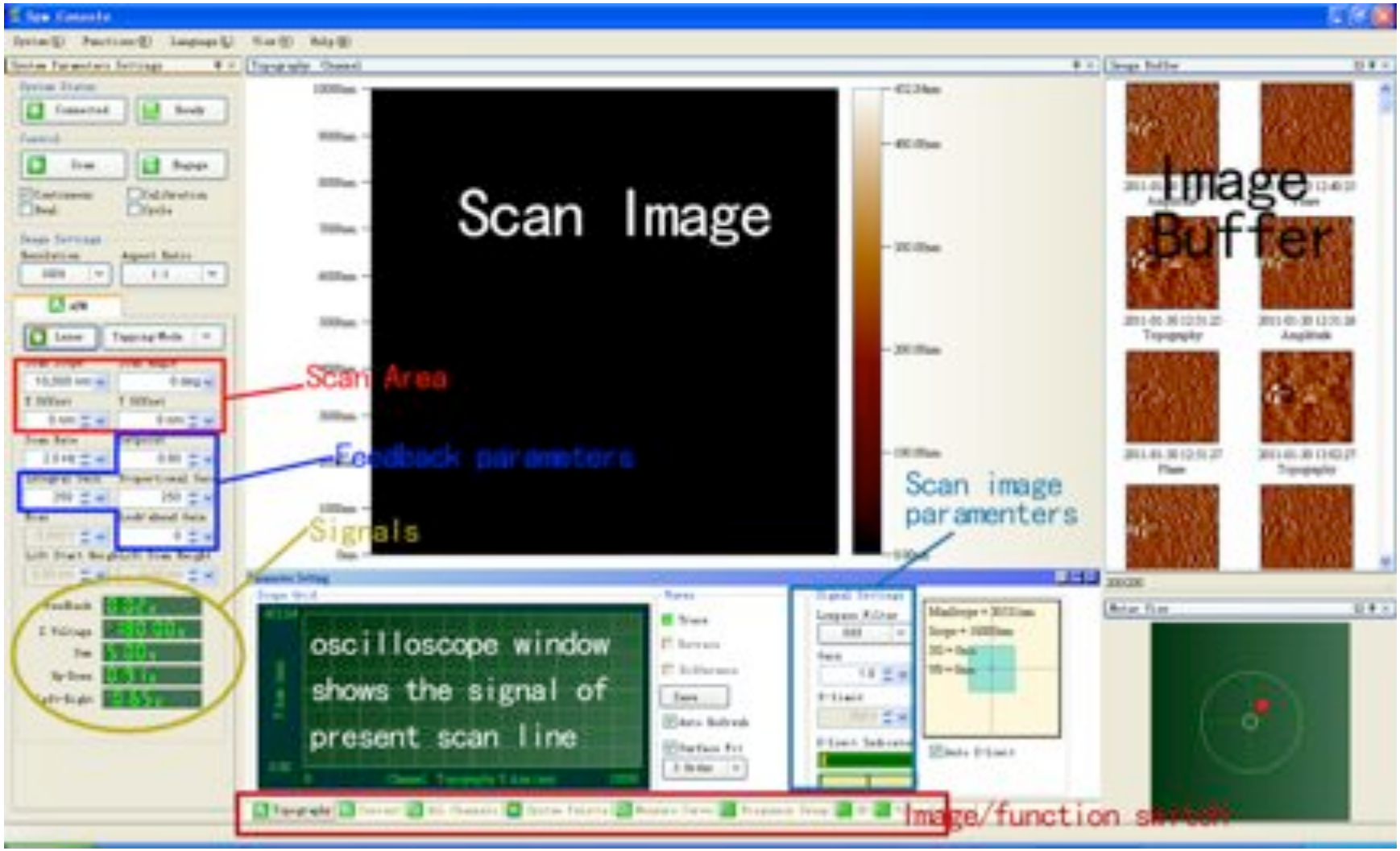
SPM control software



Imager software for post-processing

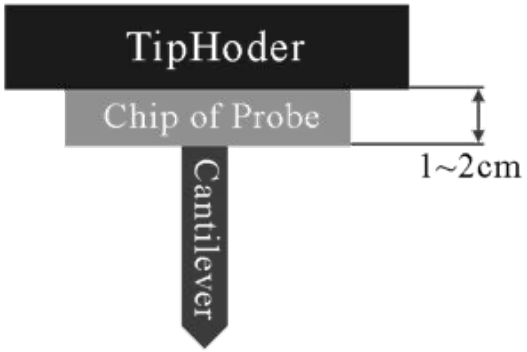


SPM control software



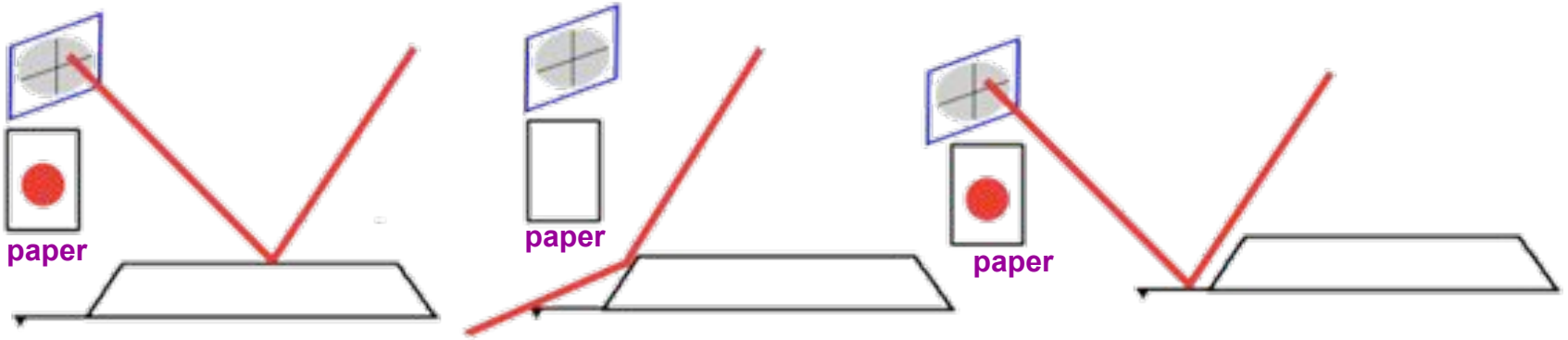
Basic Operation of AFM- Probe installation

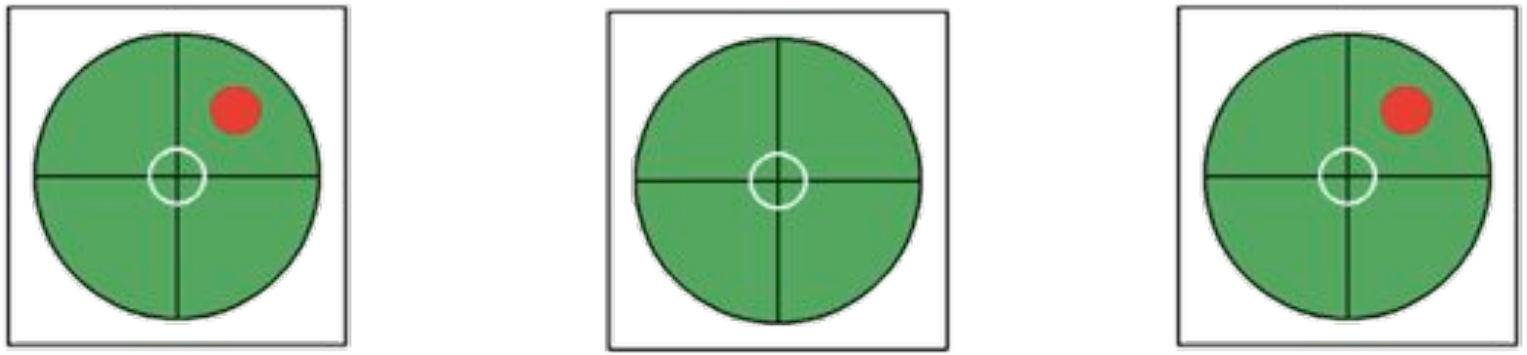
- Gently and evenly lift the spring clip with two fingers
- Insert the probe chip using tweezers



Basic Operation of AFM- Laser Alignment

This procedure is greatly enhanced using the optical microscope to view the cantilever

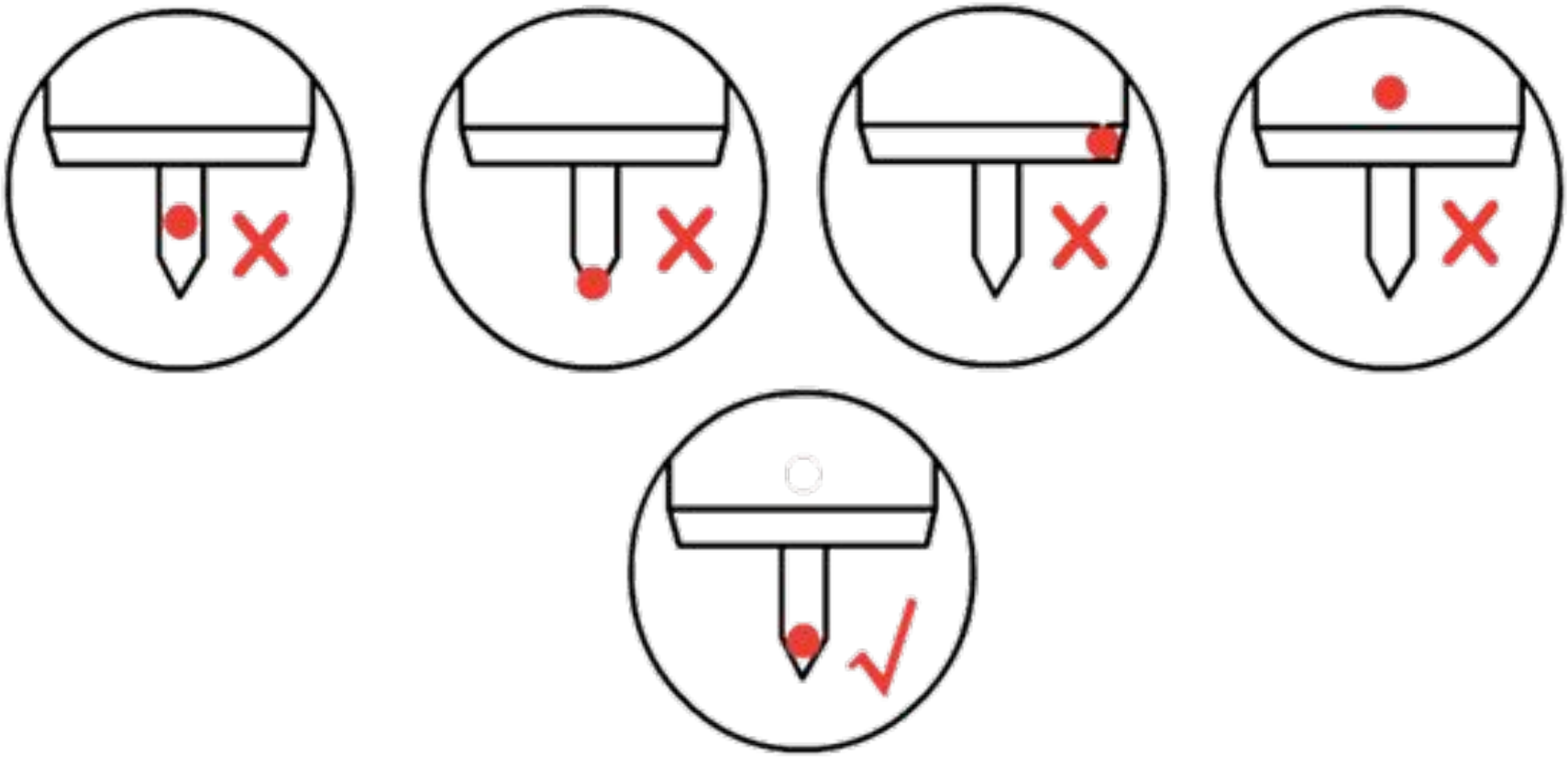




Laser on the chip | Laser on the slope | Laser on cantilever

Basic Operation of AFM- Laser Alignment

Laser must be on the backside of the TIP before engaging with the sample.



Basic Operation of AFM- Contact Mode

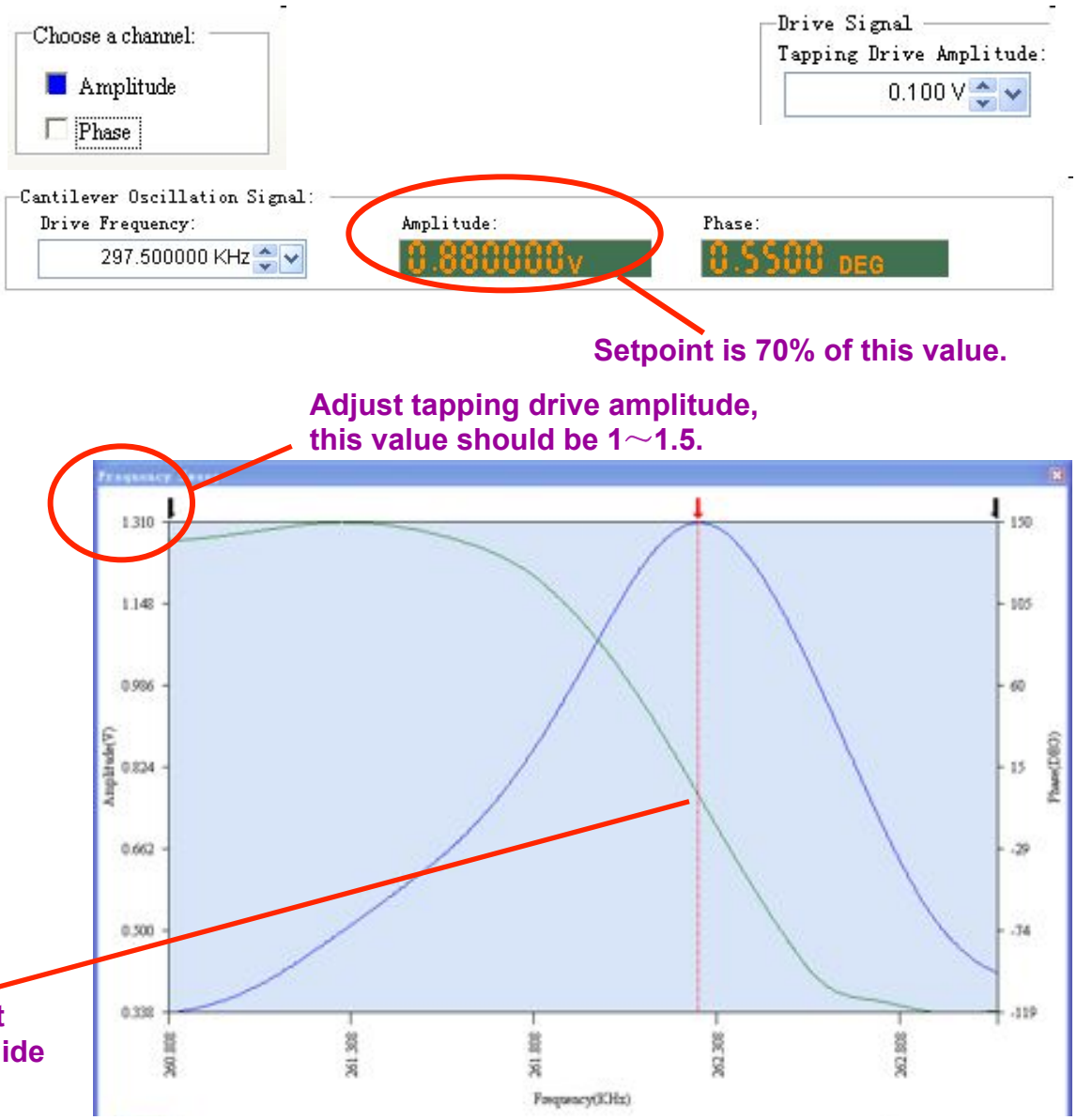
- Setpoint must be properly set before engaging:

  Setpoint: 0.1～0.3.

- Proportional gain and Integral gain must be properly set before engage: ~200

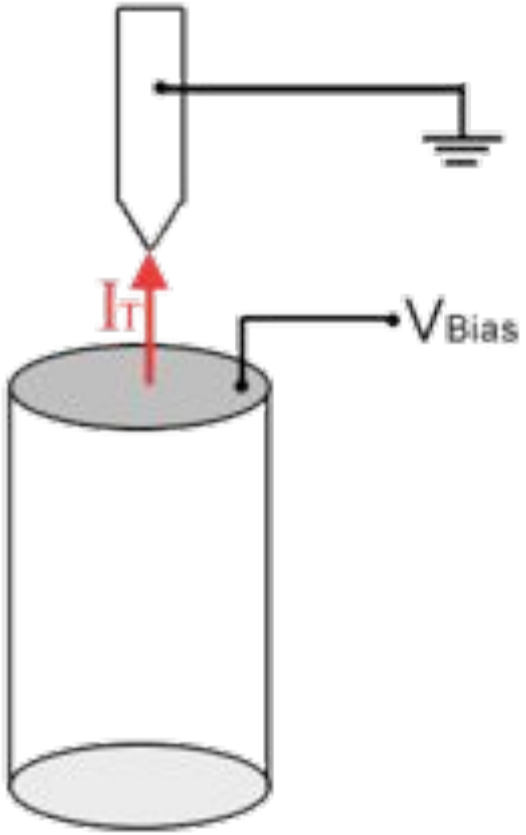
Basic Operation of AFM- Dynamic Mode

- Open the Frequency sweep window, choose Amplitude channel.
- Adjust drive amplitude, peak value should be 1～1.5.
- Red cursor sets the working frequency and amplitude, it must be set to the left side of the peak.
- Setpoint is typically 70% of the amplitude value.
- Proportional gain and Integral gain must be properly set before engage: ~200.



Basic Operation of STM

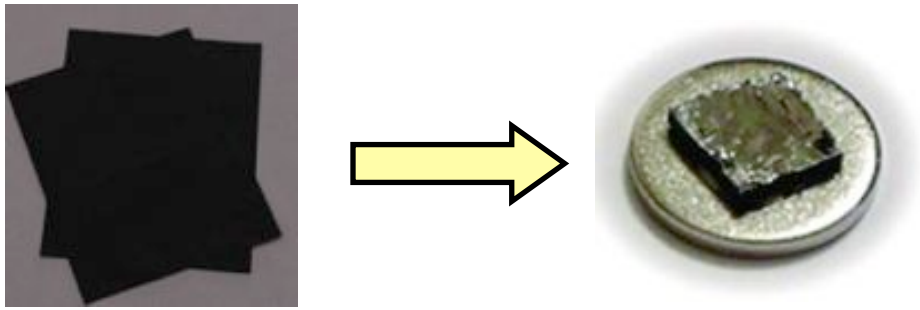
- Bias Voltage is applied to the disk of scanner (where the sample is mounted), so the sample's conductive side must be connected to the disk.
- Log mode is commonly used for most samples.
- Conductivity is affected by sample contamination, sample cleaning is necessary before experimentation.
- For metallic samples, the bias should be about 0.05V.
- The setpoint must be properly set before engaging the tip with the sample; and is usually set to 0.1-1nA.
- Proportional gain and Integral gain must be properly set before engage: ~200 (linear mode), ~3000 (Log mode).



Sample Preparation

Thin film or slice

- Soft samples should be mounted onto a sample disk with double-sided adhesive tape;
- Hard samples can be directly mounted onto scanner for scanning



soft samples should be put onto a sample disk with double-sided adhesive tape

Powder

- Powder samples are best to be treated as in SEM (Scanning Electron Microscope) scans, ultrasound-scatter and tablet press are commonly used methods;

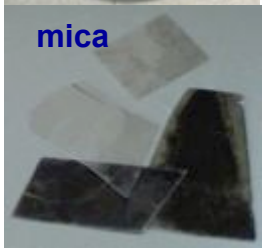
Powder- ultrasound-scatter technique

- Put a trace of powder sample into liquid; Note: the sample must not be dissolved in the liquid used!
- The most commonly used liquid is distilled water or absolute ethyl alcohol;
- The optimum concentration is usually 0.1～1g/L;
- Use an ultrasonic cleaner to scatter the powder solution thoroughly;
- Use a clean dropper to drop the scattered solution on a substrate; A mechanically-polished slice of silicon or freshly-cleaved mica surface are commonly used substrates;
- When the substrate is dried, powder particles will be attached to the substrate;
- Dynamic mode is usually used for this kind of sample.

Powder- ultrasound-scatter

Put a trace of powder sample into liquid, the concentration is usually 0.1～1g/L.

Use an ultrasonic cleaner to scatter the powder solution thoroughly for about 5~15 minutes.



Drop the scattered solution on a substrate (silicon or mica), use dynamic mode AFM to image when dried.

Powder- ultrasound- tablet press

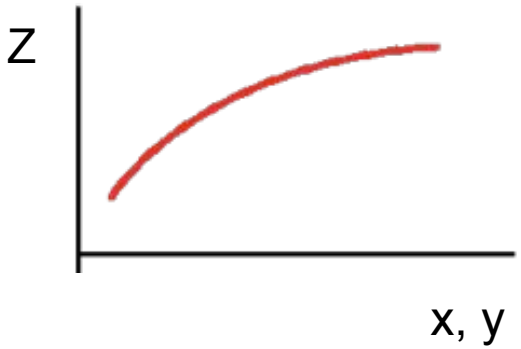
A tablet press machine

Use a tablet press machine to make the powder sample to a smooth slice; Dynamic mode AFM is usually applied to this kind of sample.

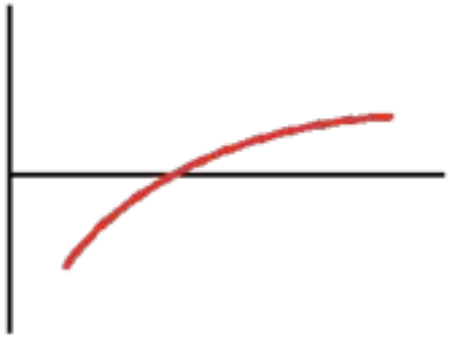
Tablet press method is usually applied to the powder sample which has large particle size or can not be put into liquid.

Scanning Parameters

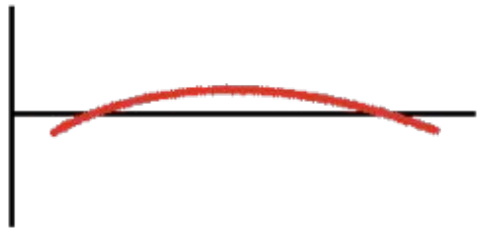
Surface Fitting –software compensation for bowing of scanner during imaging



Original Signal

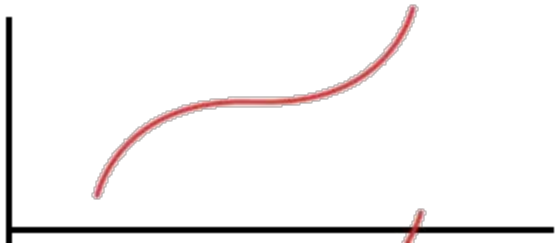


0 Order



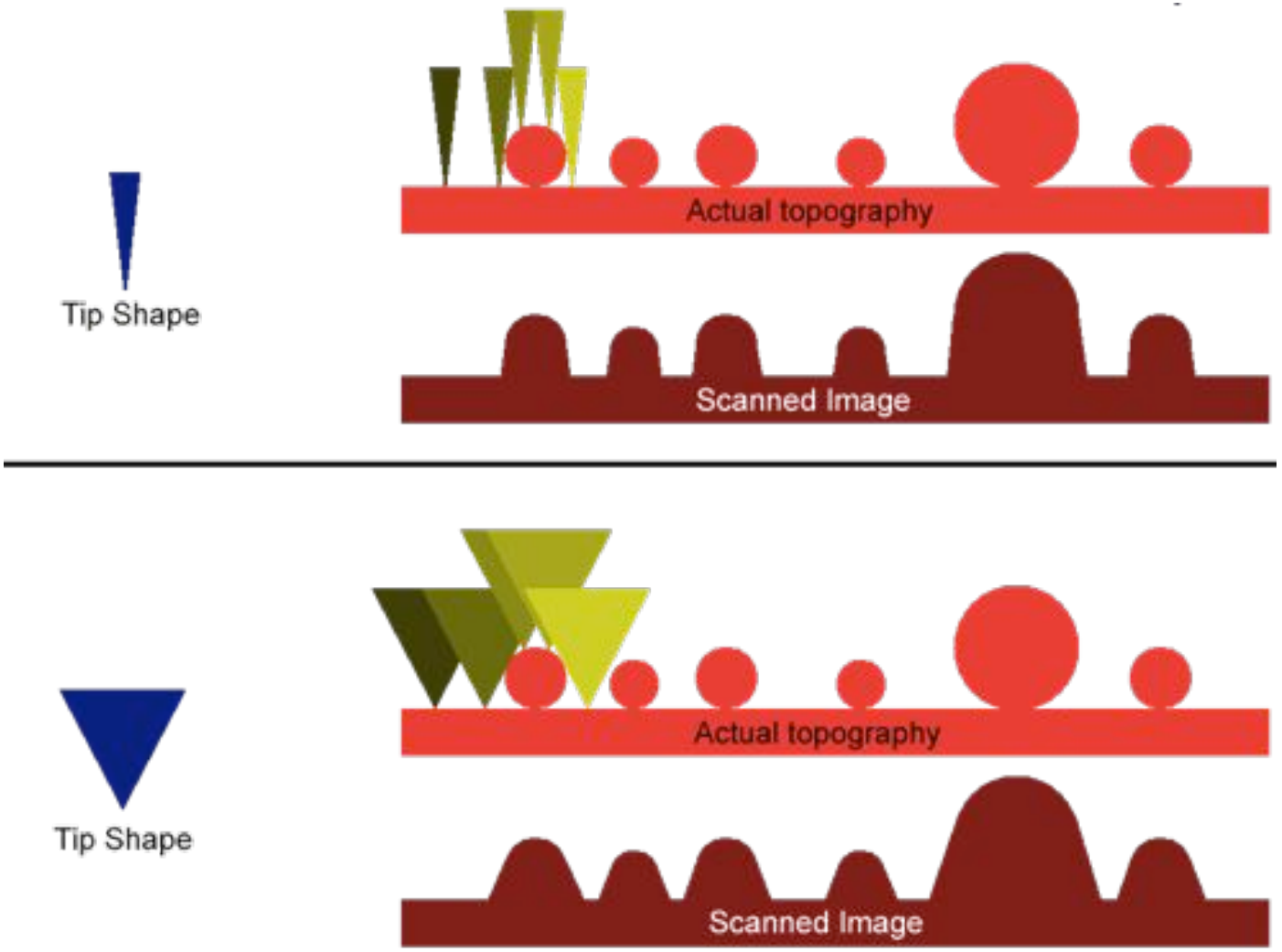
1st Order

2nd Order

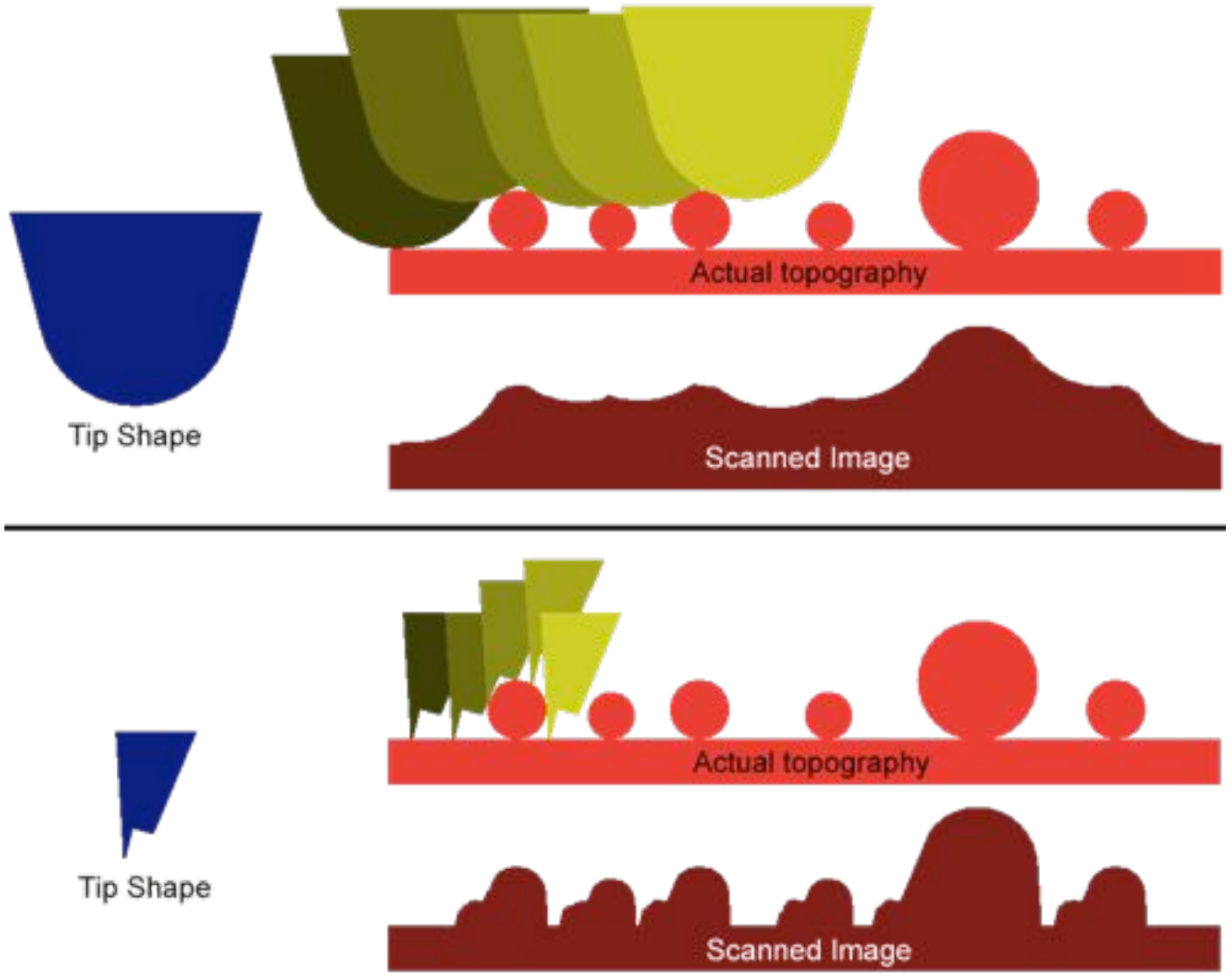


3rd or higher Order

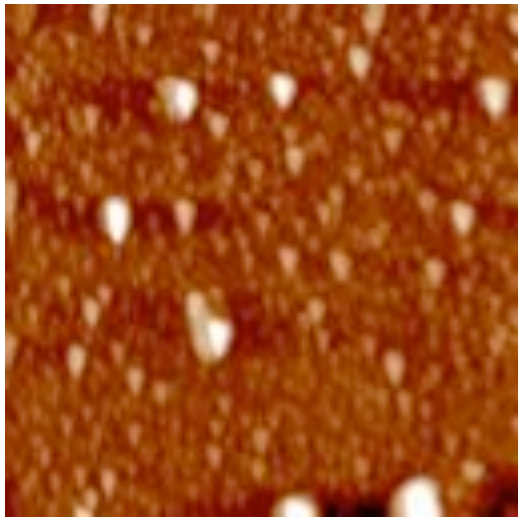
Influence of Tip

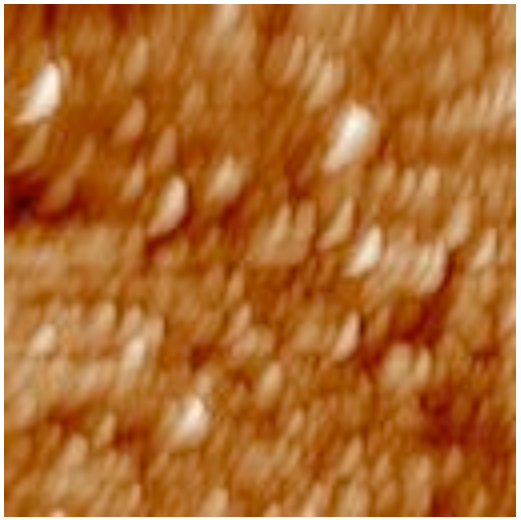


Influence of Tip

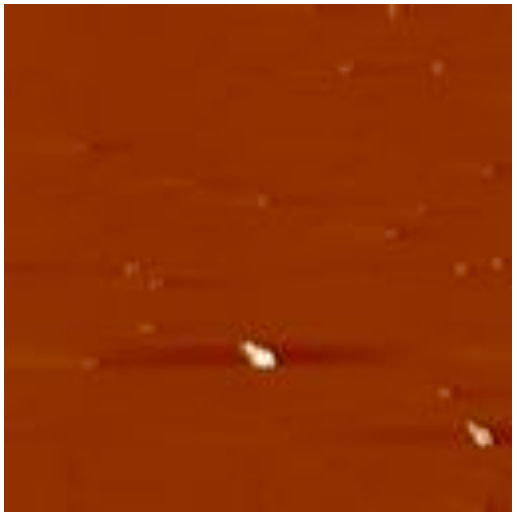


Influence of Tip





Tip shape is also scanned in the image.



Multi-tip

Imager software for processing

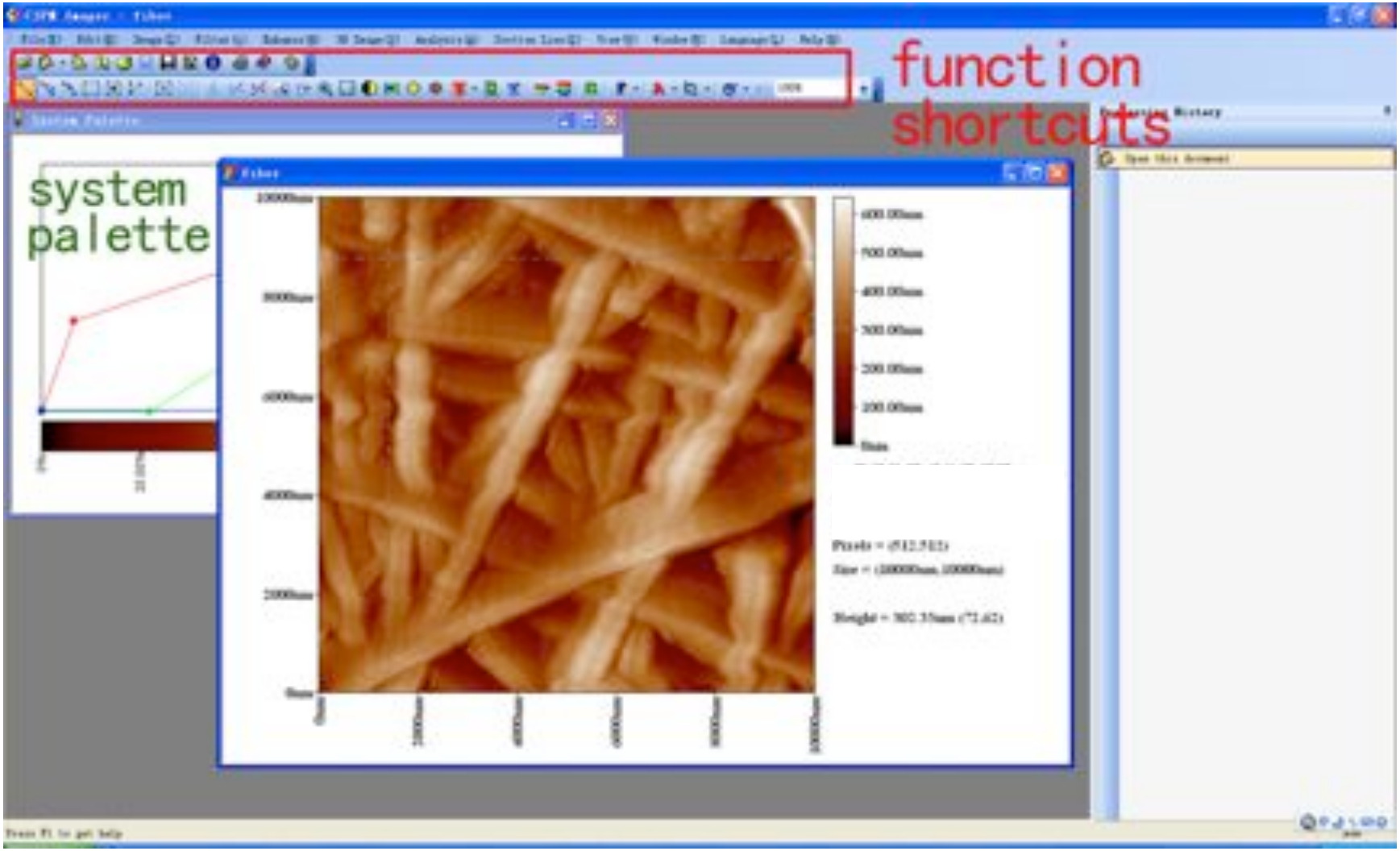
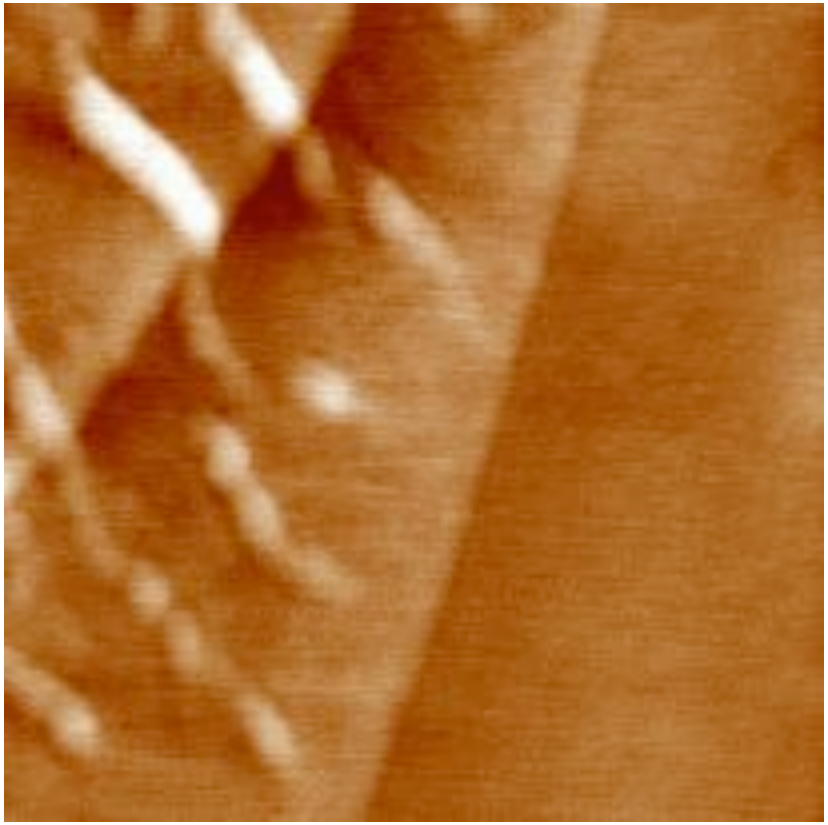


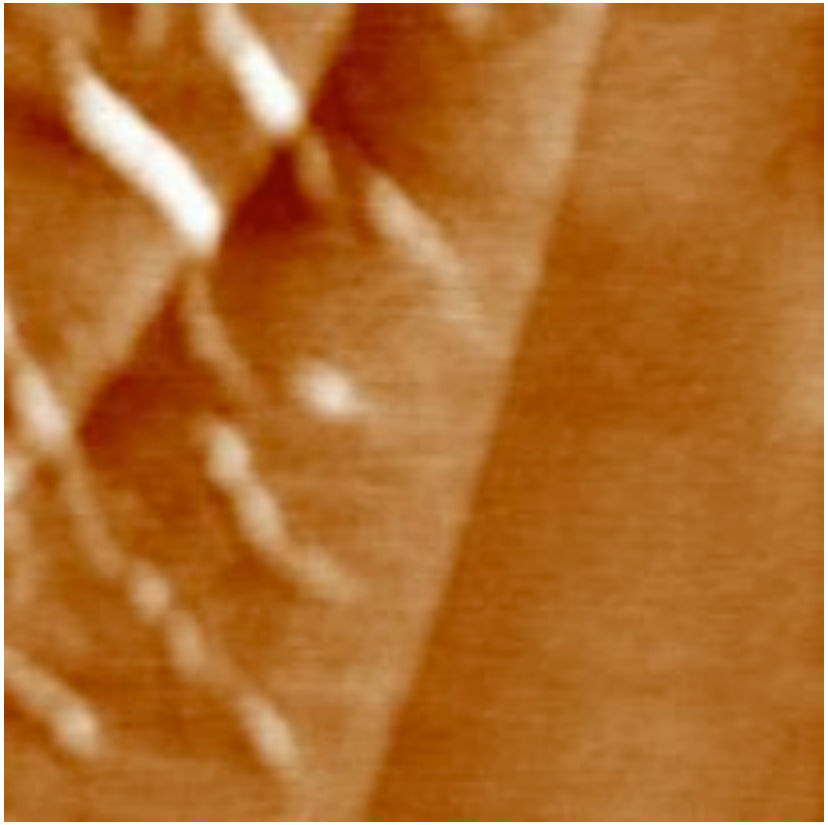
Image formats

Operations	Document suffix	Note
Scan results	.csm	Scan parameters included, can be opened only with Imager software
Save with axes	.bmp	Not for re-processing
Saved 3D images	.bmp	
curves	.cur	To obtain detailed data, open with Windows “Notepad”
Composed AVI	.avi	
Analysis reports	.htm	

Image process: filters



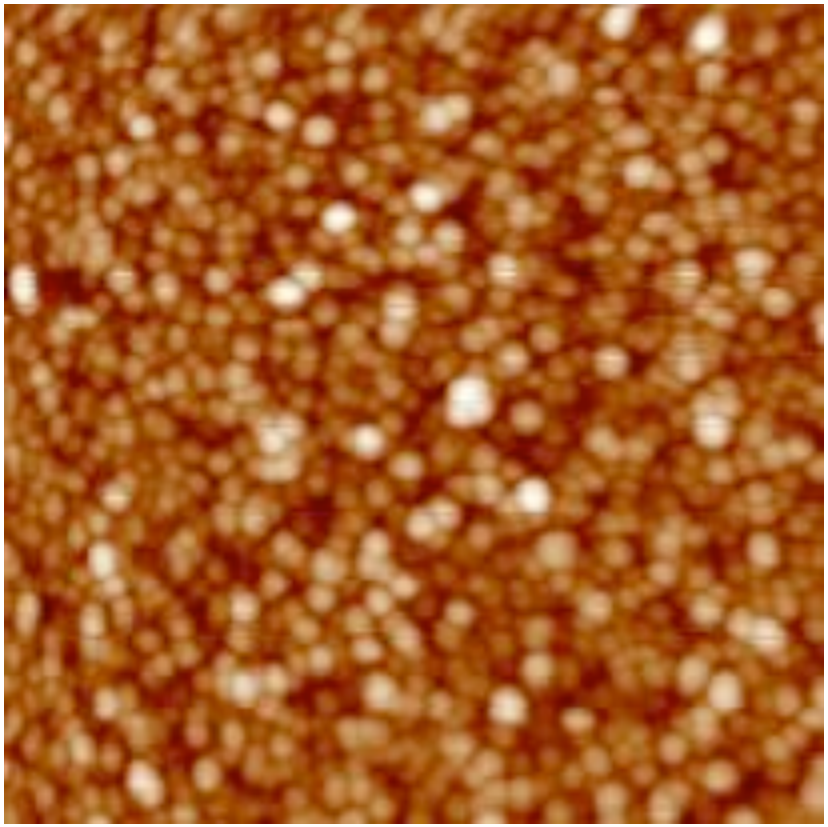
Scanned image



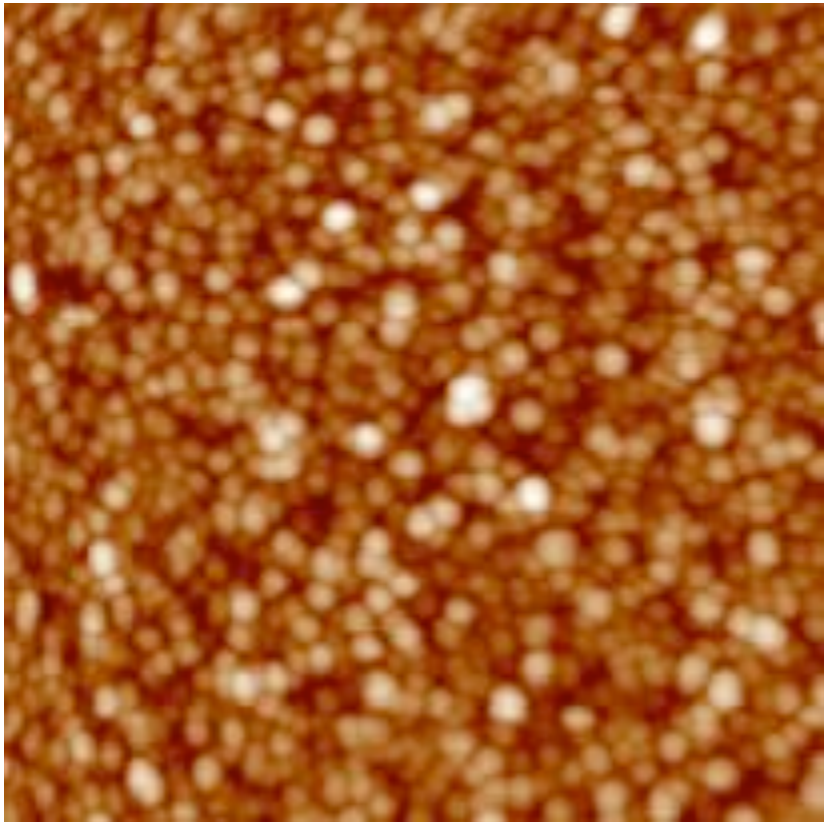
After one low-pass filter and average filter

Image process: clear scan line

Clear scan lines automatically or manually



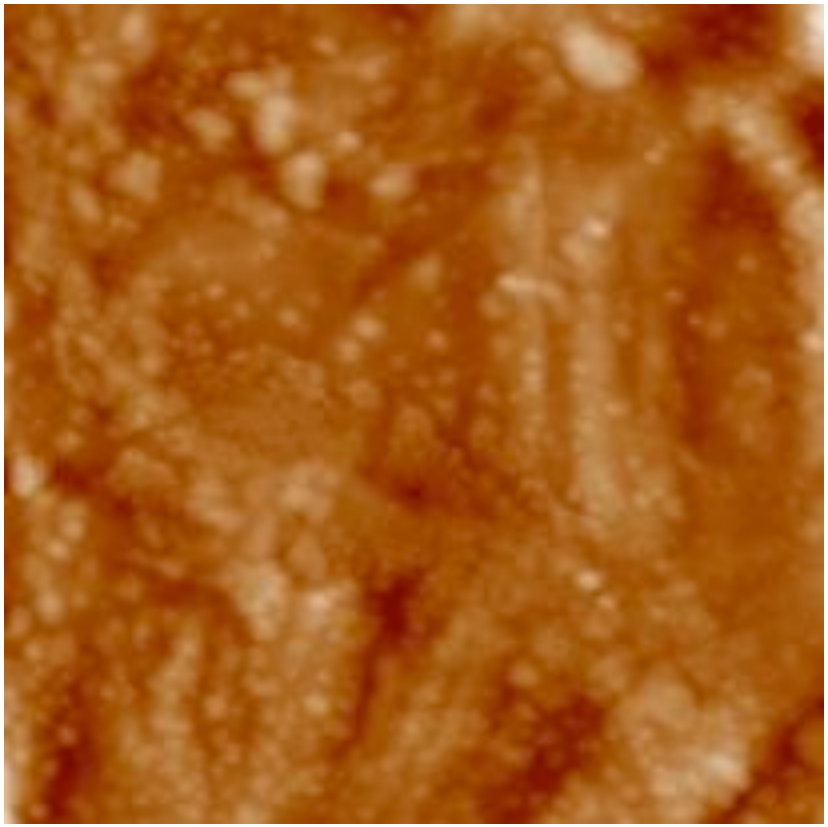
Scanned image



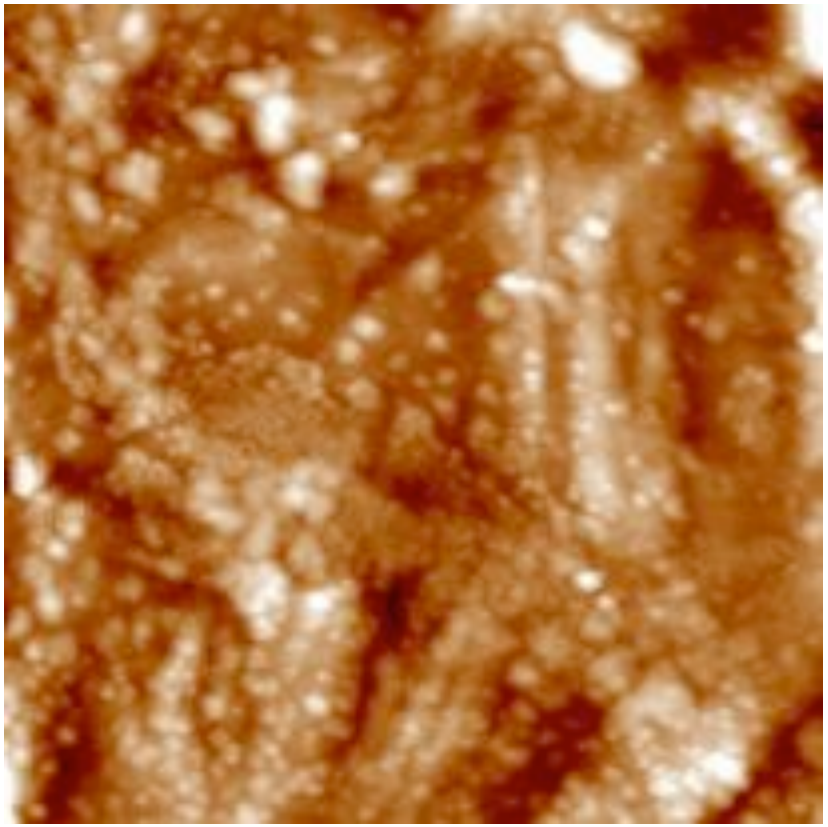
after

Image process: brightness and contrast

Best contrast, brightness and contrast adjustment

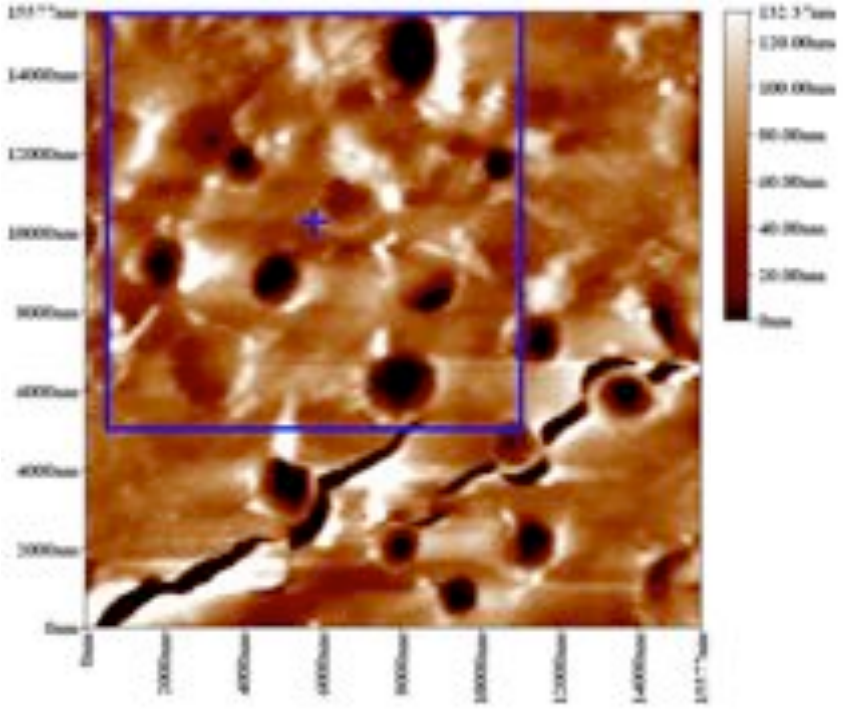


Scanned image

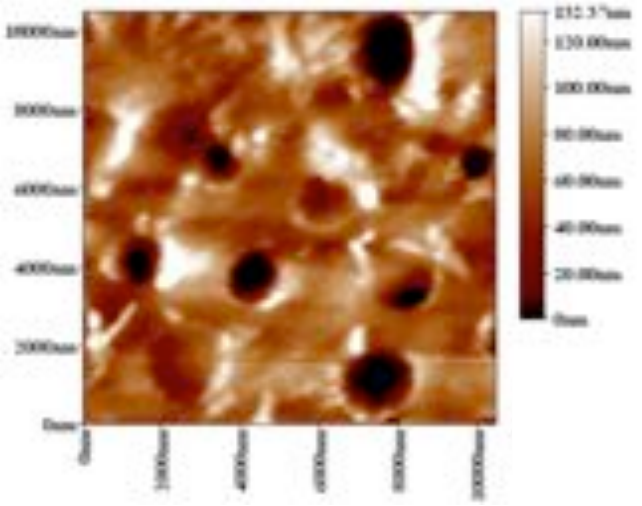


After adjustment of brightness and contrast

Image process: Zoom in/out, zone selection



Choose area



cut

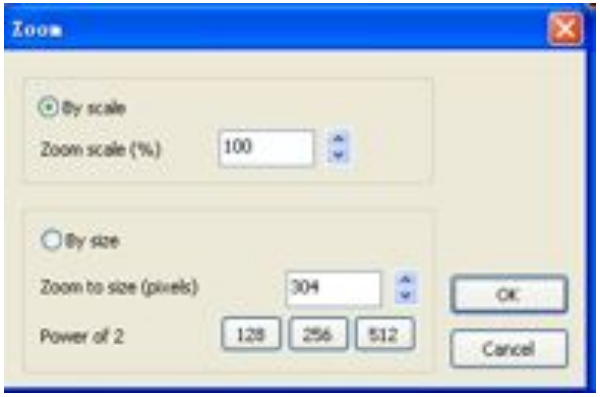
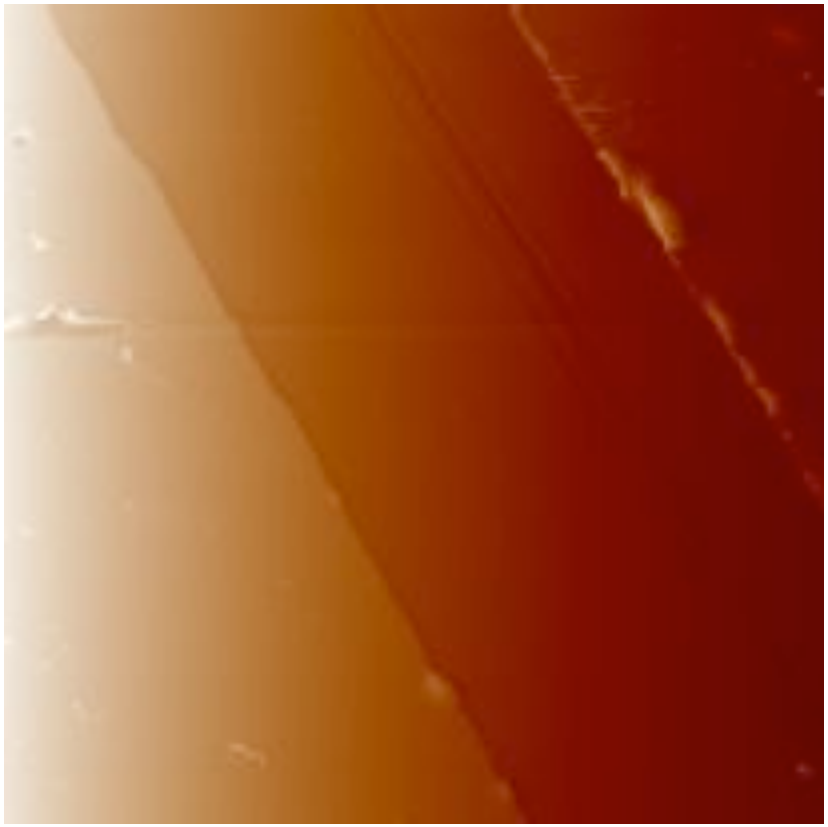
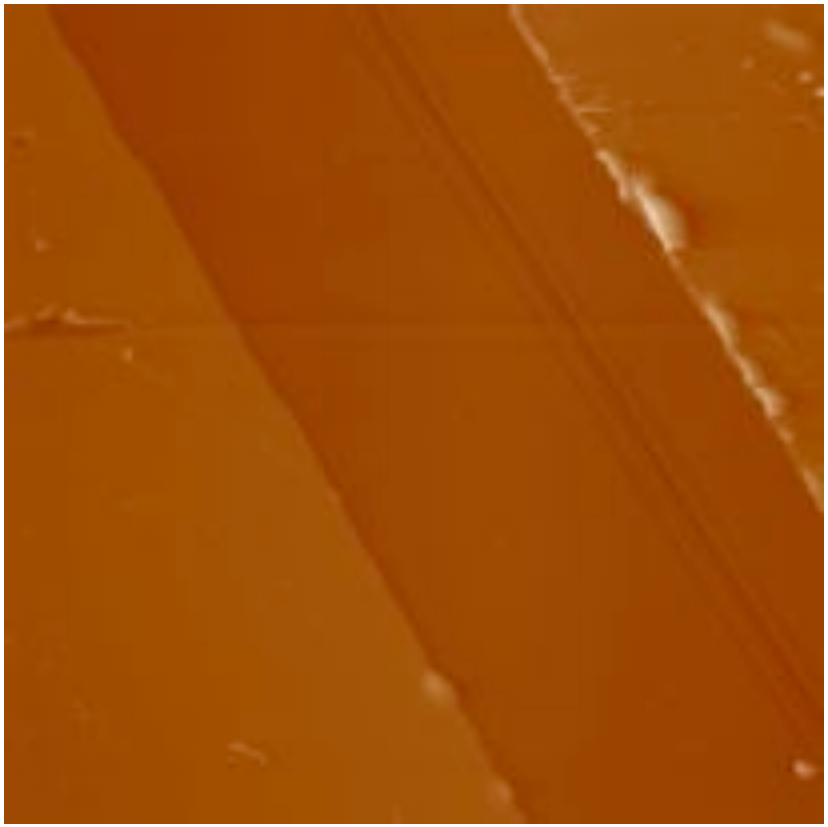


Image process: surface fit, Non-linear correction

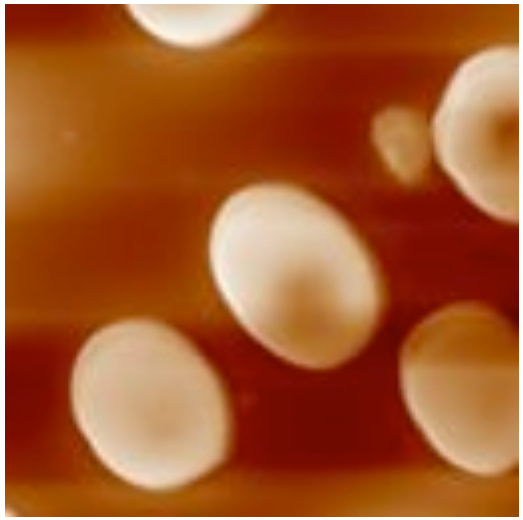


Scanned image

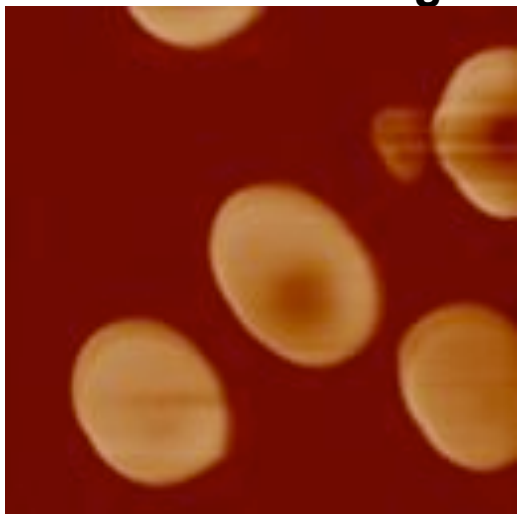


After a 3-order surface fit

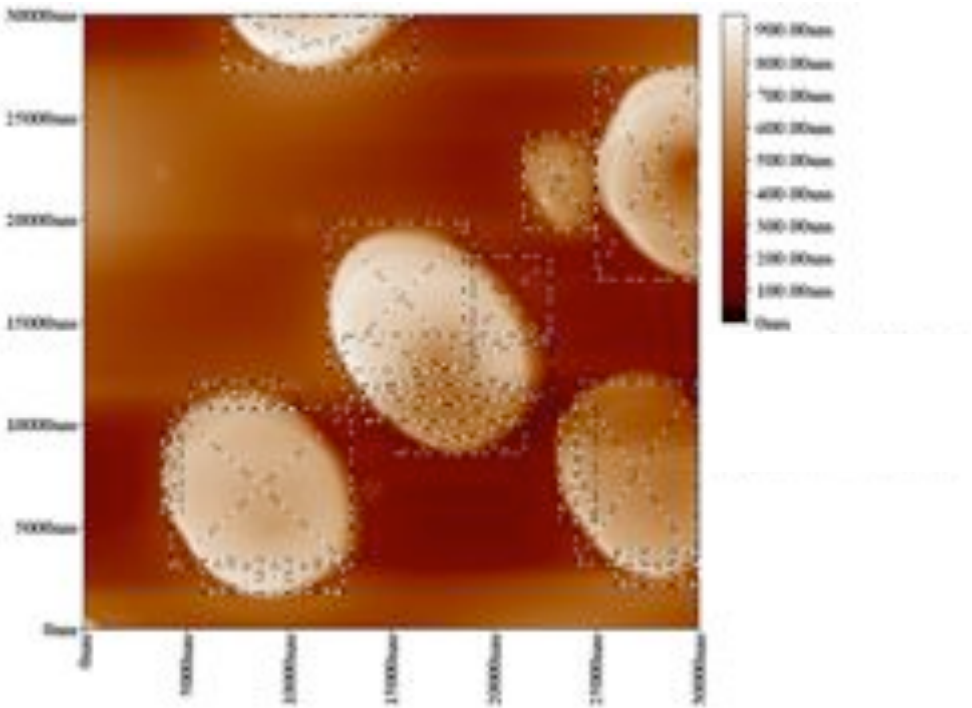
Image process: surface fit with excluded boxes



Scanned image

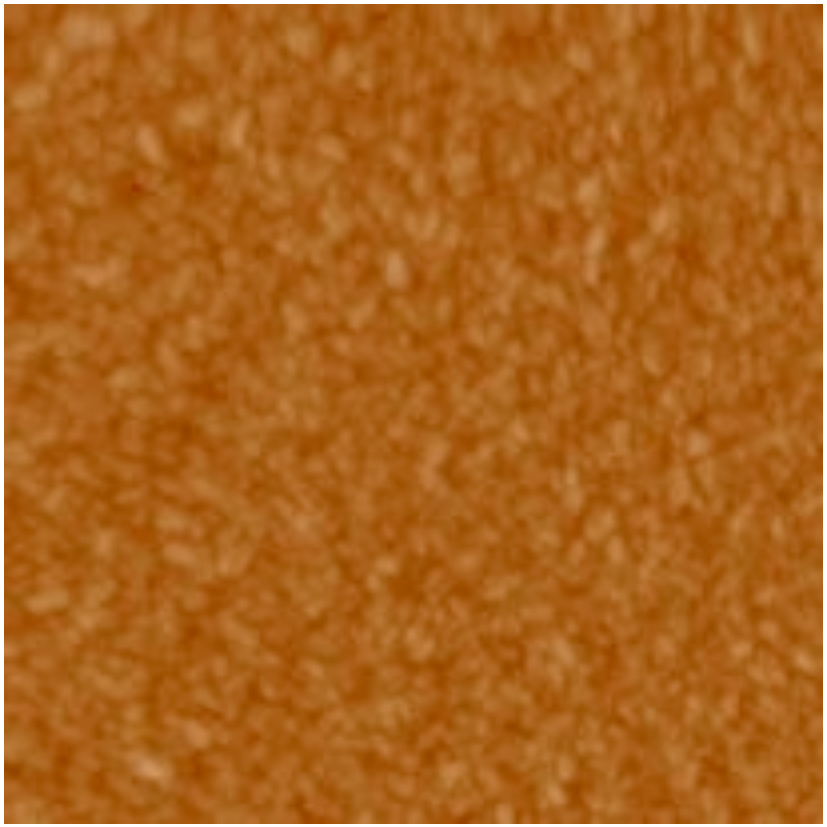


After a 3rd-order surface fit

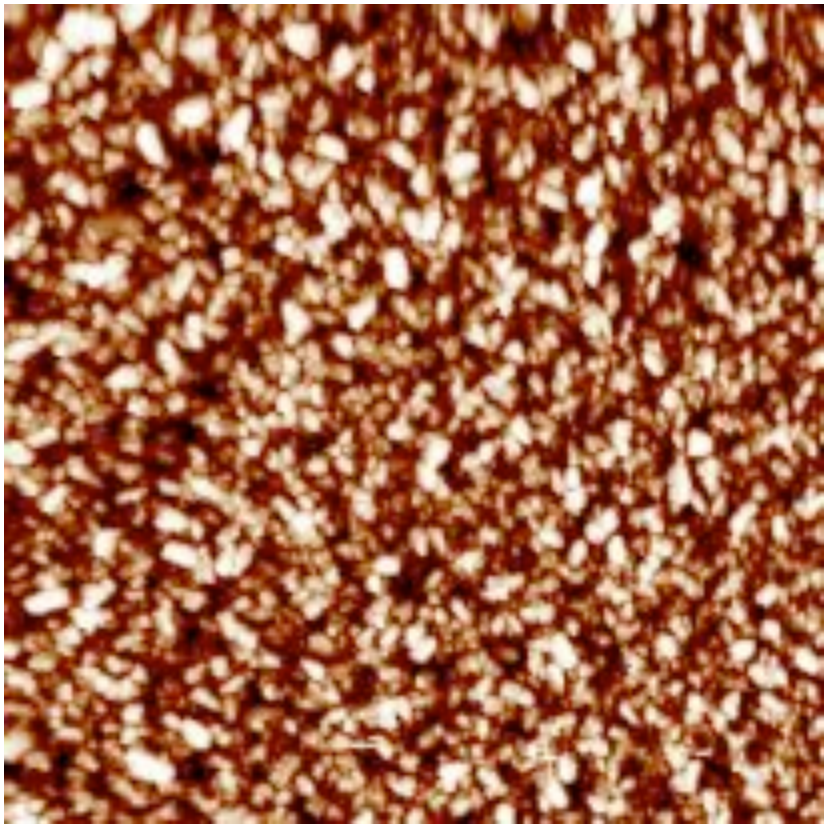


Use excluded boxes to eliminate the particles, surface fit will not include the distribution of the topography that is inside the boxes.

Image process: Equilibrate



Scanned image



after

Image Analysis: 3D image

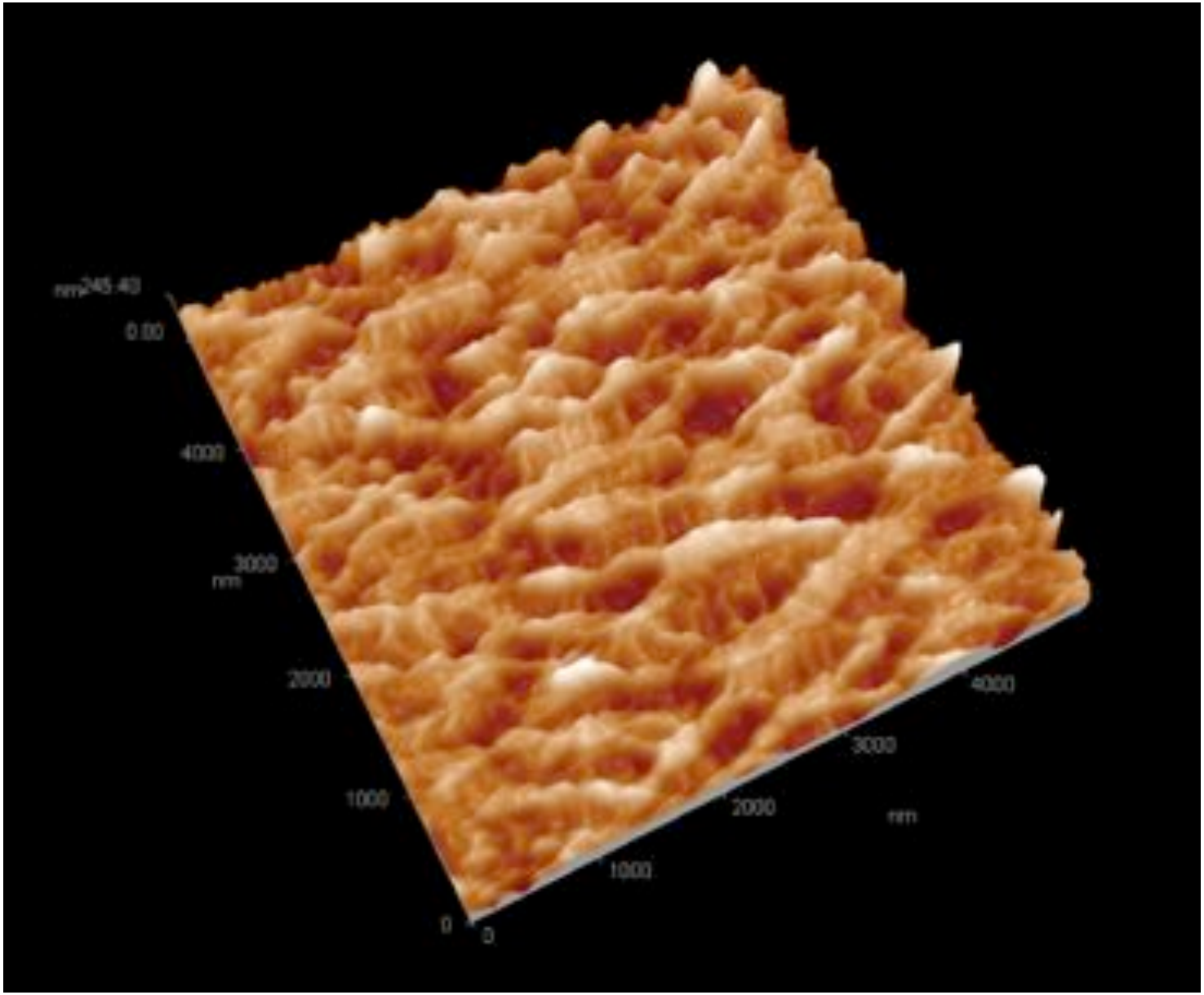
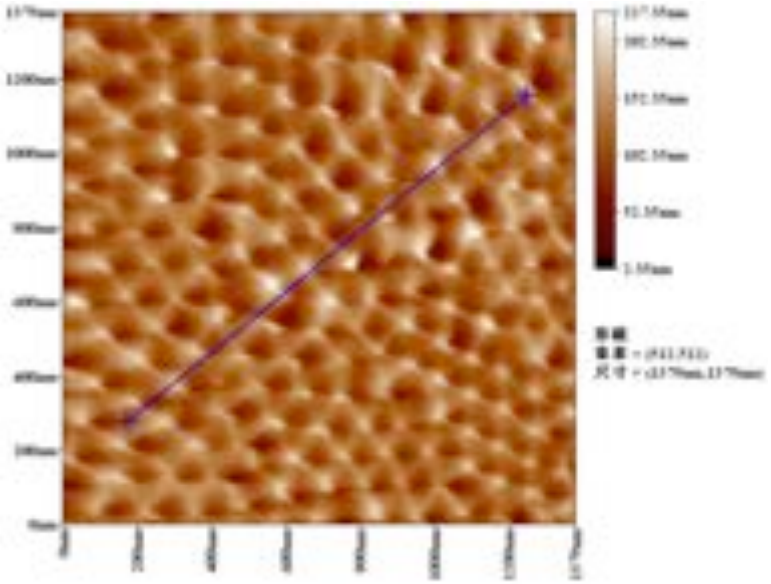
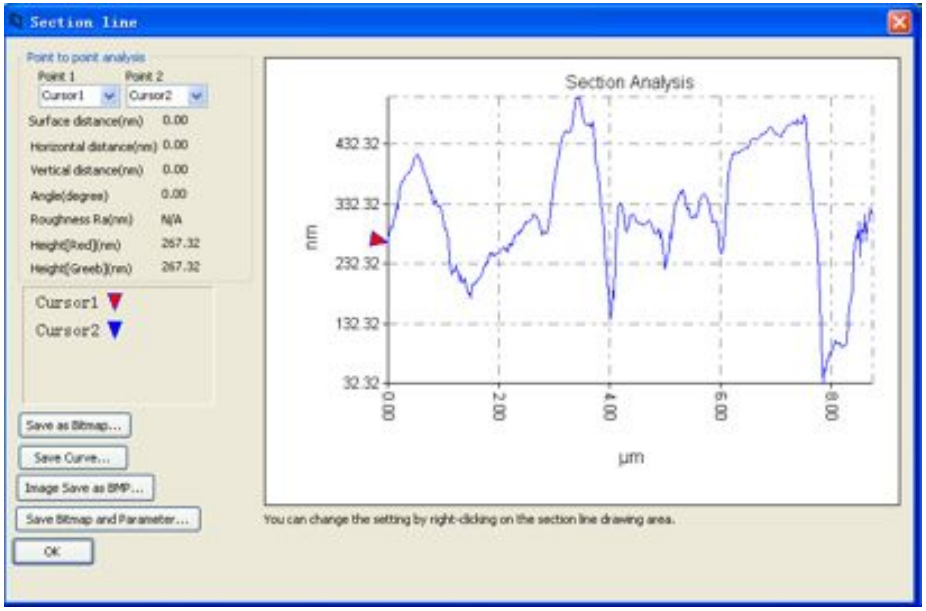


Image Analysis: Section line

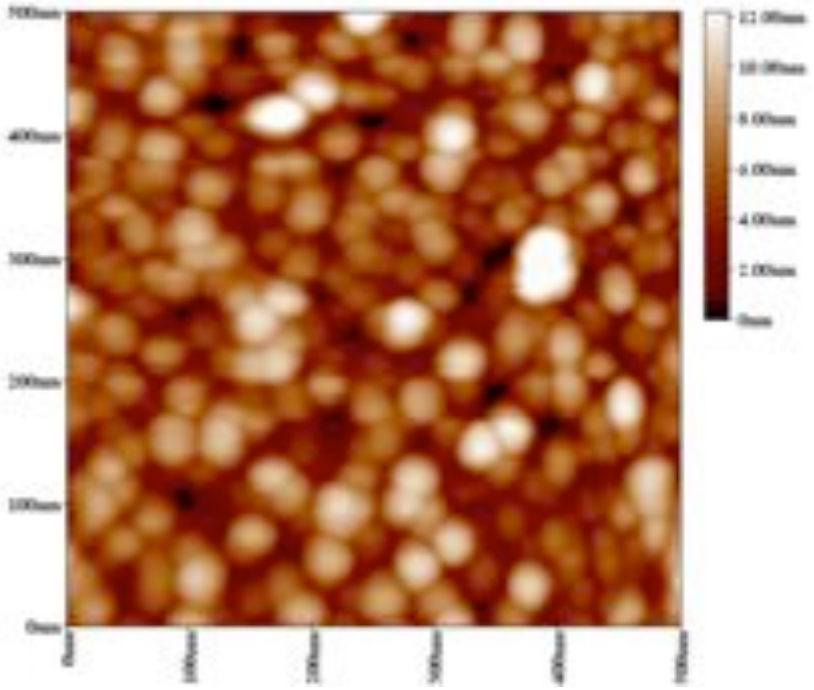


Use a line tool to make a section line

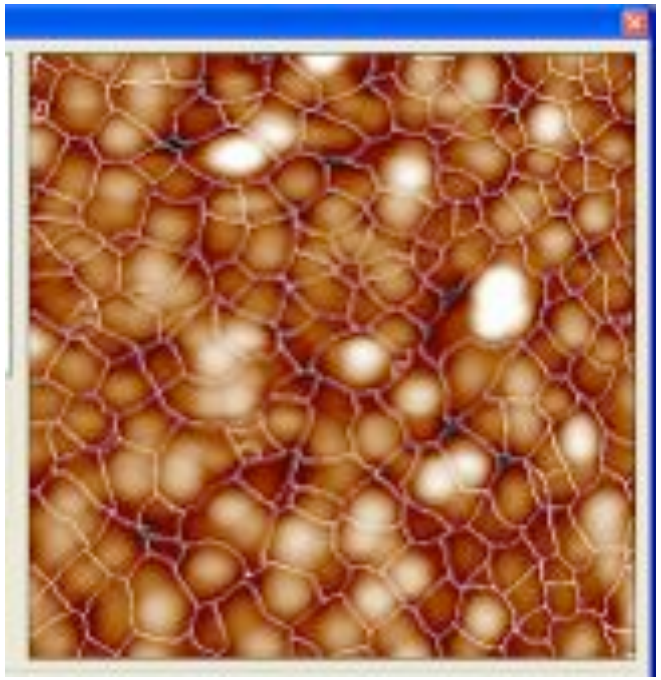


Section Analysis

Image Analysis: Grain Size Analysis

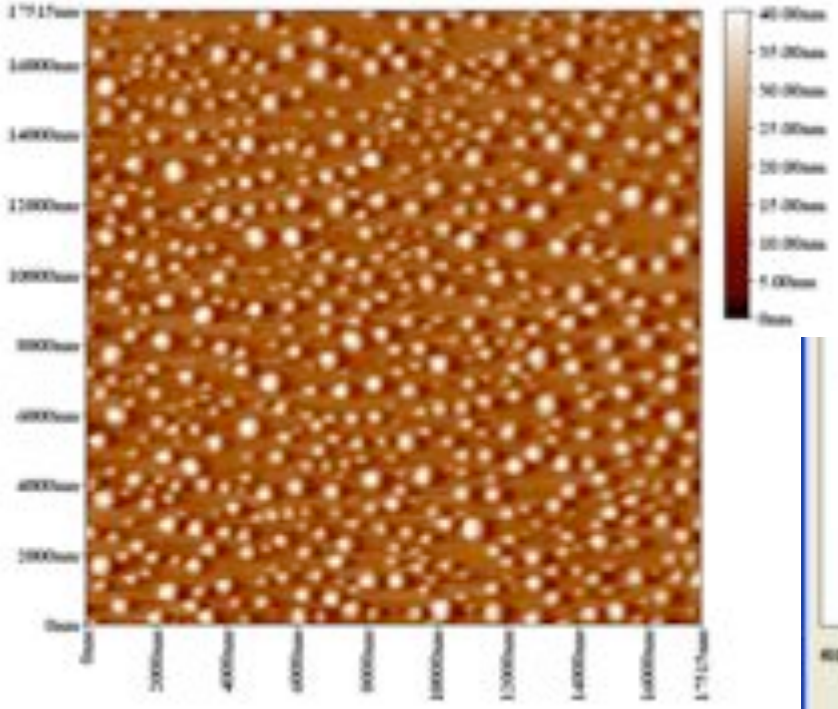


No obvious substrate on sample image

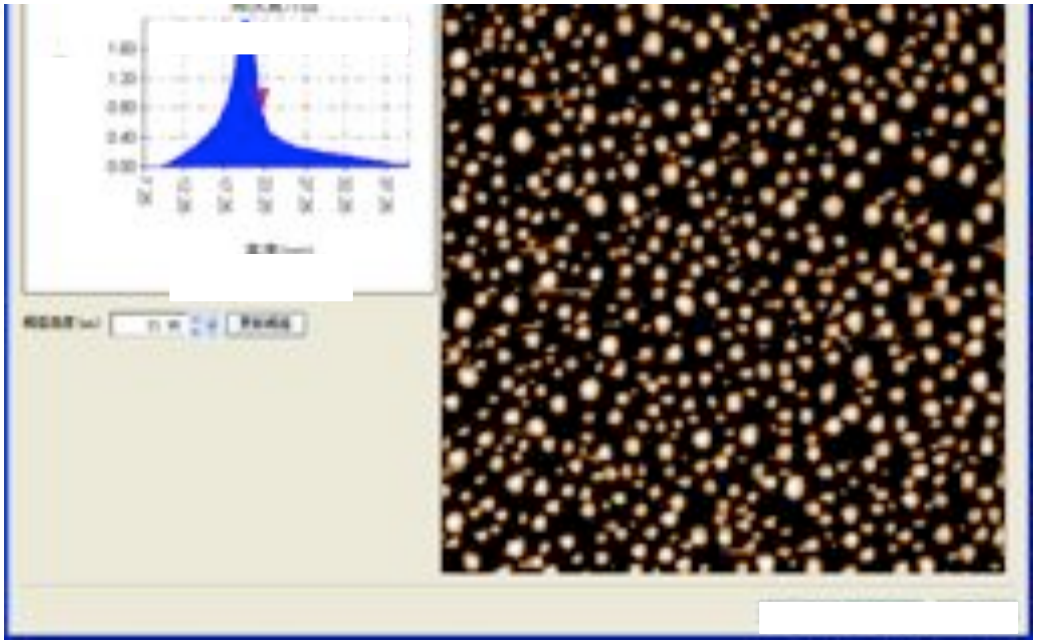


Automatic grain size analysis

Image Analysis: Grain Size Analysis

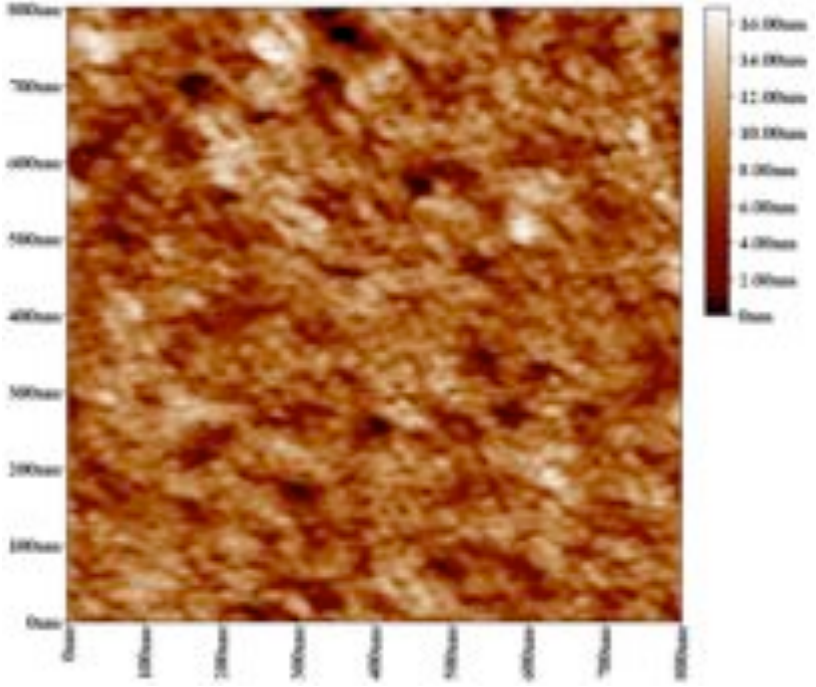


Obvious substrate can be observed on sample image



Set height threshold

Image Analysis: Surface Roughness Analysis



Scanned image

Surface Roughness Analysis