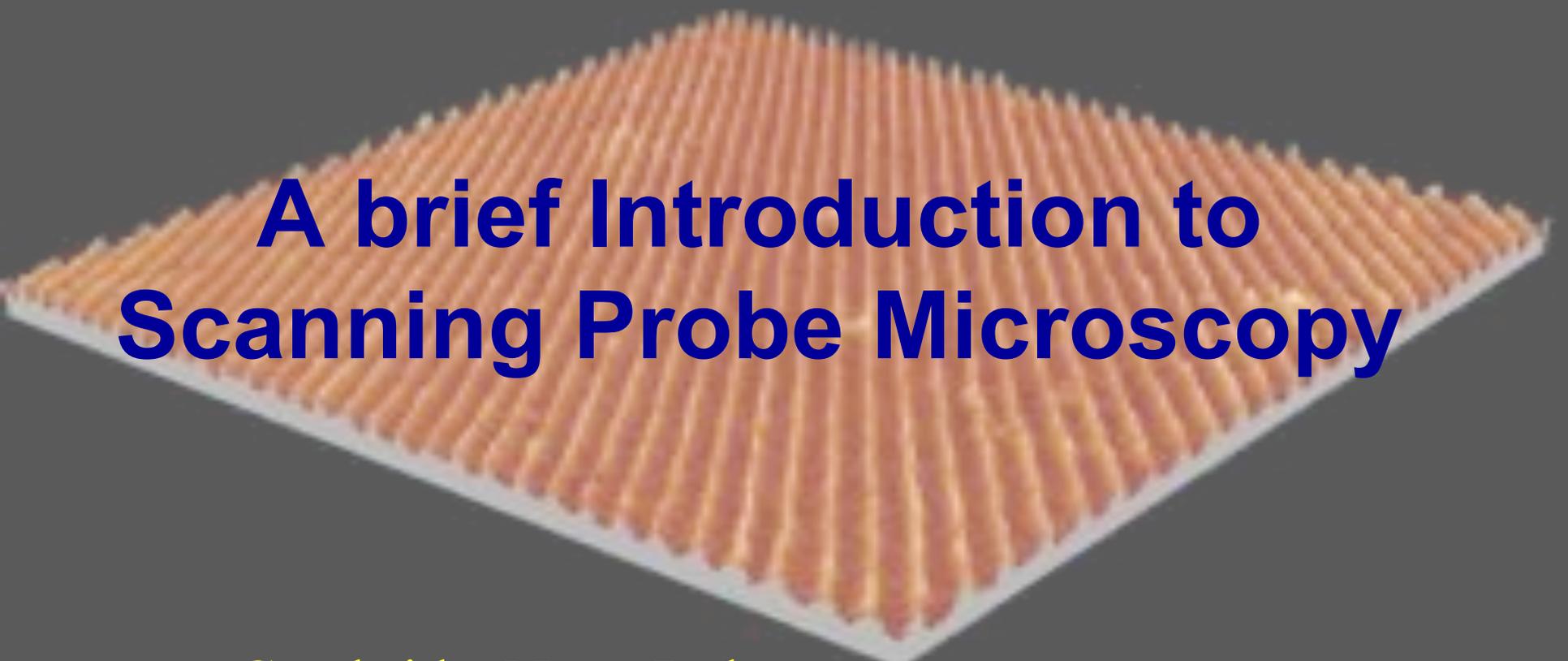




CambridgeNano

*Showing You the World*



**A brief Introduction to  
Scanning Probe Microscopy**

CambridgeNano Ltd.

[www.cambridgenano.co.uk](http://www.cambridgenano.co.uk)

# A brief history of Scanning Probe Microscopy

- The first Scanning Tunnelling Microscope (STM) was invented by Binnig and Rohrer of IBM in 1981, Zurich, Switzerland.
- On 1986, Binnig 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**



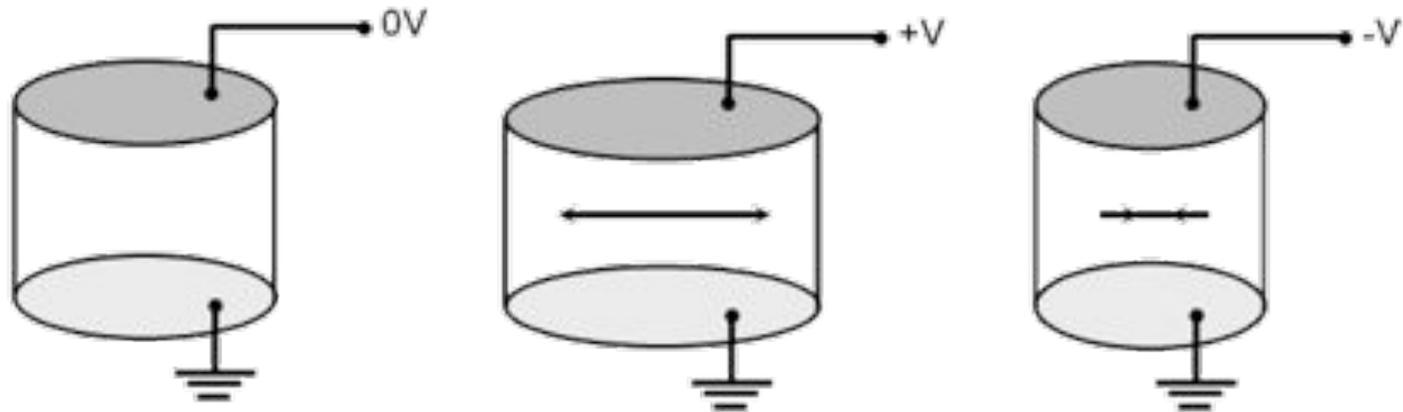
**The first AFM**

# 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

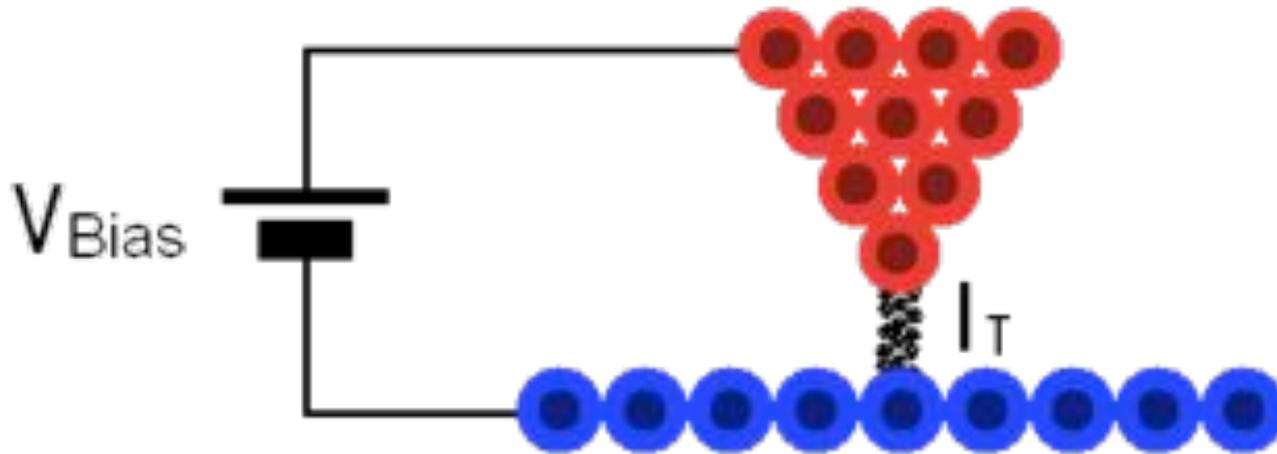


Length changes according to the applied voltage

Scanners are made of piezo materials:

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

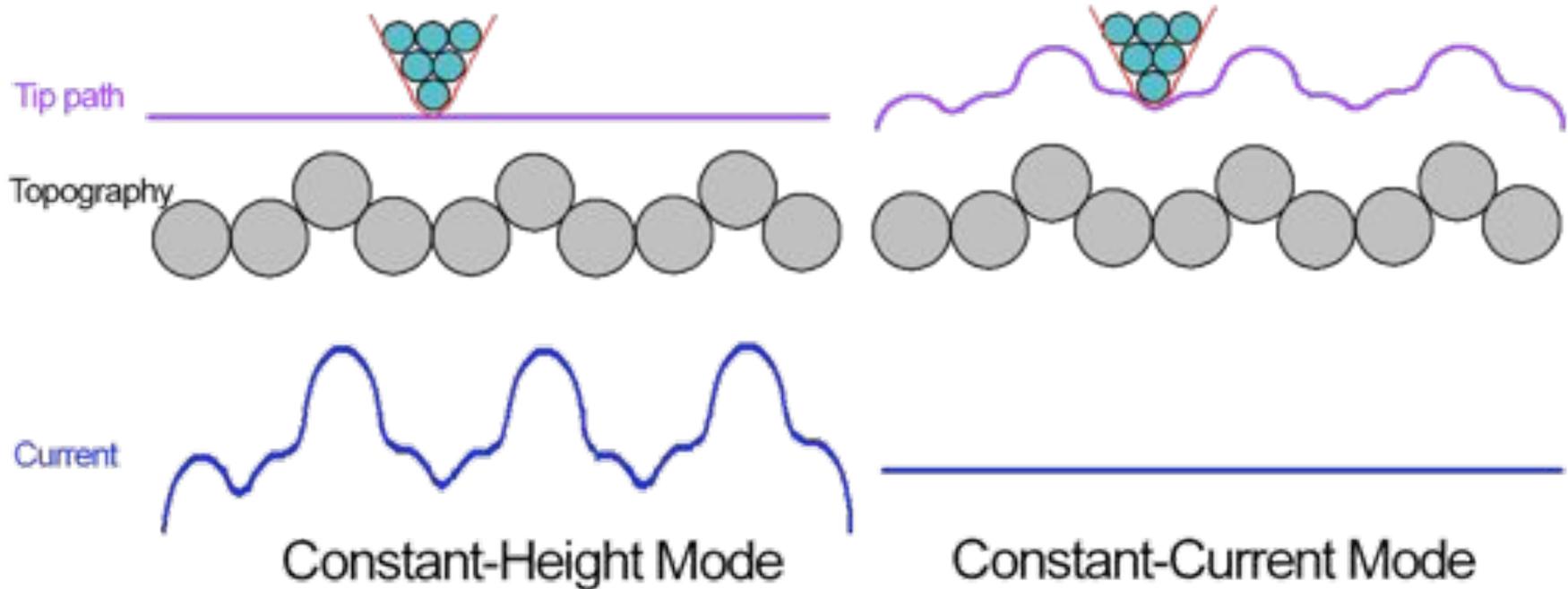
# Quantum tunneling and STM



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

- $I_T$ : Tunneling Current
- $V_{Bias}$ : Constant Bias voltage applied to tip and sample
- $C$ : Constant
- $S$ : Distance between tip and sample

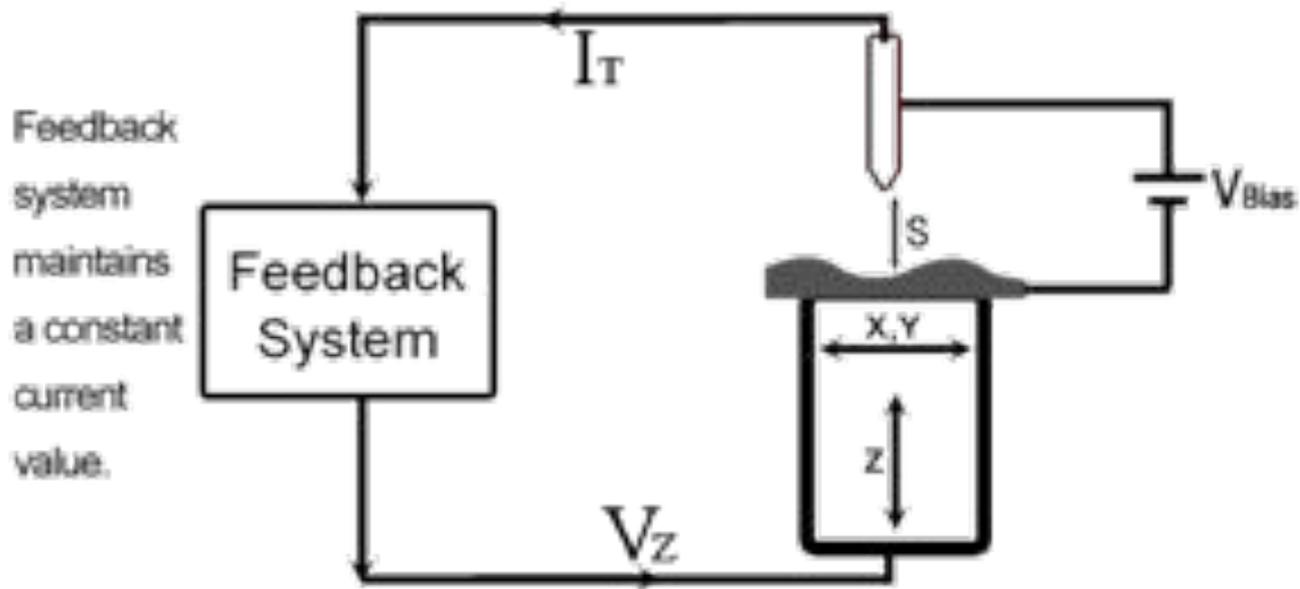
# Operation modes of STM



**Constant-Height mode: No feed-back control.**

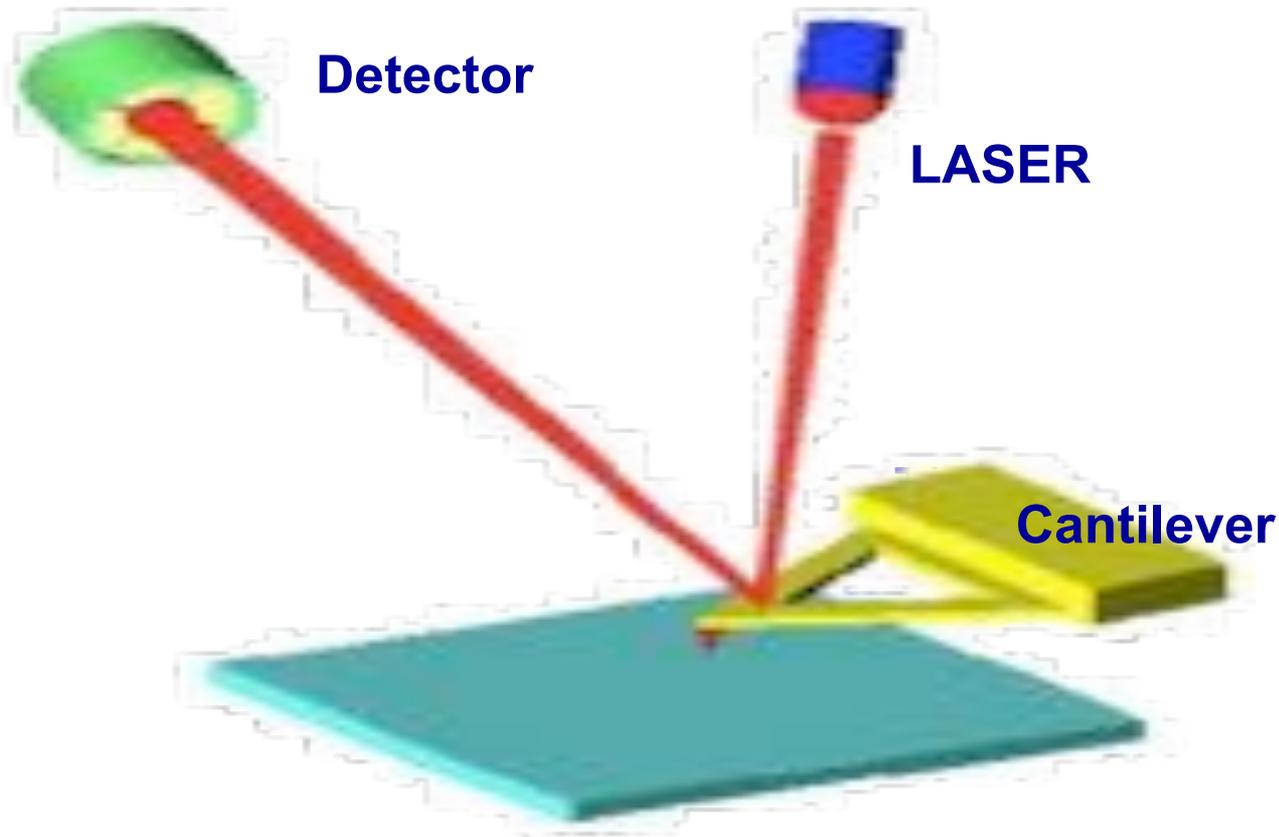
**Constant-Current mode: Feed-back control activated. Tip path equals the sample topography.**

# STM System



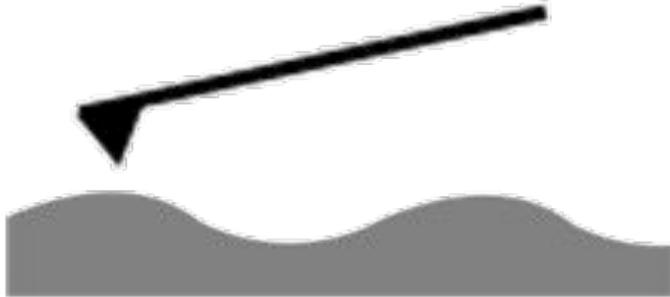
- Tip approaches to sample, current occurs.
- $V_z$  is controlled by feedback system to maintain a constant current which is called Setpoint.
- $V(x,y)$  of each scanning point  $(x,y)$  is recorded.
- Sample topography  $T(x,y)$  can be calculated by  $V(x,y)$ .

# 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.



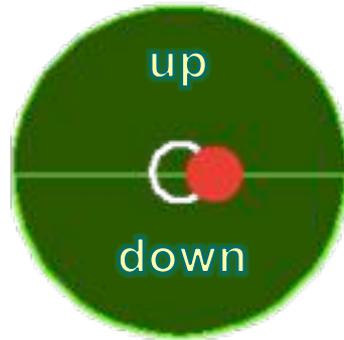
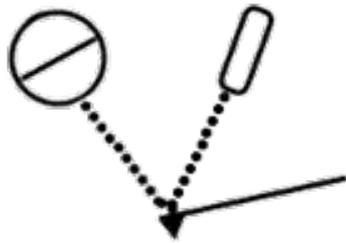
Repulsion between tip and sample, cantilever deflects upwards.



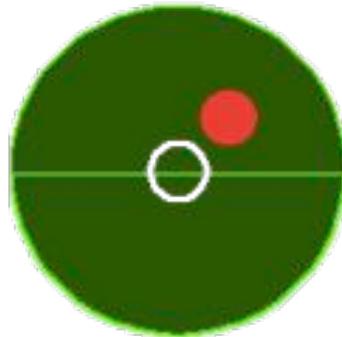
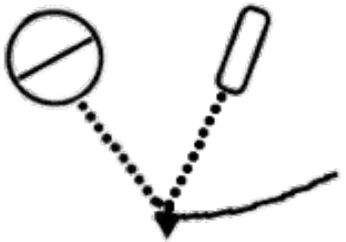
Repulsion between tip and sample, cantilever deflects downwards.

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

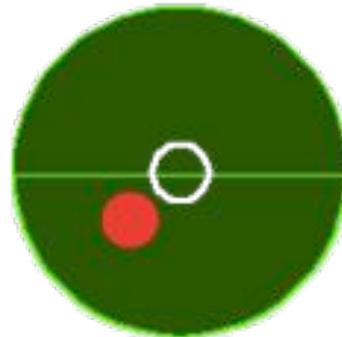
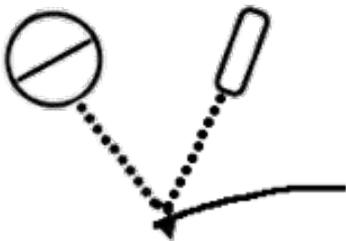
# Cantilever Deflection on Detector by Laser



No deflection: Up-Down=0

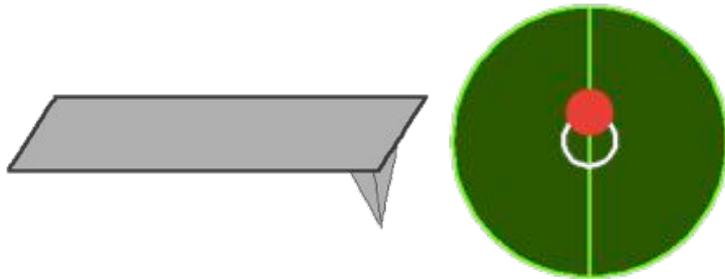


Upwards: Up-Down>0

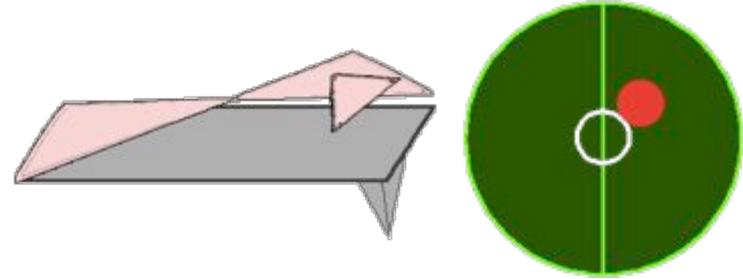


Downwards: Up-Down<0

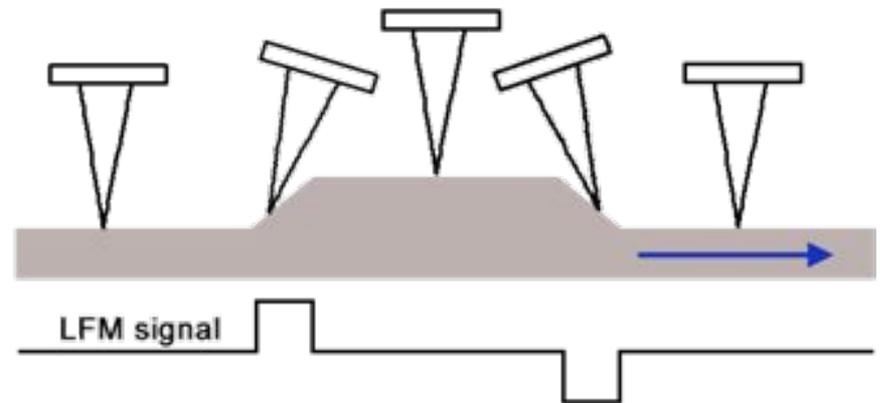
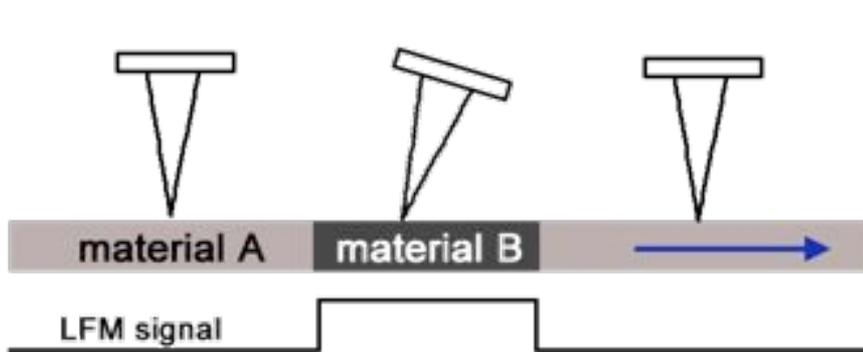
# Lateral deflection of cantilever



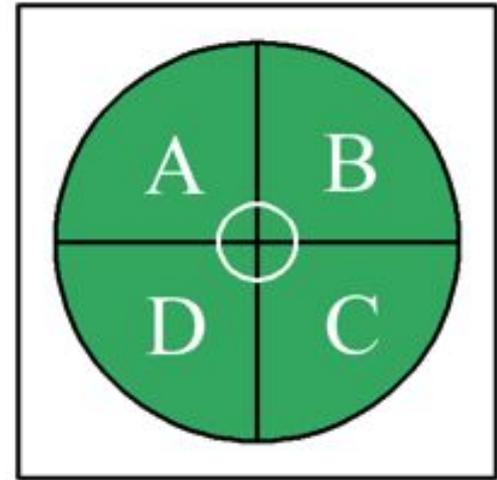
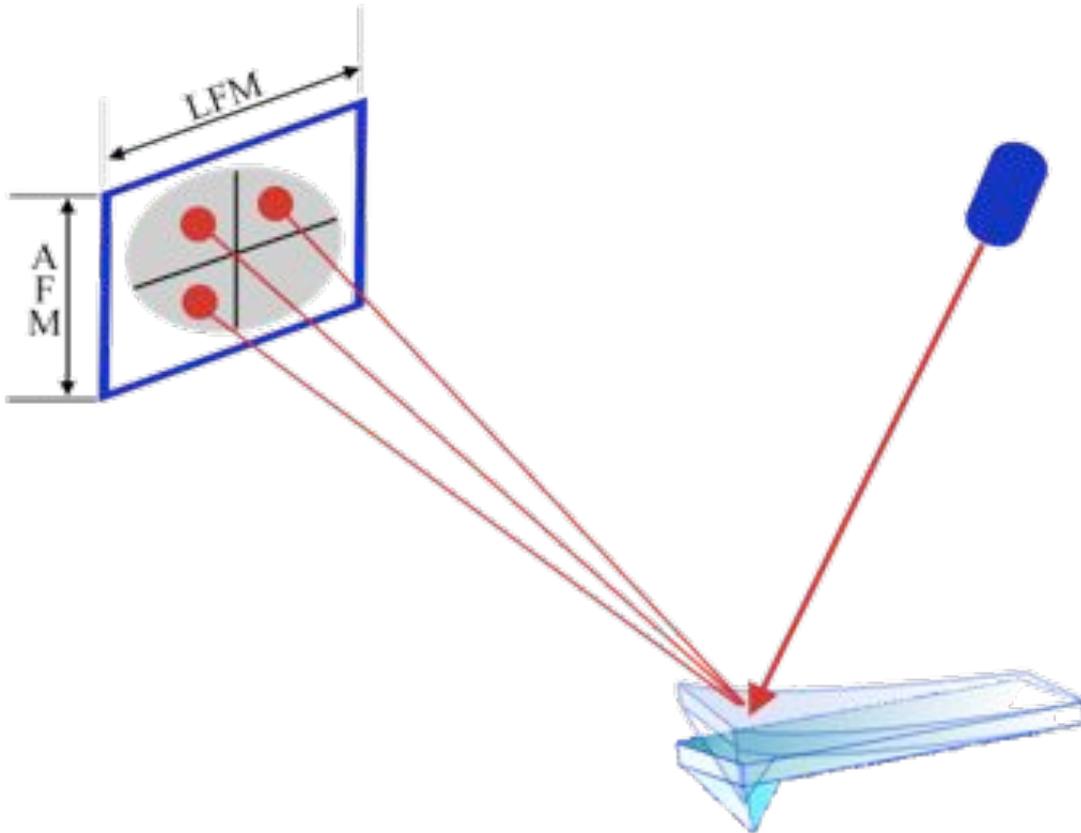
No friction: Left-Right=0



Friction: Left-Right  $\neq$  0



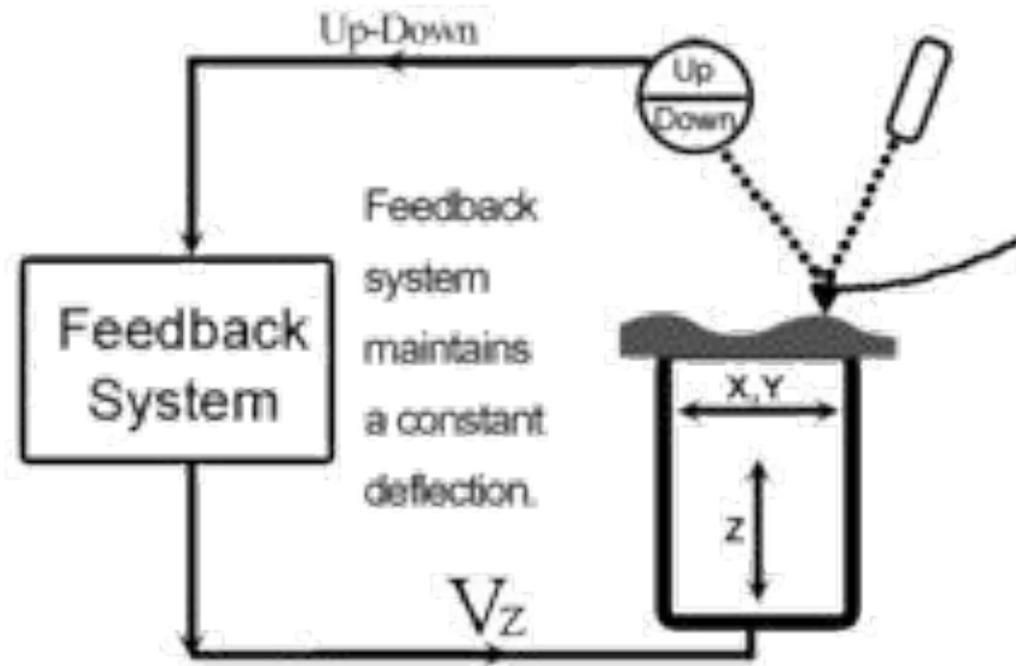
# AFM and LFM



$(A+B)-(C+D)$ : AFM signal

$(A+D)-(B+C)$ : LFM signal

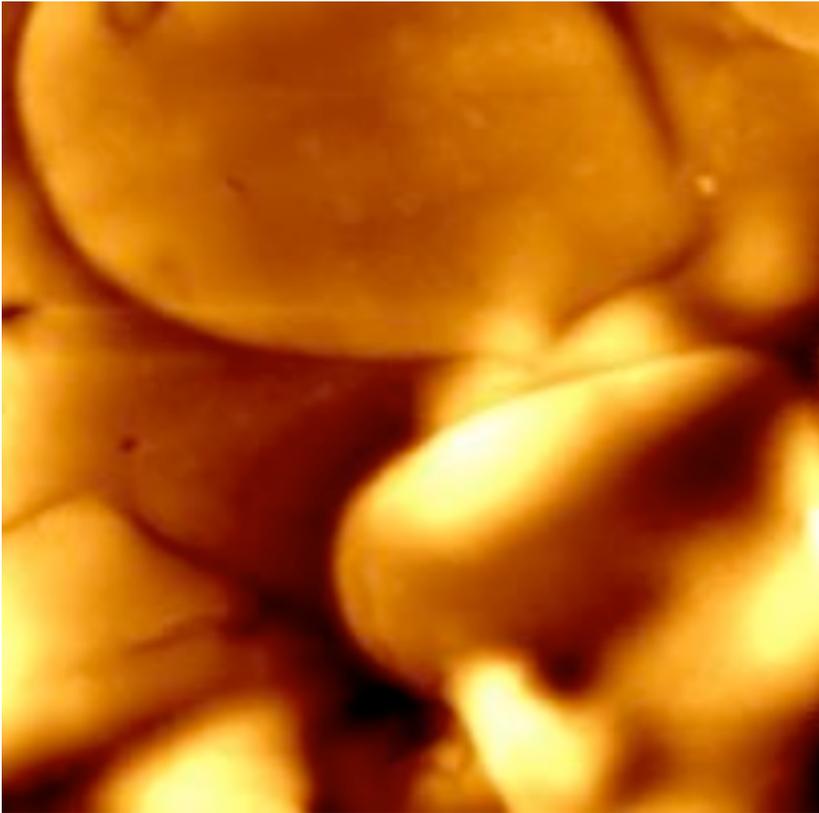
# Contact Mode AFM



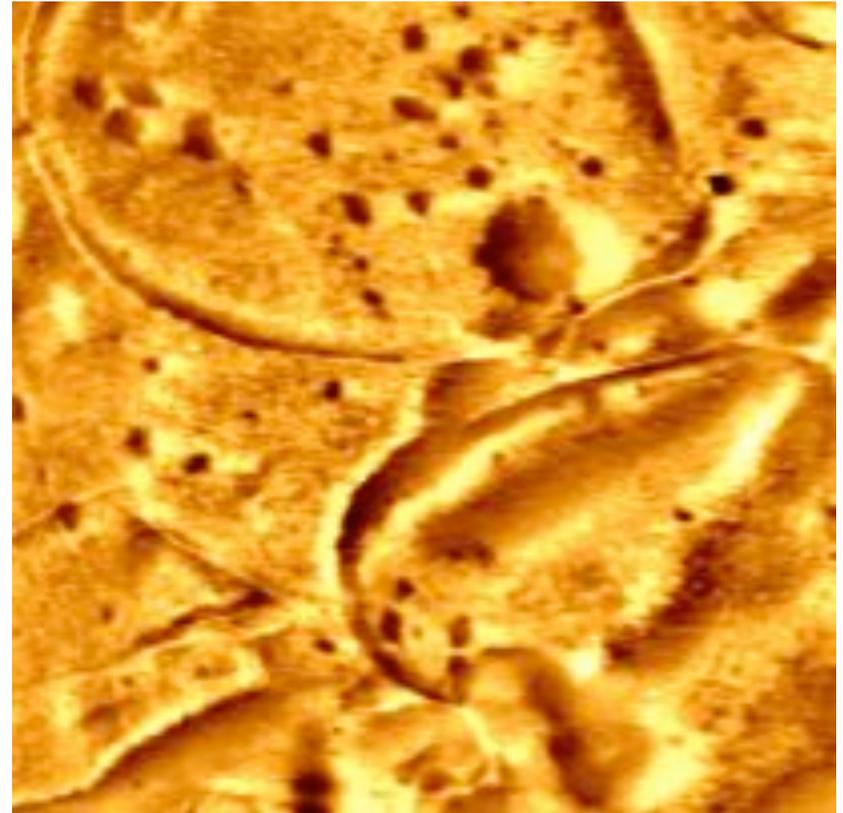
- The tip comes into contact with the sample, and deflects upwards.
- Up-Down Signal of the detector changes.
- $V_z$  is controlled by the feedback system to maintain a constant Up-Down signal, which is called the "Setpoint".
- $V(x,y)$  of each scanning point  $(x,y)$  is recorded.
- Sample topography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

# Samples of AFM and LFM

Blood Cells (12 $\mu$ m)



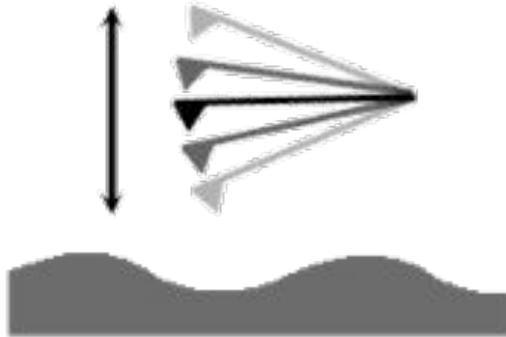
Topography



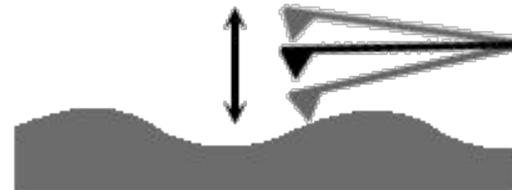
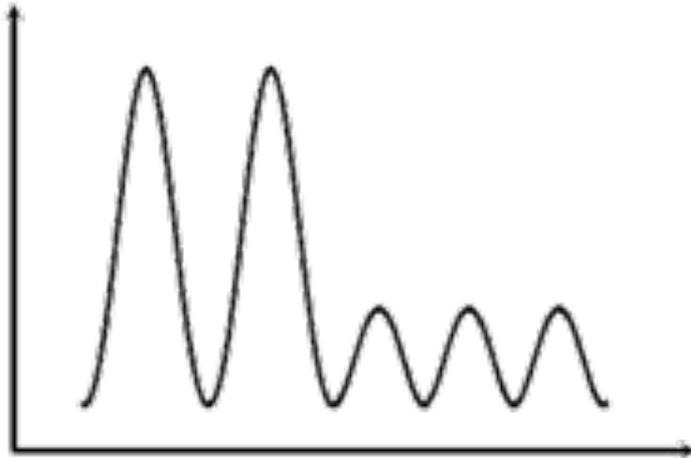
LFM

**Low friction parts can be observed on LFM image.**

# AFM- dynamic Mode



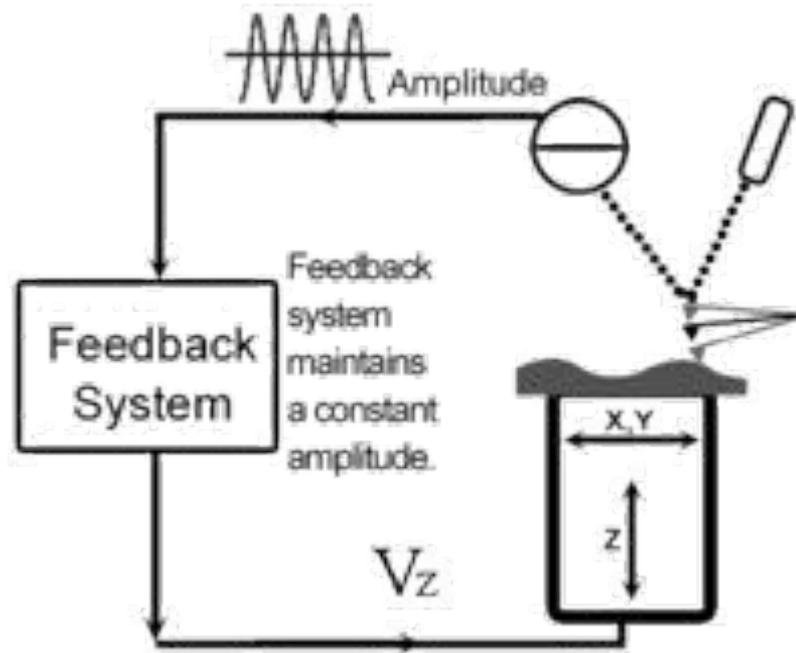
**Cantilever is vibrating normal to the surface.**



**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.**

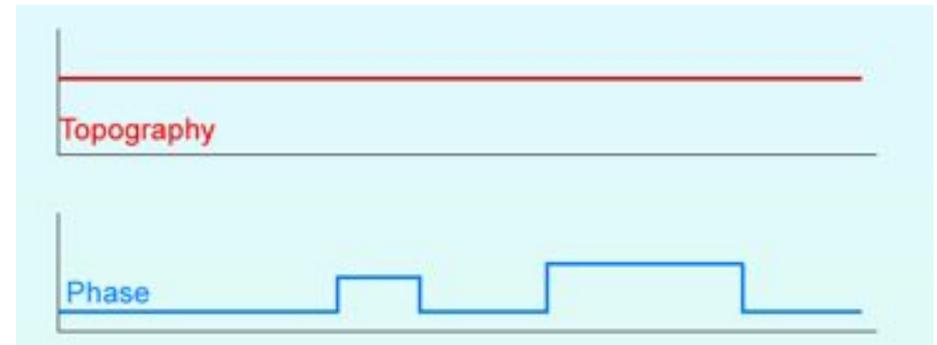
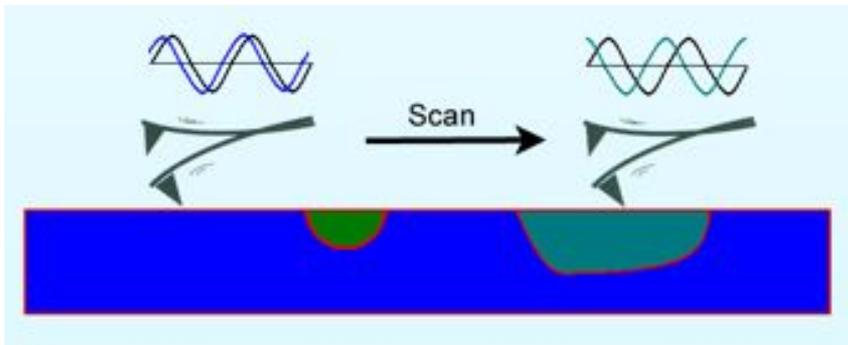
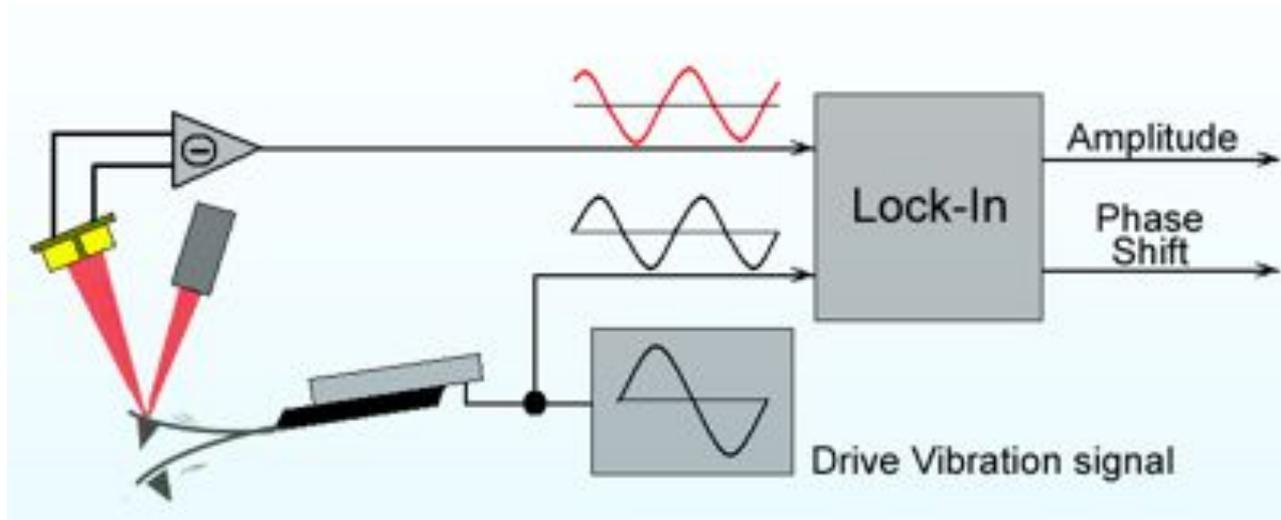
# Dynamic Mode in more detail



- An internal oscillator drives the cantilever via a small piezo plate.
- The oscillation amplitude is reduced when tip taps the sample.
- $V_z$  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

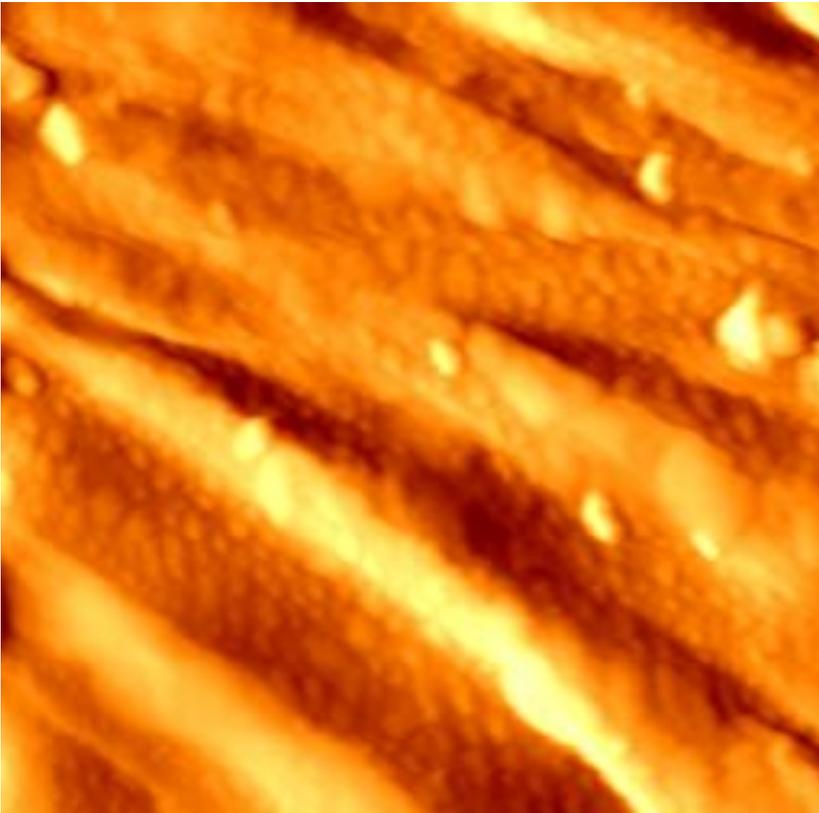
- As the tip scans over the sample surface, changes in the force gradient experienced by the tip (which have many possible origins, including stray electric & magnetic fields, and differences in the mechanical properties of the sample) cause the frequency and phase of the oscillating cantilever to shift. 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.



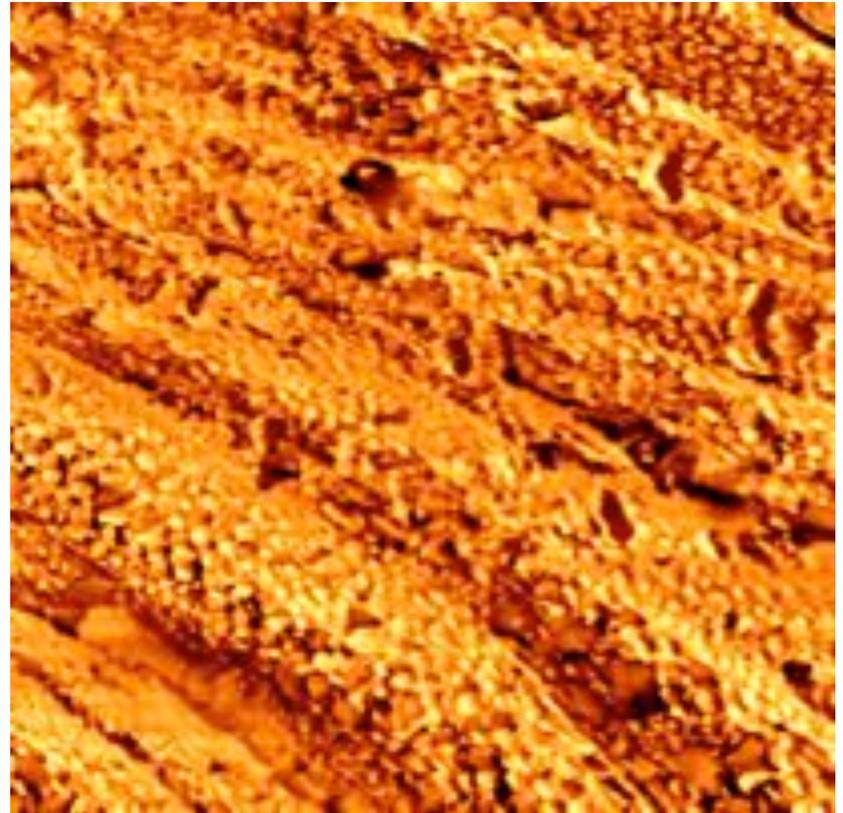
The relative phase of the oscillator and the cantilever depends on local sample properties

# Example of dynamic Mode

Organic Film (9.38 $\mu\text{m}$  area)



**Topography**

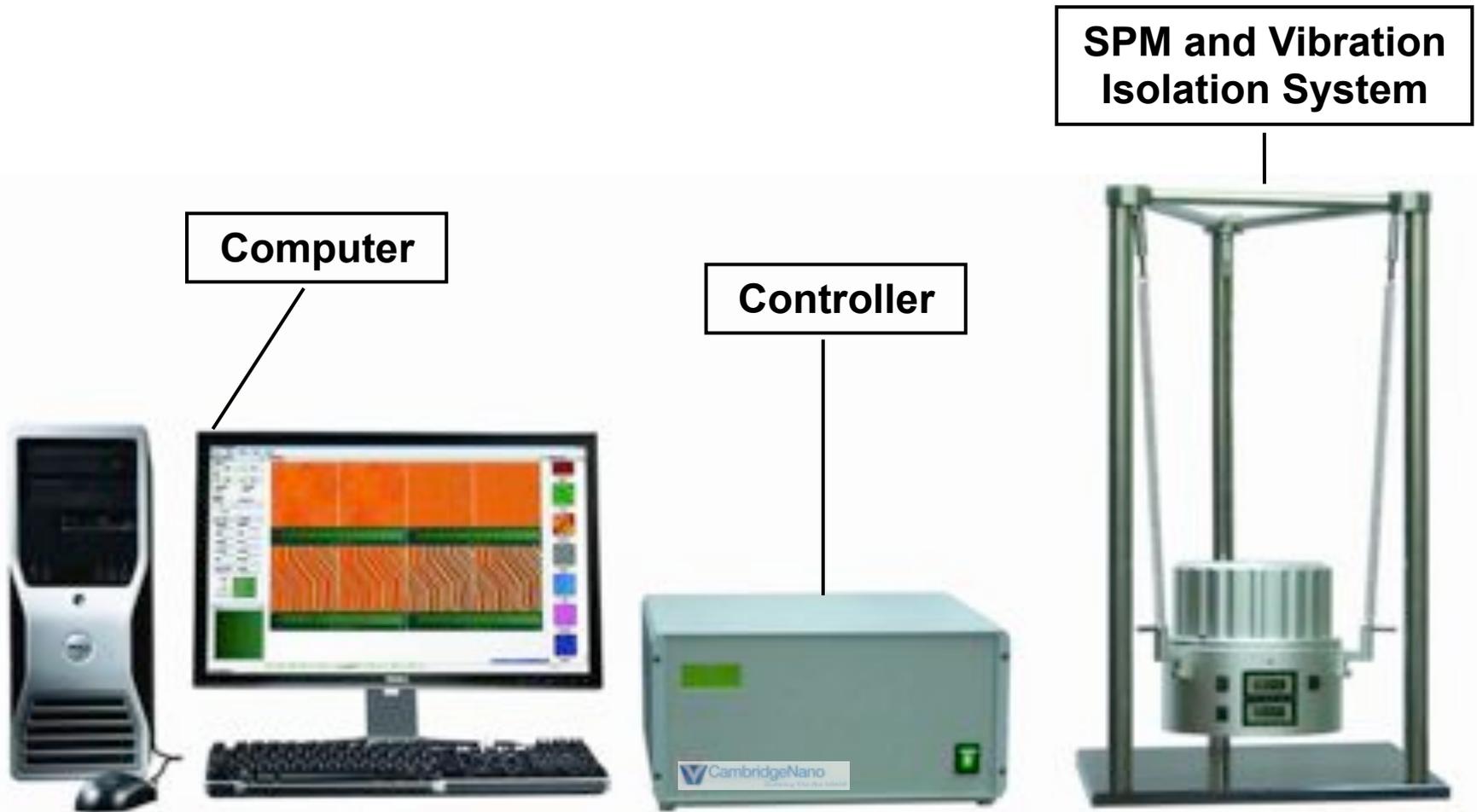


**Phase Image**

# 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



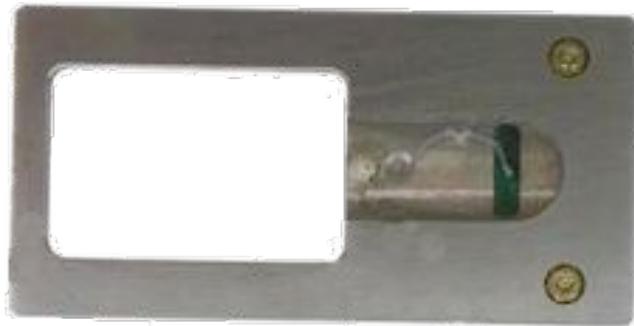
**SPM Head**



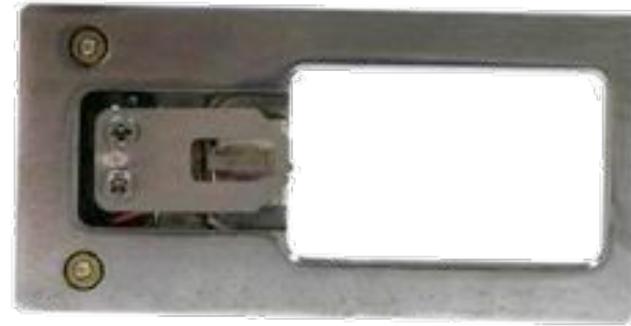
**SPM Base**



**Interchangeable  
Scanners**



**STM Tipholder**

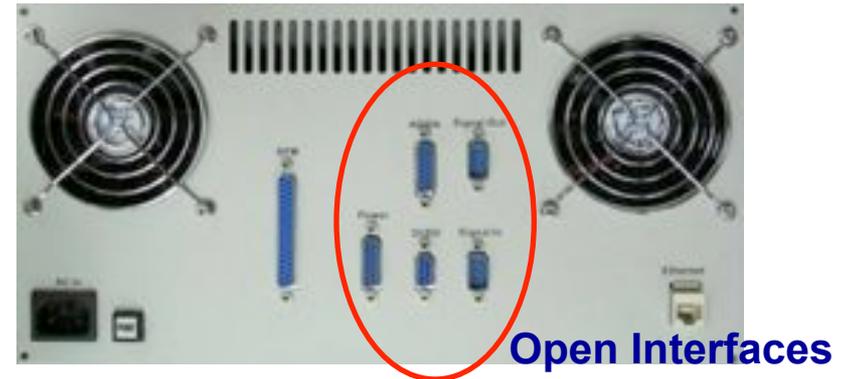


**AFM Tipholder**

# 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

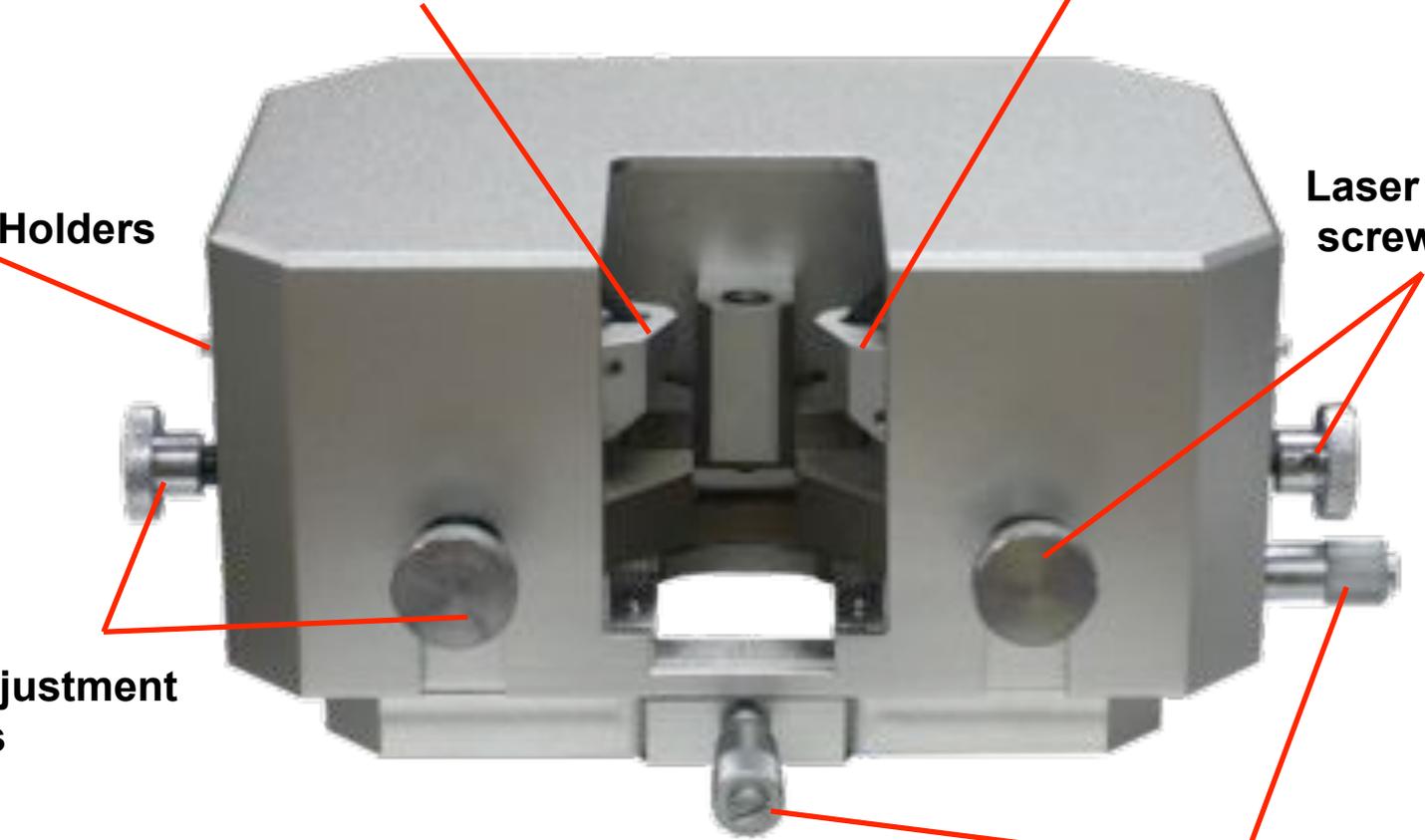
Laser

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.

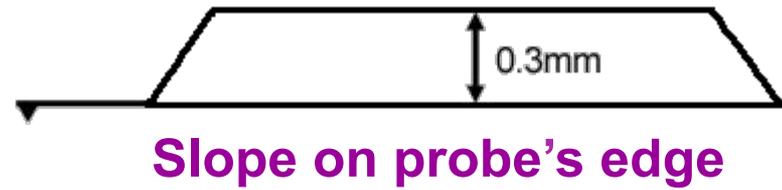
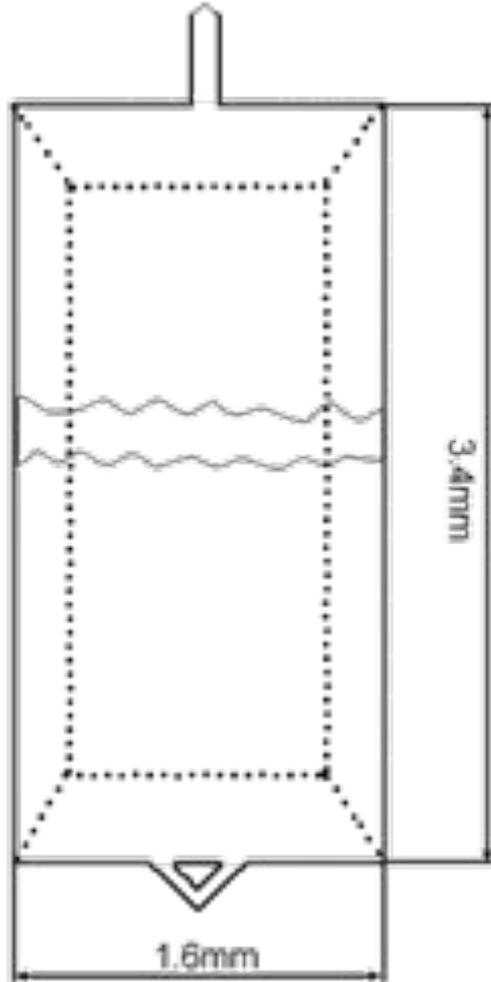
Be very careful when installing or removing the scanner.

- **Use**

**Cover must be removed before use.**



# Probes for AFM



**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...

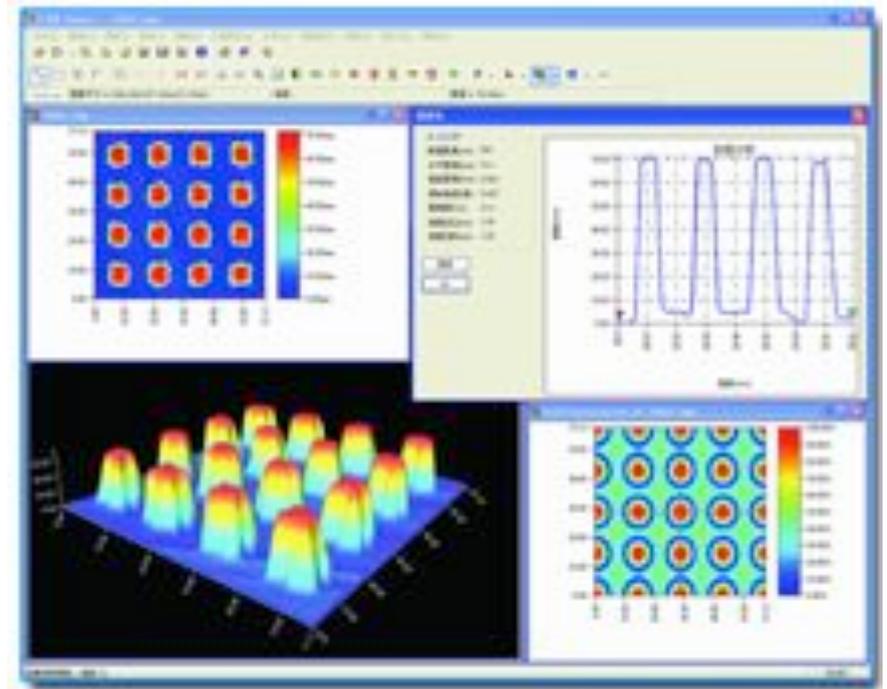
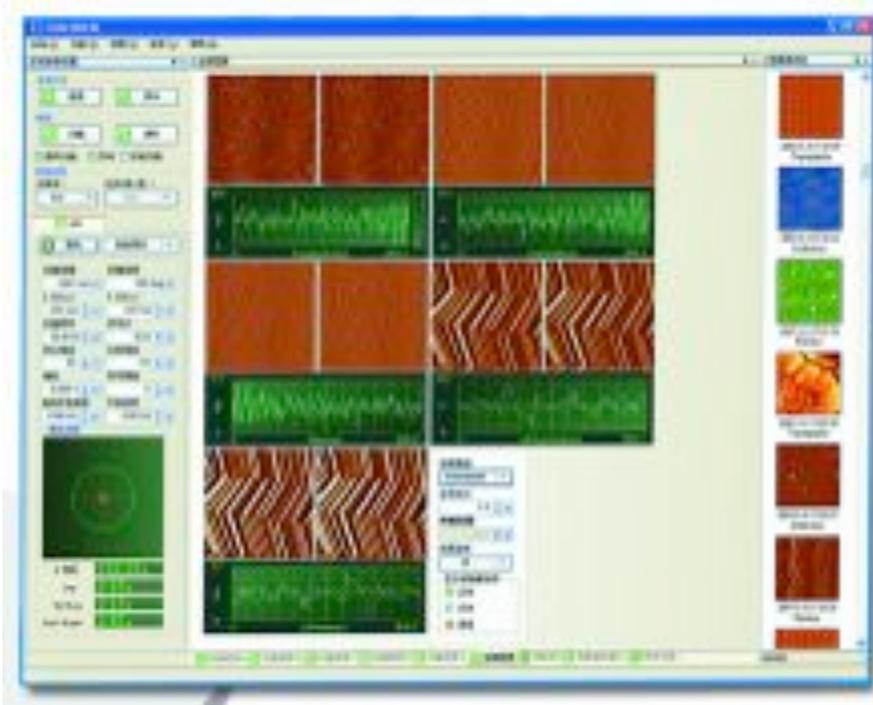
## Difference between probes for contact and dynamic mode

	Contact Mode	Dynamic Mode
Length	450 $\mu\text{m}$	125 $\mu\text{m}$
Width	50 $\mu\text{m}$	30 $\mu\text{m}$
Thickness	2 $\mu\text{m}$	4 $\mu\text{m}$
Force Constant	0.2N/m	40N/m
Frequency	13kHz	300kHz

# Software

SPM control software

Imager software for post-processing



# SPM control software

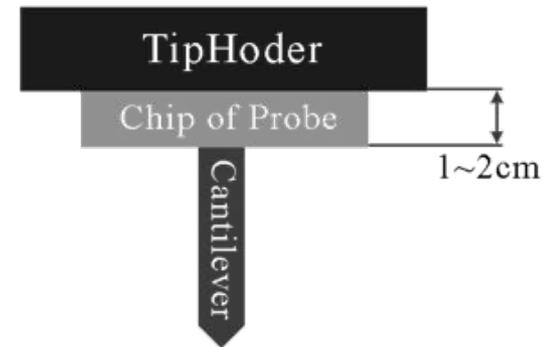
The screenshot displays the SPM control software interface, which is divided into several functional areas:

- System Parameters Settings:** Located on the left, it contains various control buttons such as "Connected", "Ready", "Start", "Stop", "Pause", and "Resume". It also includes "Scan Settings" and "Acquire Settings" sections.
- Scan Image:** A large central window showing a dark scan area with a color scale on the right. The text "Scan Image" is overlaid on this window.
- Image Buffer:** A grid of small image thumbnails on the right side, each with a timestamp and label like "Topography" or "Amplitude". The text "Image Buffer" is overlaid on this grid.
- Feedback Parameters:** A section in the lower-left area showing numerical values for "Feedback", "Z Voltage", "Piezo", "Piezo Drive", and "Piezo Drive". A yellow circle highlights these values, with a label "Signals" pointing to it.
- Oscilloscope Window:** A green window at the bottom center displaying the text "oscilloscope window shows the signal of present scan line".
- Image/function sketch:** A small window at the bottom right showing a circular diagram with a red dot, representing a scan area or function. A red box highlights this window with the label "Image/function sketch".
- Scan image parameters:** A section in the lower-right area showing parameters like "MaxImage = 2000lines", "Step = 2000lines", "Z = 0mm", and "Z = 0mm". A blue arrow points to this section with the label "Scan image parameters".
- Scan Area:** A red box highlights a specific area in the "Scan Settings" section, with a red arrow pointing to it and the label "Scan Area".
- Feedback parameters:** A blue box highlights a section in the "Scan Settings" area, with a blue arrow pointing to it and the label "Feedback parameters".

# Basic Operation of AFM- Probe installation

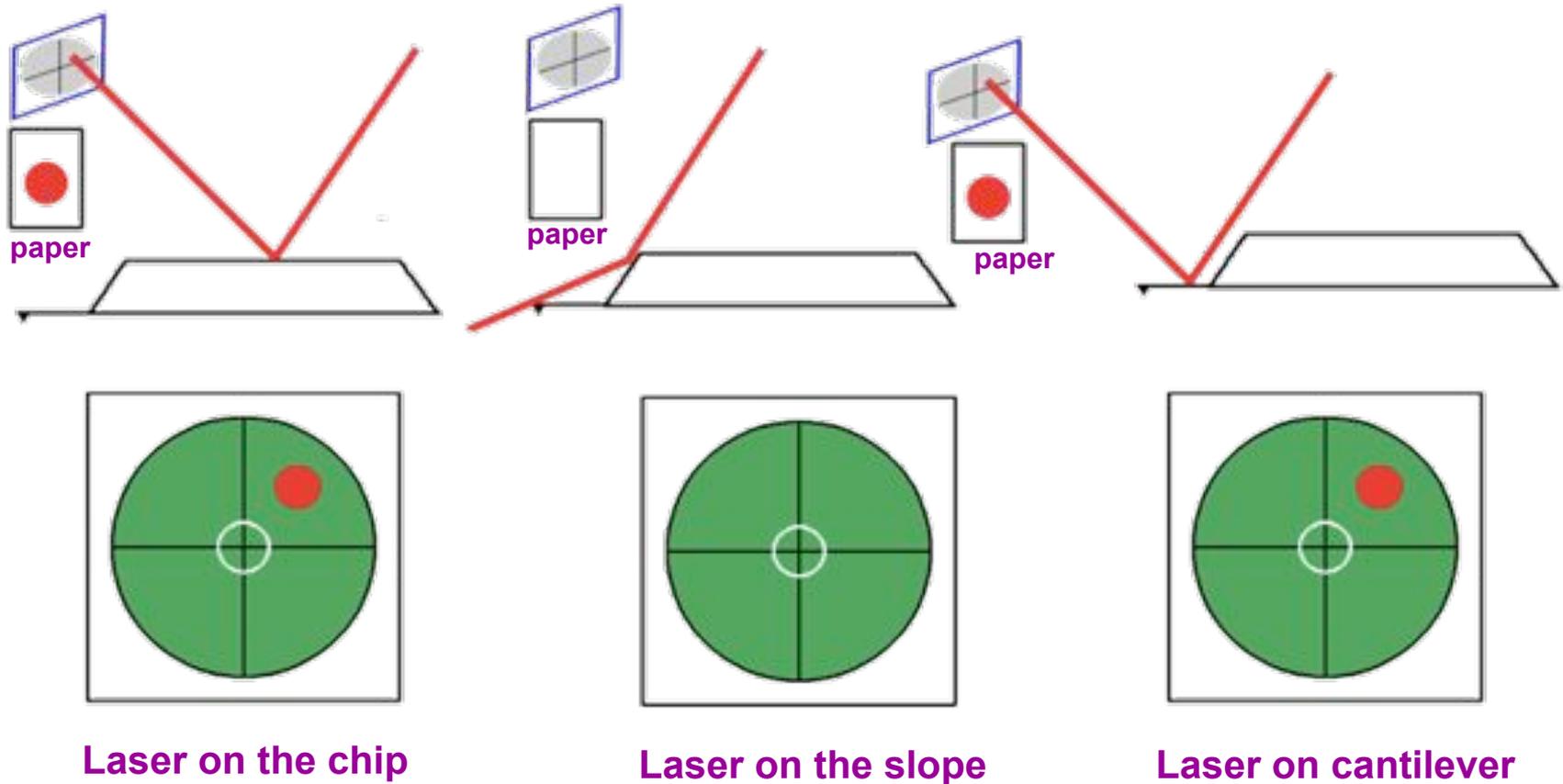


- Gently and evenly lift the spring clip with two fingers
- Insert the probe chip using tweezers



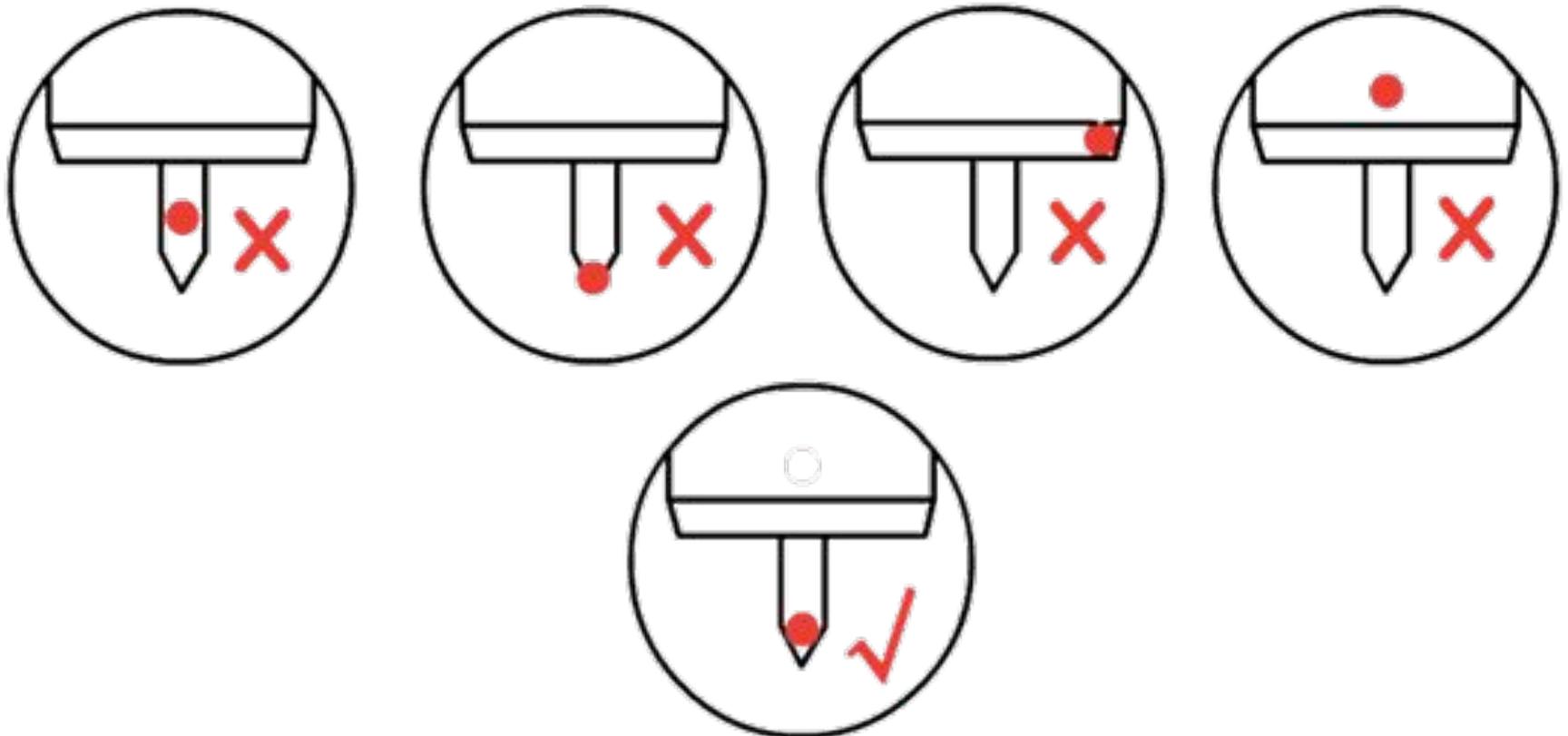
# Basic Operation of AFM- Laser Alignment

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



# 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

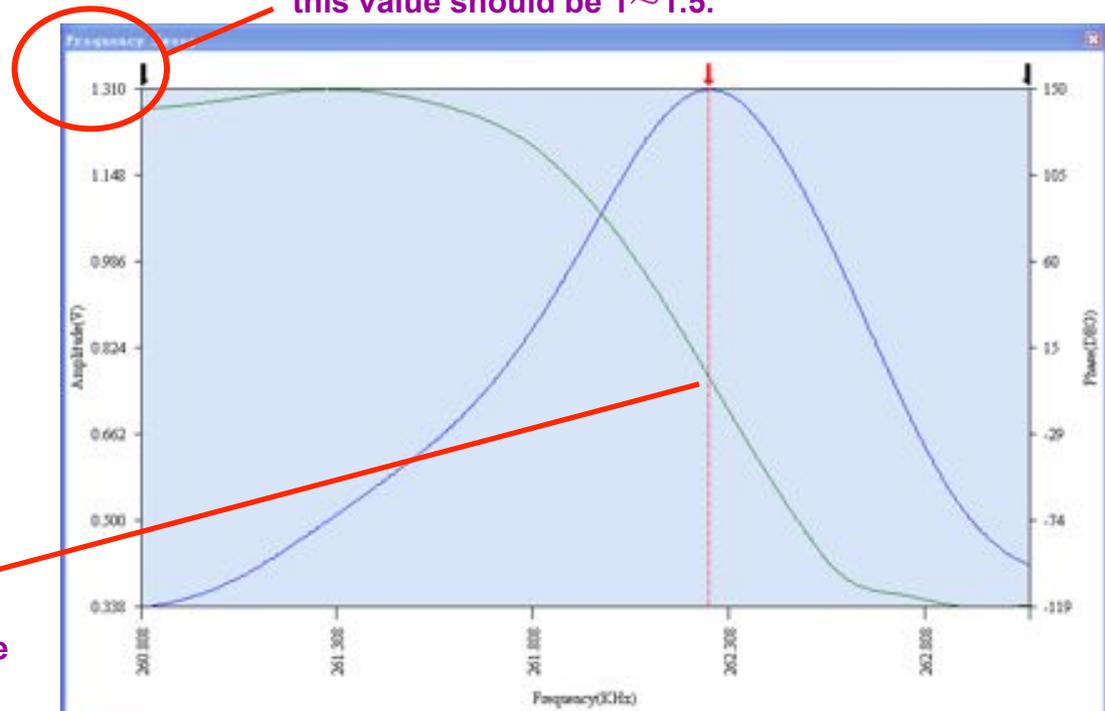
- Open the Frequency sweep window, choose Amplitude channel.
- Adjust drive amplitude, peak value should be 1~1.5.
- Red cursor sets the working frequency and amplitude, it must be set to the left side of the peak.
- Setpoint is typically 70% of the amplitude value.
- Proportional gain and Integral gain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



Setpoint is 70% of this value.

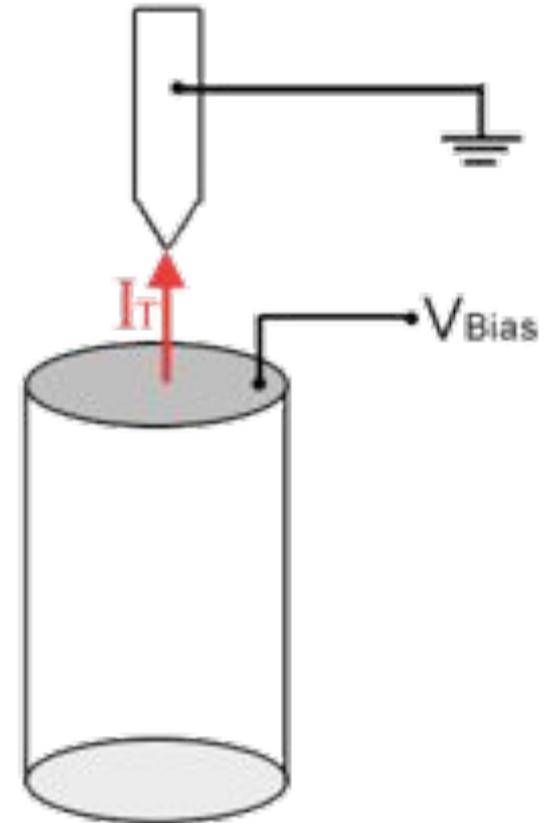
Adjust tapping drive amplitude, this value should be 1~1.5.

Red cursor just below the left side of the peak.



# Basic Operation of STM

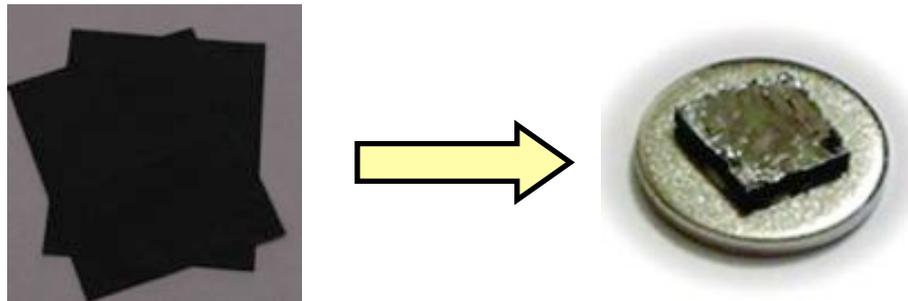
- Bias Voltage is applied to the disk of scanner (where the sample is mounted), so the sample's conductive side must be connected to the disk.
- Log mode is commonly used for most samples.
- Conductivity is affected by sample contamination, sample cleaning is necessary before experimentation.
- For metallic 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

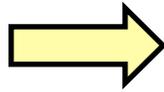
● Powder samples are best to be treated as in SEM (Scanning Electron Microscope) scans, ultrasound-scatter and tablet press are commonly used methods;



# 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  $0.1 \sim 1\text{g/L}$ ;
- 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



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



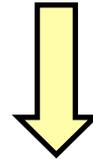
Drop the scattered solution on a substrate (silicon or mica), use dynamic mode AFM to image when dried.

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

# Powder- ultrasound- tablet press



**A tablet press machine**

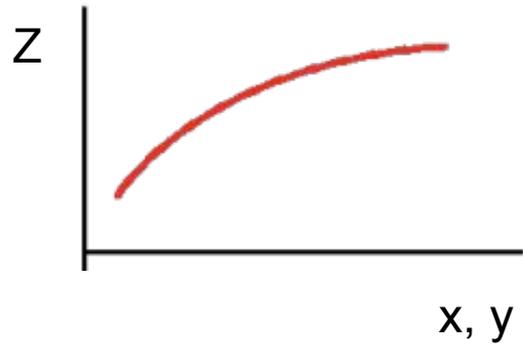


Use a tablet press machine to make the powder sample to a smooth slice; Dynamic mode AFM is usually applied to this kind of sample.

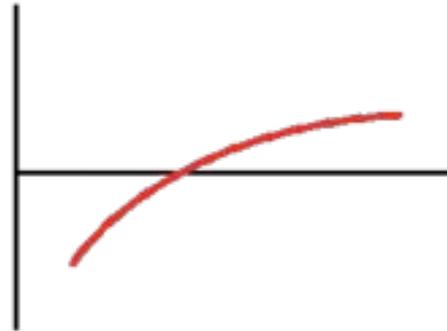
Tablet press method is usually applied to the powder sample which has large particle size or can not be put into liquid.

# Scanning Parameters

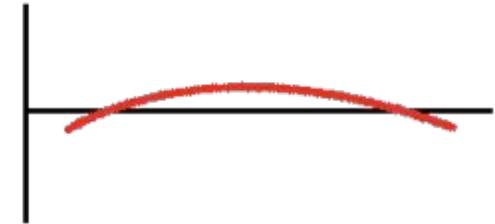
# Surface Fitting –software compensation for bowing of scanner during imaging



Original Signal



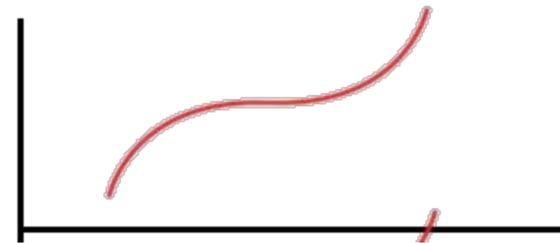
0 Order



1st Order

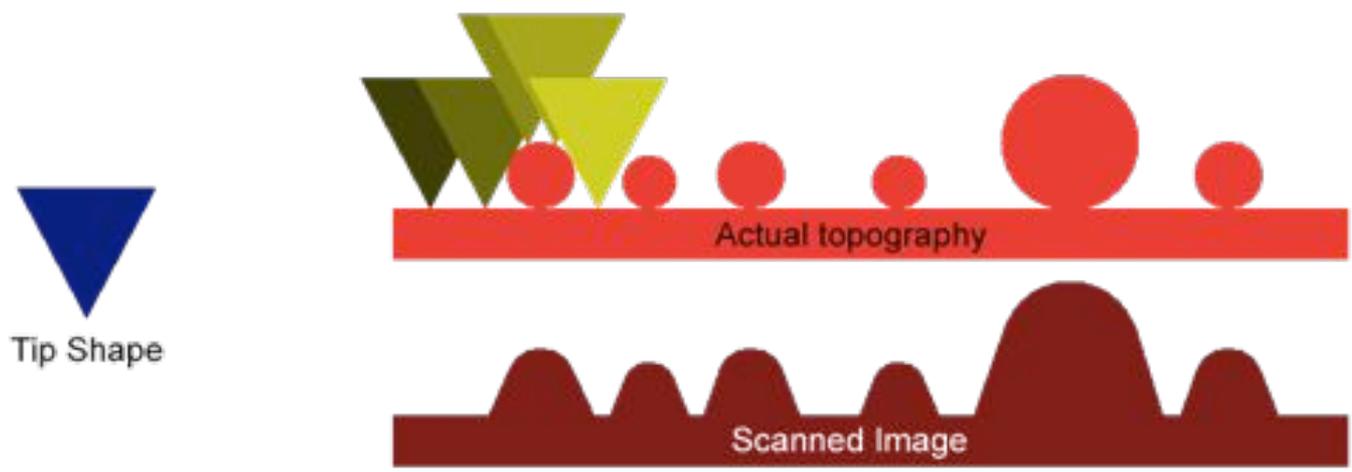
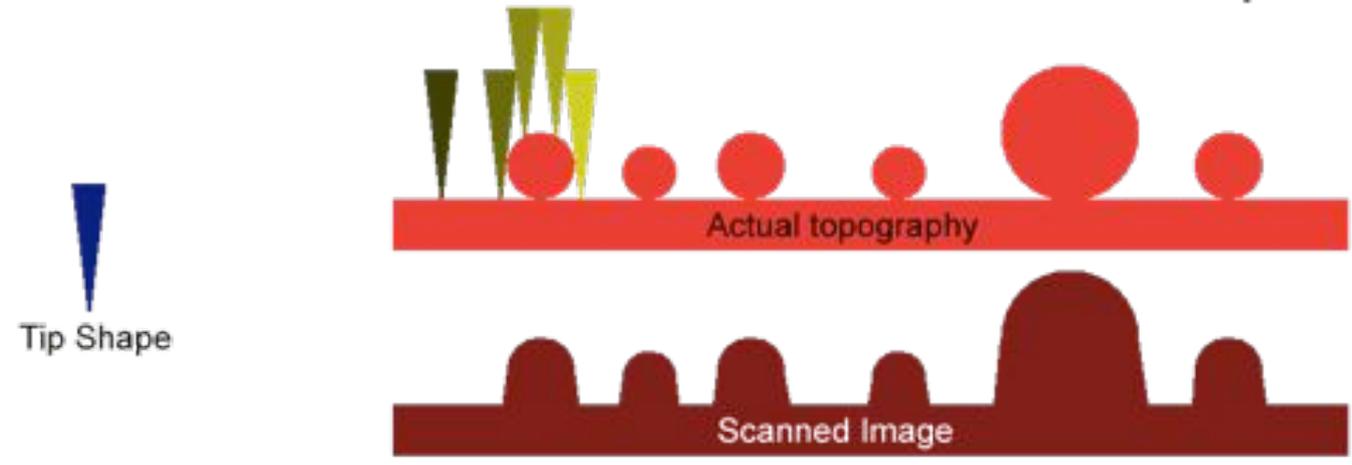


2nd Order

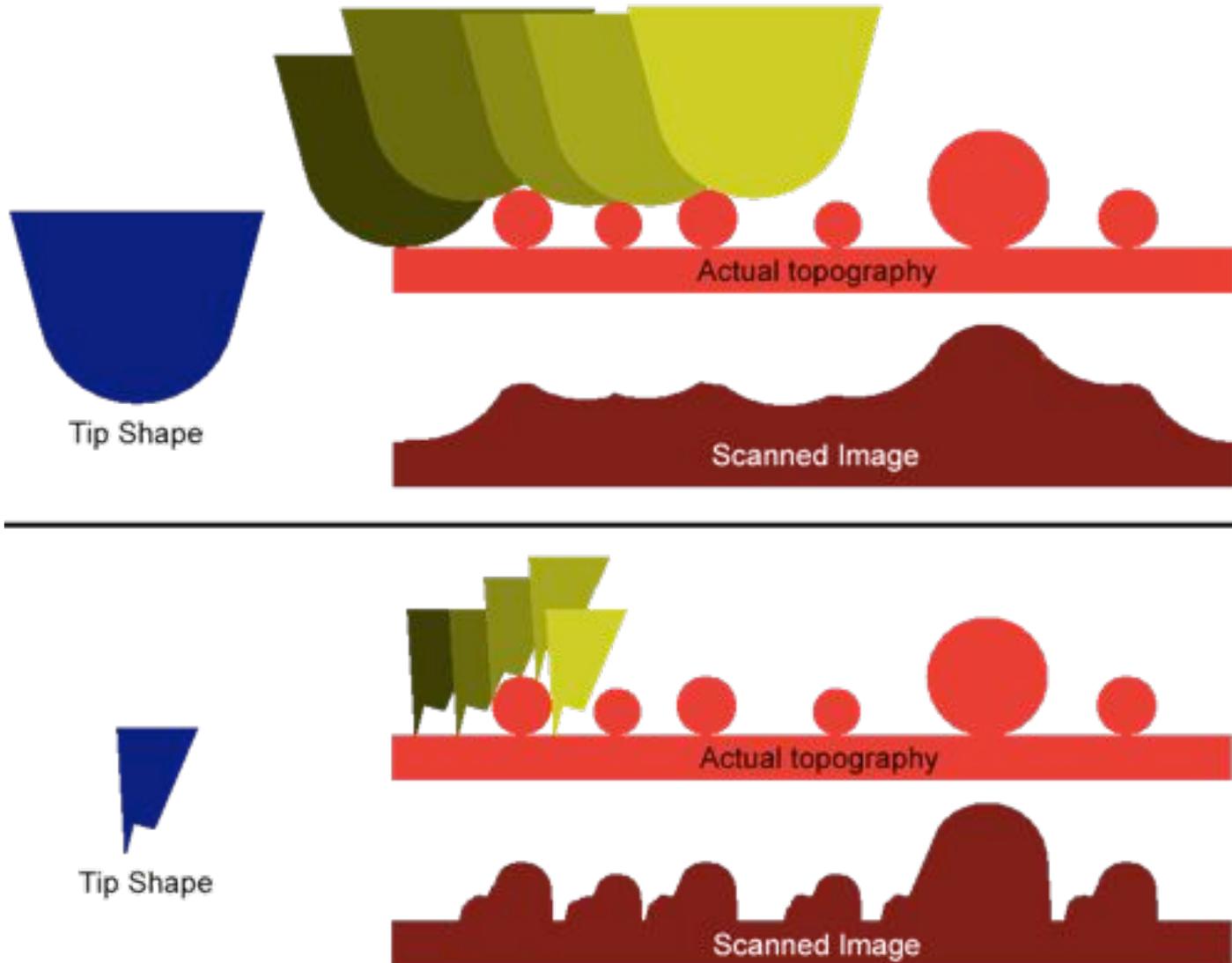


3rd or higher Order

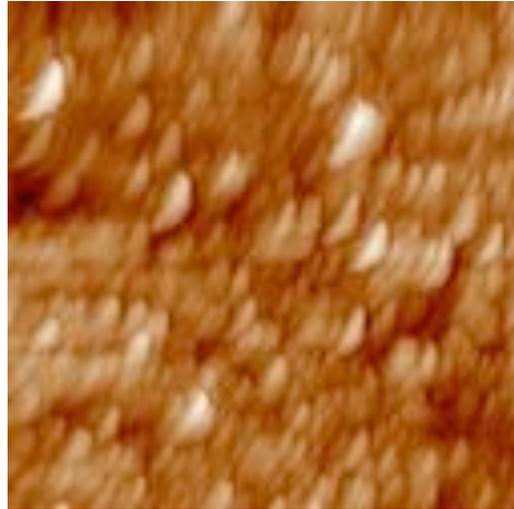
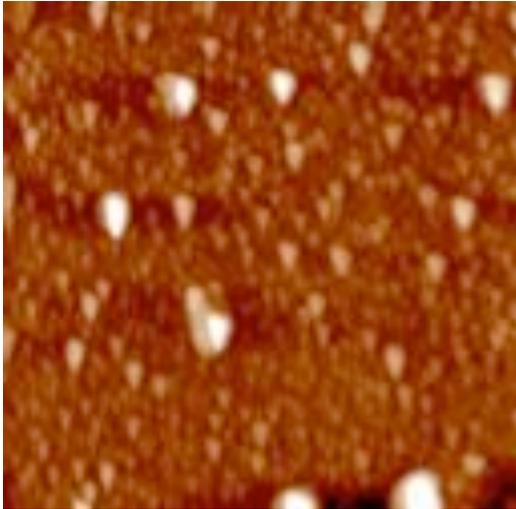
# Influence of Tip



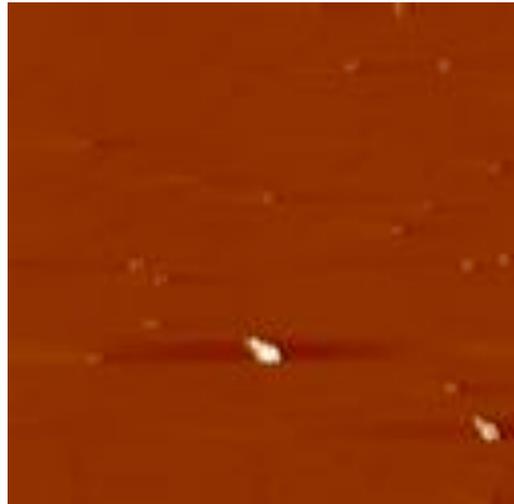
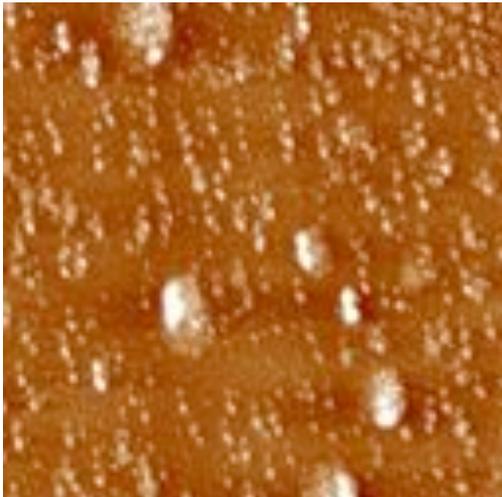
# Influence of Tip



# Influence of Tip



Tip shape is also scanned in the image.



Multi-tip



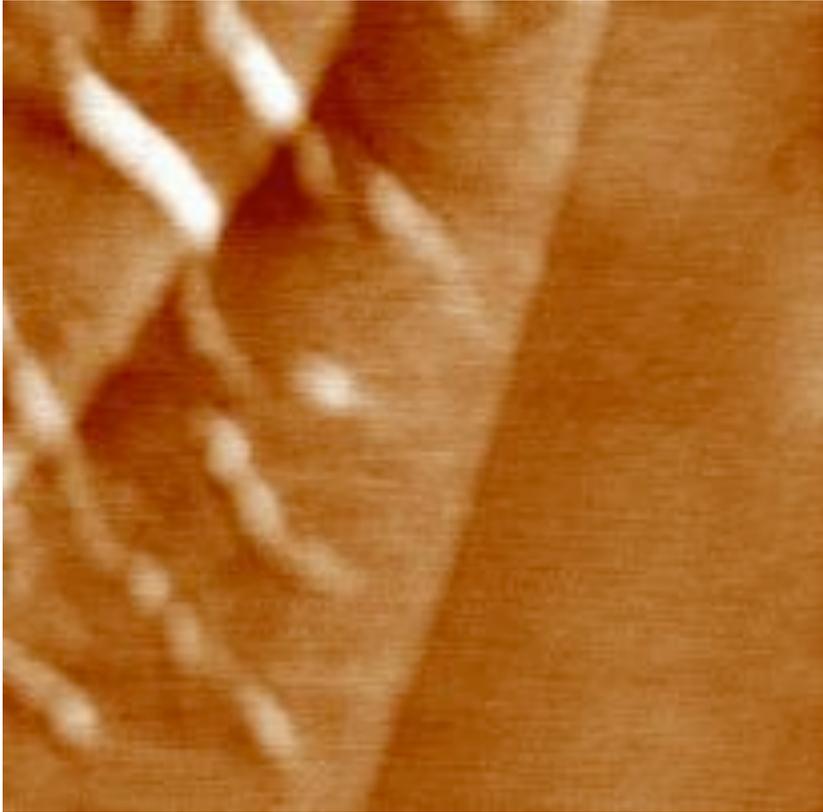
# Imager software for processing

The screenshot displays the Imager software interface. At the top, a menu bar includes File, Edit, View, Image, Filter, Process, Window, and Help. Below the menu bar is a toolbar with various icons for file operations, editing, and processing. A red box highlights a section of the toolbar, with the text "function shortcuts" written in red next to it. On the left side, there is a "System Palette" window showing a line graph with three data series (red, green, and blue) and a color bar below it. The main window, titled "Fiber", displays a large image of a fiber bundle. To the right of the image is a vertical color scale legend ranging from 0 to 800,000. Below the image, technical specifications are listed: Pixels = (512, 512), Size = (2600000, 2600000), and Weight = 300.37ms (71.62). At the bottom left, a status bar reads "Press F1 for get help".

# 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



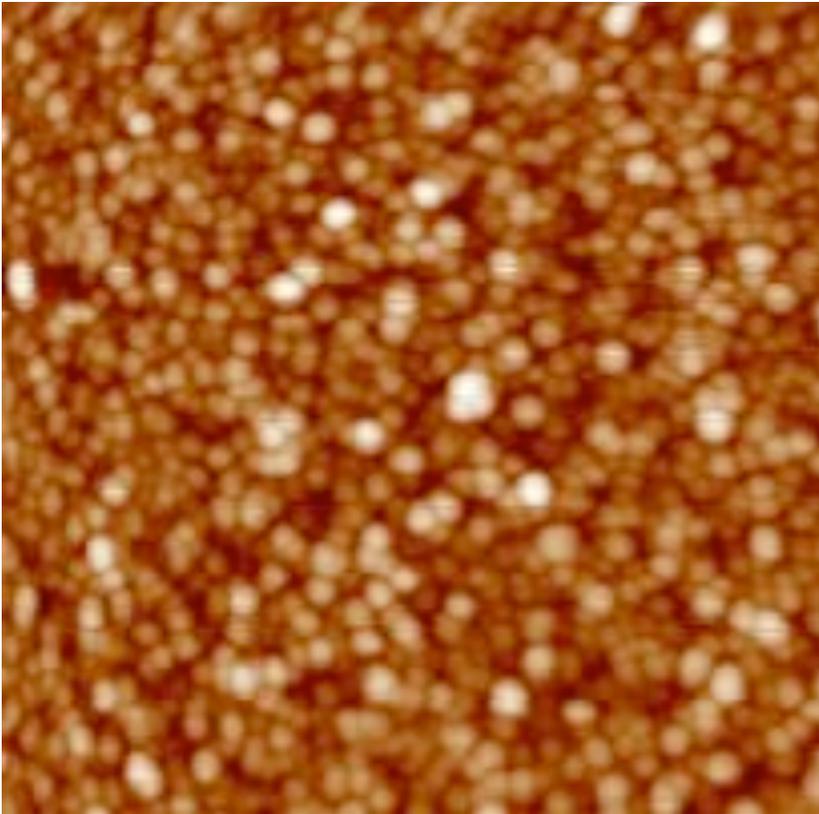
**Scanned  
image**



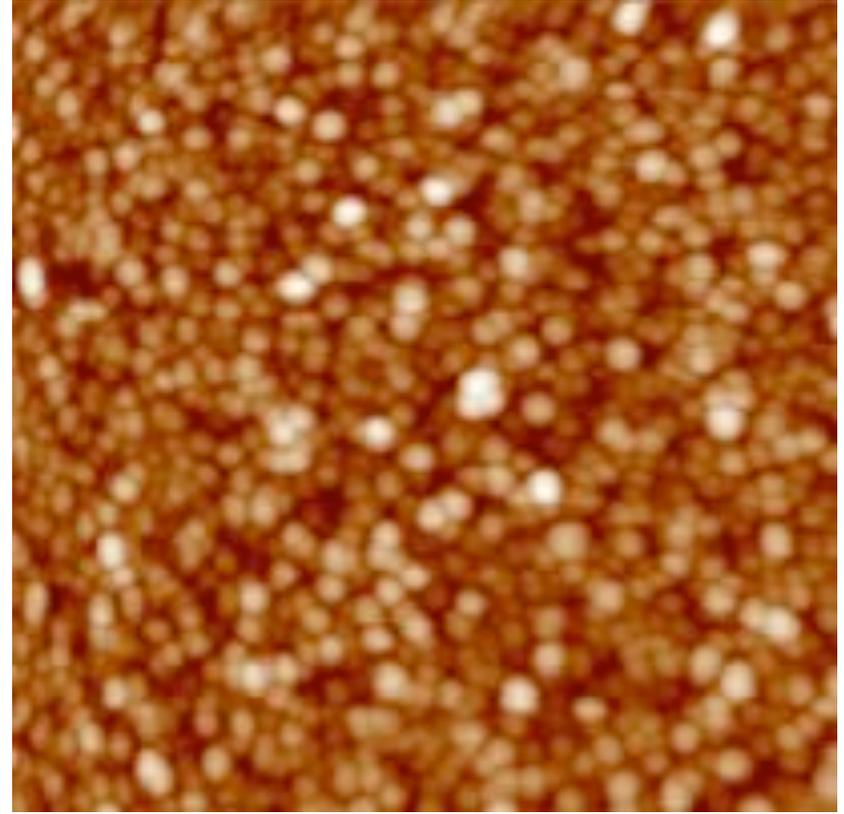
**After one low-pass filter and  
average filter**

# Image process: clear scan line

Clear scan lines automatically or manually



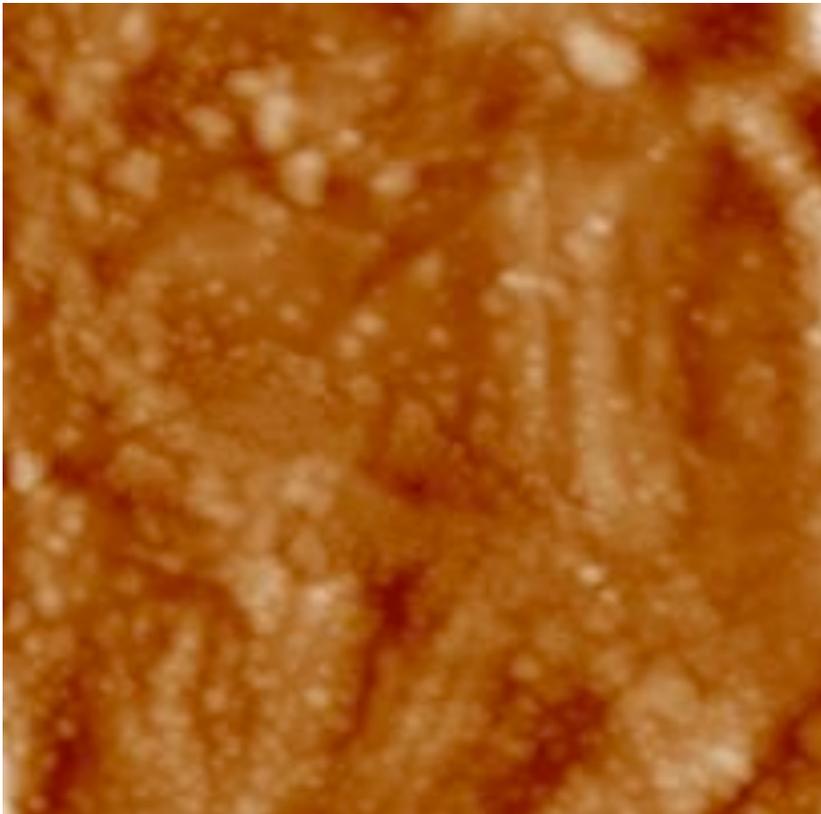
**Scanned  
image**



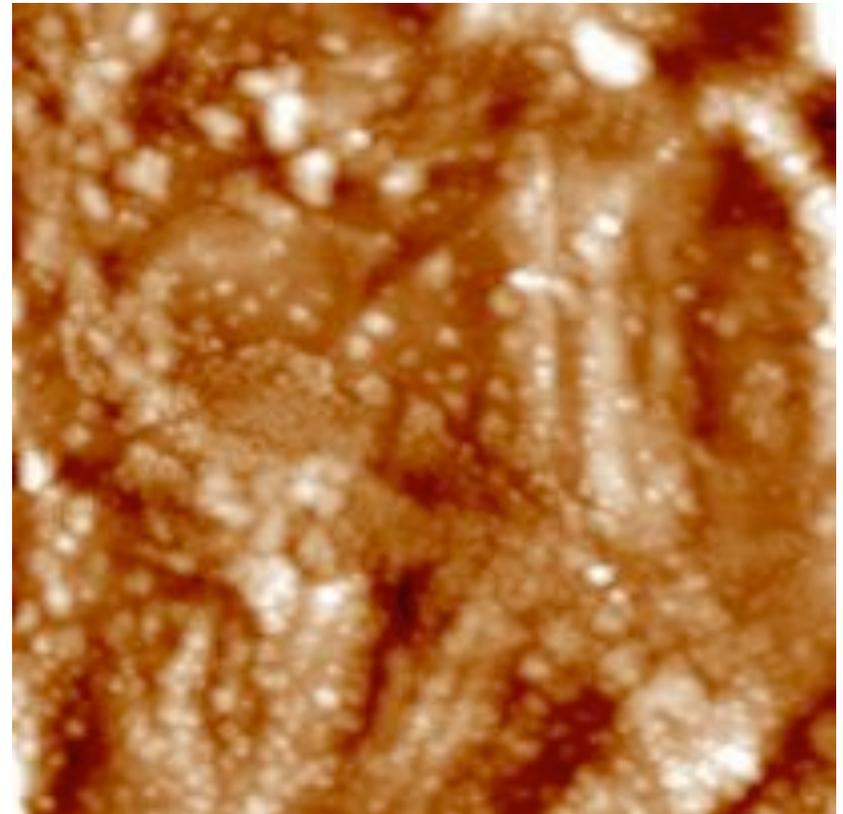
**after**

# Image process: brightness and contrast

Best contrast, brightness and contrast adjustment

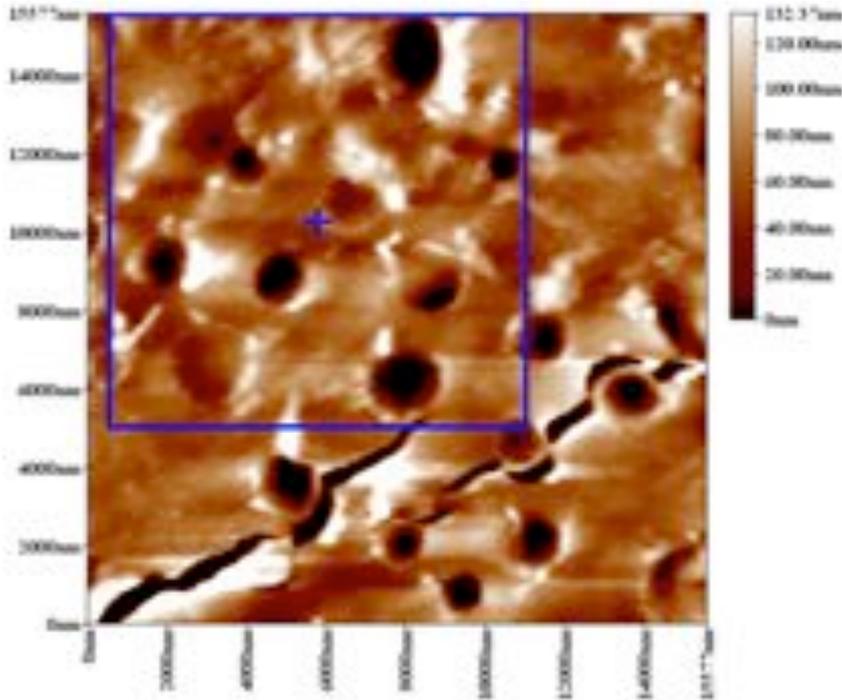


**Scanned  
image**

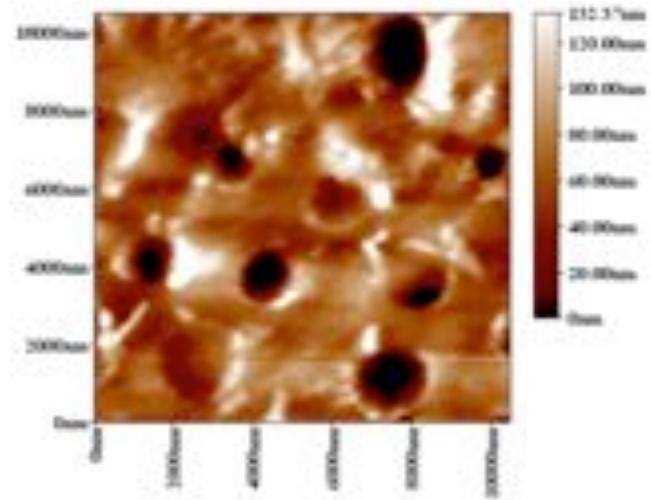


**After adjustment of brightness  
and contrast**

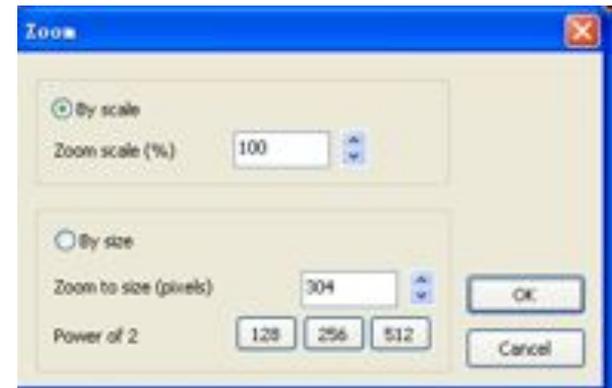
# Image process: Zoom in/out, zone selection



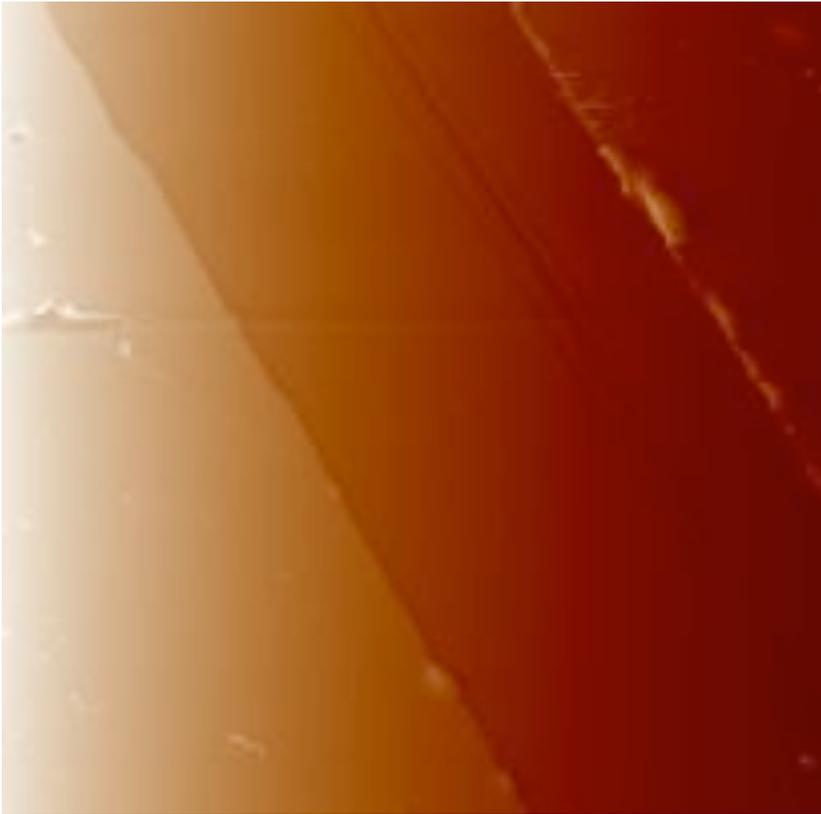
Choose area



cut



# Image process: surface fit, Non-linear correction

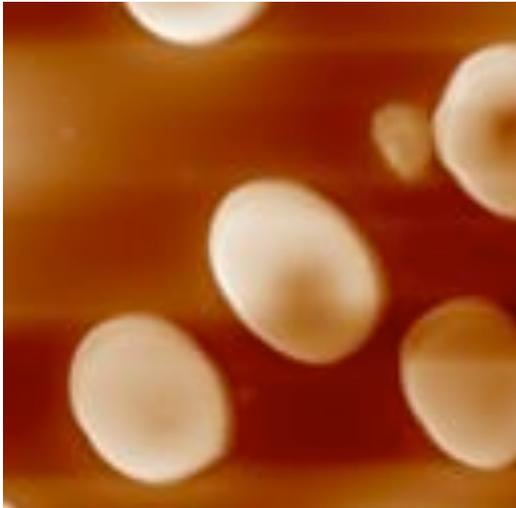


**Scanned image**

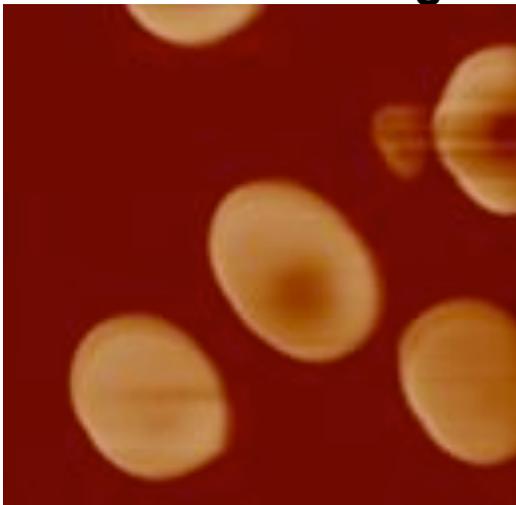


**After a 3-order surface fit**

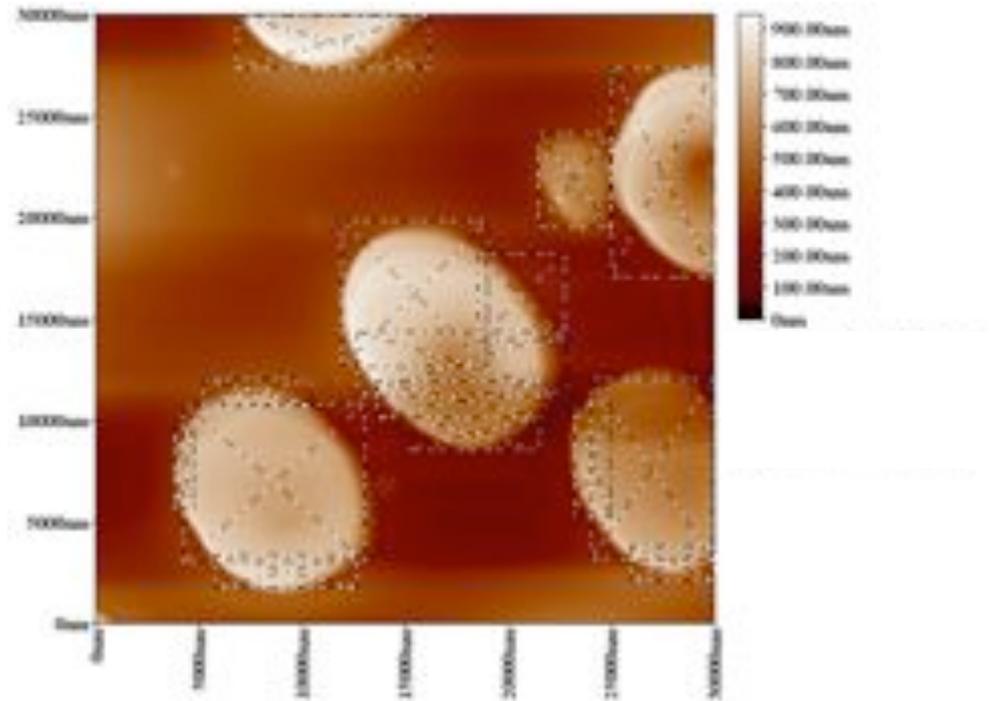
# Image process: surface fit with excluded boxes



**Scanned image**

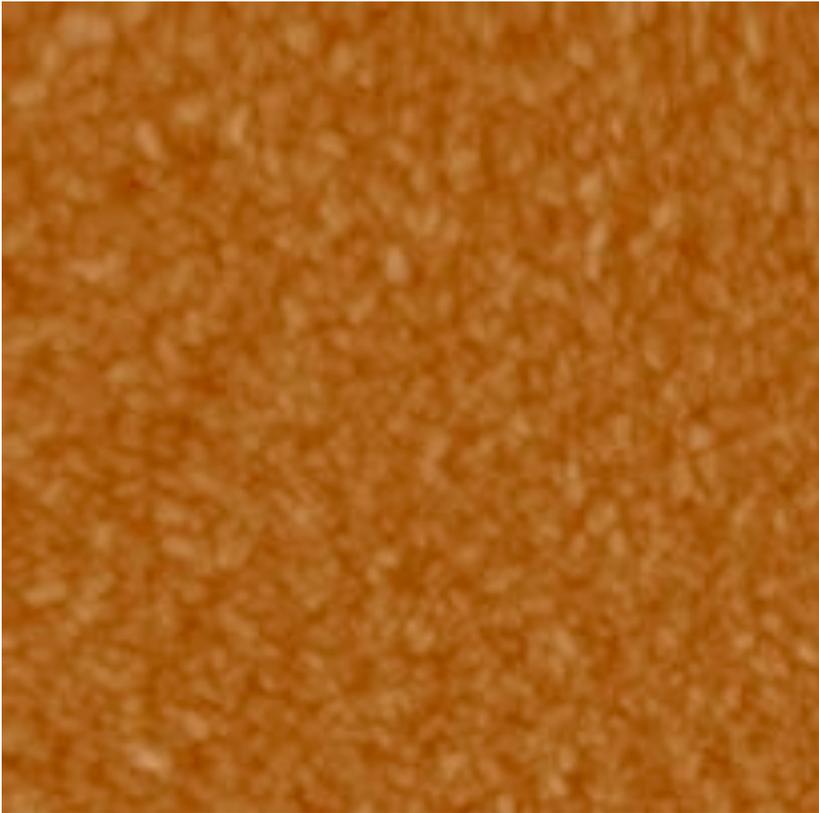


**After a 3rd-order surface fit**

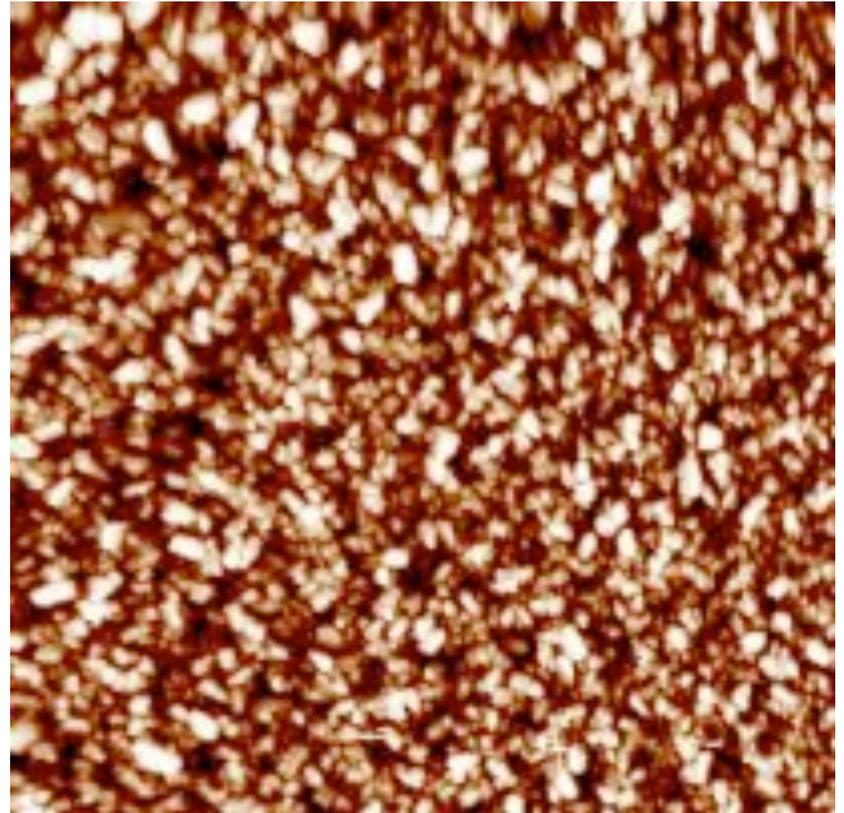


**Use excluded boxes to eliminate the particles, surface fit will not include the distribution of the topography that is inside the boxes.**

## Image process: Equilibrate

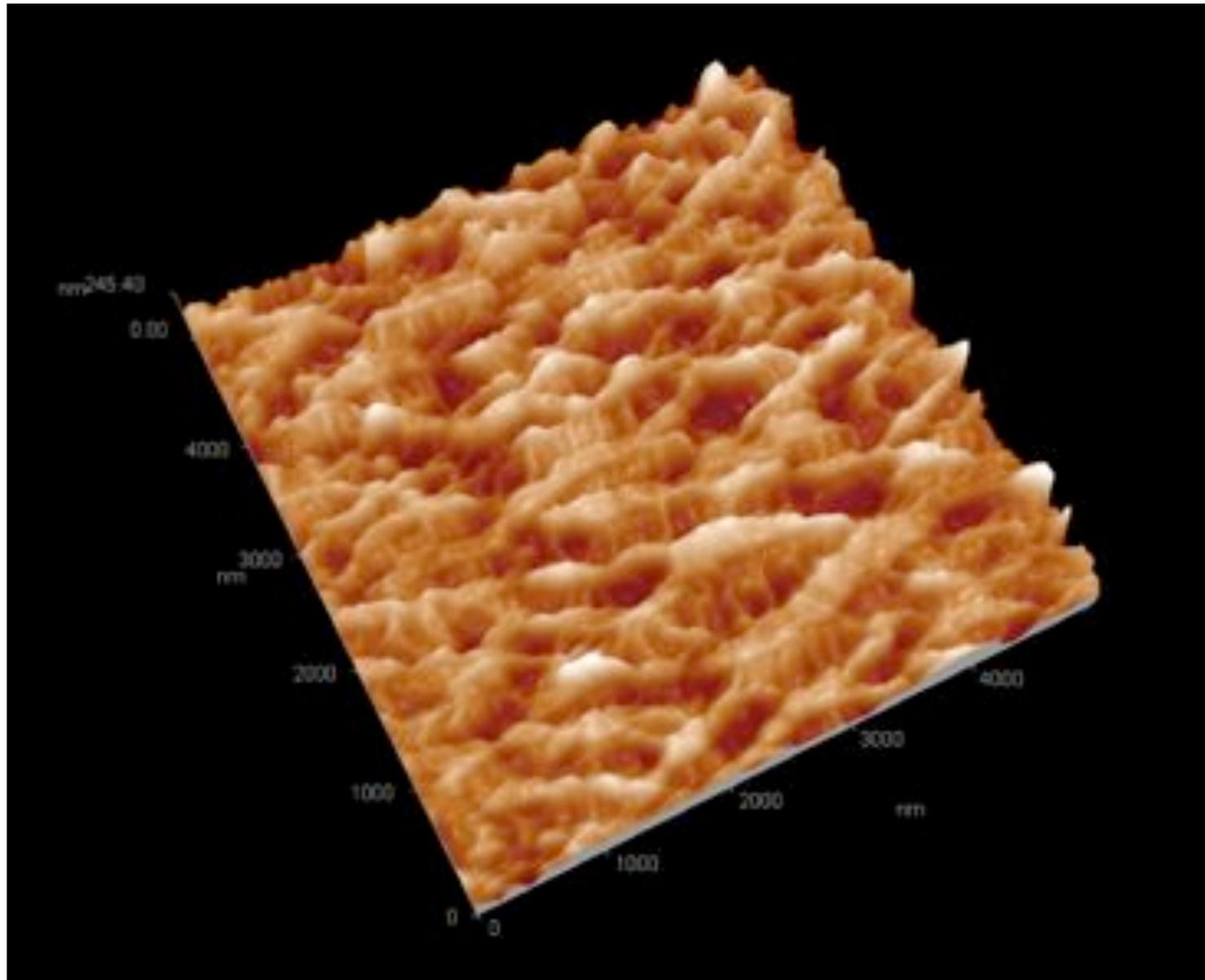


**Scanned image**

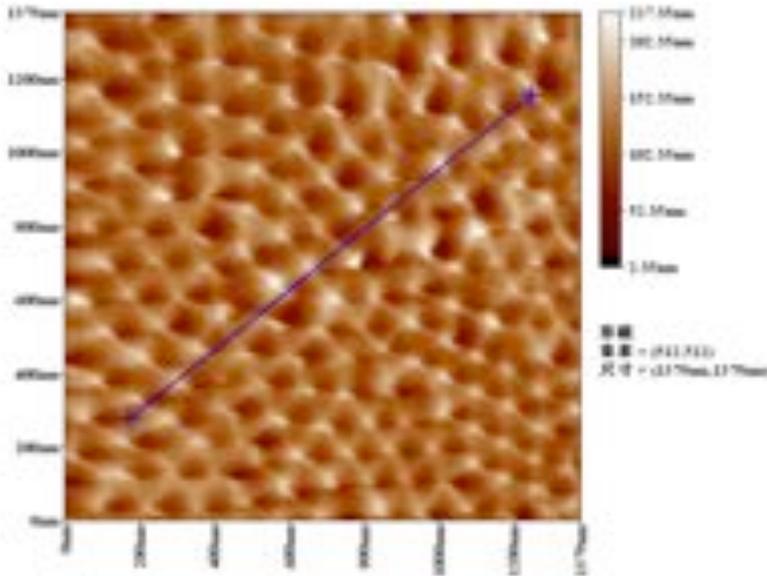


**after**

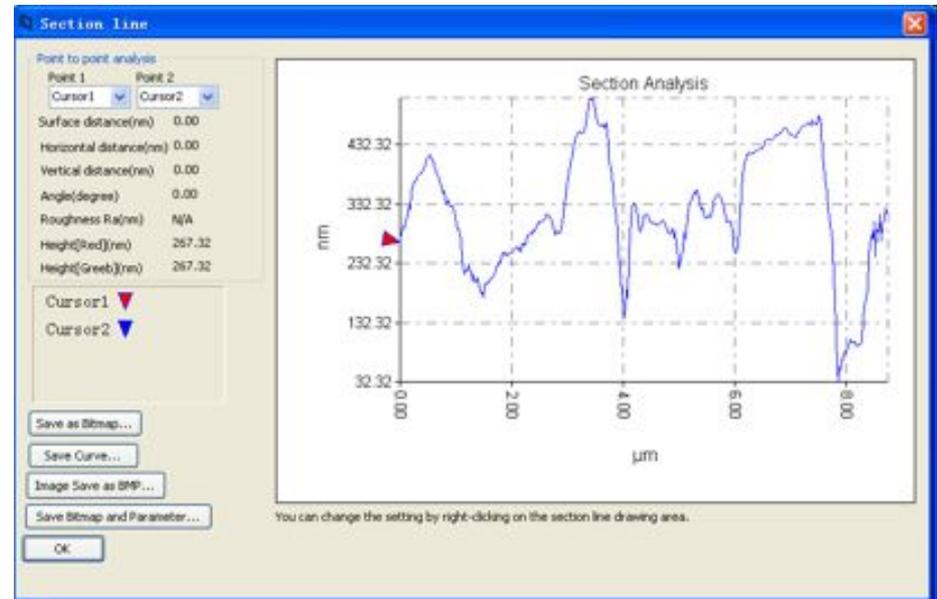
# Image Analysis: 3D image



# Image Analysis: Section line

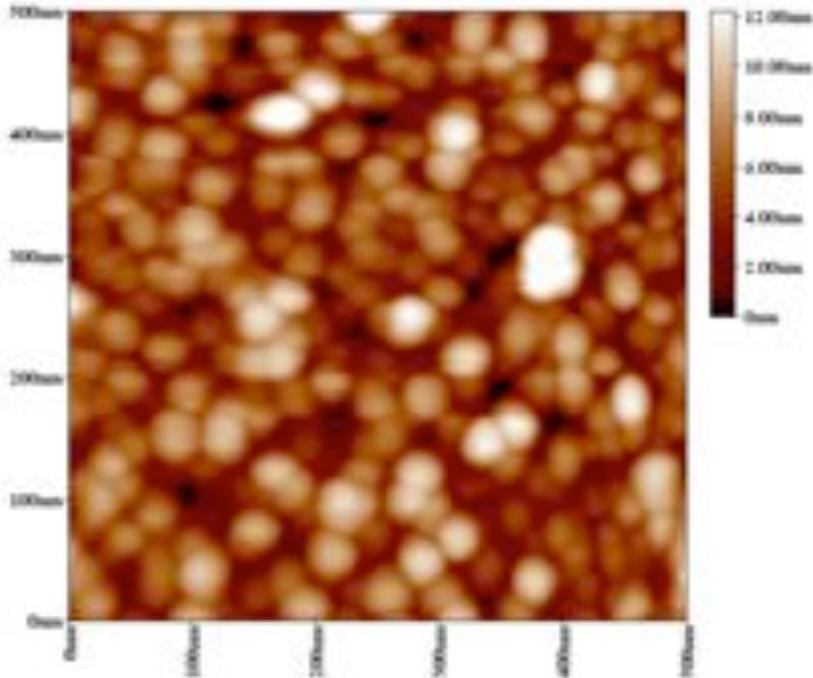


Use a line tool to make a section line

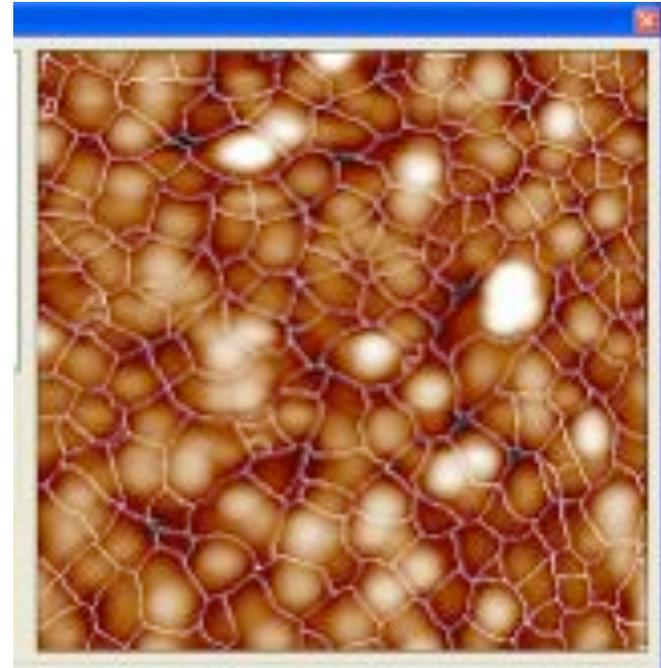


Section Analysis

# Image Analysis: Grain Size Analysis

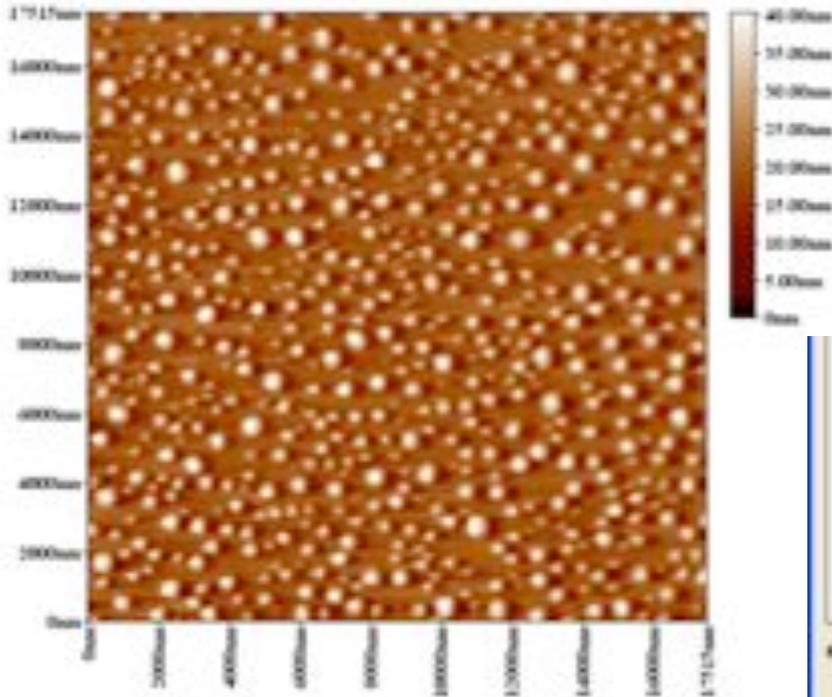


**No obvious substrate  
on sample image**

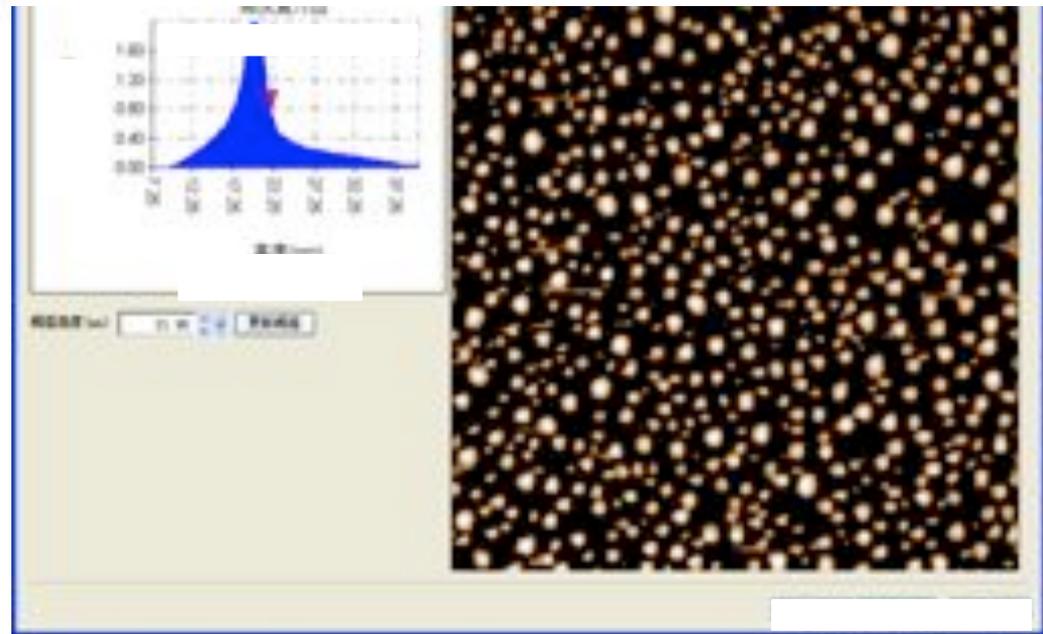


**Automatic grain size analysis**

# Image Analysis: Grain Size Analysis

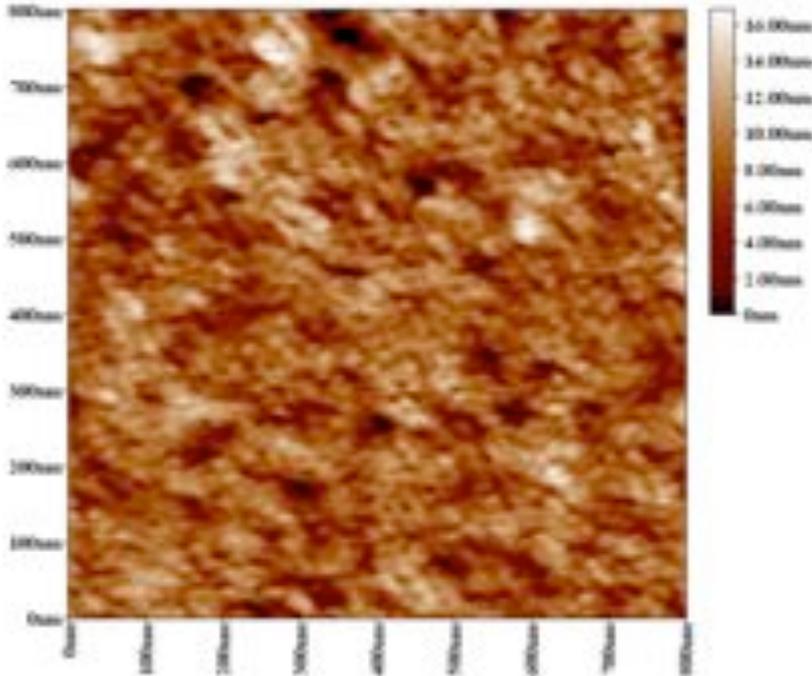


Obvious substrate can be observed on sample image



Set height threshold

# Image Analysis: Surface Roughness Analysis



Scanned image



Surface Roughness Analysis