Characterization, Testing of Nanotechnology Structures and Materials

E SC 216

Unit 4
Advanced Scanning Probe Microscopy

Lecture 1
Probe Characterization Techniques

Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

Characterization on the Nanoscale

- Using nanoscale materials and understanding them are two different things.
- Modern tools help us to manipulate and characterize materials at the nanoscale.
- Two notable innovations:

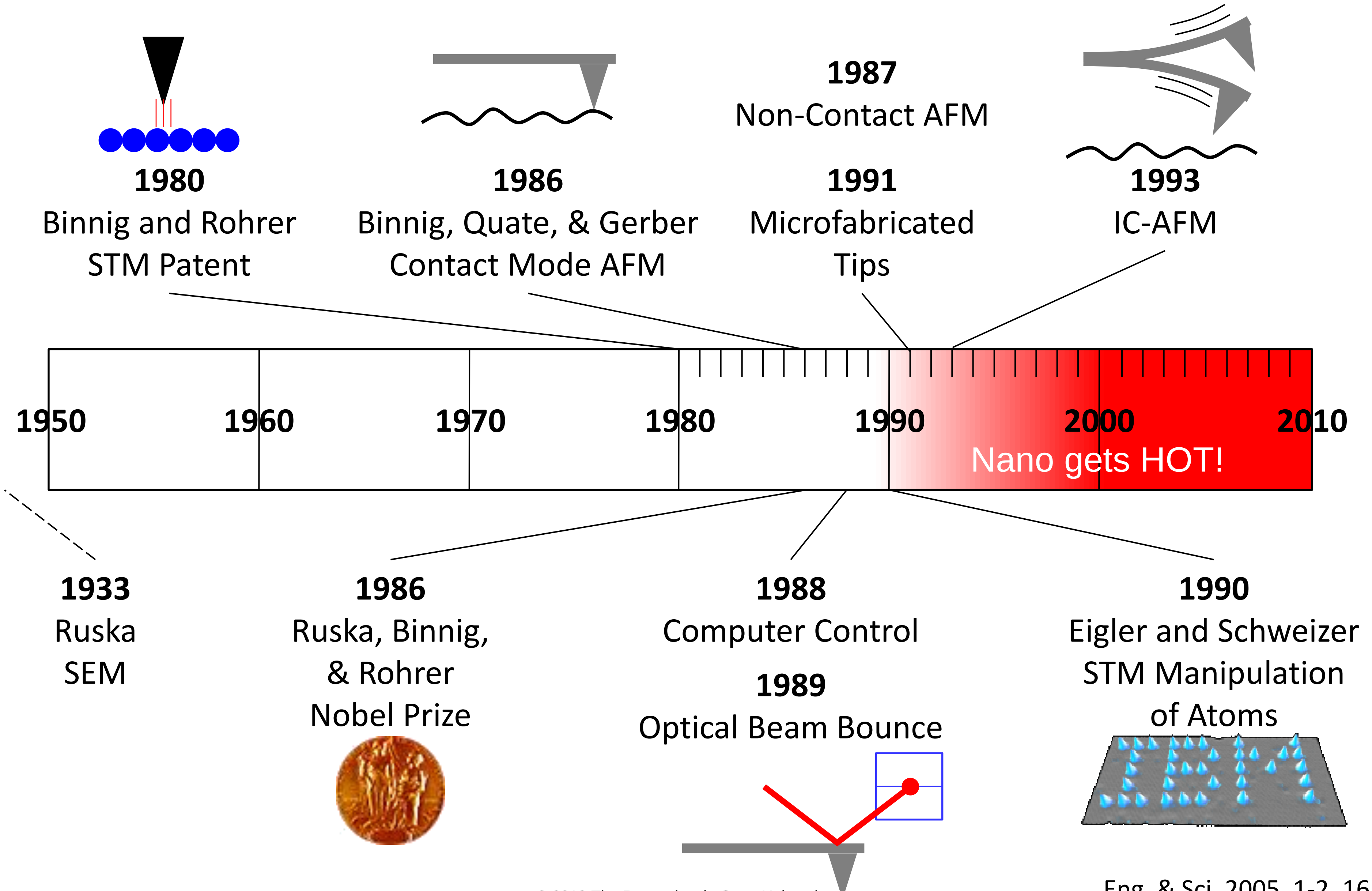
  FESEM: Field Emission Scanning Electron Microscopy

    "seeing" at the nanoscale

  SPM: Scanning Probe Microscopy (e.g., AFM)

    "feeling" at the nanoscale

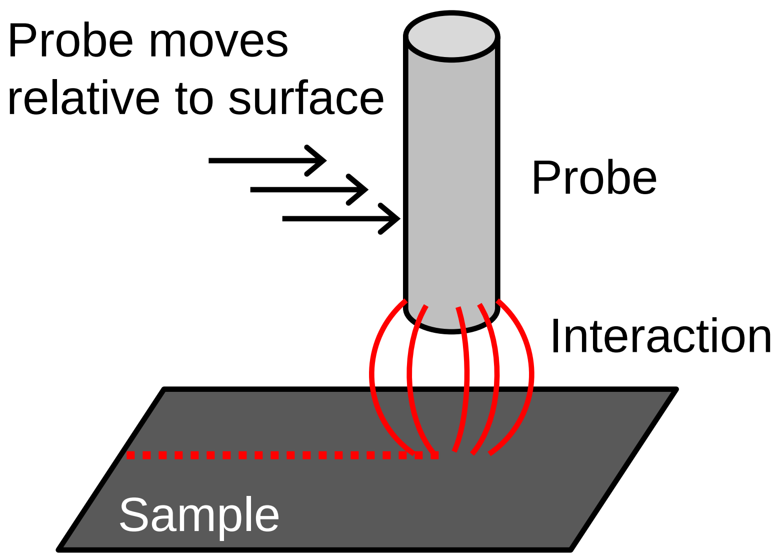
Timeline of Nanocharacterization



Eng. & Sci. 2005, 1-2, 16

A Sample of SPM Techniques

- Scanning Tunneling Microscopy (STM)
- Atomic Force Microscopy (AFM)
- Dynamic Force Microscopy (DFM)
- Scanning Near-Field Optical Microscopy (SNOM)

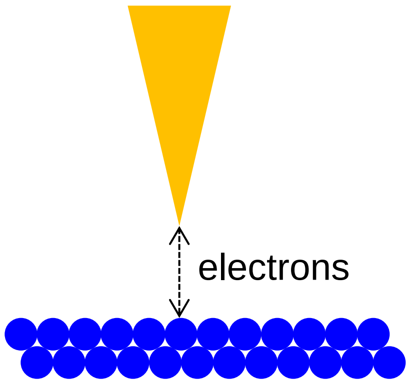


<u>Selected Possible Interactions</u>
Current Flow
Capacitance
Attractive and Repulsive Forces
Magnetic Forces
Absorption or Emission of Light
Flow of Heat

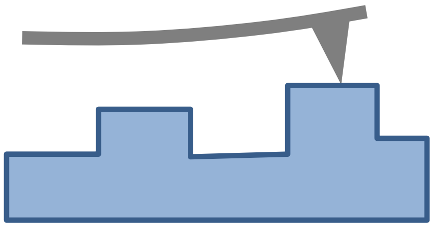
A Sample of SPM Techniques

For Example:

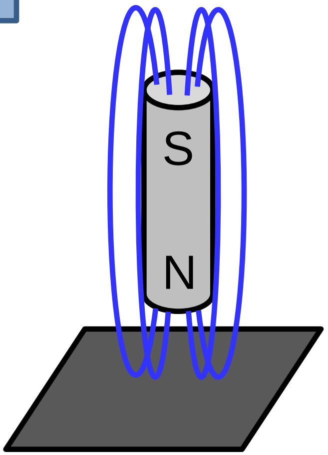
STM = electrical measurements



AFM = mechanical measurements



MFM = magnetic measurements



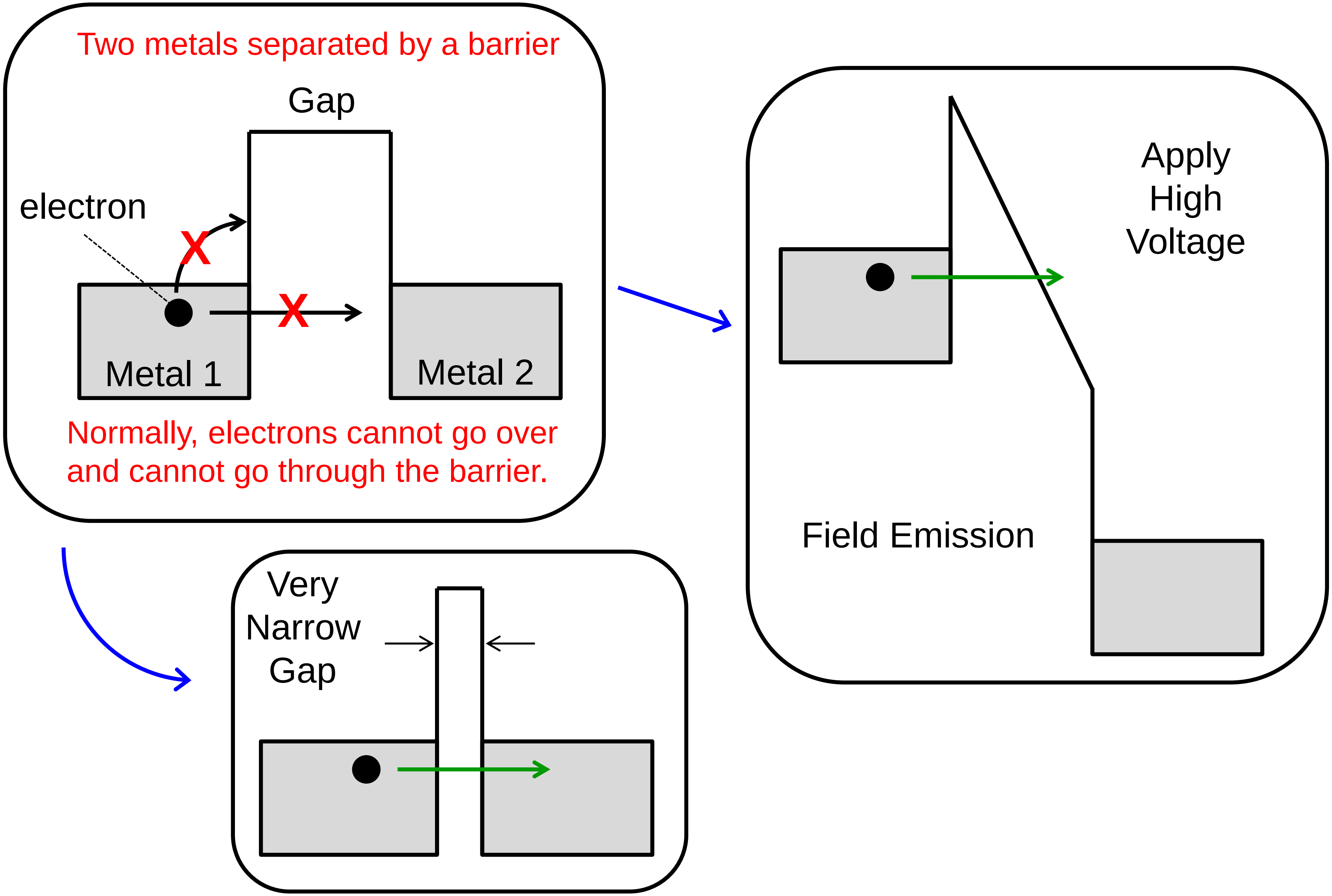
Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  – Hardware and Components
  – Tip/Sample Interactions
  – Common Modes of Operation
  – Pitfalls and Image Artifacts
- Example of Instrument Operation

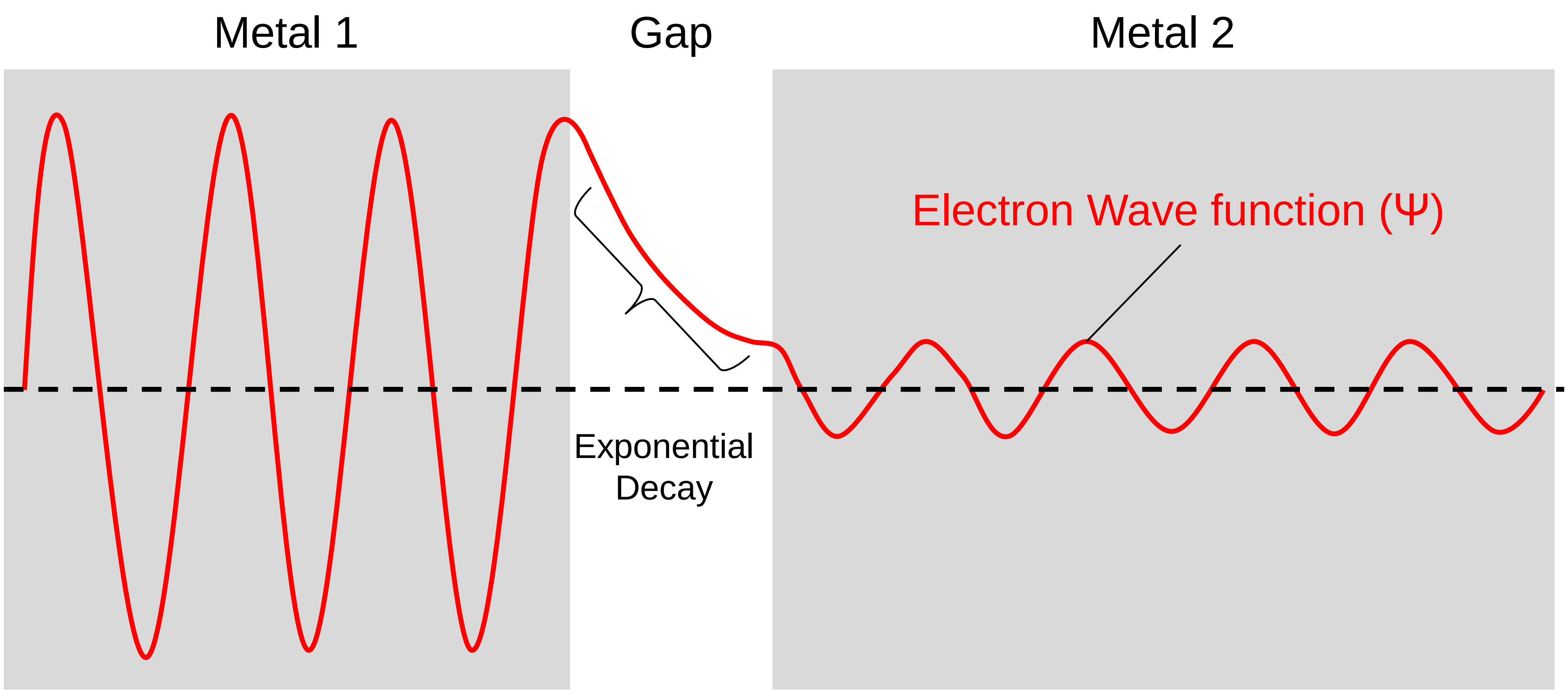
Scanning Tunneling Microscopy

- Utilizes a conducting tip, usually tungsten or gold, sharpened to the point of a single atom (ideally).
- A bias voltage is applied between the tip and the sample and the two are brought into close proximity (less than 10 Å).
- At this distance the electrons can quantum mechanically tunnel through the gap between tip and sample.

Scanning Tunneling Microscopy



Scanning Tunneling Microscopy



- The magnitude of Ψ is related to the probability of finding an electron in the material.
- If the gap is small enough, then the electron has some probability of crossing over from metal 1 to metal 2.
- In the absence of a high electric field, the gap must be less than a few nm to observe tunneling.

http://hyperphysics.phy-astr.gsu.edu

Tunneling through a controllable vacuum gap

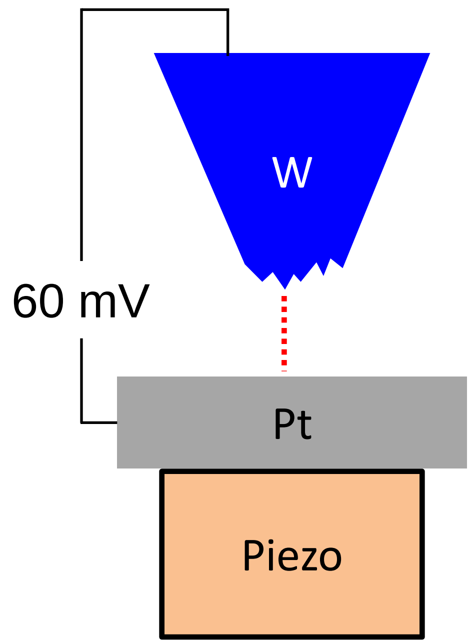
G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel
IBM Zurich Research Laboratory, 8803 Rüschlikon-ZH, Switzerland

(Received 30 September 1981; accepted for publication 4 November 1981)

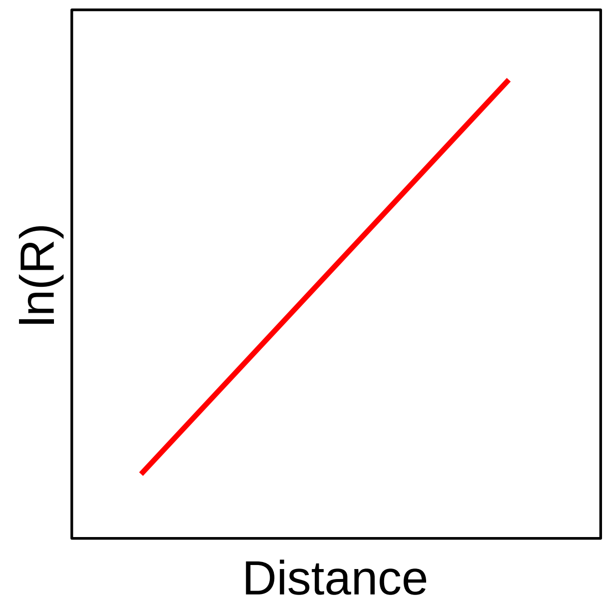
We report on the first successful tunneling experiment with an externally and reproducibly adjustable vacuum gap. The observation of vacuum tunneling is established by the exponential dependence of the tunneling resistance on the width of the gap. The experimental setup allows for simultaneous investigation and treatment of the tunnel electrode surfaces.

"The main purpose of the experiment is not to observe vacuum tunneling *per se*, but to achieve it in a configuration which allows simultaneously spatially resolved tunneling spectroscopy and other surface spectroscopy methods."

"A crucial point for the experiment is the suppression of vibrations…"



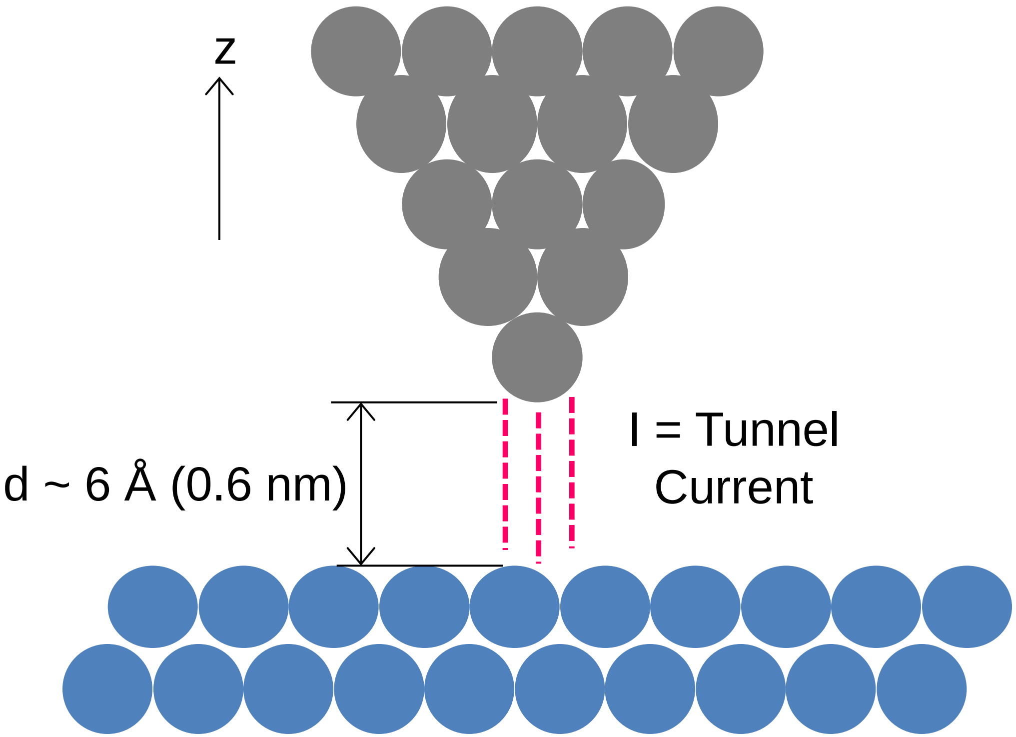
For tunneling, current flow varies exponentially with distance.

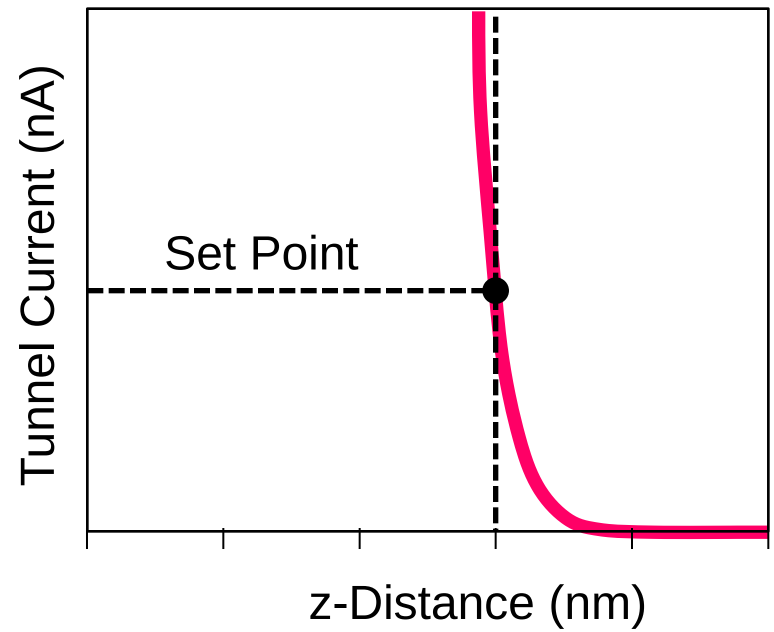


Appl. Phys. Lett. 1982, 40(2), 178.

How Can Tunnel Current Be Used?

Tunnel current is very sensitive to the tip-sample separation distance.

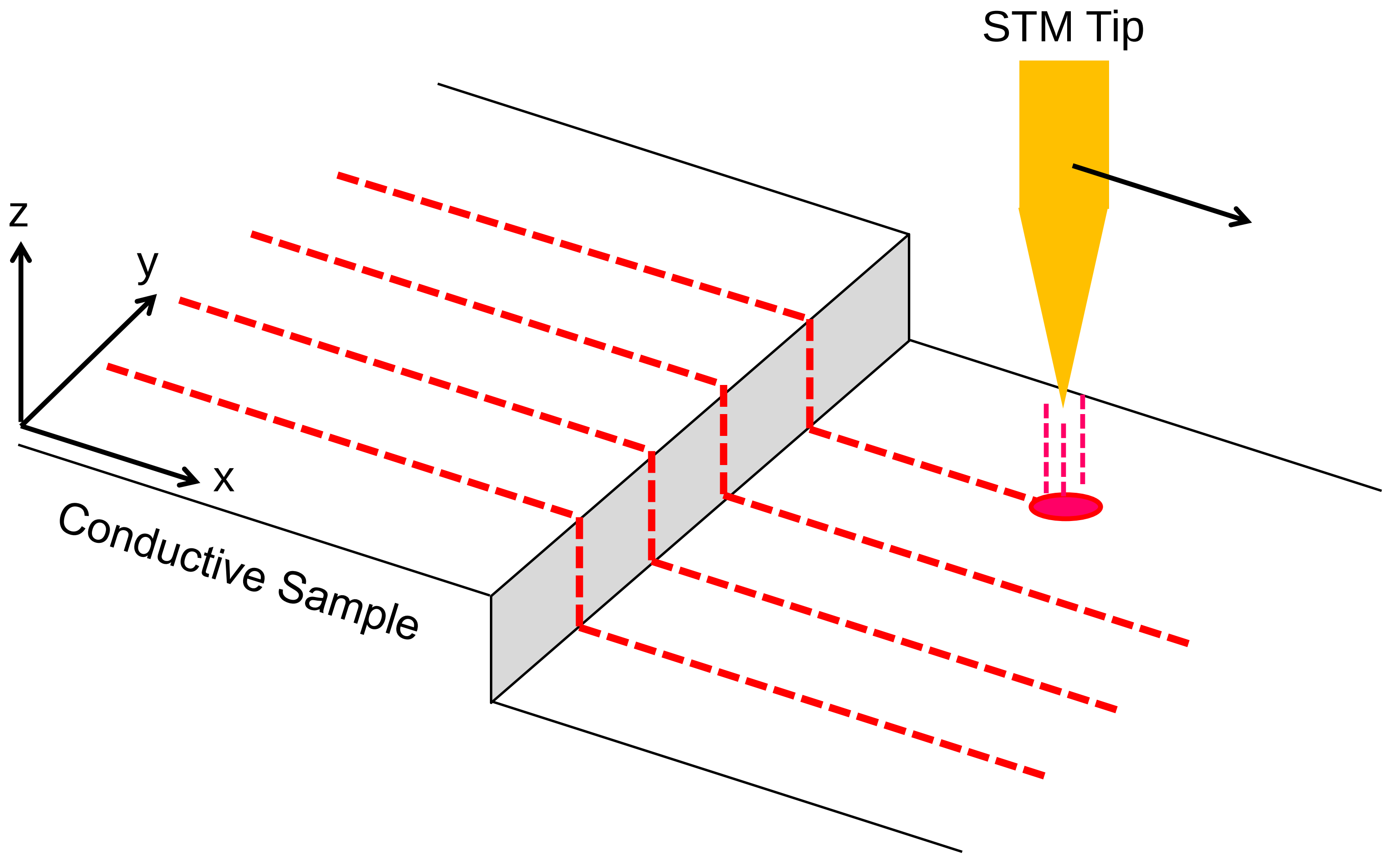




<u>Under Ideal Circumstances</u>

I(z) ~ exp[-2ad]

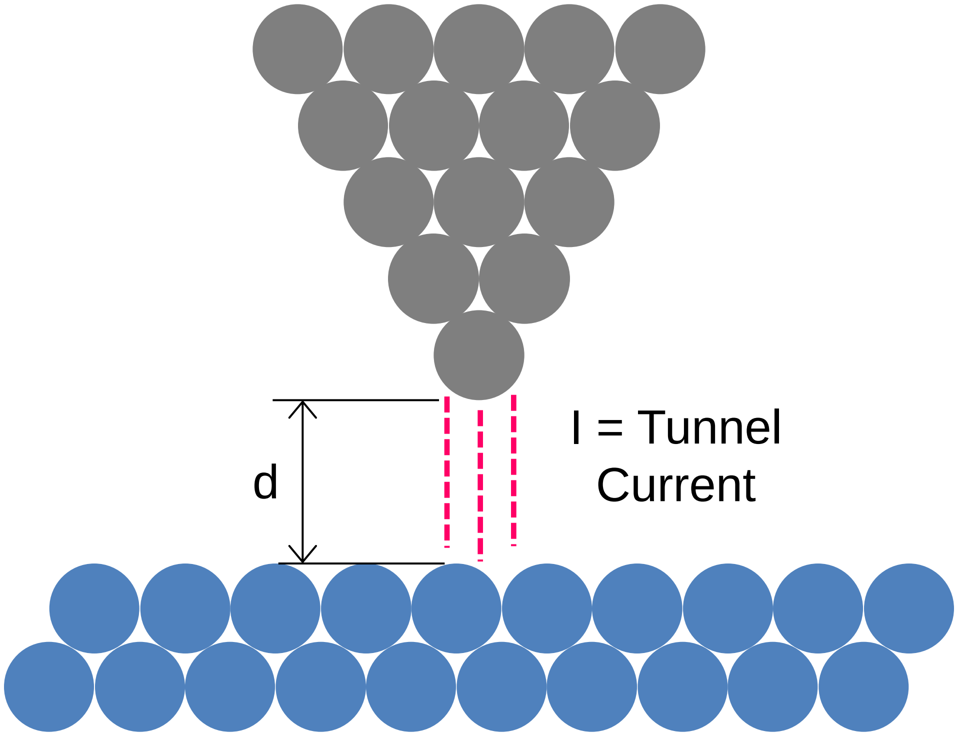
STM: Principle of Operation



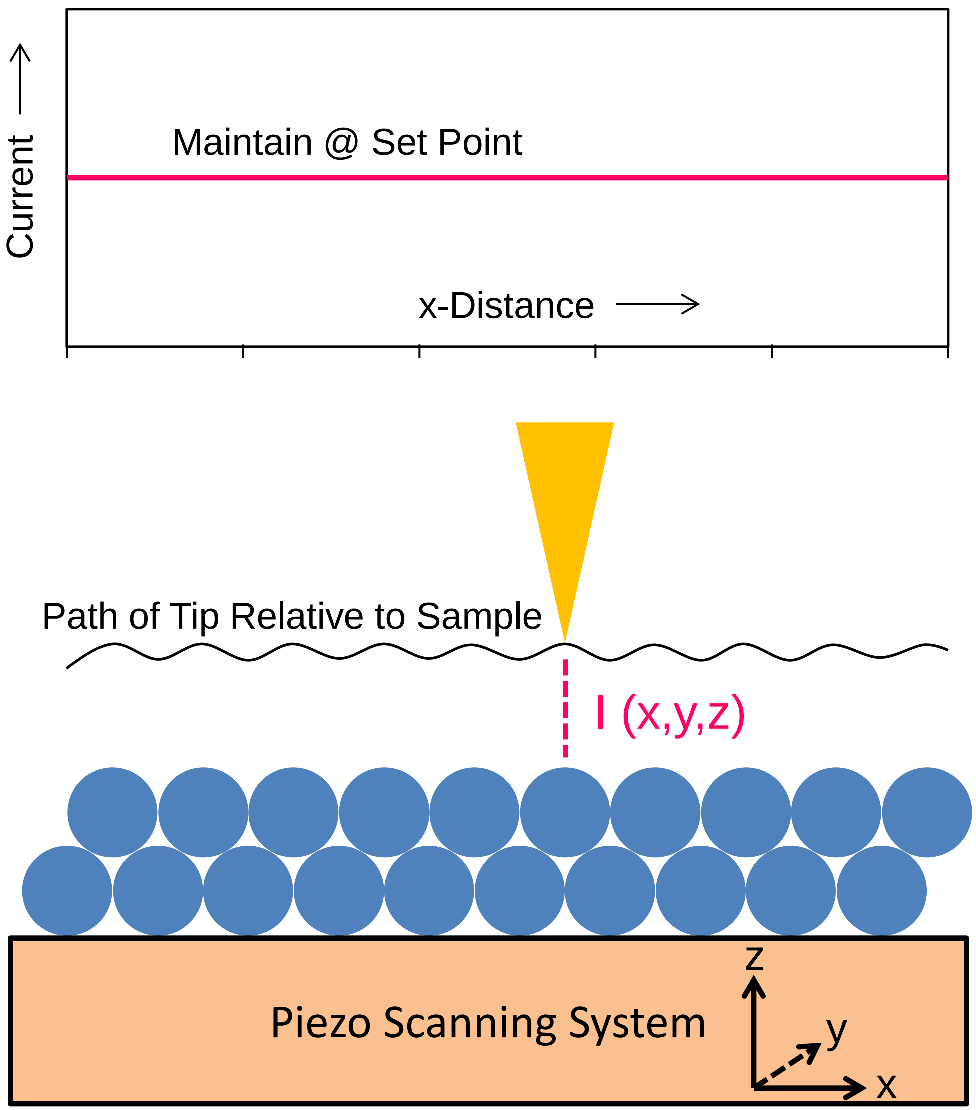
Gap is adjustable using piezoelectric material.
Tunnel current is maintained at set point by adjusting gap.
STM functions by scanning in a controlled manner while adjusting tip-sample distance.

Scanning Tunneling Microscopy

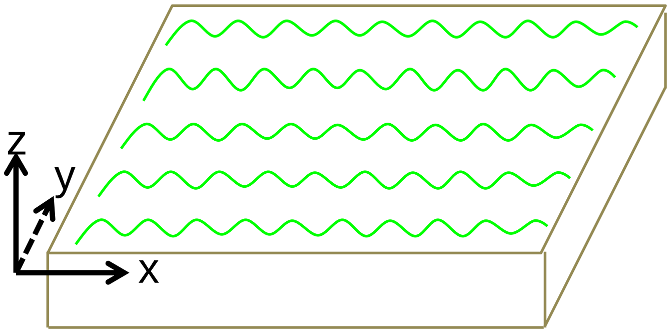
- The tunneling current changes exponentially with the distance between the tip and the sample.
- The changes are detected by a feedback loop which keeps the tunneling current constant by adjusting the tip-surface separation distance (d).
- The signal provides high resolution information about the surface topography and/or the electronic states of the surface.



STM: Principle of Operation



Data from many scans are compiled to form a 3-D representation of the surface.



STM: What can be measured?

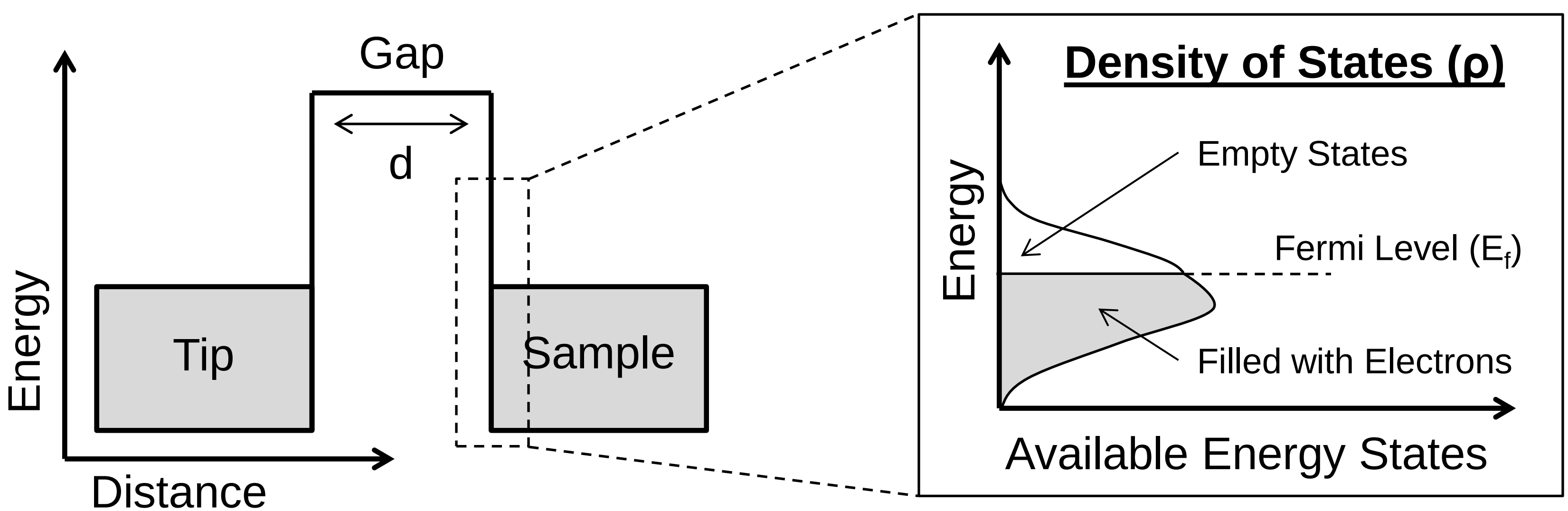
Bias Applied Between Tip and Sample

Tunnel Current

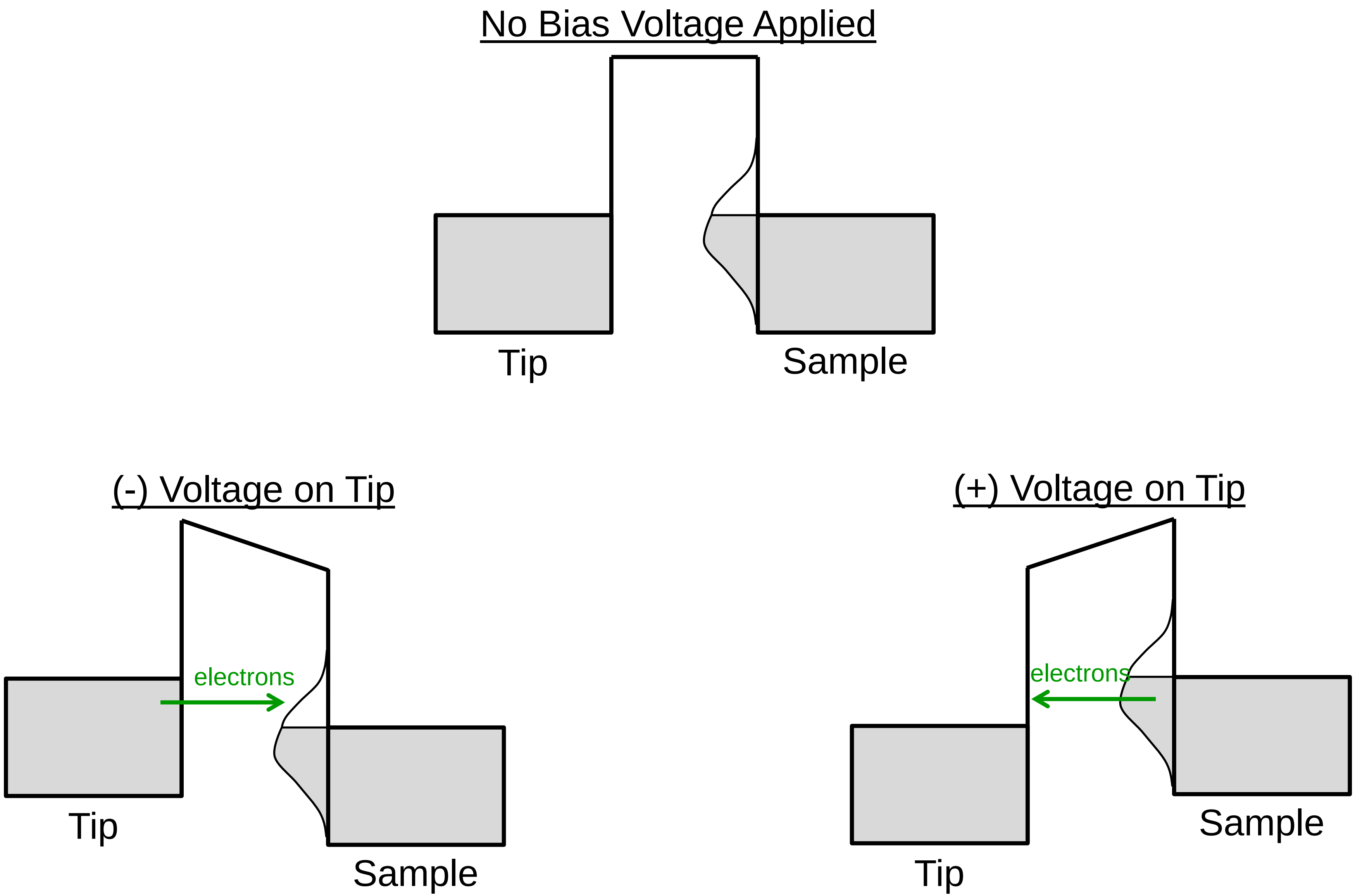
$$I = e\Delta V\rho$$

Charge on Electron

Complex Function That Describes the Electronic Structure of the Sample



STM: What can be measured?



Current Imaging Tunneling Spectroscopy (CITS)

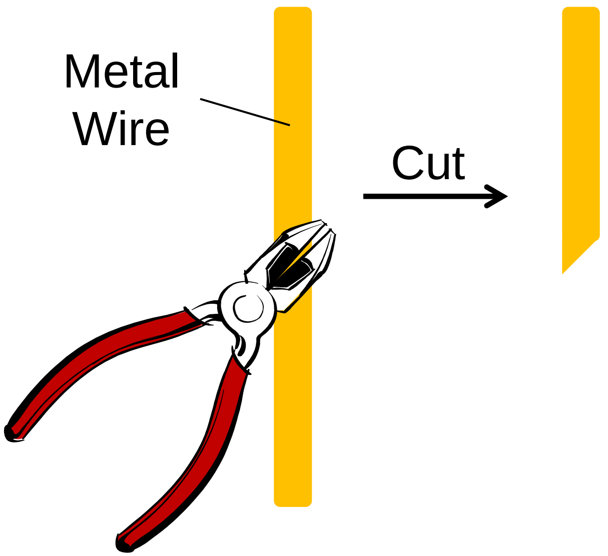
- Filled and unfilled electronic states can be examined by varying the tip-sample bias.
  - Negative bias on tip: electrons flow into unfilled states of the sample.
  - Positive bias on tip: electrons flow out of filled states of the sample.
- CITS = At each (x,y) point, acquire data at multiple (+) and (-) biases. This allows visualization of the local electronic structure of the sample.

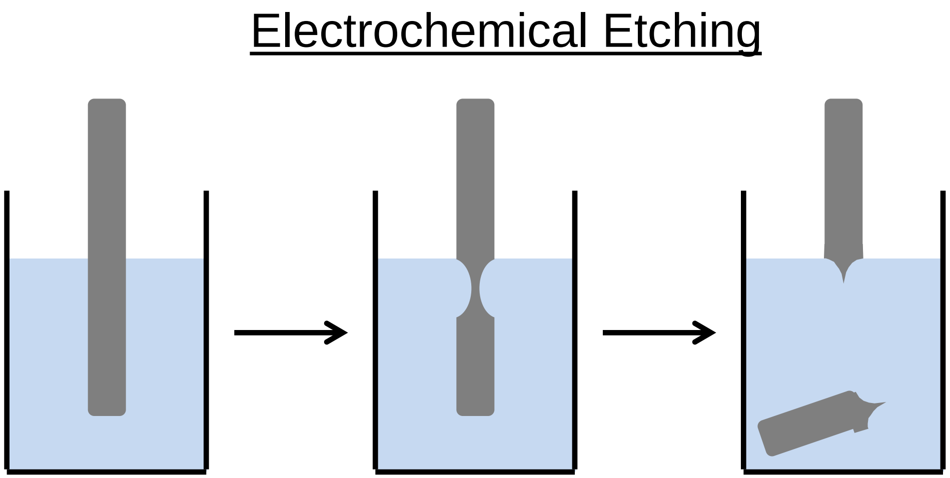
STM: Requirements and Limitations

- Sample and tip must be conductive.
- Requires isolation from vibrations.
- Precise positioning mechanisms.
- Ability to measure very small currents.
- Samples must be flat on nm scale.
- Sample surfaces should be well-defined and stable over time.

STM: Requirements and Limitations

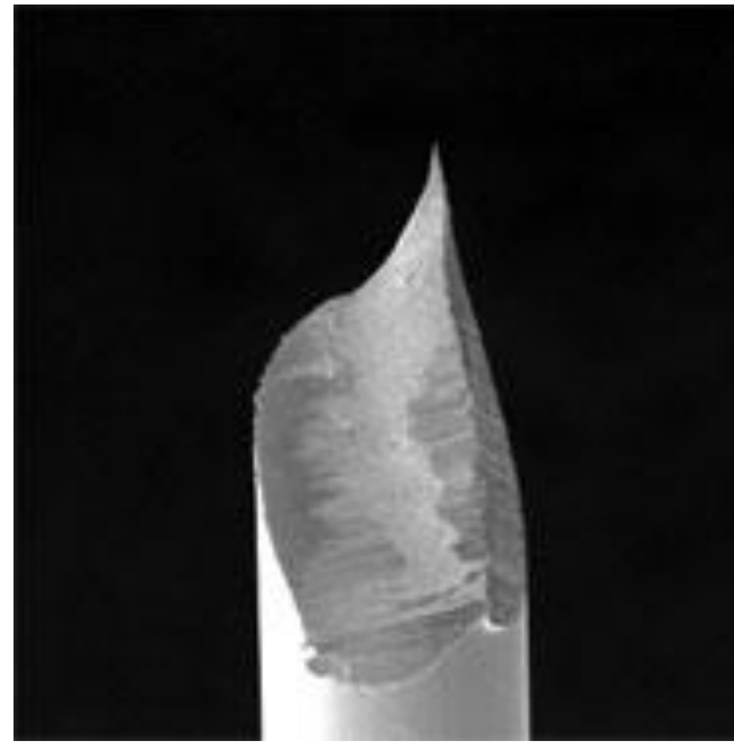
- "Good" tips are difficult to prepare.
- Only about 20% of tips produce meaningful results.
- Two ways to prepare tips:





Scanning Tunneling Microscopy

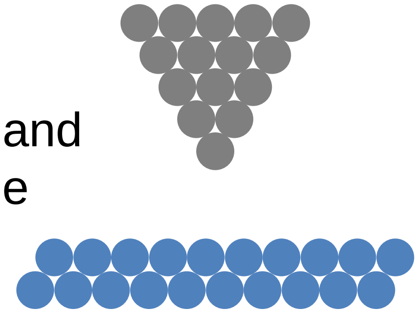
- Sharpened, conducting tip with a bias voltage applied between the tip and the sample.
- Sample and tip must be conductors or semiconductors.
- How sharp is an actual tip?

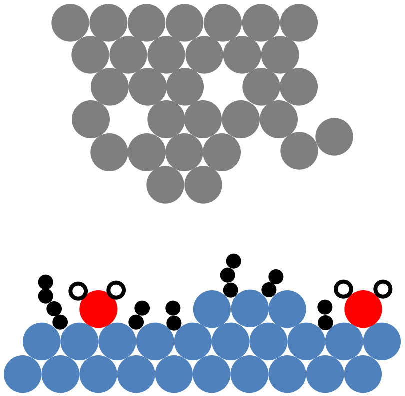


STM: Requirements and Limitations

- STM can be done in air, but best results are obtained in ultra high vacuum (UHV).
- Samples and tips must be stable:
  - Oxide layers do not form
  - UHV prevents adsorption of contaminates from ambient air

Ideal tip and sample





Real tip and contaminated surface

Interesting Surfaces for STM

- Surface reconstruction of Si(111) 7x7.
  - An early victory for STM.
  - STM images clearly showed the arrangement of atoms on the reconstructed surface for the first time.
  - Measurements done after annealing in UHV.
- HOPG (Highly-Oriented Pyrolytic Graphite)
  - STM images used to explain the packing of layers relative to each other.
  - How do the individual layers of graphene lay on top of each other? Do the atoms line up or not?

Phys. Rev. Lett. 1983, 50(2), 120.
Video of Si(111) Surface Reconstruction: http://www.vimeo.com/1086112

Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

From STM to AFM

Many interesting surfaces and materials are not conductive. Can a technique similar to STM be used to characterize them? What other tip-sample interactions can be used and how can they be measured?

VOLUME 56, NUMBER 9 PHYSICAL REVIEW LETTERS 3 MARCH 1986

Atomic Force Microscope

G. Binnig[a] and C. F. Quate[b]
Edward L. Ginzton Laboratory, Stanford University, Stanford, California 94305

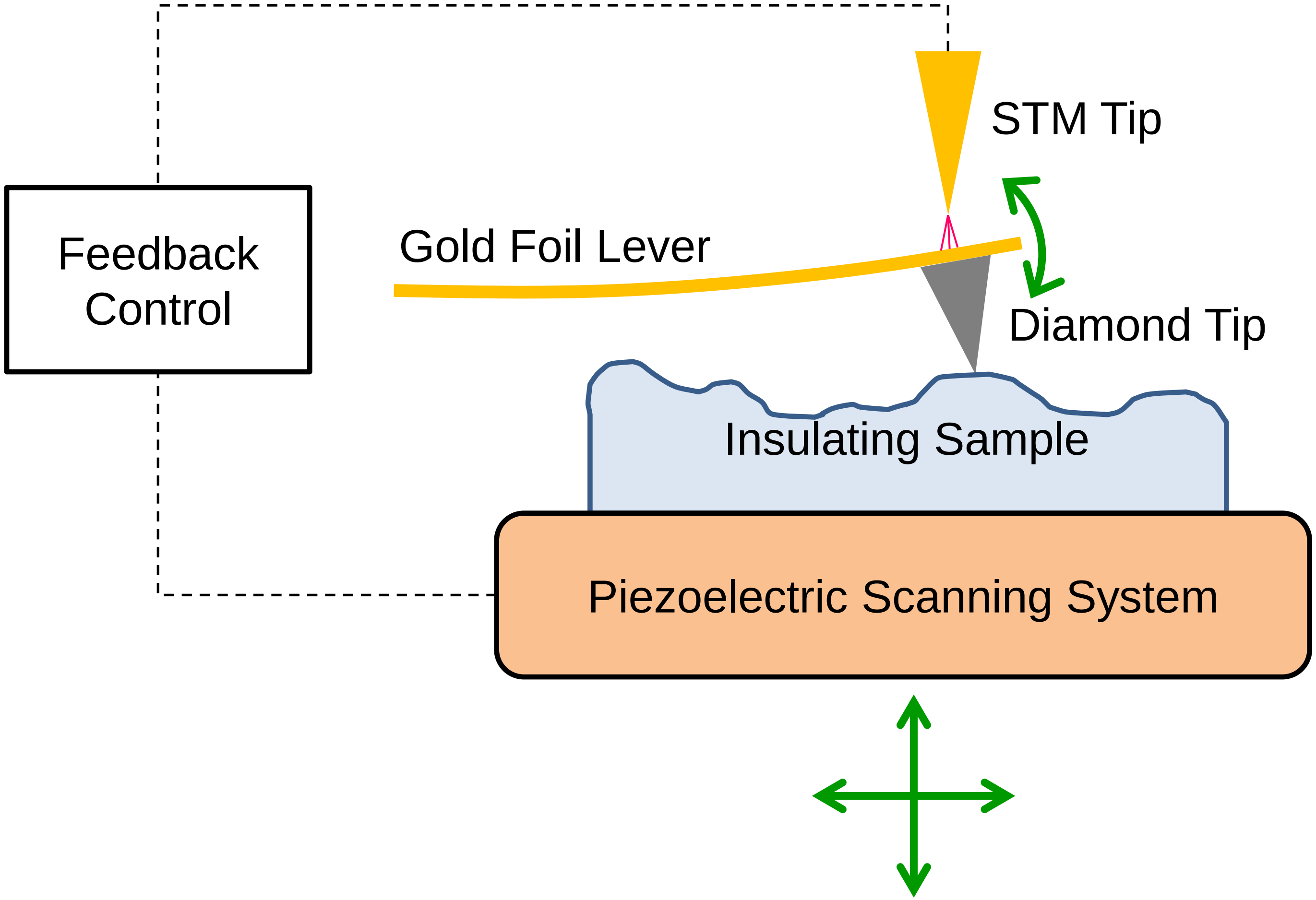
and

Ch. Gerber[c]
IBM San Jose Research Laboratory, San Jose, California 95193
(Received 5 December 1985)

The scanning tunneling microscope is proposed as a method to measure forces as small as 10^{-18} N. As one application for this concept, we introduce a new type of microscope capable of investigating surfaces of insulators on an atomic scale. The atomic force microscope is a combination of the principles of the scanning tunneling microscope and the stylus profilometer. It incorporates a probe that does not damage the surface. Our preliminary results *in air* demonstrate a lateral resolution of 30 Å and a vertical resolution less than 1 Å.

Phys. Rev. Lett. 1986, 56(9), 930.

From STM to AFM



STM current is monitored and used to keep the diamond tip in contact with the sample surface.

Voltages are applied to the piezoelectric to move the sample relative to the tip.

Phys. Rev. Lett. 1986, 56(9), 930.

Atomic Force Microscopy (AFM)

- Traditional microscopes are limited by lens quality and diffraction loss.
- Scanning-probe instruments (like an AFM) do not use lenses to focus light.
- Instead, a small probe is rastered across the sample while a representative signal is monitored.
- Information gathered from the probe (e.g., height) can be reconstructed to form an "image" of the sample surface.

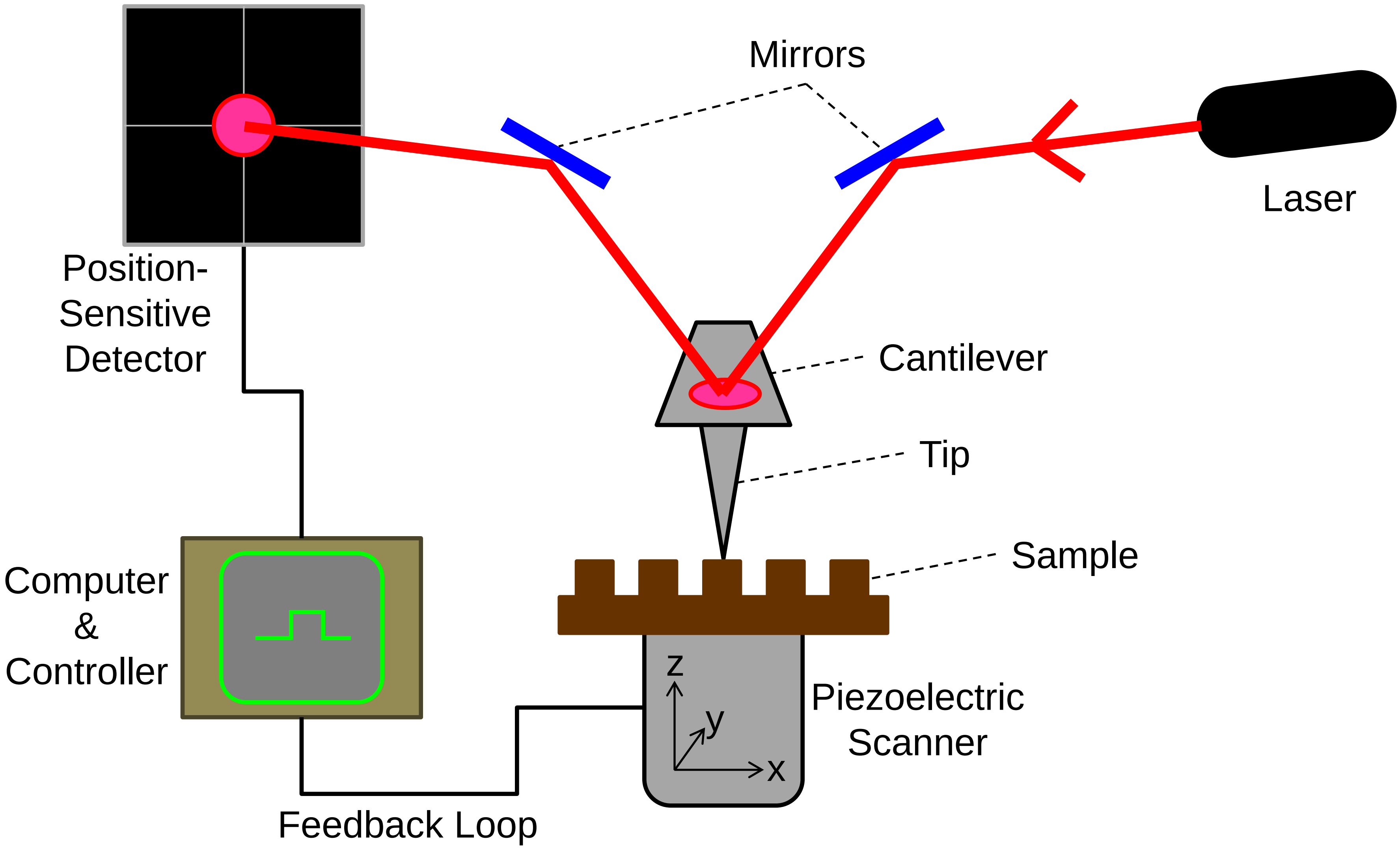
Atomic Force Microscopy

- The AFM is one of many scanning probe tools. All use a nanoscale probe to scan over a surface.
- An Atomic Force Microscope (AFM) is a tool for characterizing surfaces on a scale of about 0.1 nm to 100 microns.
- As the name implies, the instrument measures the forces acting between the tip and the sample.
- It also is commonly used as a type of microscope, due to its ability to gather high-resolution topographic information.
- The combination of force measurement and spatial resolution makes AFM a very versatile characterization technique.

Outline

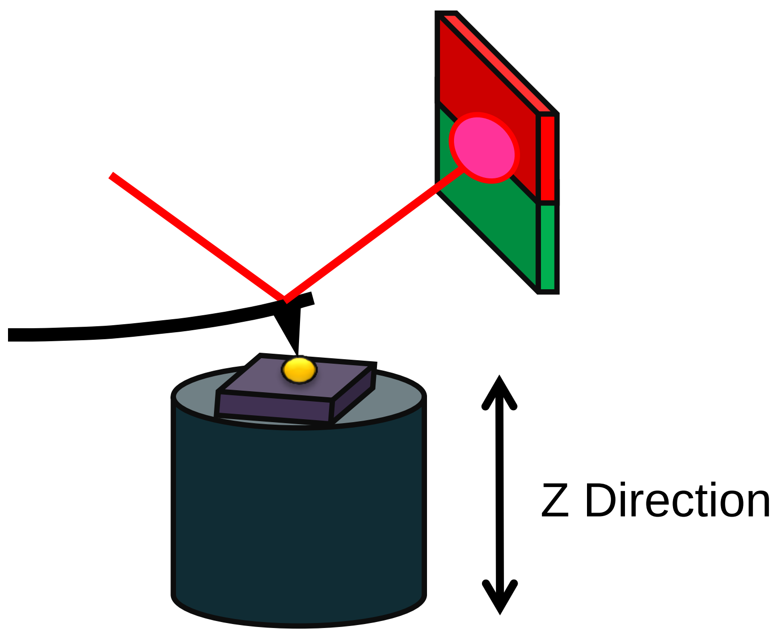
- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

Principle of Operation



Principle of Operation

In its most basic mode of operation, AFM measures the surface by monitoring the deflection of the cantilever in the ***Z direction***. Detection is done by measuring the signal produced by a segmented photo-detector.

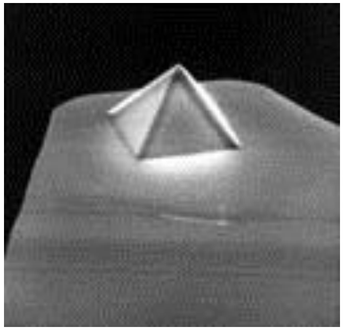


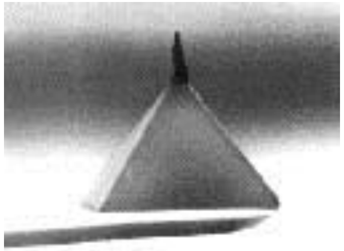
Hardware and Components

1. Sharpened tip on a cantilever.
2. A way of monitoring the deflection of the tip/cantilever (e.g., laser beam & detector).
3. Piezoelectric positioning and scanning system.
4. Computer-controlled feedback loop.

AFM Tip Technology

- Micro machining techniques can produce inexpensive, reasonably sharp tips.
- The photos below show three common tip configurations.
- All AFM tips have a finite radius. This “end radius” generally determines the resolution of the AFM.

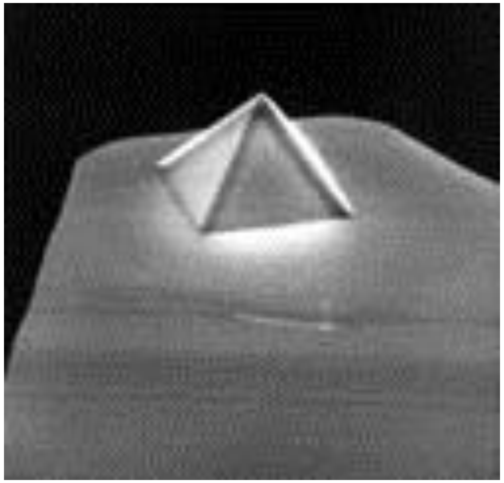


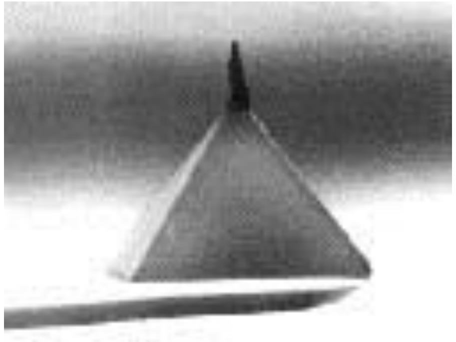


David Baselt, California Institute of Technology, Copyright © 1993 by David Baselt.

AFM Tip Technology

- The “standard” tip is about 3 um tall pyramid with a 30 nm end radius
- The electron-beam-deposited tip (EBD) is an improvement. An e-beam deposits carbonaceous material to effectively sharpen the radius.





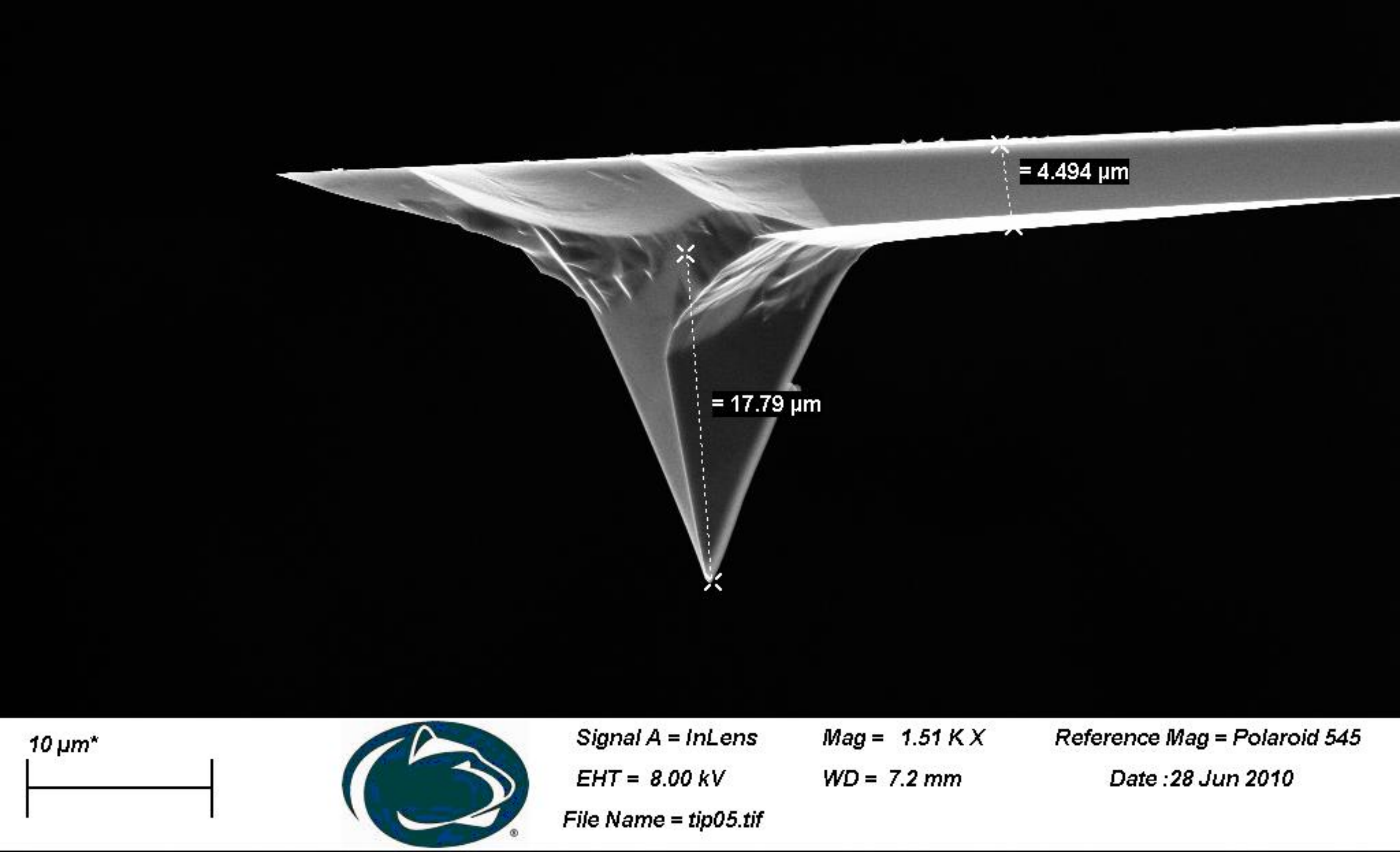
David Baselt, California Institute of Technology, Copyright © 1993 by David Baselt.

AFM Tip Technology

- The "ultralever" is manufactured using improved microlithography techniques. It offers high aspect ratio, and a nominal 10 nm end radius.

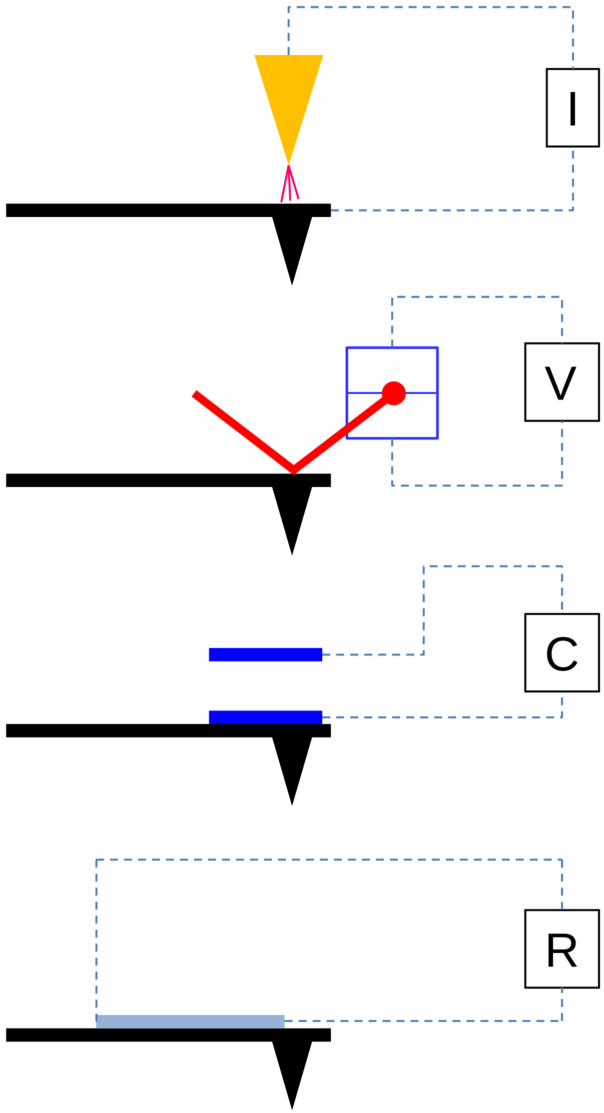
David Baselt, California Institute of Technology, Copyright © 1993 by David Baselt.

FESEM image of a typical tip/cantilever.



Can you propose a process to make an AFM tip out of silicon?

Detecting Cantilever Deflection



STM — Tunneling Current
Phys. Rev. Lett. 1986, 56(9), 930.

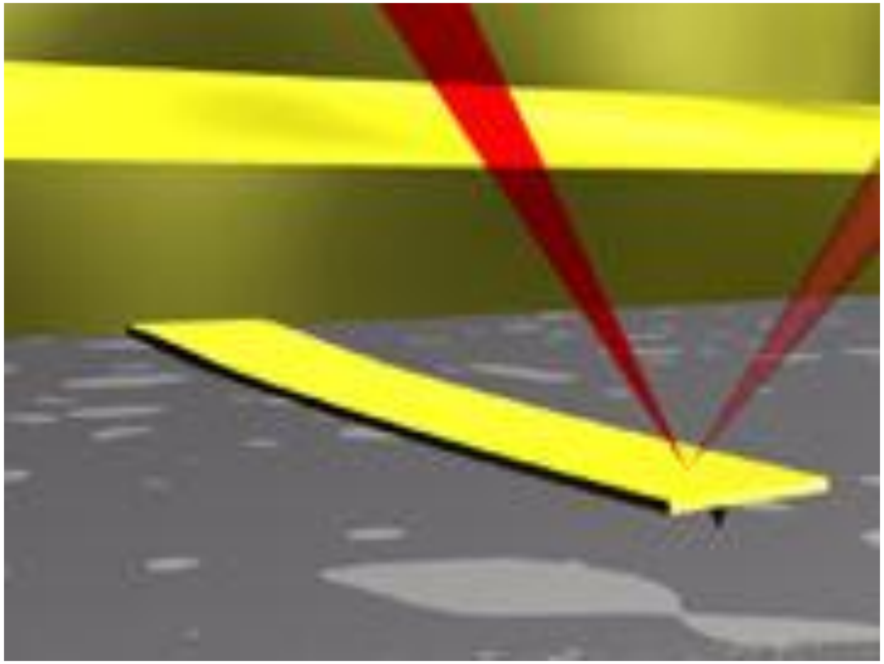
Optical Lever (Beam Bounce) — Voltage from Photodiodes
Appl. Phys. Lett. 1988, 53(12), 1045.

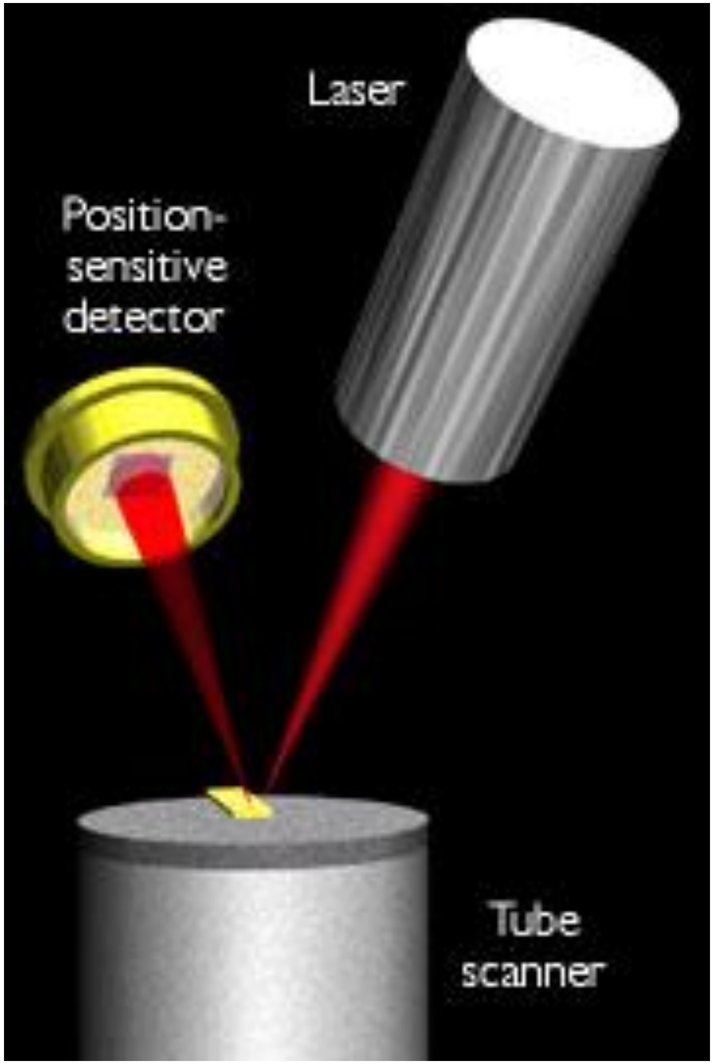
Capacitance — Change in Capacitance
J. Vac. Sci. Technol. A 1990, 8(1), 383.

Piezoresistance — Change in Resistance
Appl. Phys. Lett. 1993, 62(8), 834.

AFM Optical Lever

Data from the detector is used to maintain or control tip-sample interactions.





David Baselt, California Institute of Technology, Copyright © 1993 by David Baselt.

AFM Optical Lever

- The optical lever magnifies motions of the tip.
- The cantilever–detector distance is thousands of times the magnitude of the cantilever deflection.
- This magnification can approach 2000x, and the optical lever can theoretically obtain very low noise levels.
- This technique is inexpensive and accurate compared to other methods of measuring samples on the nanoscale.

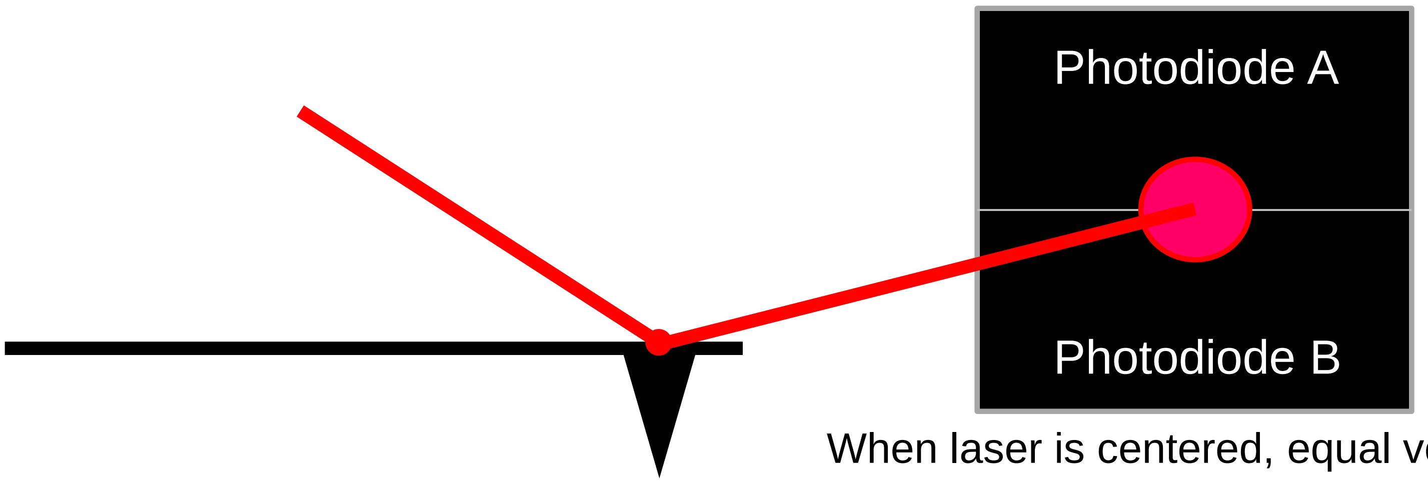
Photodiode Detector

Photodiode: A device that converts incident light into current or voltage.

The incident light comes from the laser beam reflecting off the cantilever.

The detector contains multiple (2 – 4) photodiodes.

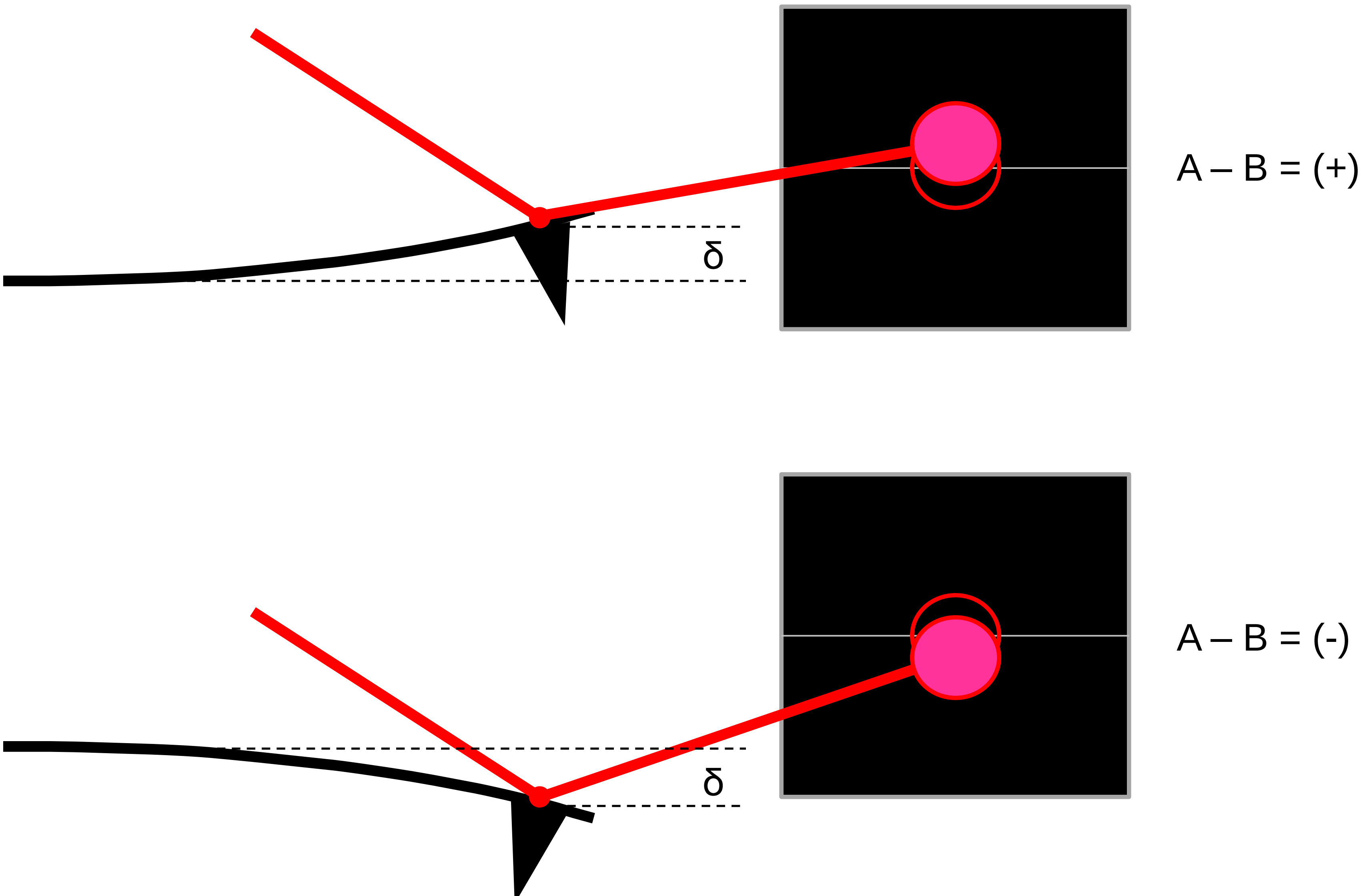
Comparison of the signals from the photodiodes creates a position-sensitive signal that measures the deflection (δ) of the cantilever.



When laser is centered, equal voltages from top (A) and bottom (B) photodiodes.

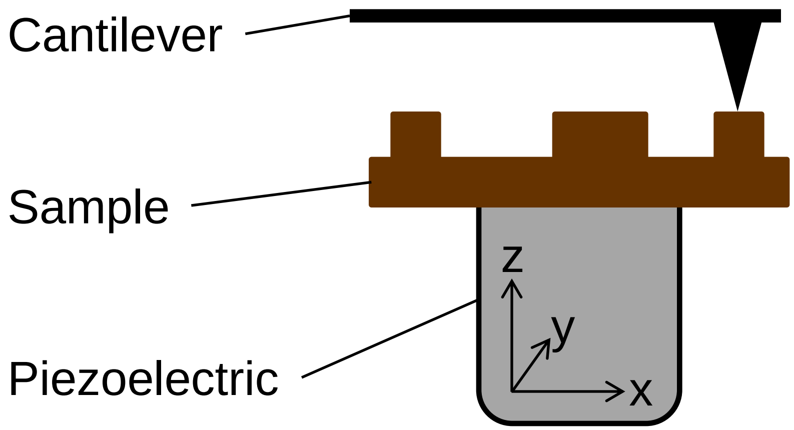
$$A - B = 0$$

Photodiode Detector



Piezoelectric Scanner

<u>Piezoelectric Material</u>: A material that changes shape in the presence of an electric field (Voltage).



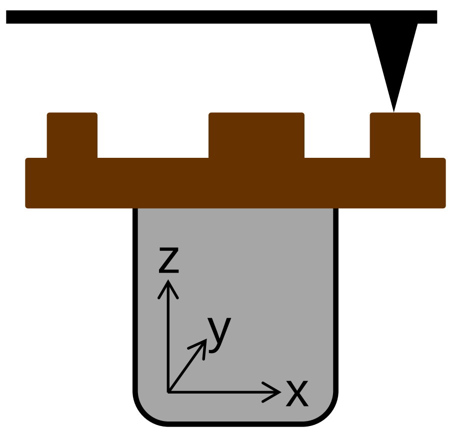
High electric fields produce only small changes in the size of the material (microns or less).

Since applied voltages can be controlled, this effect can be used to precisely position the sample.

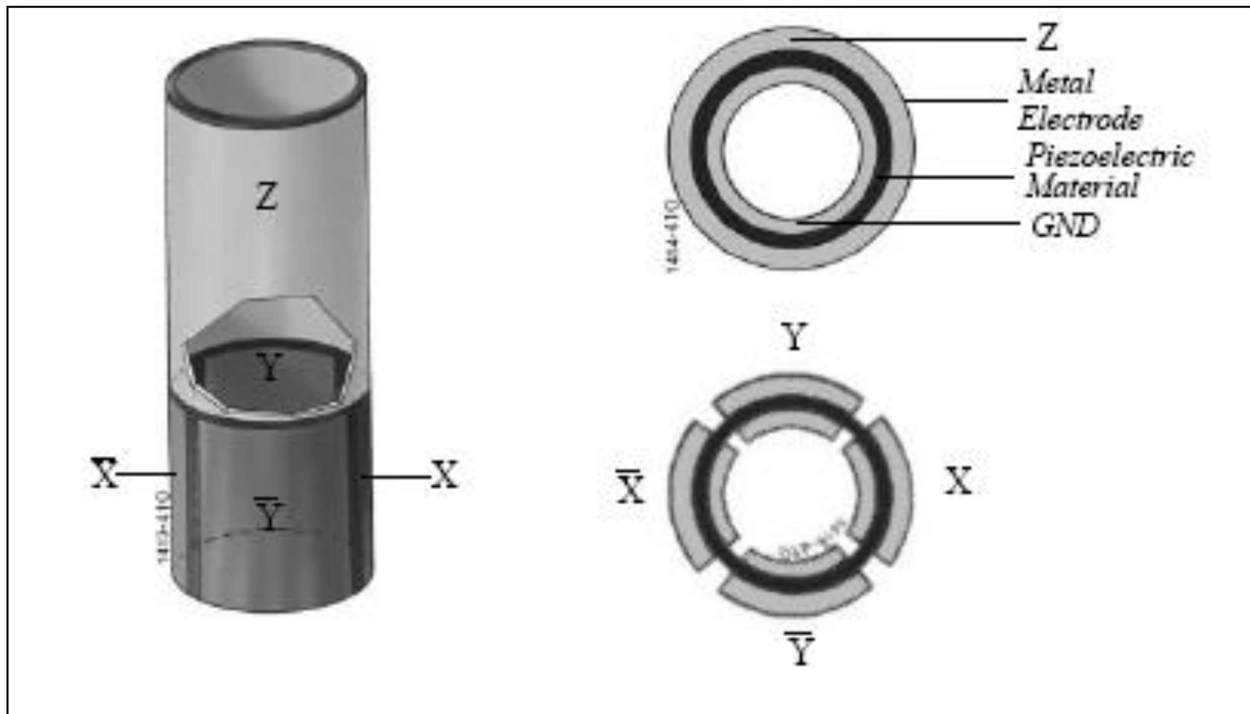
Piezoelectric Scanner

The tube-shaped scanner allows for movement in all three directions (x,y,z).

Metal electrodes on the tube of piezoelectric material allow for voltages to be applied.



Piezoelectric Tube Scanner



Positioning the Sample

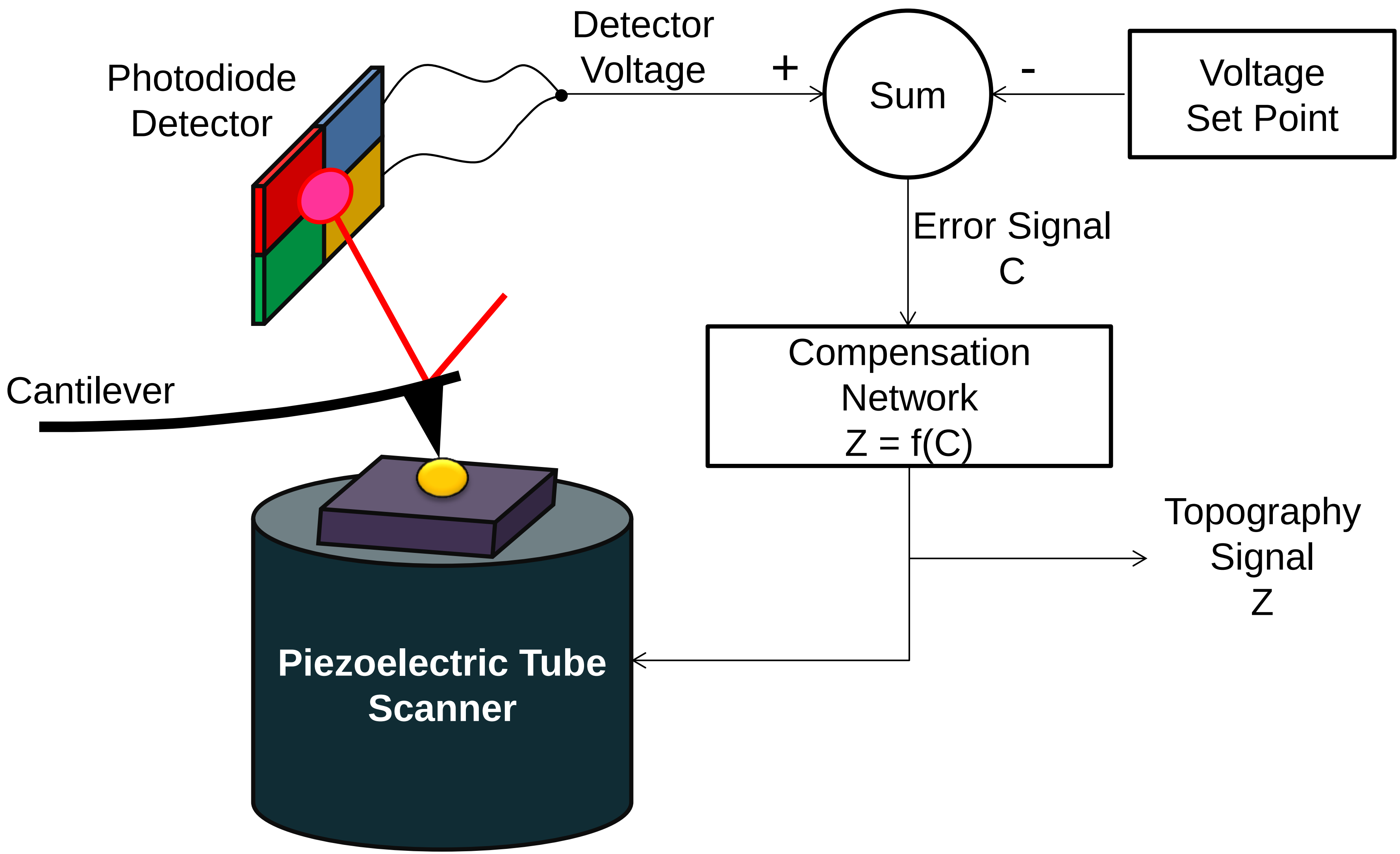
- An active platform is used to control the position of the sample, this is a distinct improvement compared to profilometers.
- The active platform is known as a tube scanner. It is made of a piezoelectric material that expands or contracts according to applied voltages.
- 4 outer electrodes control XY motion, while an inner electrode controls the height (Z).
- A compensation network (feedback loop) monitors the cantilever deflection and keeps it constant by adjusting the height of the sample or cantilever.

Positioning the Sample

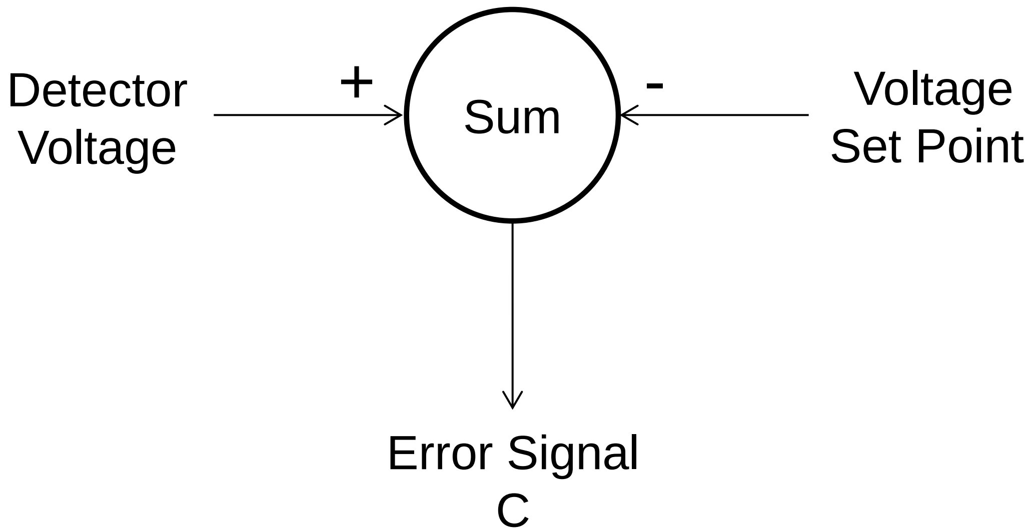
This system can be analyzed in three areas.

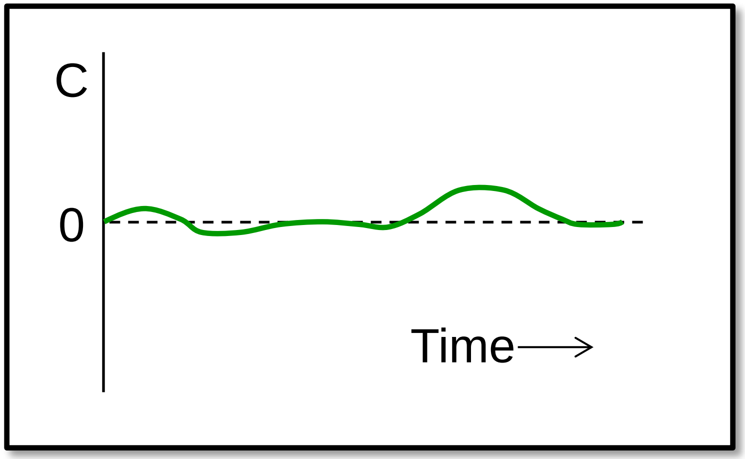
1. Tube scanner controls the height of the sample.
2. The cantilever and optical lever measure the height of the sample.
3. A feedback loop keeps the cantilever deflection constant by adjusting the applied voltages to the scanner.

Feedback Loop



Feedback Loop





If Detector Voltage = Voltage Set Point, then Error Signal is zero.
→ No action is required by the control system.

If Detector Voltage ≠ Voltage Set Point, then Error Signal is not zero.
→ Control system must respond to bring the Error Signal back to zero.

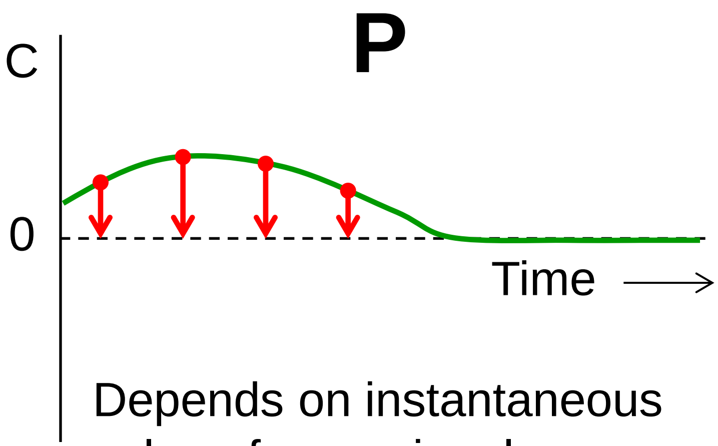
Feedback Loop: PID Control

Three components to control system:

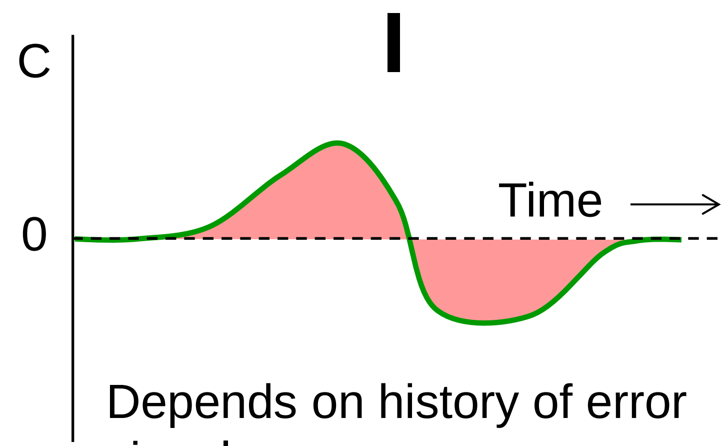
- P - Proportional: Reacts to current value of error signal
- I - Integral: Reacts to sum of recent errors
- D - Derivative: Reacts to instantaneous rate of change of the error signal

How would each of these components respond in real time?

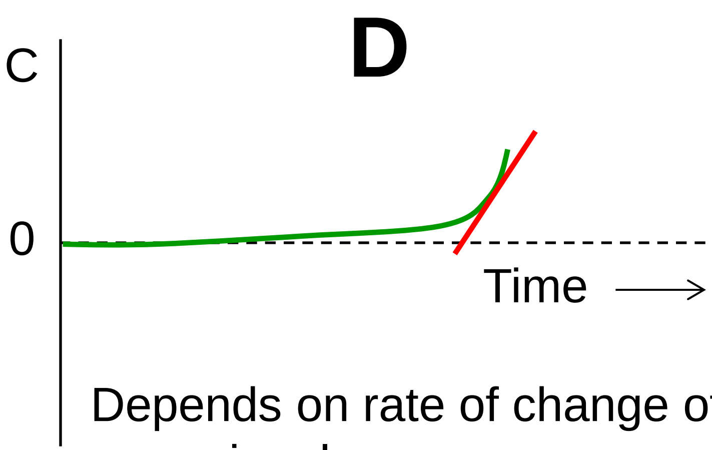
Feedback Loop: PID Control



Depends on instantaneous value of error signal



Depends on history of error signal



Depends on rate of change of error signal

Each type of control addresses a unique aspect of the system response.

Tuning of the PID parameters is usually required to achieve robust control.

Tuning the Feedback Loop

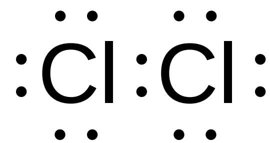
- Start with P = 1.00 and I = D = 0.00
- Increase P until overshoot begins to occur
- Then decrease P to just below maximum stable value (with no overshoot)
- Increase I and determine effect.
- Rule of Thumb: I = 0.3 x P
- D generally not needed. Keep D = 0.00

Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

Bonding and Forces Revisited

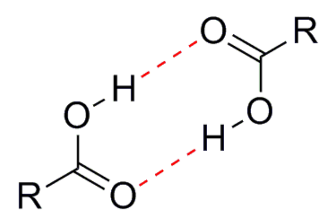
- Covalent Bonds



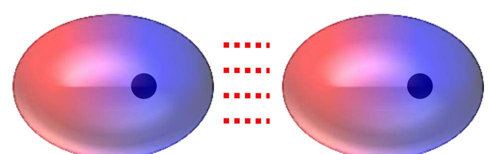
- Ionic (electrostatic)

$$F = \frac{Z_{-}Z_{+}e^2}{r^2}$$

- Hydrogen Bonding

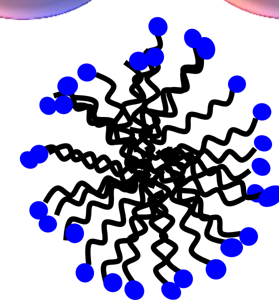


- van der Waals Forces



$$F \propto \frac{1}{r^6}$$

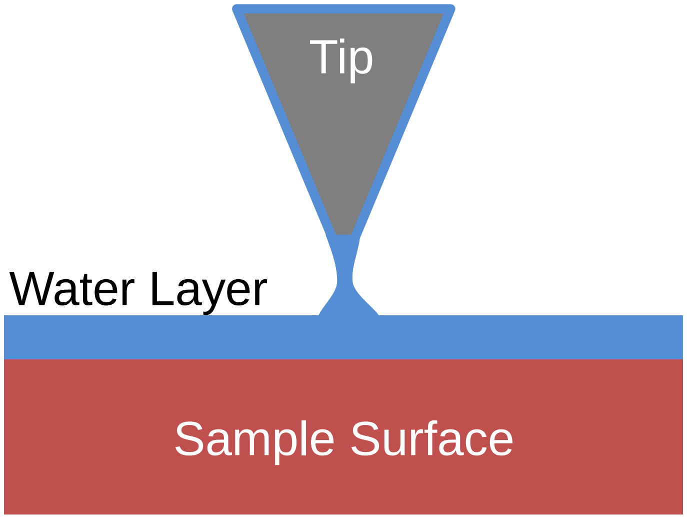
- Hydrophobic Interactions



Tip-Sample Interactions

Tip and sample may experience many possible forces and interactions. Each force acts over a characteristic length scale. Some are attractive, some are repulsive.

- Long-range electrostatic (up to 100 nm)
- Short range electrostatic
- Short-range polarization
- Dispersion forces (few nm)
- van der Waals forces
- Pauli repulsion (electrons)
- Capillary forces



Tip-Sample Interactions

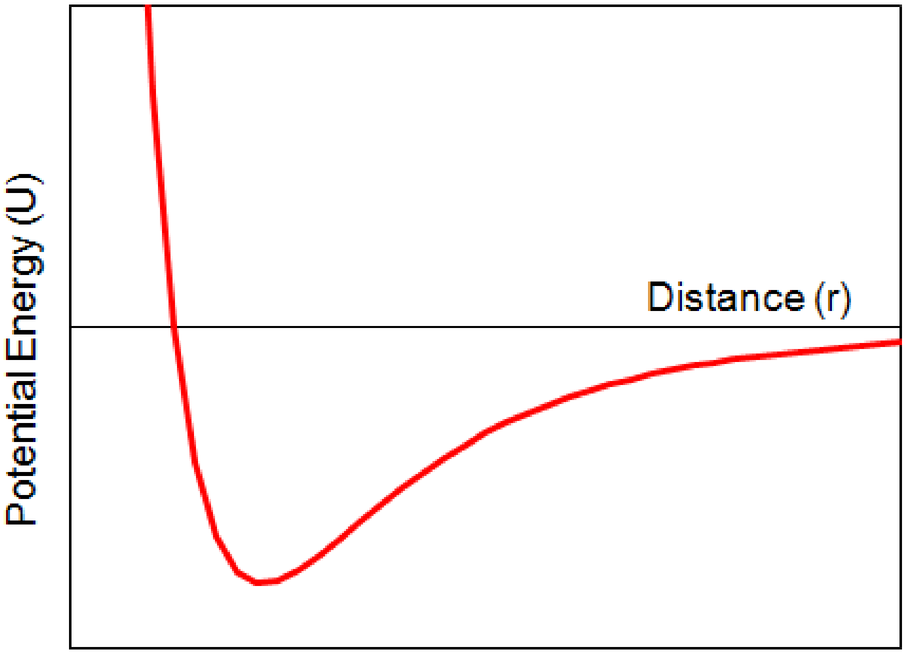
In addition to being a type of microscope, AFM can be used to measure the forces acting between the tip and the sample surface.

This data is useful because it can be related to surface energy of the sample.

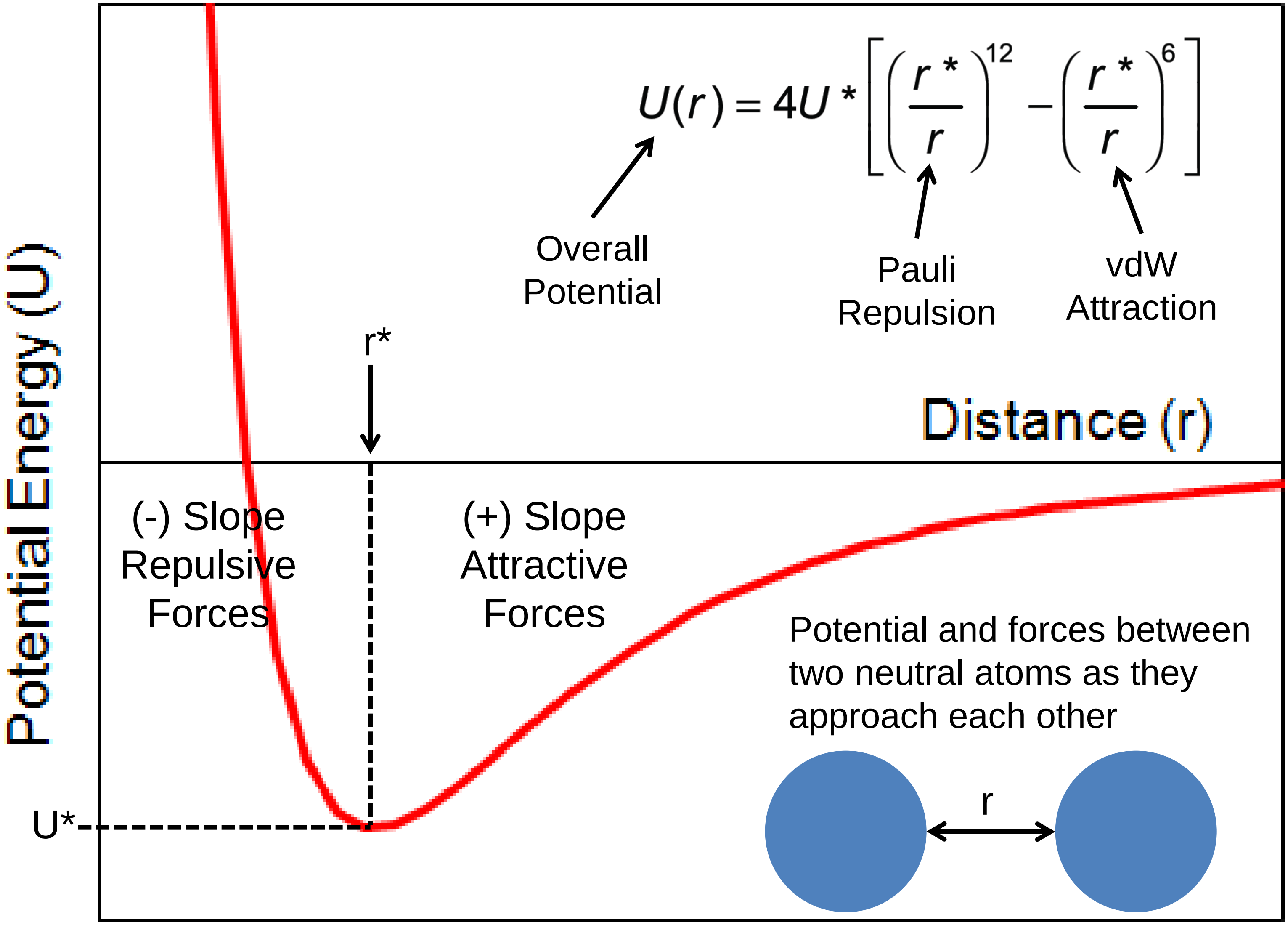
There are many theoretical approaches to modeling tip-sample interactions. Each model treats interactions in a slightly different way.

Results depend heavily on the geometry of the tip and the types of forces considered.

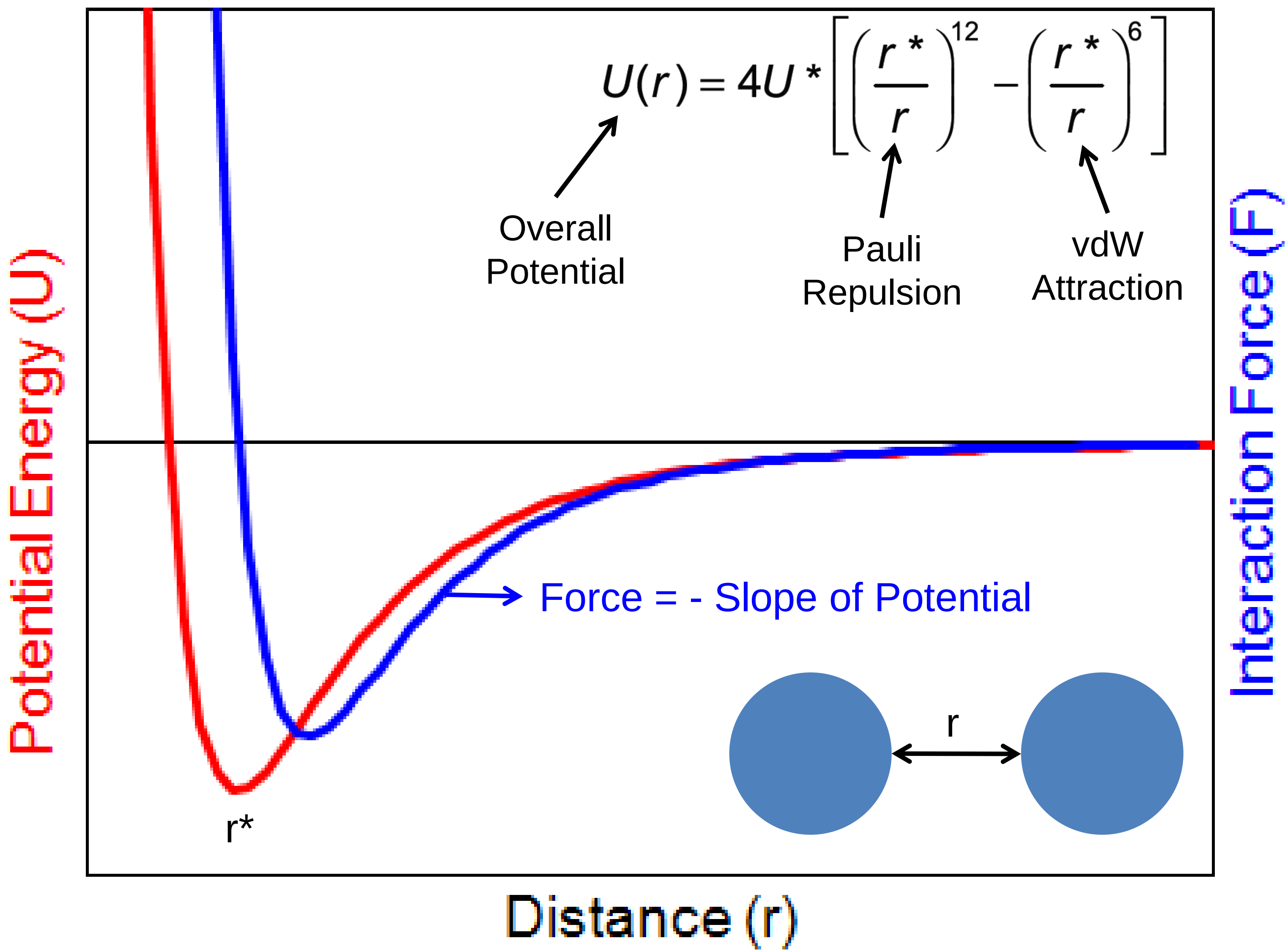
The Lennard-Jones potential is a simple model that combines dispersion (attractive vdW force) and Pauli repulsion (repulsion at very short distances due to overlapping electron clouds).



Lennard-Jones Potential



Lennard-Jones Potential



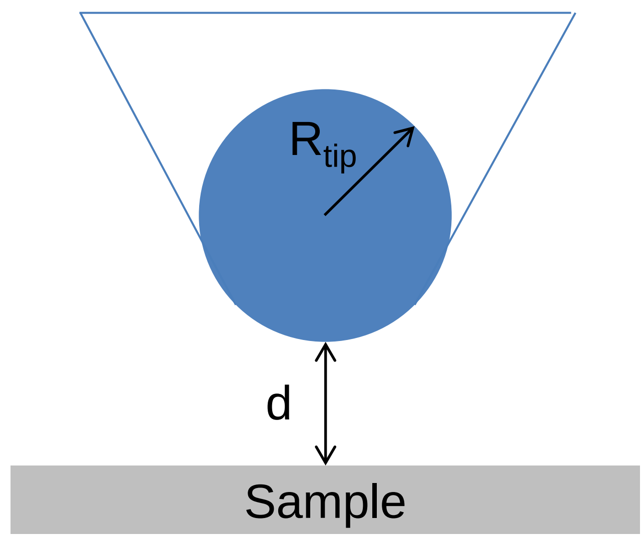
Tip-Sample Interactions

The simple Lennard-Jones potential on the previous slide considers what happens when two neutral atoms approach one another.

Actual tips and samples are comprised of many atoms arranged in a specific (unknown) geometry.

Often, the problem is simplified to a planar sample being approached by a spherical tip.

When the interactions between all atoms are considered, the potential varies as 1/d and the force varies with $1/d^2$.



$$U_{vdW} = \frac{-HR_{tip}}{6d}$$

Tip-Sample Interactions

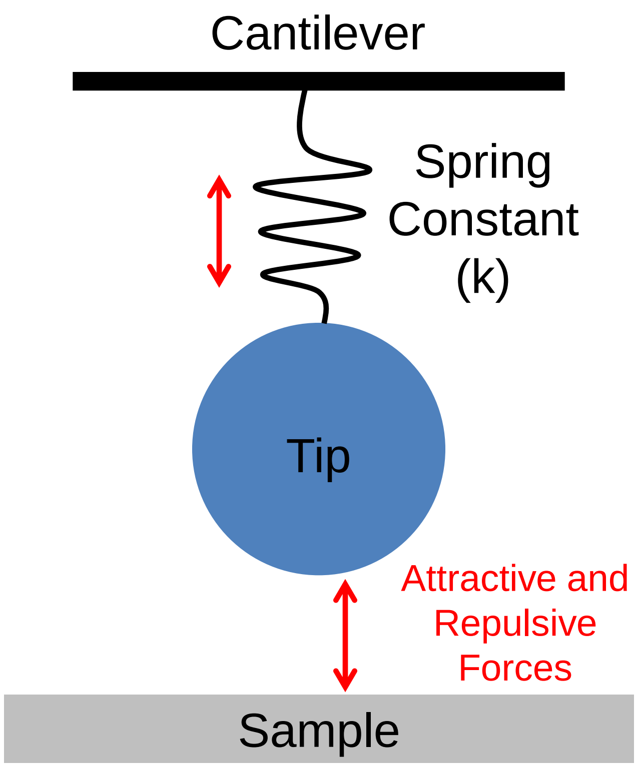
The tip is mounted at the end of a cantilever.

The cantilever is flexible and behaves like a spring.

As the tip approaches the surface, it will experience forces due to tip-sample interactions.

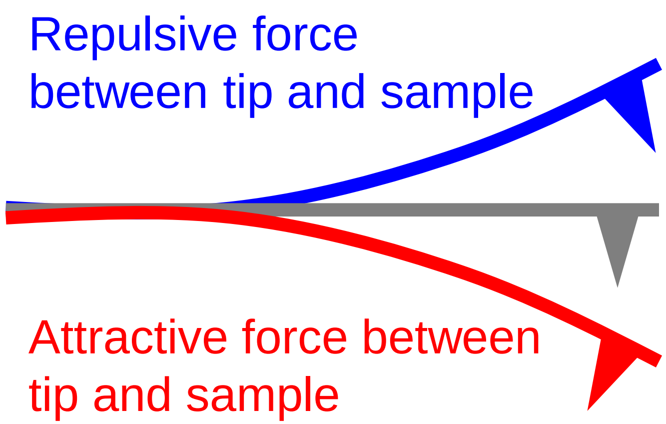
The spring-like action of the cantilever also acts on the tip.

A force balance can be set up that models all forces acting on the tip as it approaches the sample.

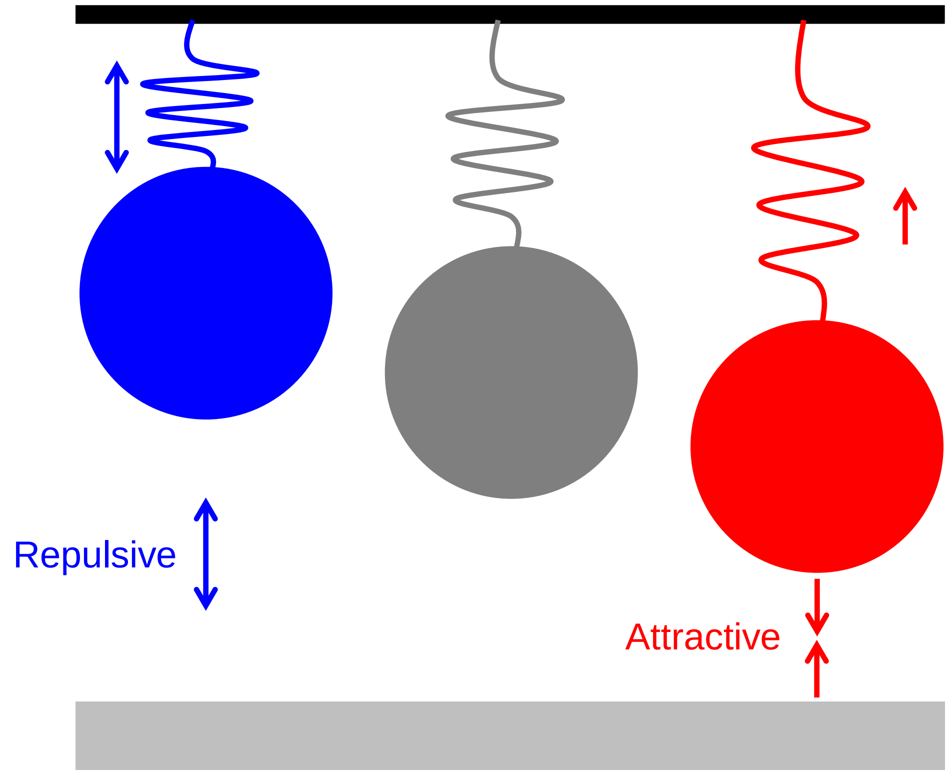


Tip-Sample Interactions

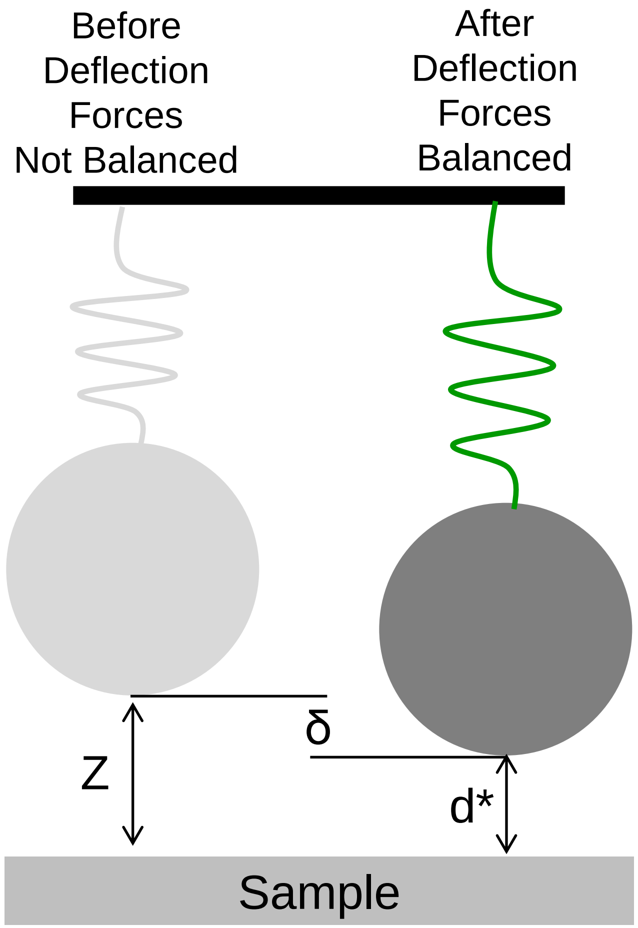
Examples of Cantilever Deflection

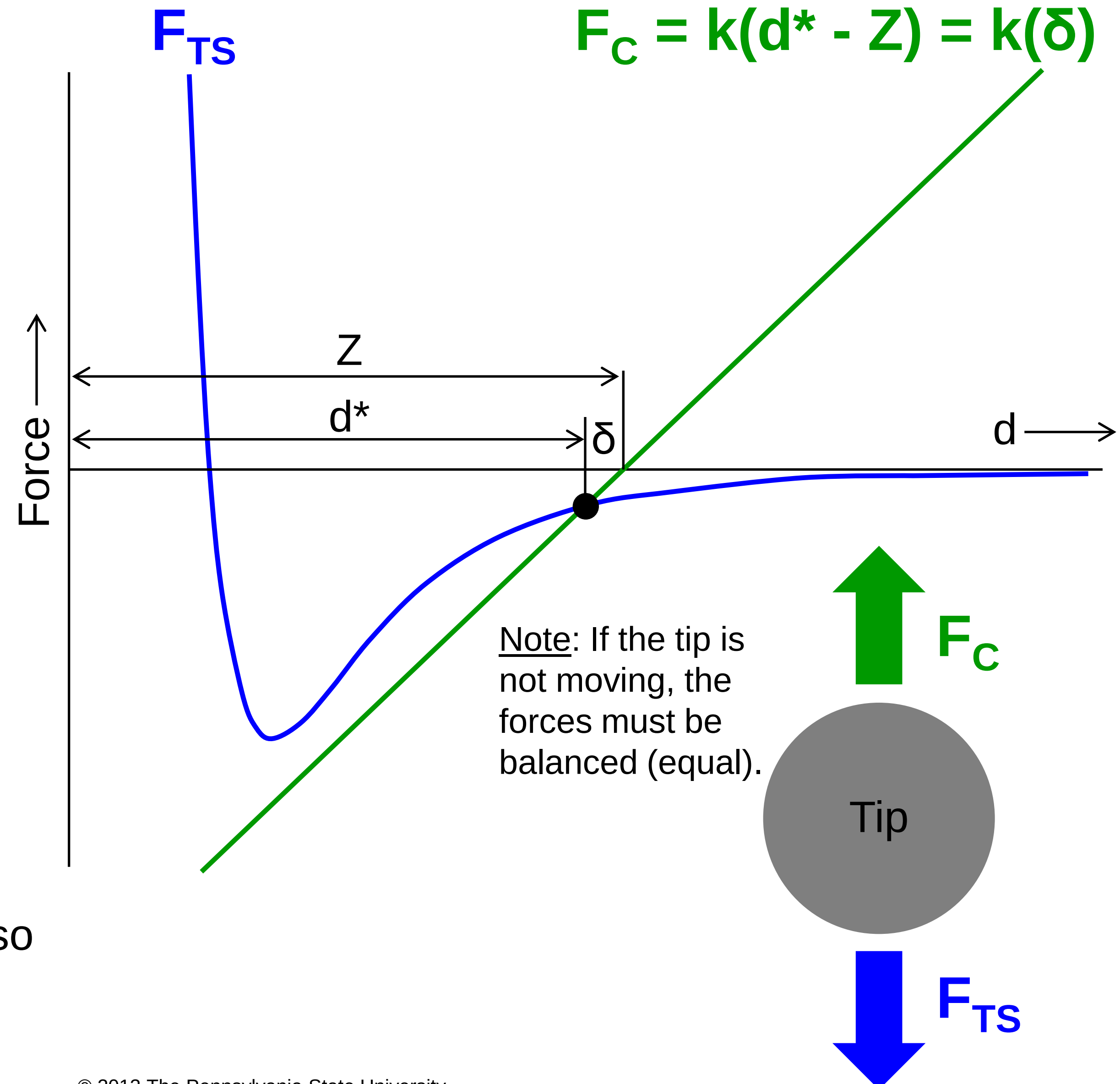


Note: If the tip is not moving, the forces must be balanced.



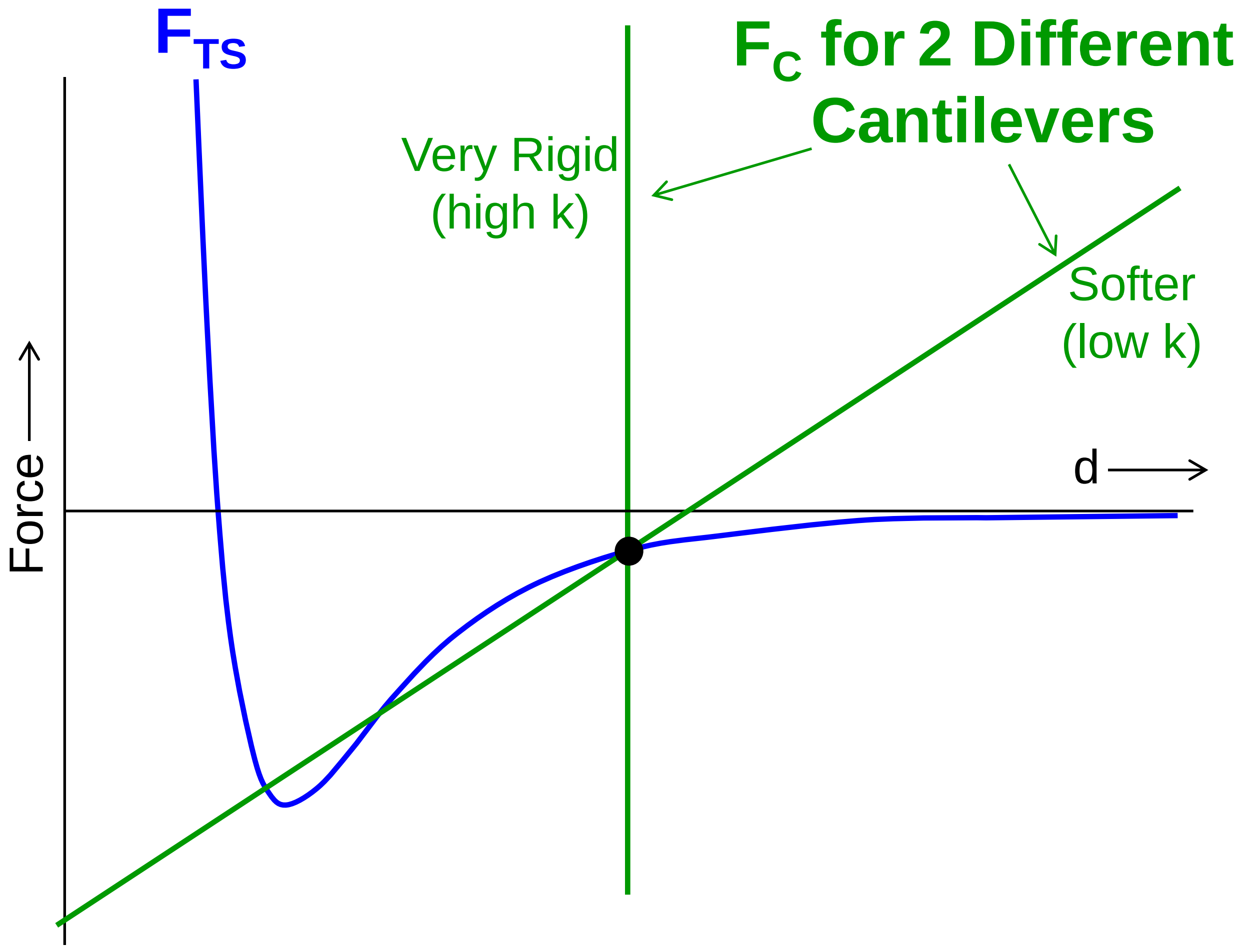
Tip-Sample Interactions



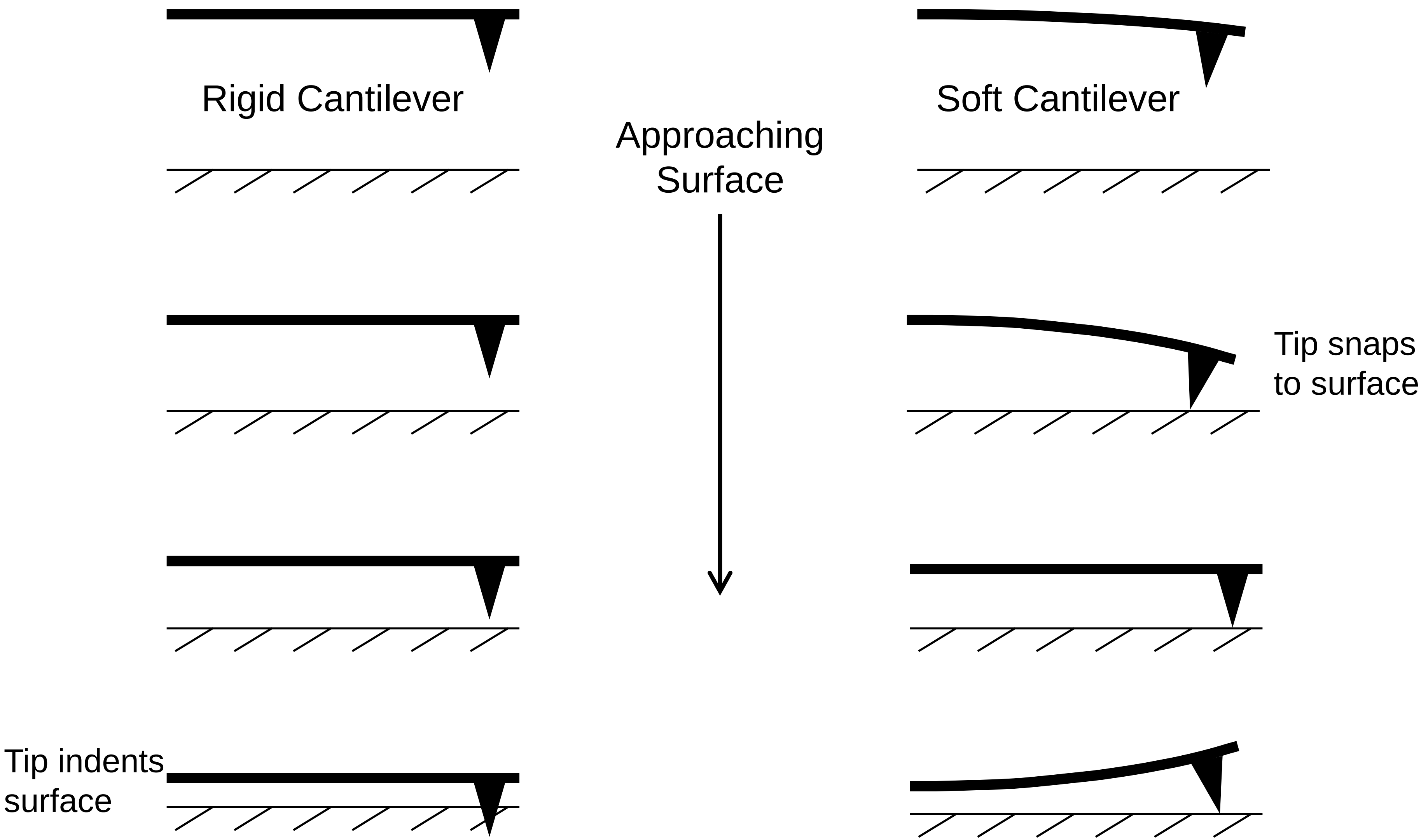


Note: Up is the (+) direction, so this picture shows a negative deflection (δ is negative).

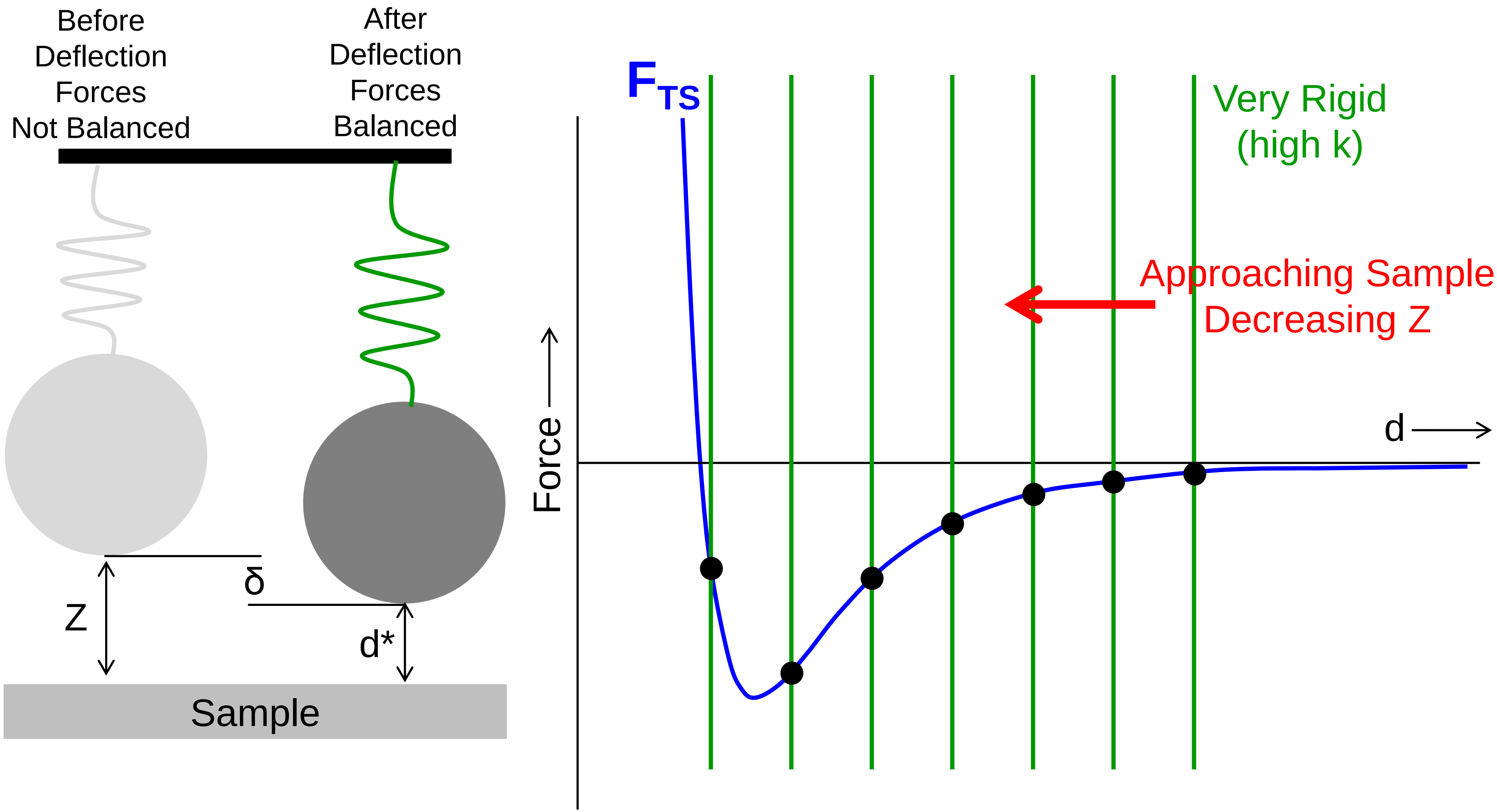
Cantilever Spring Constant



Tip-Sample Interactions

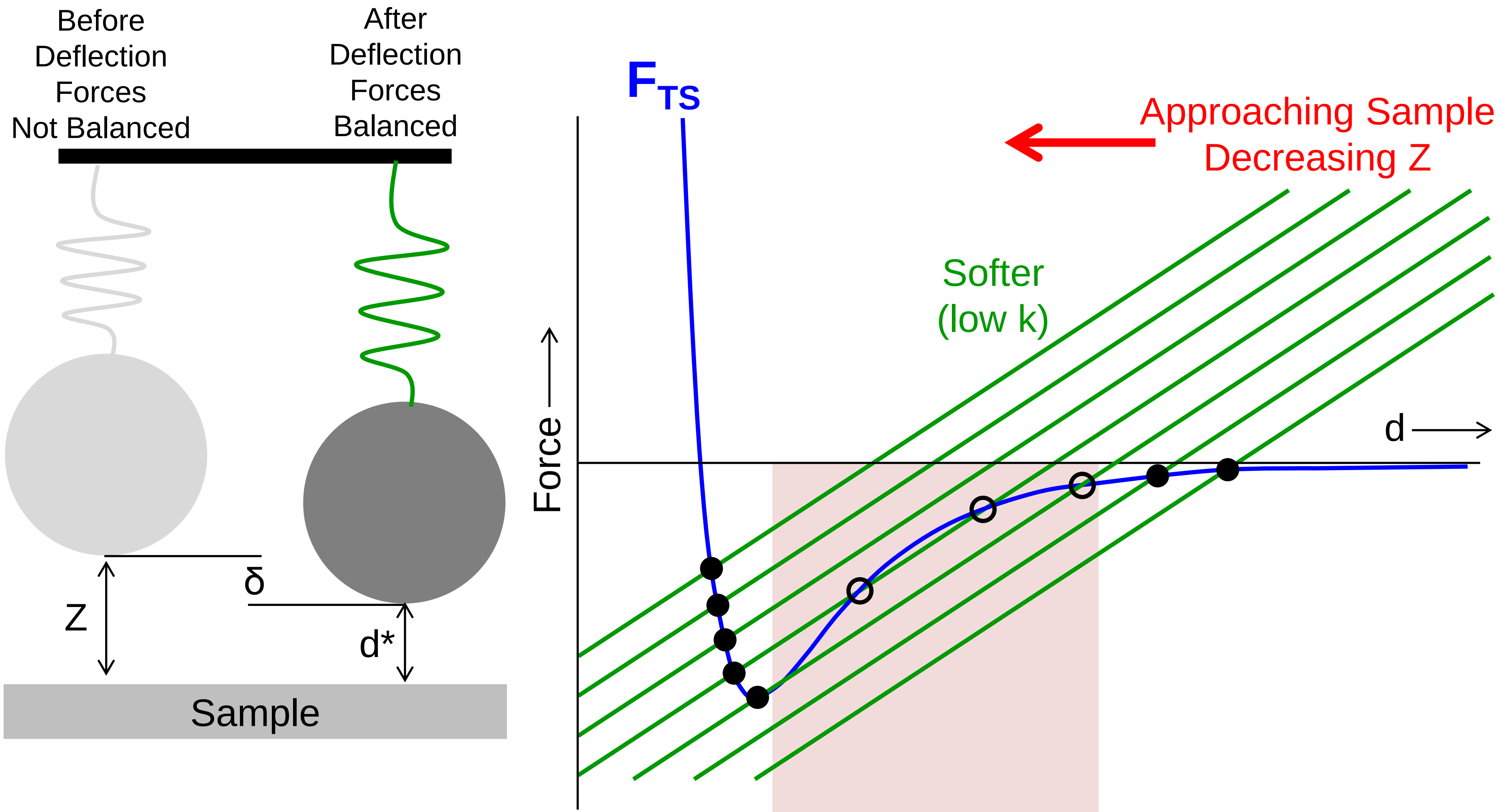


Cantilever Spring Constant



Cantilever is so rigid that the tip does not snap onto the surface. All d's are accessible.

Cantilever Spring Constant

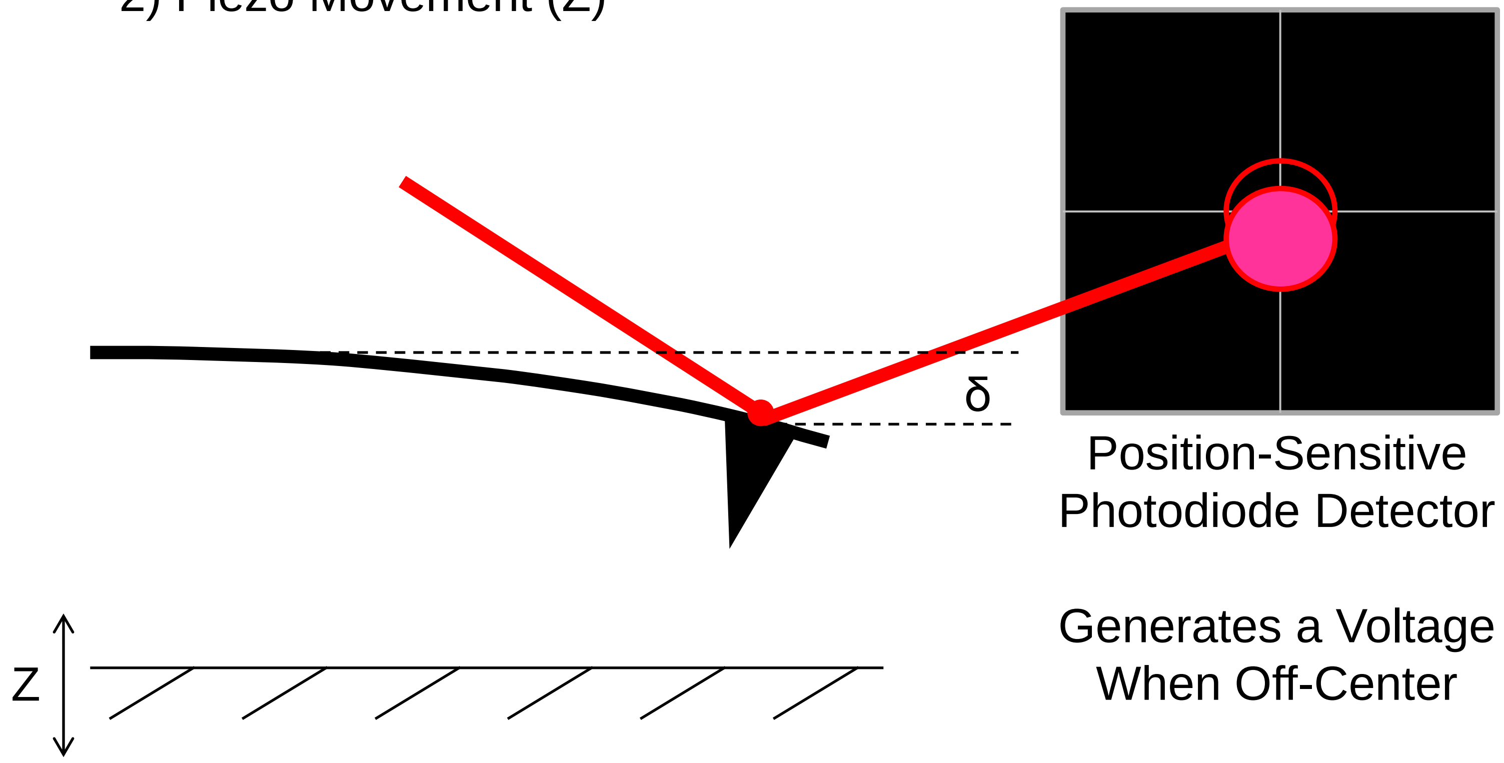


Tip snaps onto surface.
Some d's are not accessible.

AFM Signals and Measurements

What signals or variables are measured by the AFM instrument?

1) Cantilever Deflection (δ)
2) Piezo Movement (Z)



Generates a Voltage
When Off-Center

What do these signals look like as the tip approaches the sample surface?

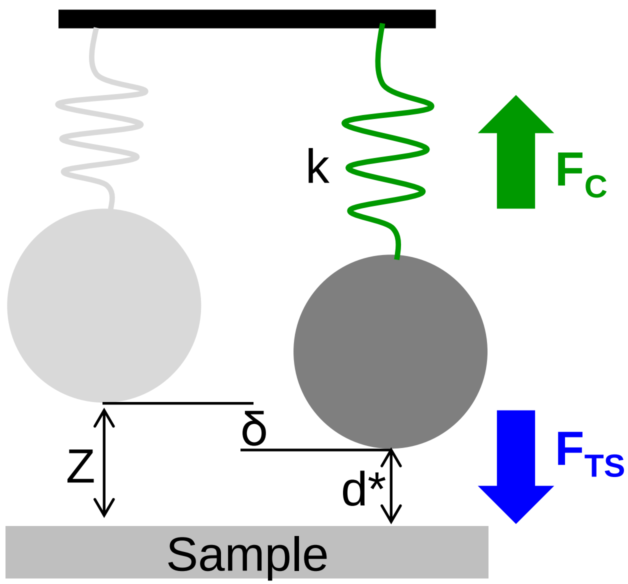
Force-Distance Curves

The data collected as the tip approaches, contacts, and retracts from the surface can be used to construct Force-Distance curves.

The collected data is a series of (Z, δ) data points.

These curves give information about the interaction forces between the tip and the surface.

The data points can be converted to (d, F_{TS}) data as follows:



At equilibrium

$$F_{TS} = F_C$$

$$F_{TS} = F_C = k\delta$$

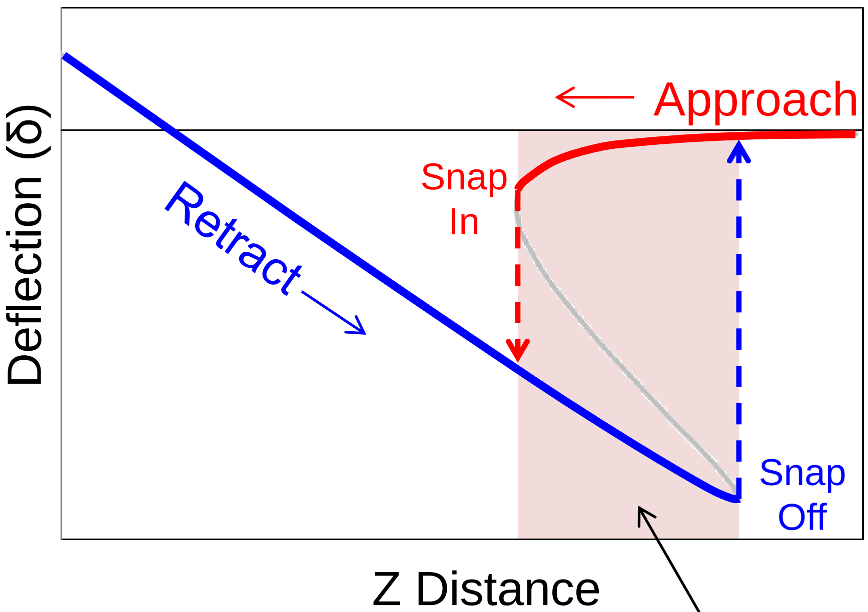
$$d^* = d = \delta + Z$$

Plug each (Z, δ) into these equations to convert to (d, F_{TS}).

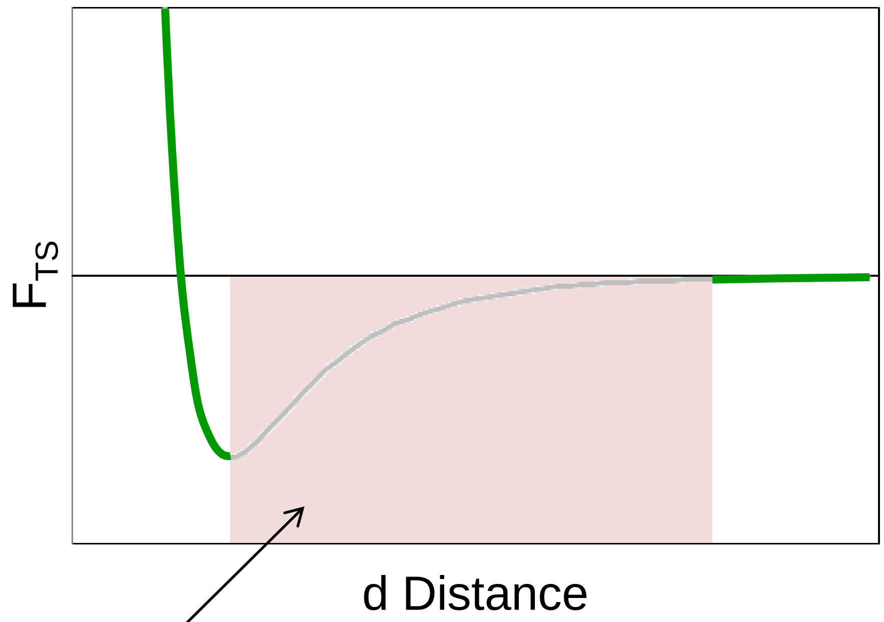
This generates a Force-Distance curve.

Force-Distance Curves

(Z, δ) Data

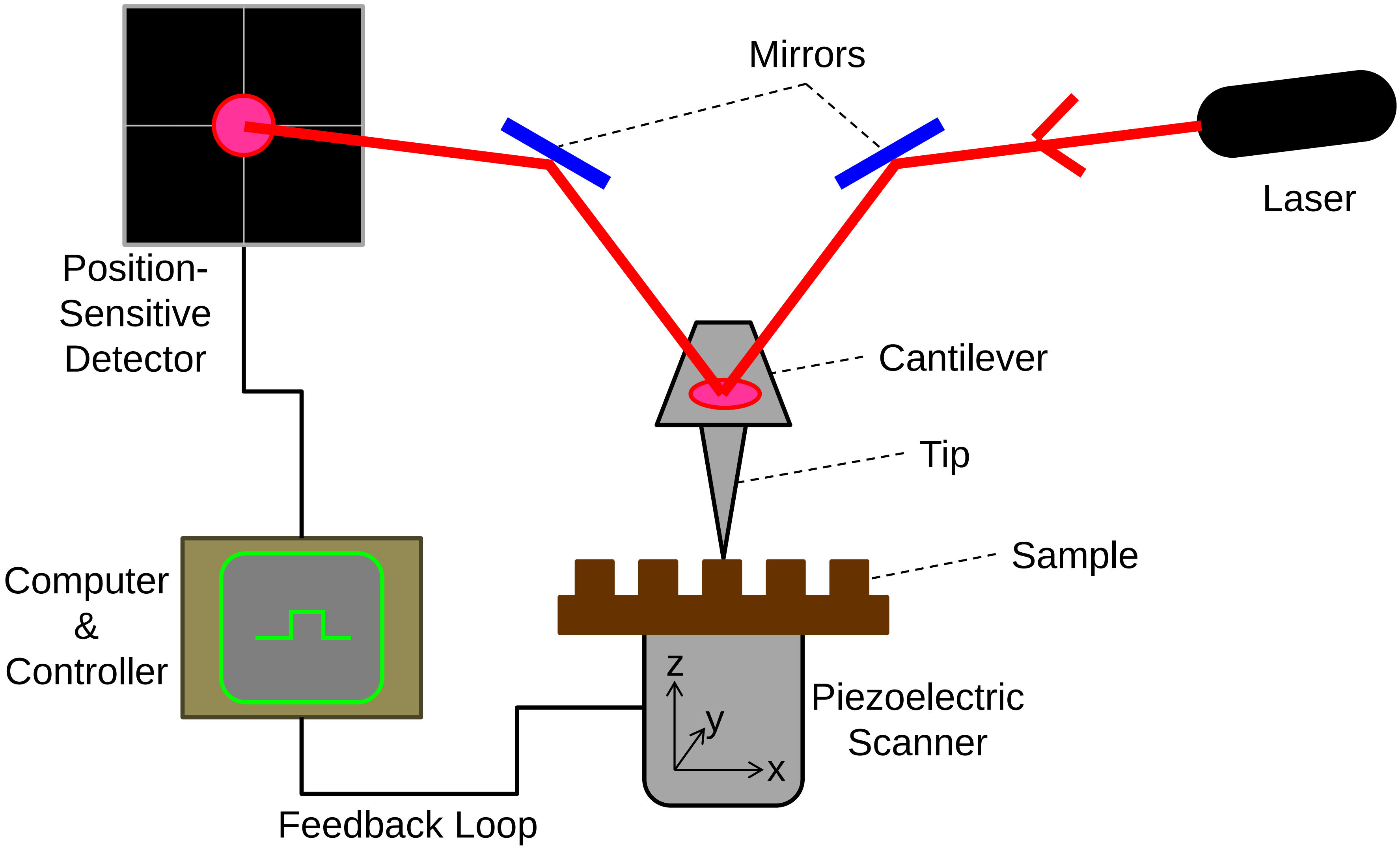


(d, F_{TS}) Data



Depending on the cantilever spring constant, some areas are not measurable.

Principle of Operation



Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

AFM Modes of Operation

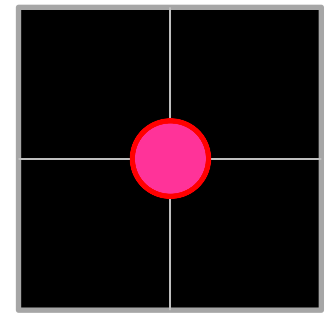
- An AFM can be operated in many different modes
- 2 modes will be overviewed:
  - contact
  - tapping mode
- Each mode has advantages and trade-offs.

Contact Mode

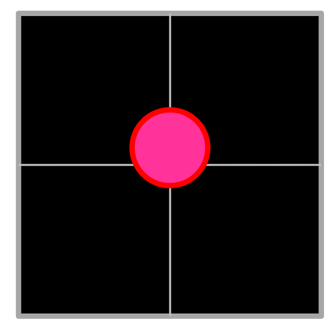
- In contact mode, the sample topography is measured by scanning the tip, which contacts the surface, across the sample.
- This is a popular mode of operation for durable samples.
- Since the tip is in contact with the surface, damage to tip and sample may occur as scanning proceeds.
- Frictional forces may also exist between the sample and tip.
- Tips can become contaminated and wear down over time.

Contact Mode

- Detector: DC signal
- Voltage Set Point: how hard/soft the tip touches surface
- Higher Set Point = Harder Contact (greater cantilever deflection)
- Must watch for damage to sample (horizontal streaks)

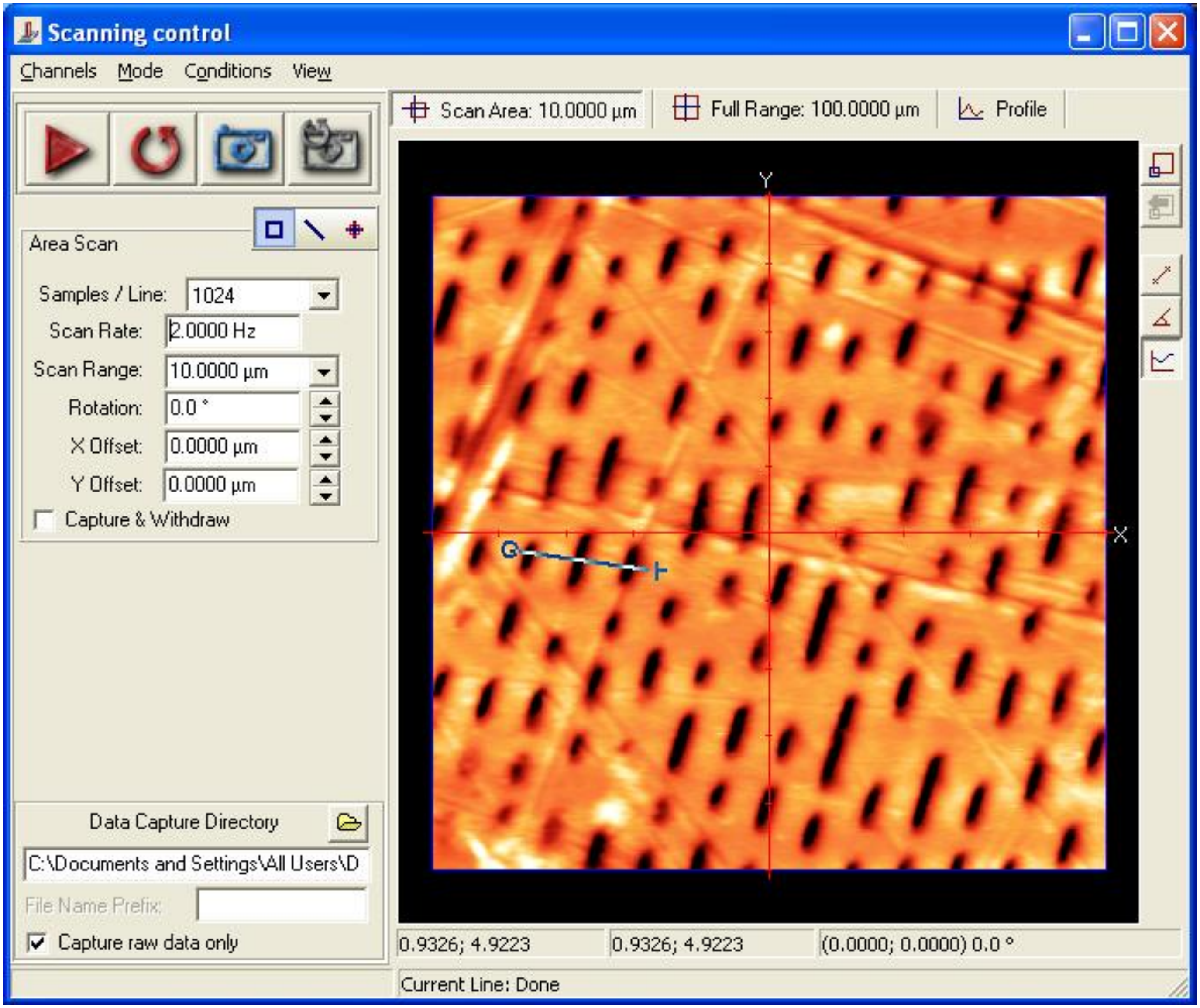


Top – Bottom = Small V



Top – Bottom = Large V

Contact Mode



Noncontact Mode

- In noncontact mode, the instrument senses the van der Waals attractive forces between the surface and the probe that is held above the surface.
- Eliminates damage to the surface that could occur in contact mode.
- Best results are obtained in UHV since moisture on surfaces creates capillary forces in ambient conditions.

Tapping Mode

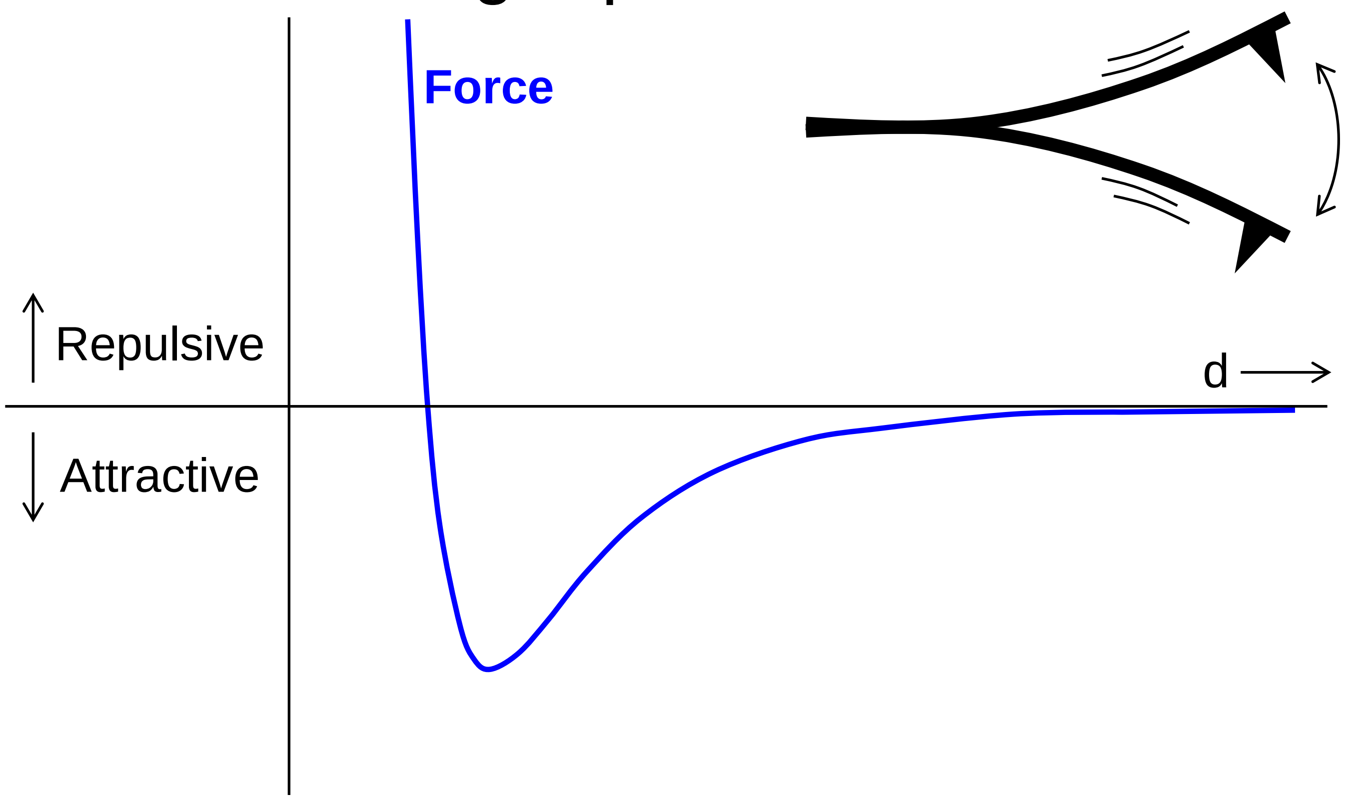
- Also called intermittent contact mode.
- In this mode the cantilever is excited close to its resonant frequency by an external piezoelectric ceramic attachment.
- Resonant frequencies are typically from 15 kHz to 500 kHz. (Compare to rate of scanning.)
- The amplitude of cantilever vibration is usually in the 10 – 100 nm range.

Tapping Mode

- Tapping mode works well for many samples including soft, adhesive, or fragile surfaces.
- Tapping mode overcomes problems associated with friction, adhesion, electrostatic forces, and other difficulties associated with conventional contact mode techniques.

Tapping Mode

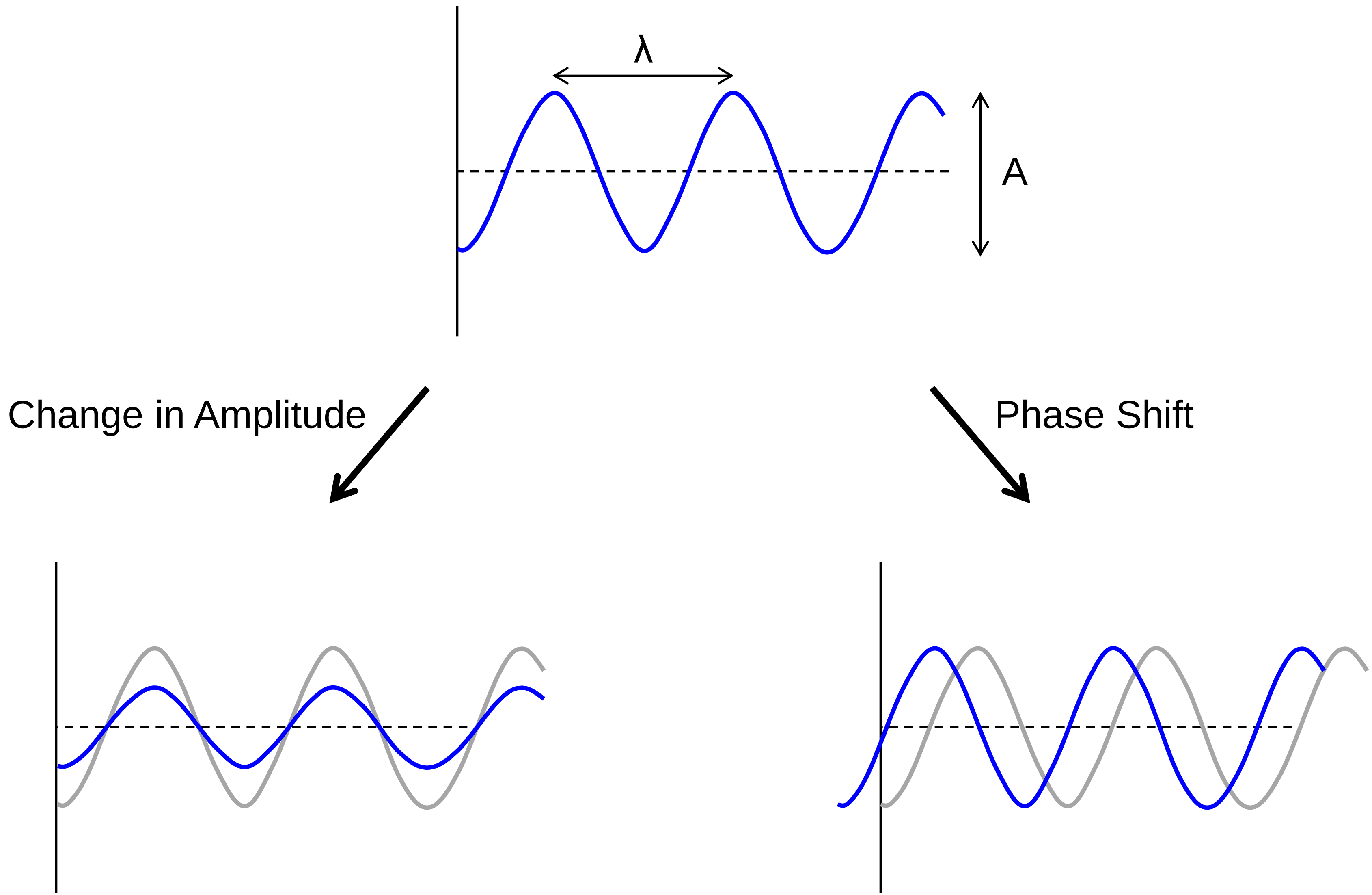
- At the bottom of each vibration, the tip comes close to the surface and may contact it briefly.
- Therefore, there is an abrupt change from weak attraction to strong repulsion.



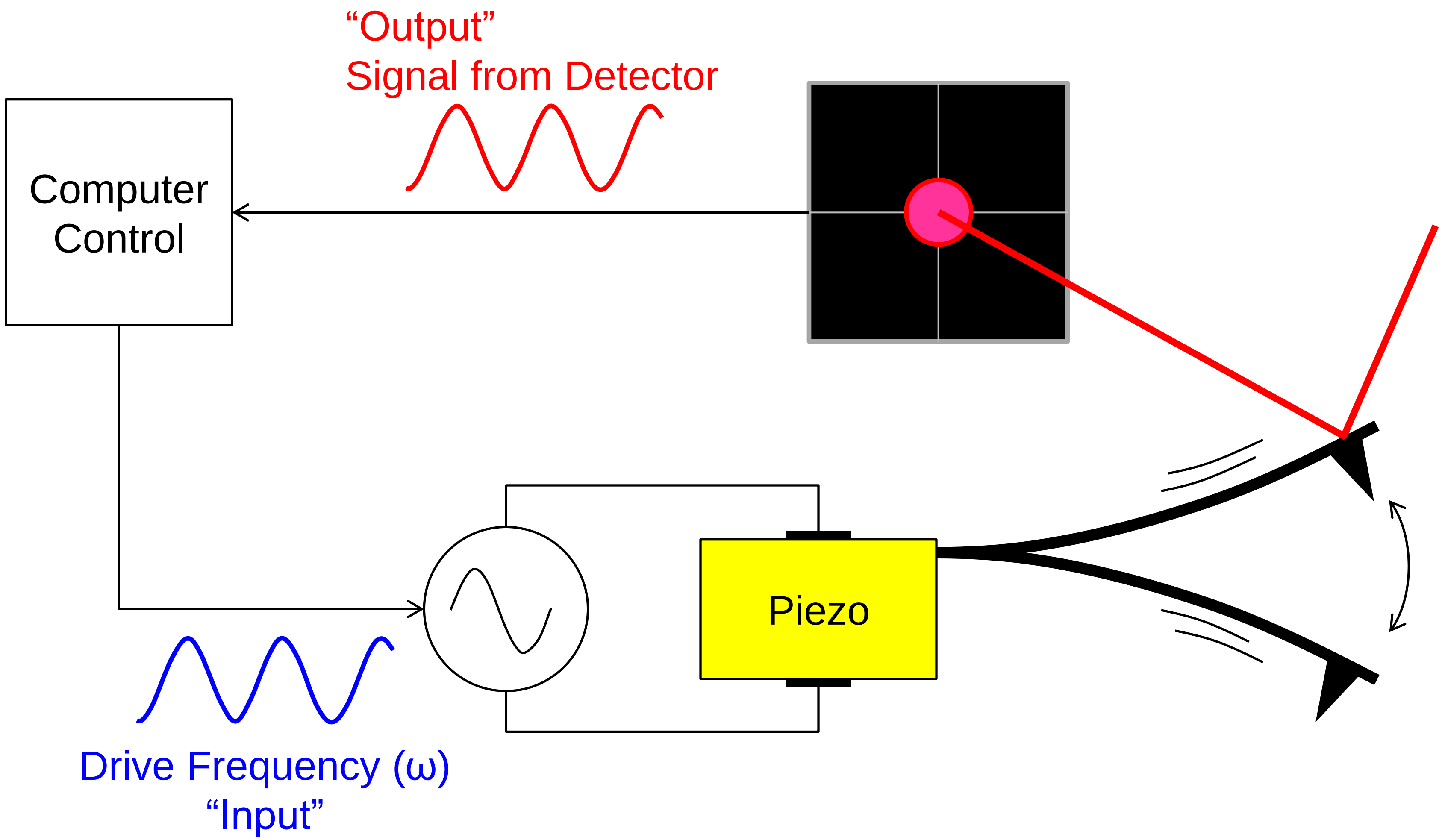
Tapping Mode

- Cantilever is tuned in free space (not contacting the sample) to determine its fundamental frequency (ω_0).
- Vibration at ω_0 produces the maximum amplitude of cantilever/tip movement.
- As vibrating tip approaches the sample, tip-sample interactions dampen the amplitude of vibration (amplitude decreases).
- The phase lag (or phase shift) can also be measured and gives information about the energy dissipated by the interactions.

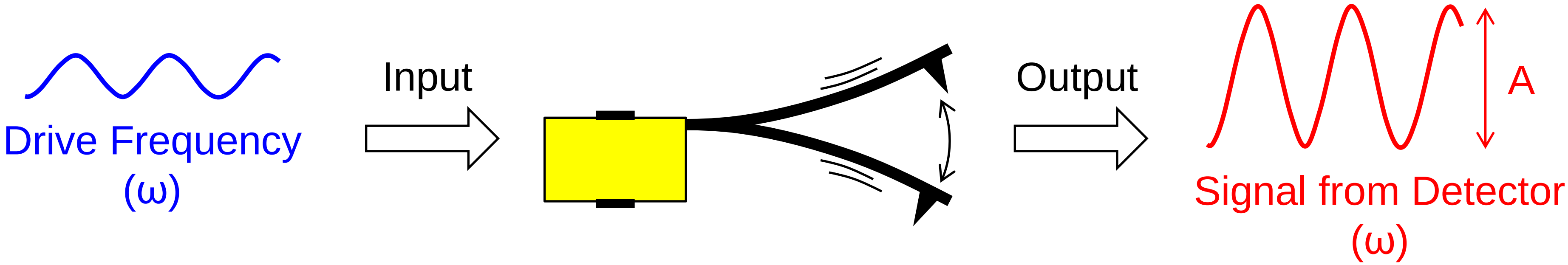
Amplitude and Phase Shift

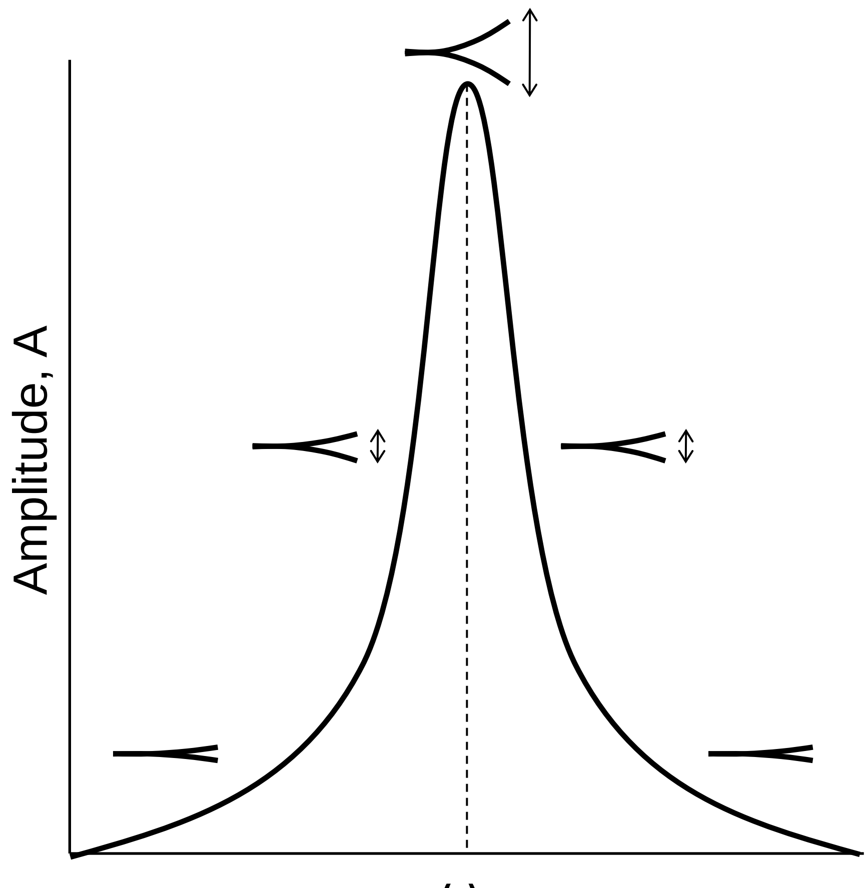


Tapping Mode



Tuning the Cantilever

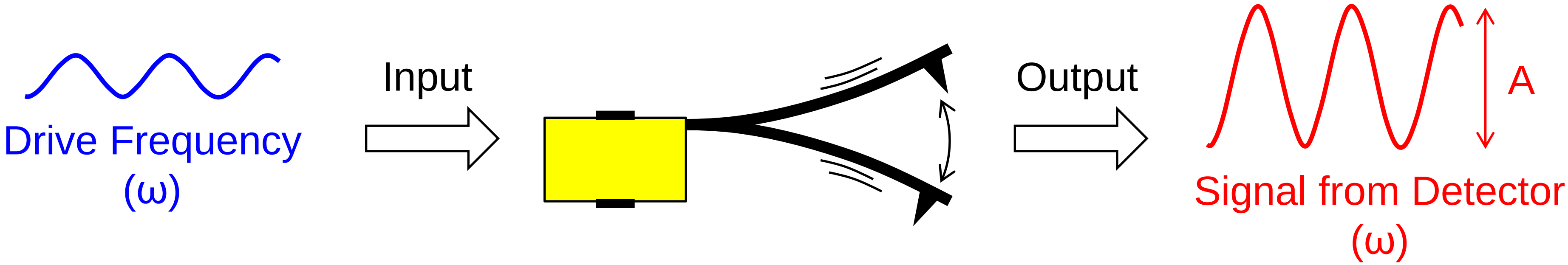


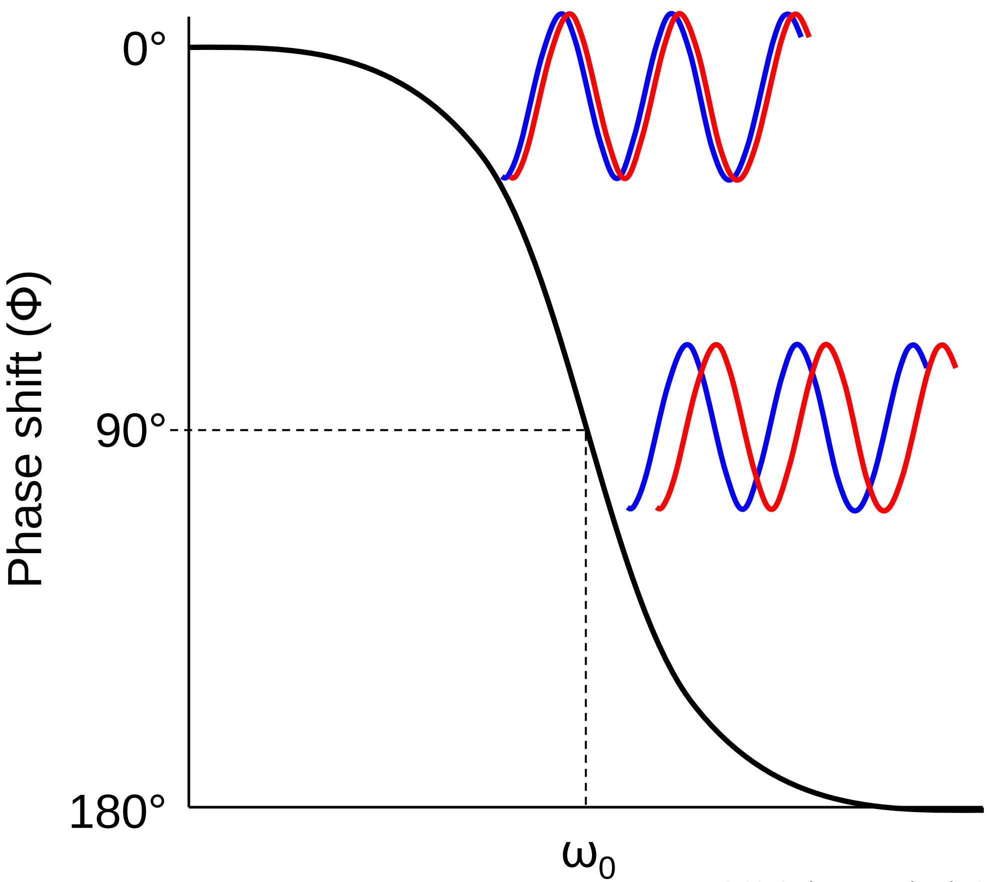


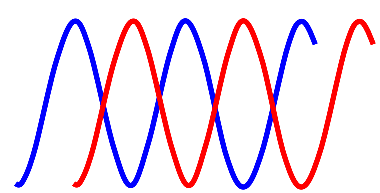
Sweep drive frequency and measure amplitude of cantilever vibration.

There will be a frequency (ω_0) at which the amplitude is maximized.

Tuning the Cantilever





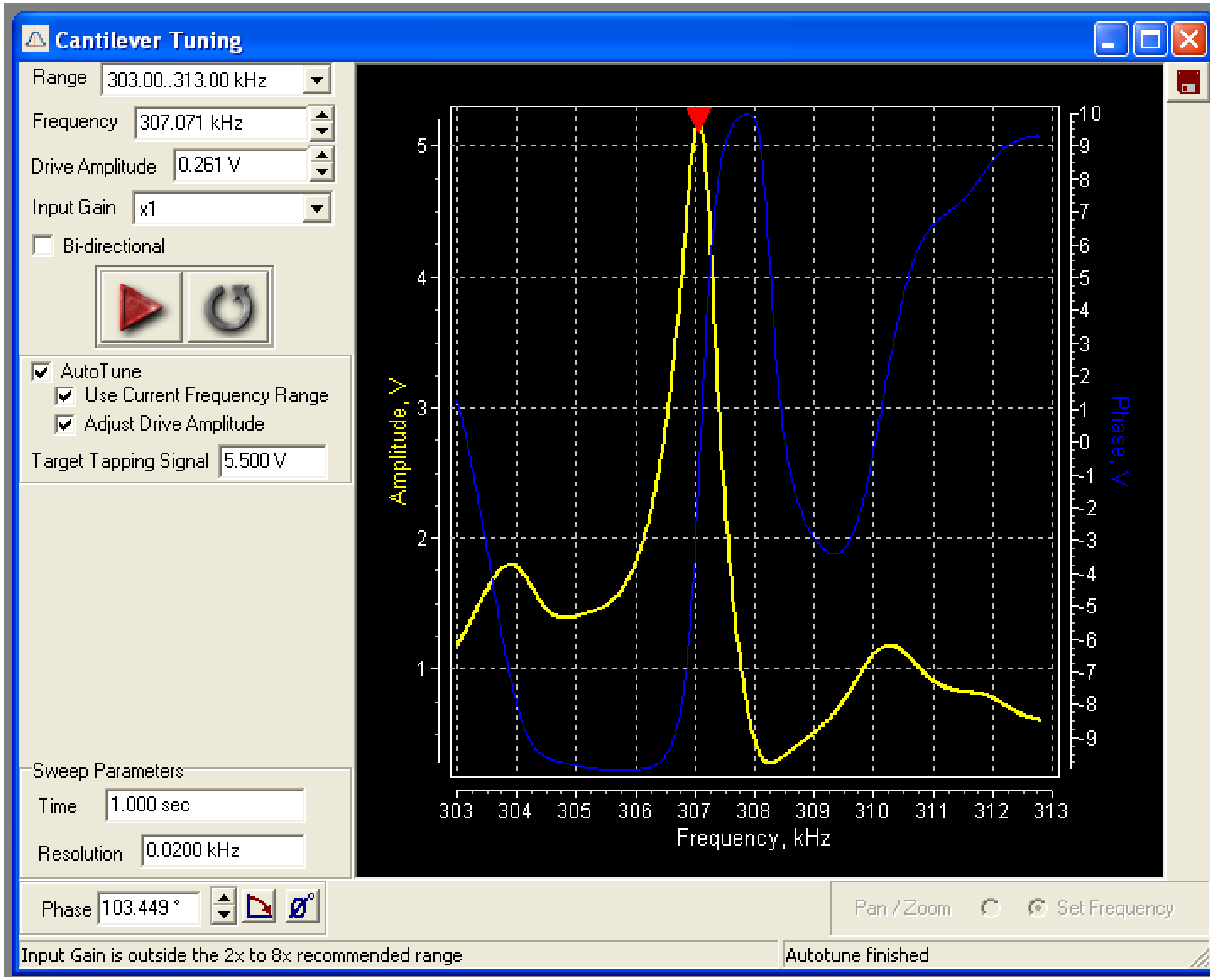


Phase shift (Φ) is an additional signal that contains information about the sample surface.

Phase shift is caused by the dissipation of energy as the tip interacts with the sample surface.

This information is not available in contact mode. Why?

Tuning the Cantilever



Actual data gathered while tuning a cantilever.

Data sets reported as voltages.

Curves have complex shape compared to idealized case shown previously.

Approaching the Sample

The cantilever is tuned above the sample (not interacting with the sample surface).

The drive frequency (ω) is selected during tuning and does not change during approach or scanning.

Therefore, the maximum amplitude of oscillation occurs when the tip is not affected by tip-sample interactions.

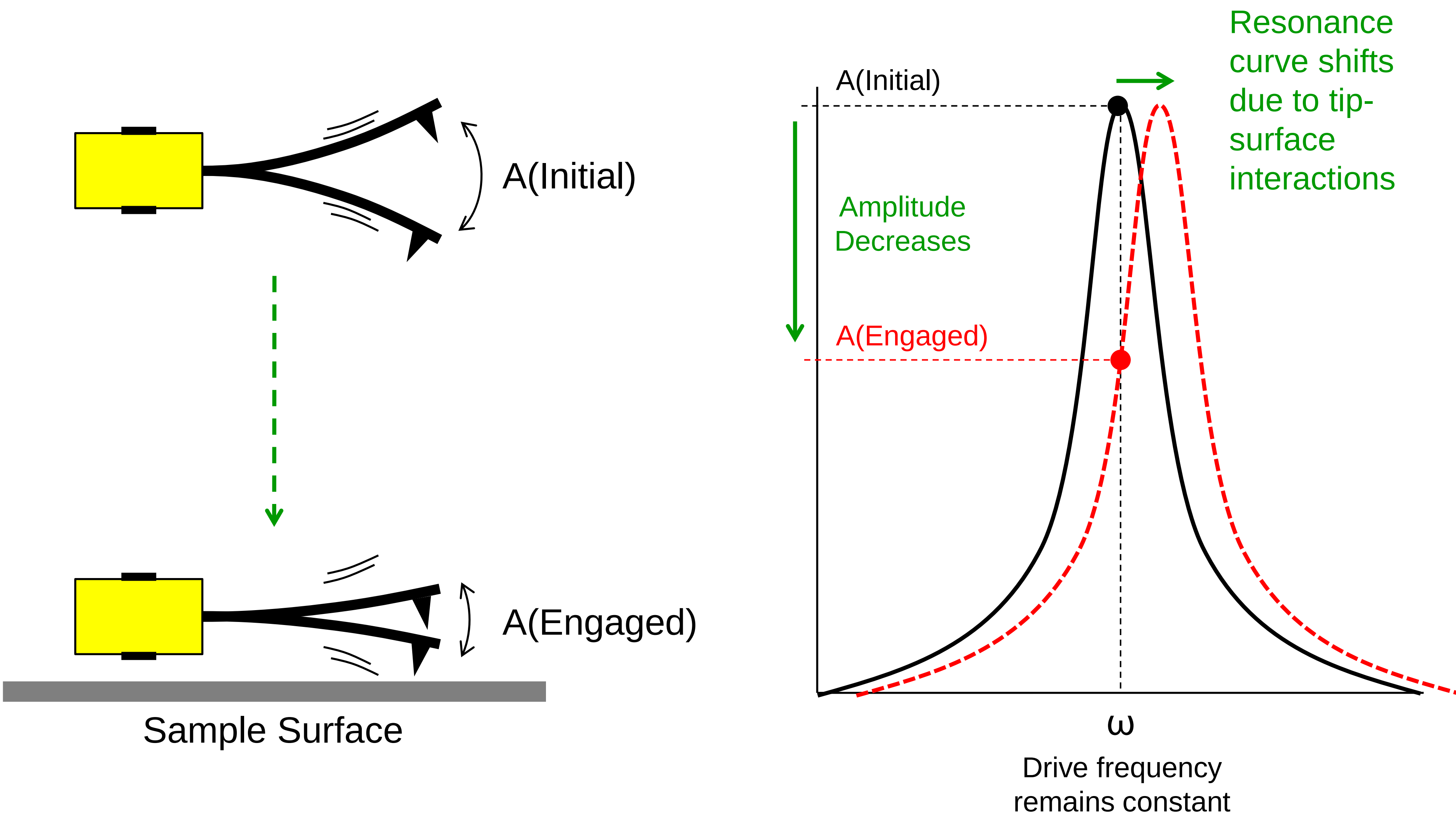
Interactions with the surface dampen the oscillation of the cantilever.

Tip-sample forces change (ω_0) such that the drive frequency $\omega \neq \omega_0$ when the tip approaches the surface.

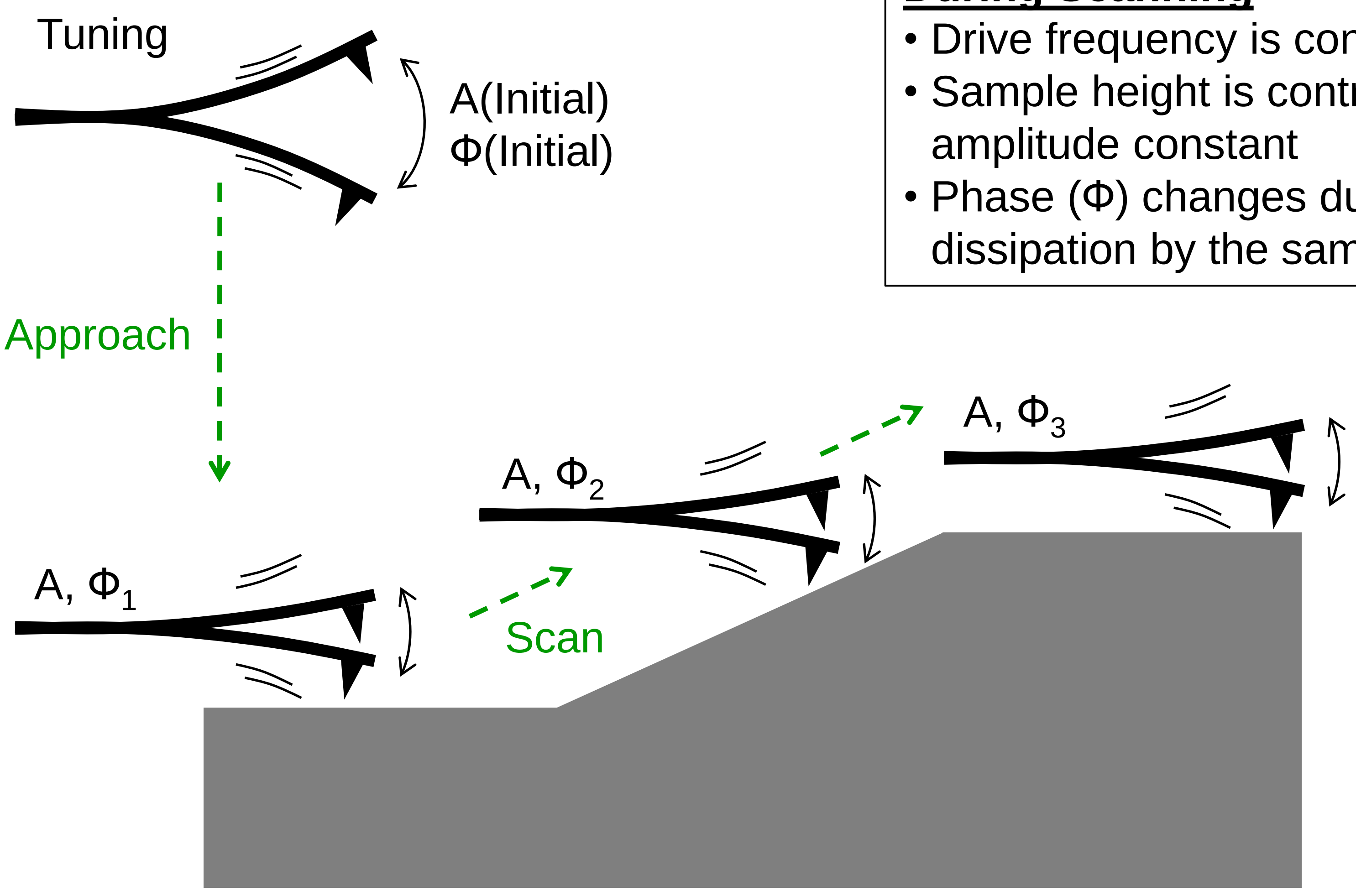
This effectively "de-tunes" the cantilever and lowers the amplitude of oscillation.

The system detects the lowered amplitude as a decreased voltage produced by the detector (compared to the higher voltage produced during tuning).

Approaching the Sample



Scanning the Surface

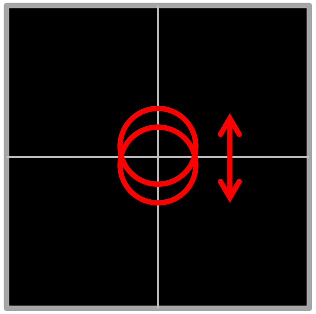


During Scanning
- Drive frequency is constant
- Sample height is controlled to keep amplitude constant
- Phase (Φ) changes due to energy dissipation by the sample surface

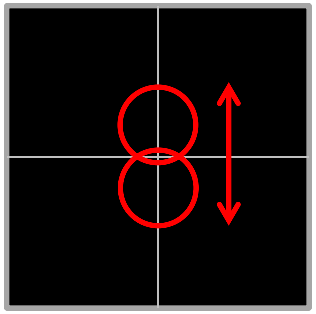
Note: A < A(Initial) and A is chosen by adjusting the Voltage Set Point.

Tapping Mode: Amplitude

- Detector: AC signal
- Cantilever is oscillating at drive amplitude
- Tries to maintain set point voltage which corresponds to an oscillation amplitude (A)
- Interaction with sample decreases the amplitude
- Higher V = Lighter Tapping



Top – Bottom = Small V
Lower Amplitude
Harder Tapping

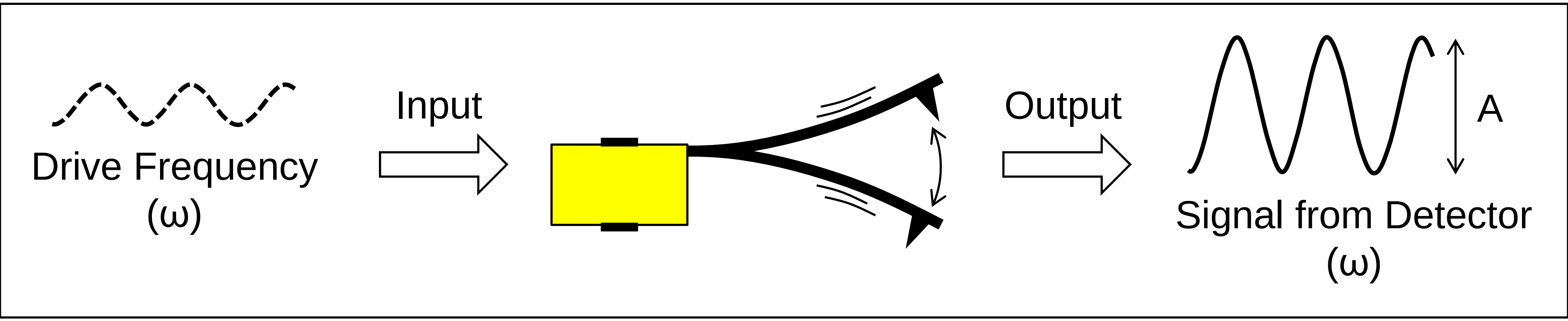


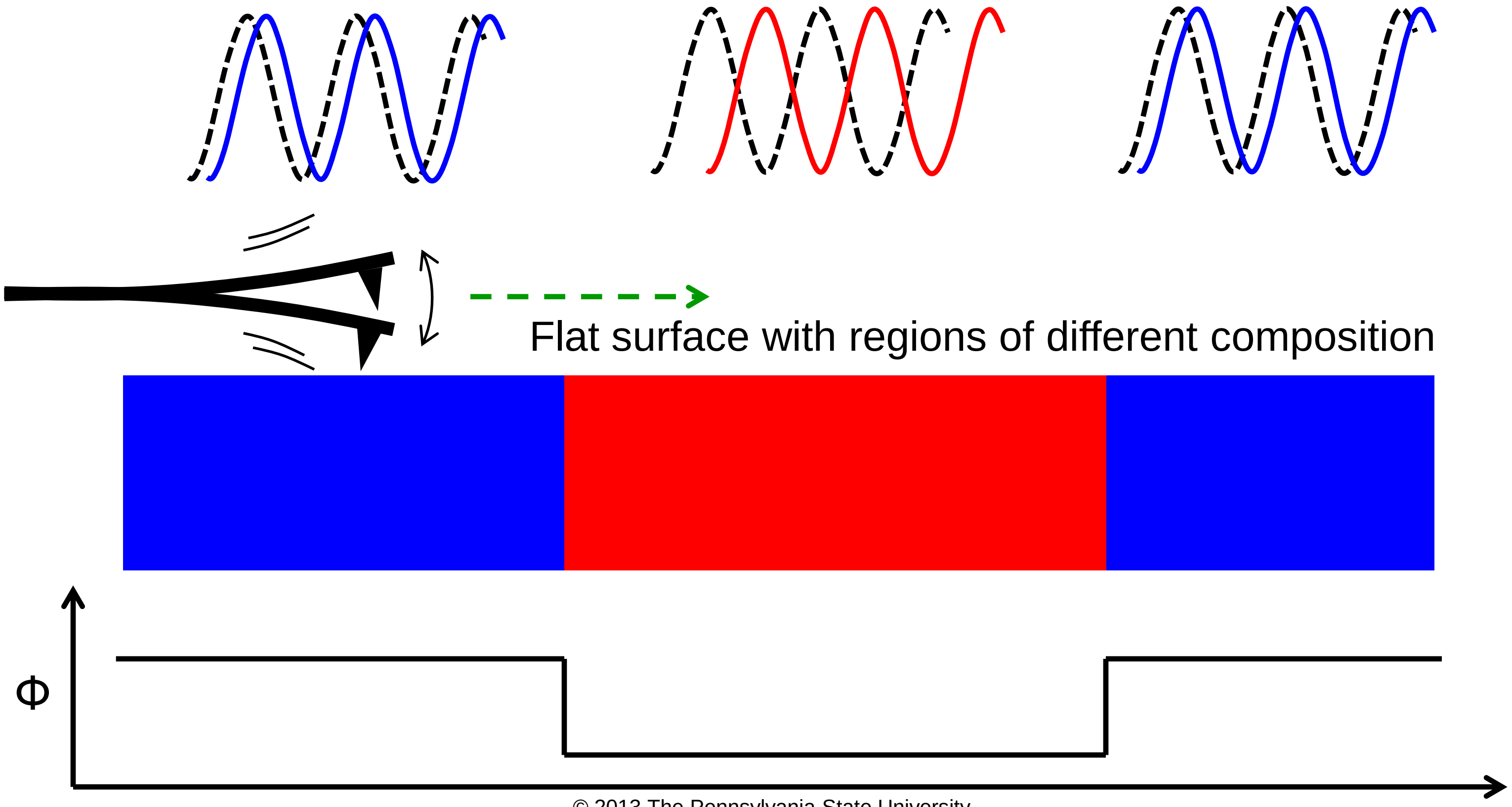
Top – Bottom = Large V
Higher Amplitude
Softer Tapping

Tapping Mode: Phase

- In addition to height information, tapping mode provides information about the interaction of the tip with the sample
- Viscoelasticity: “rubbery-ness”
- Energy from the vibrating tip is absorbed and dissipated by the sample
- Causes phase shift of output AC signal relative to input driving AC signal
- Phase Image gives information about tip-sample interactions even when there is no change in height

Phase Contrast

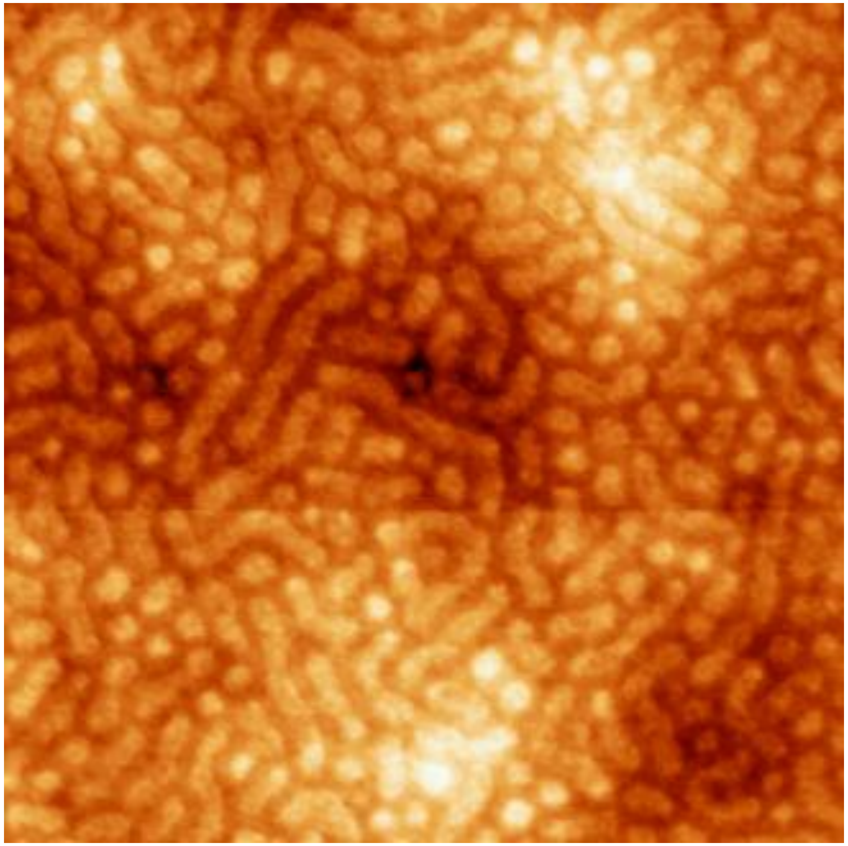




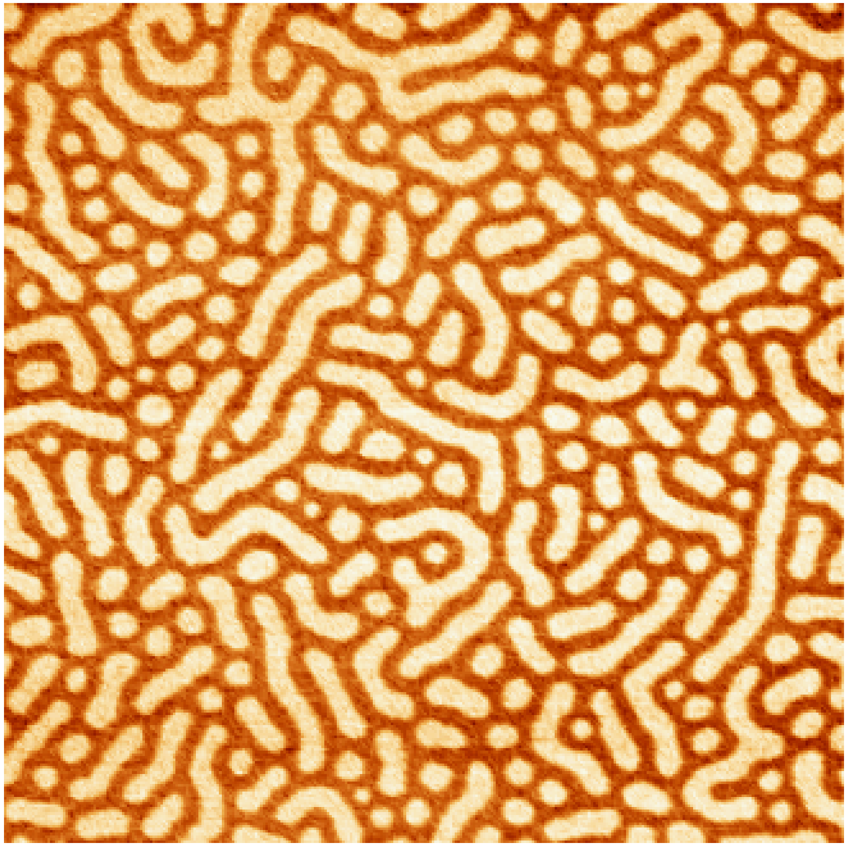
Example Images: Tapping Mode

The two sets of data below were simultaneously collected while scanning a block copolymer film (PS-*b*-PMMA). Scan area is 2 um x 2 um. The film is very flat but the phase image detects differences in the material properties of PS versus PMMA.

Height Data



Phase Shift Data



Outline

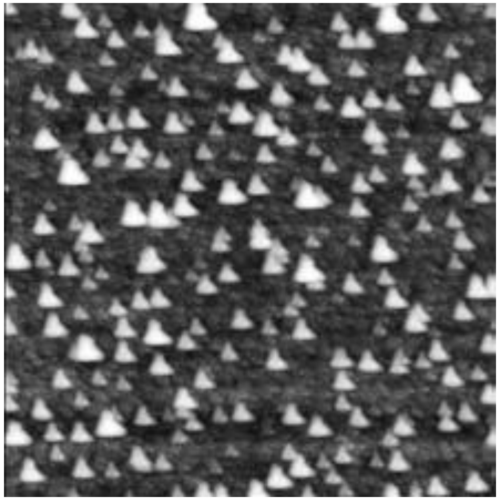
- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

Common Problems

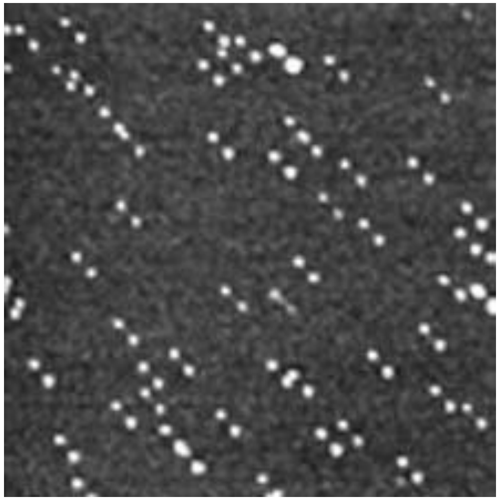
- If sample is not clean, dust particles will interfere with scanning.
- Improperly aligned laser and/or detector gives low signal (want > 2.00 V on detector).
- Image artifacts due to damaged or dirty tip
- Feedback Loop not tuned properly. This causes overshoot and “ringing.”

Image Artifacts

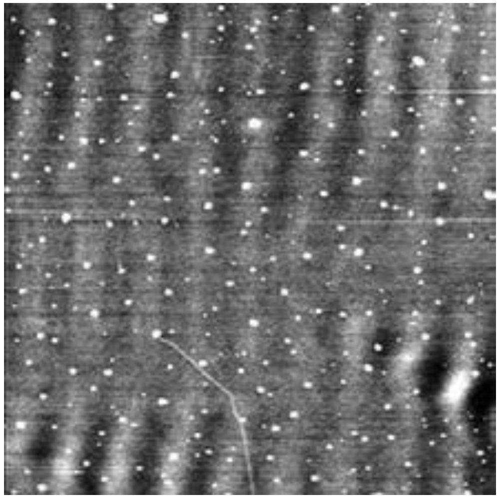
Dull or Dirty Tip



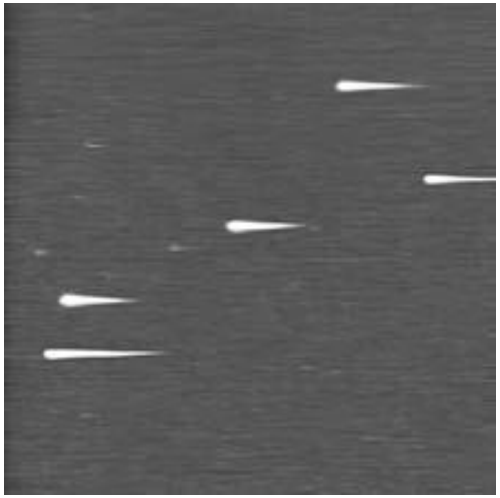
Multiple Tips



Optical Interference Effects



Tip Not Tracking The Surface



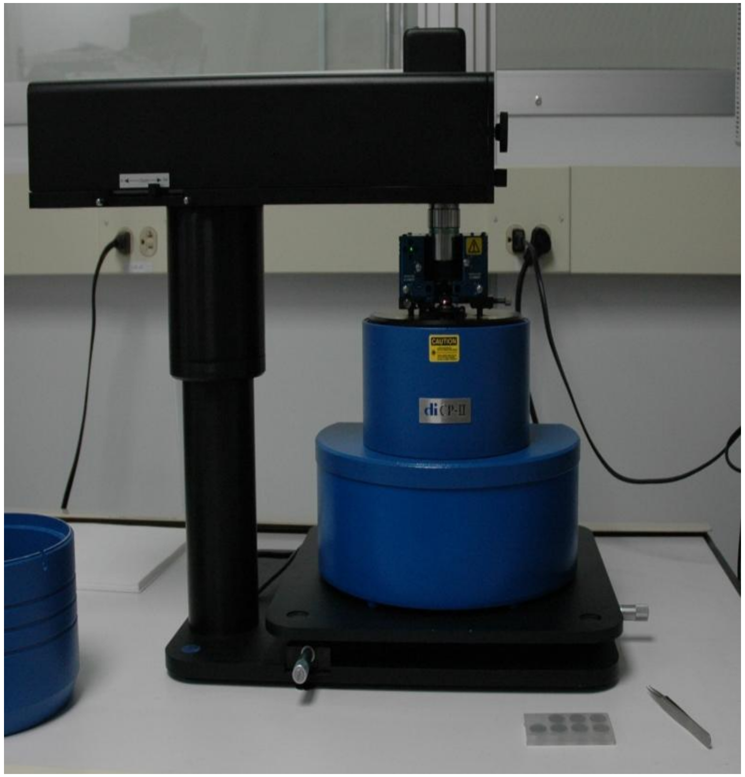
Veeco: “A Practical Guide to SPM” http://www.veeco.com/pdfs/library/SPM_Guide_0829_05_166.pdf

Outline

- Overview of Scanning Probe Techniques
- Scanning Tunneling Microscopy
- Atomic Force Microscopy
  - Hardware and Components
  - Tip/Sample Interactions
  - Common Modes of Operation
  - Pitfalls and Image Artifacts
- Example of Instrument Operation

SPMs in The Teaching Cleanroom

Veeco CP-II



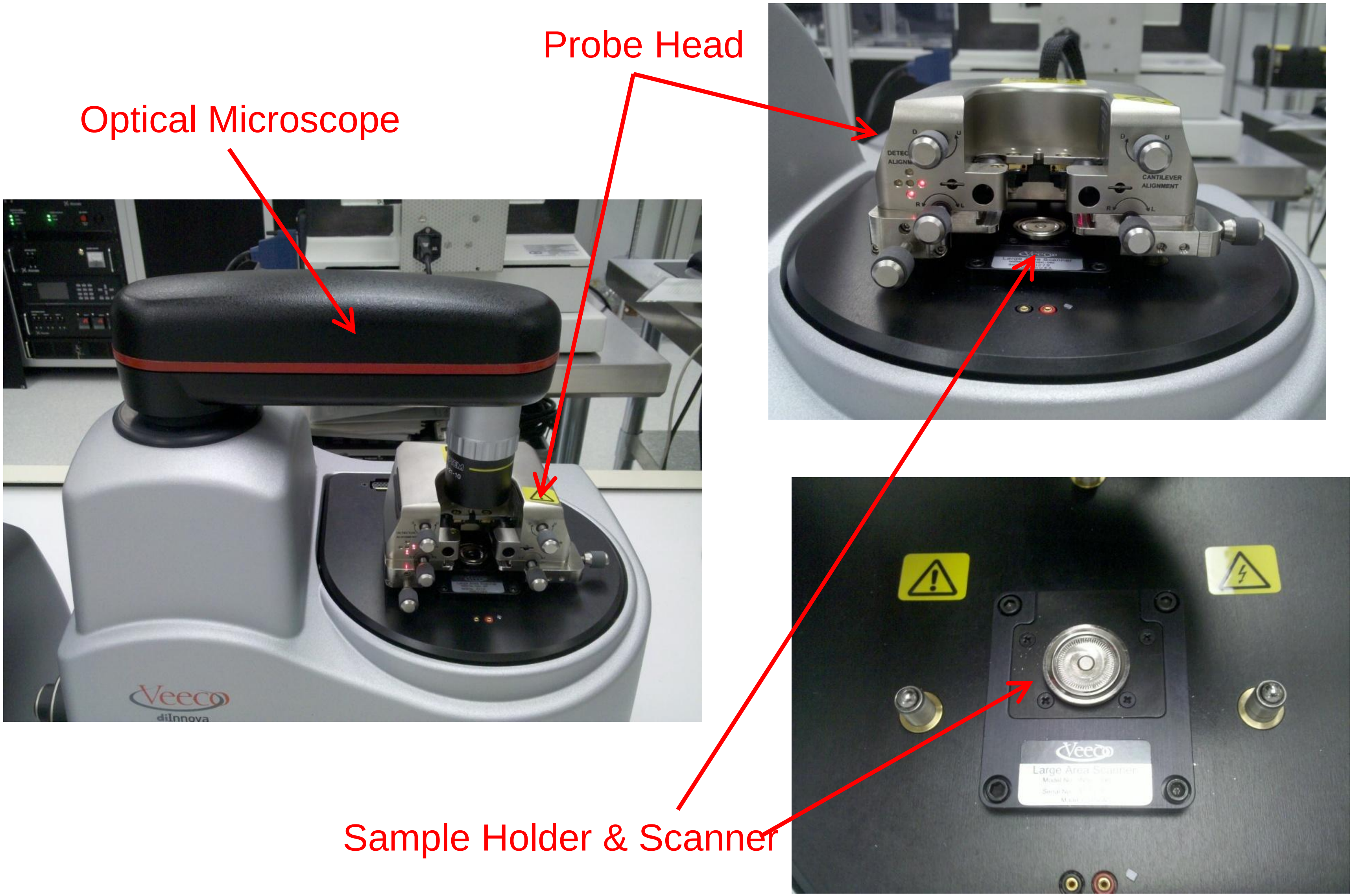
Veeco Innova

Penn Sate Center for Nanotechnology Education and Utilization

Example Specifications Veeco Innova SPM

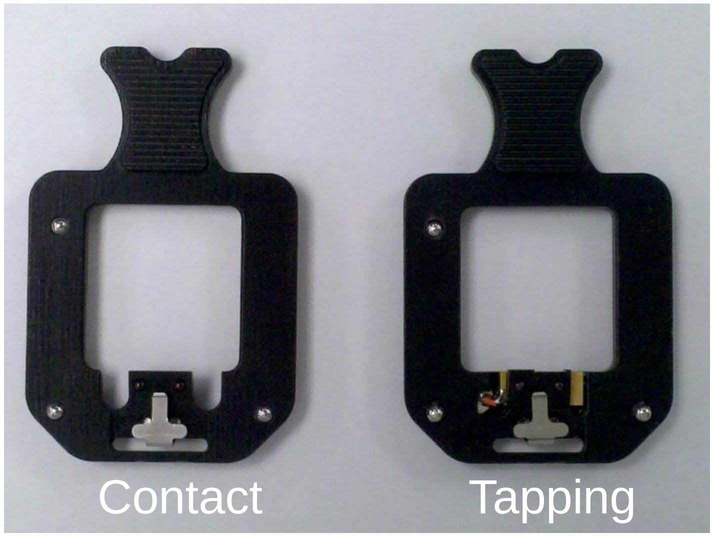
- **Closed-Loop Scanner:** X,Y > 90 μm, Z > 7.5 μm
- **Open-Loop Scanner:** X,Y > 5 μm, Z > 1.5 μm
- **Sample size:** 45 mm x 45 mm x 18 mm (X, Y, Z)
- **Motorized Z Axis Stage:** Z Travel: 18 mm
- **Optics:**
  - Camera: on-axis color CCD with motorized zoom
  - Field of view: 1.25 mm – 0.25 mm (motorized zoom, with 10x objective)
  - Resolution: <2 μm with standard 10x objective
- **Electronics:** 20-bit DAC control, 100 kHz ±10V ADCs, digital feedback
- **System software:** SPMLab™ v7 for data acquisition & analysis, Windows XP

Innova SPM Parts

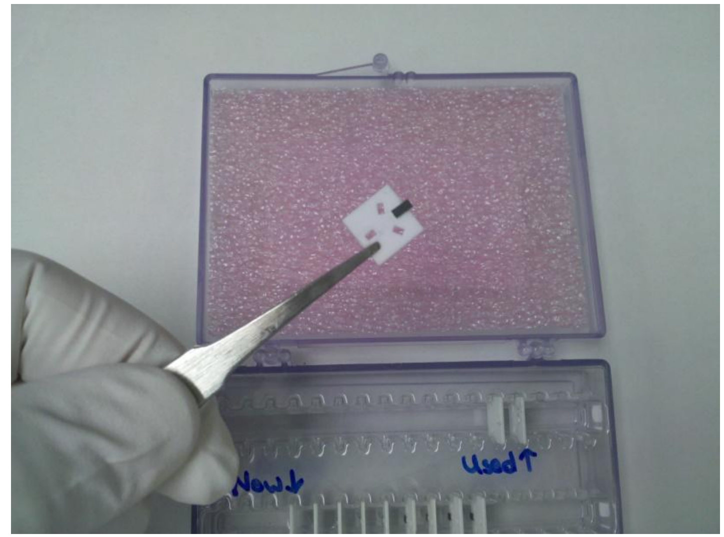


Innova SPM Parts

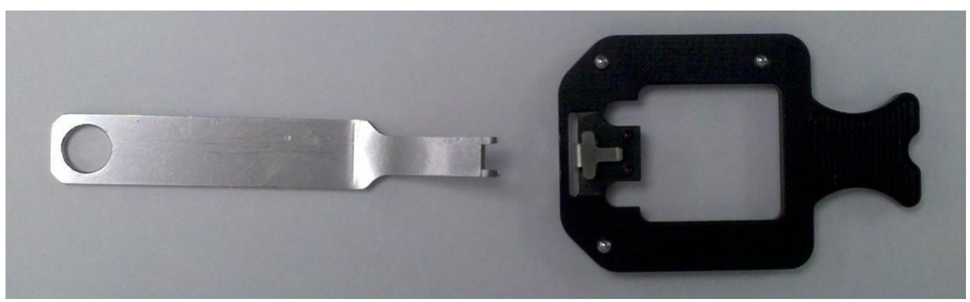
Probe Cartridges



Pre-Mounted Tips



Spring Tool



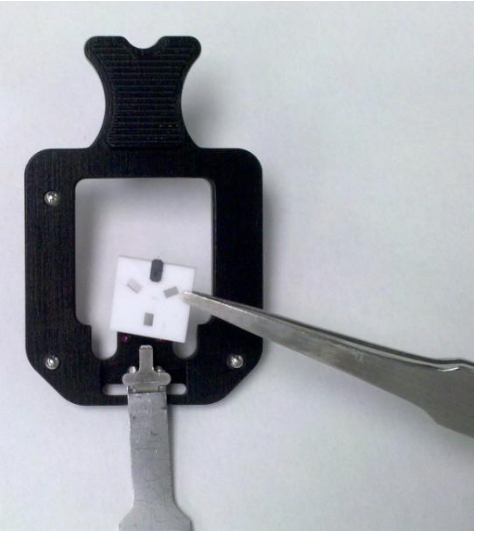
Loading a Pre-Mounted Tip

- Use the spring tool to place a ceramic chip carrier into the probe cartridge.

1. Insert spring tool under metal clip.

2. Gently apply pressure to open the clip.

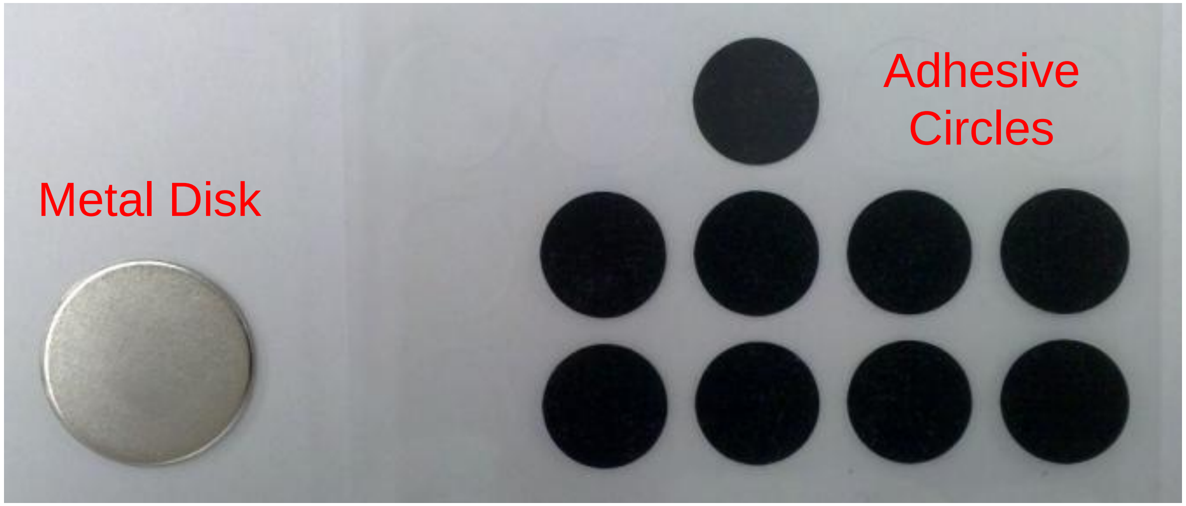
3. Insert pre-mounted tip.

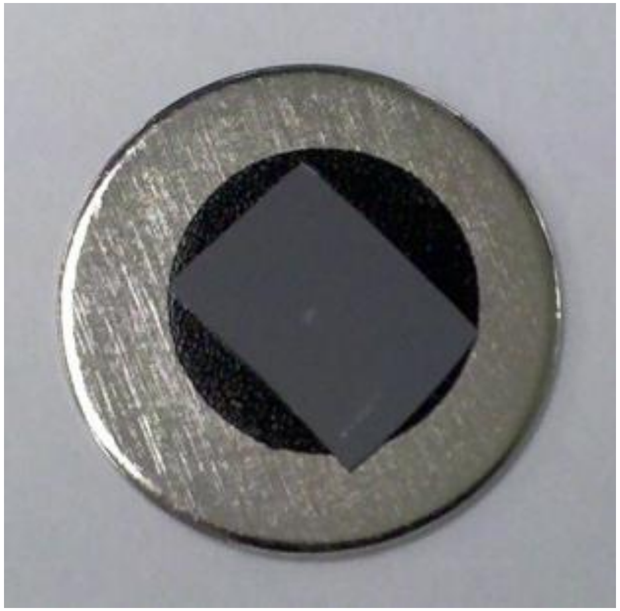


4. Carefully, use fingers to ensure proper positioning of the ceramic carrier chip.

Sample Preparation and Mounting

- Mount the sample onto a metal disk.
- Sample should be flat and free of dust and debris.
- Sample may need to be cleaved to fit into probe head.

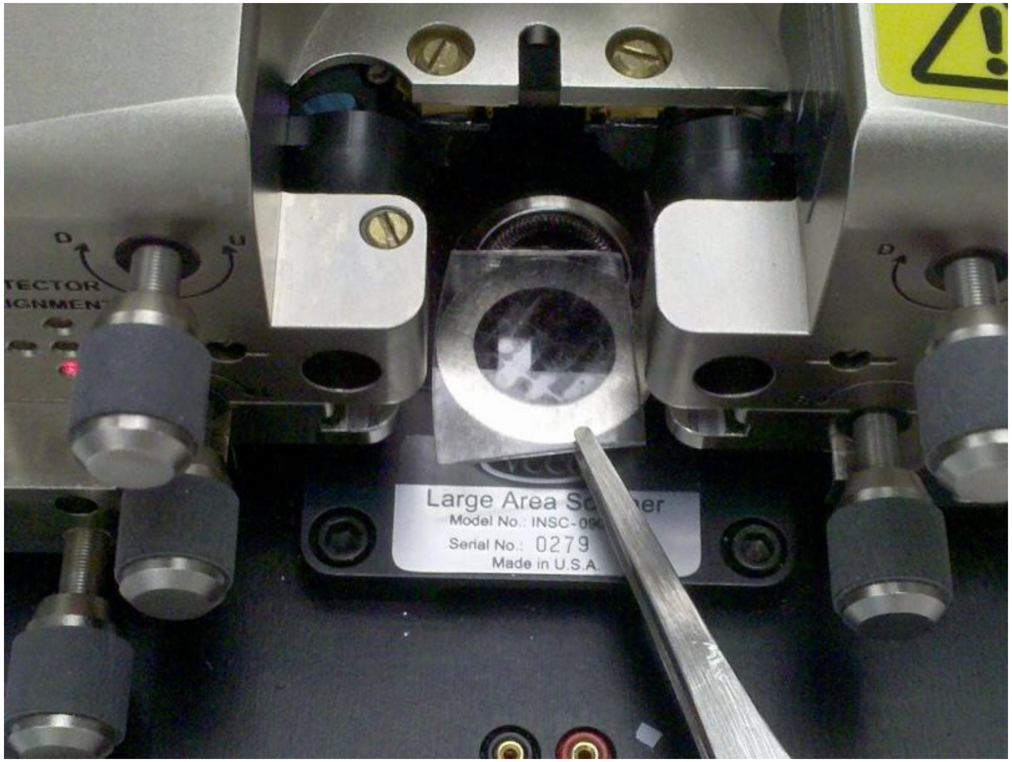




Properly Mounted
Sample

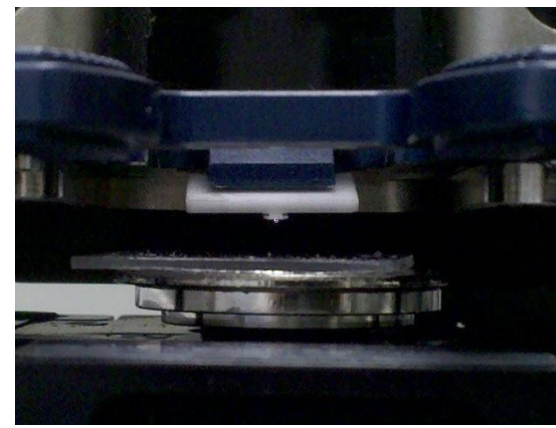
Loading the Sample

- Use forceps to gently slide the sample onto the sample holder.
- Magnet on sample holder attracts metal disk.
- Be careful not to “snap” metal disk onto the holder as this may damage the scanner.
- Check for adequate clearance between the sample and the probe head.



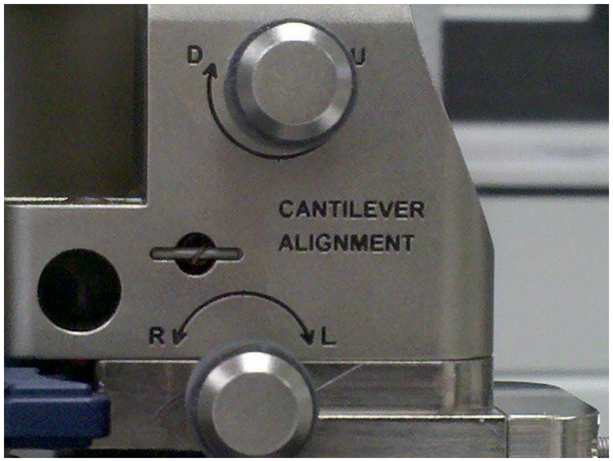
Preparing the Instrument

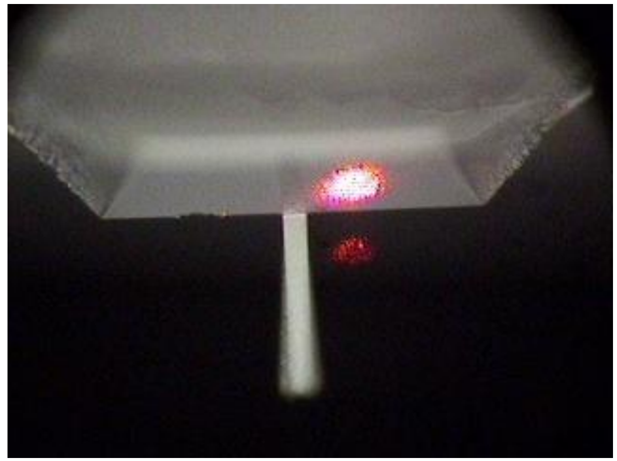
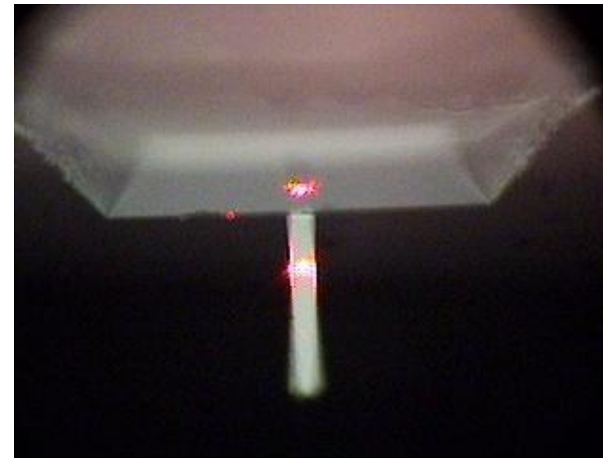
- Check probe head height to make sure the tip won't collide with the sample.
- Slide the probe cartridge into the probe head.
- Swing the optical microscope back into place over the sample.



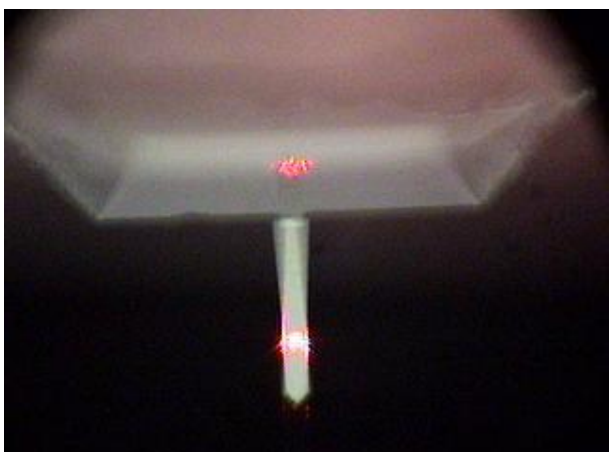
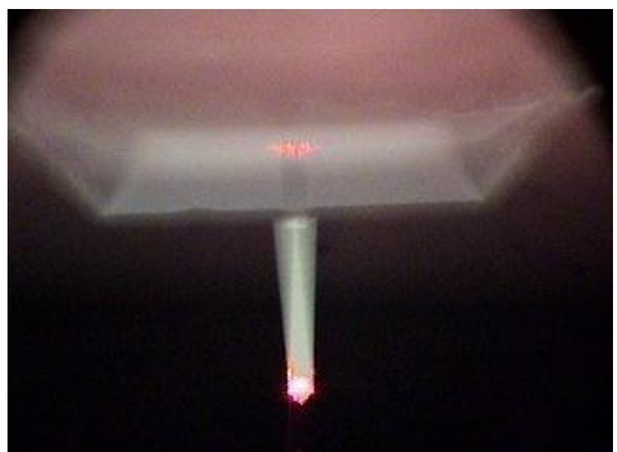
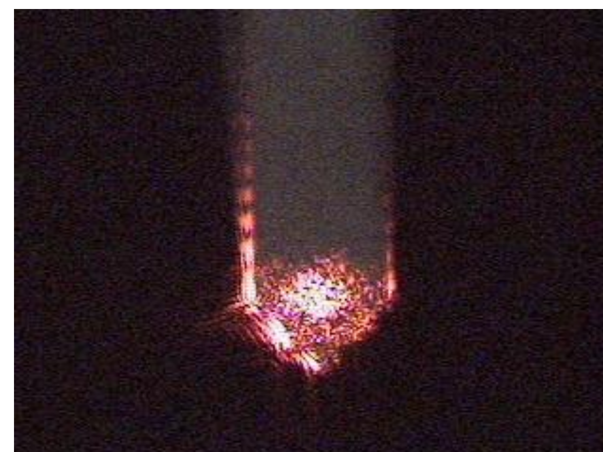
Laser Alignment on Cantilever

- Use “Cantilever Alignment” knobs to move the laser onto the end of the cantilever.



- Roughly position the laser spot, then walk it out to the correct position

- Zoom in and refocus as necessary.

Detector Alignment

- Click on the 4-quadrant detector icon.
- A new window will appear which shows the position of the laser on the detector.
- Adjust the detector alignment knobs to center the spot.

Before Fine Adjustment

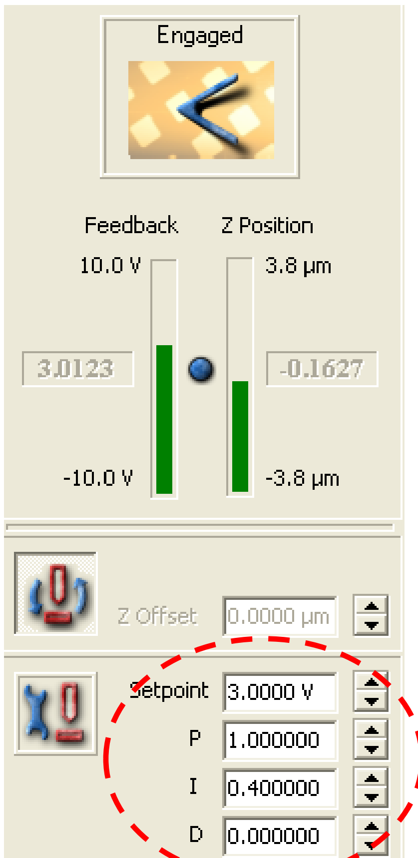
After Fine Adjustment

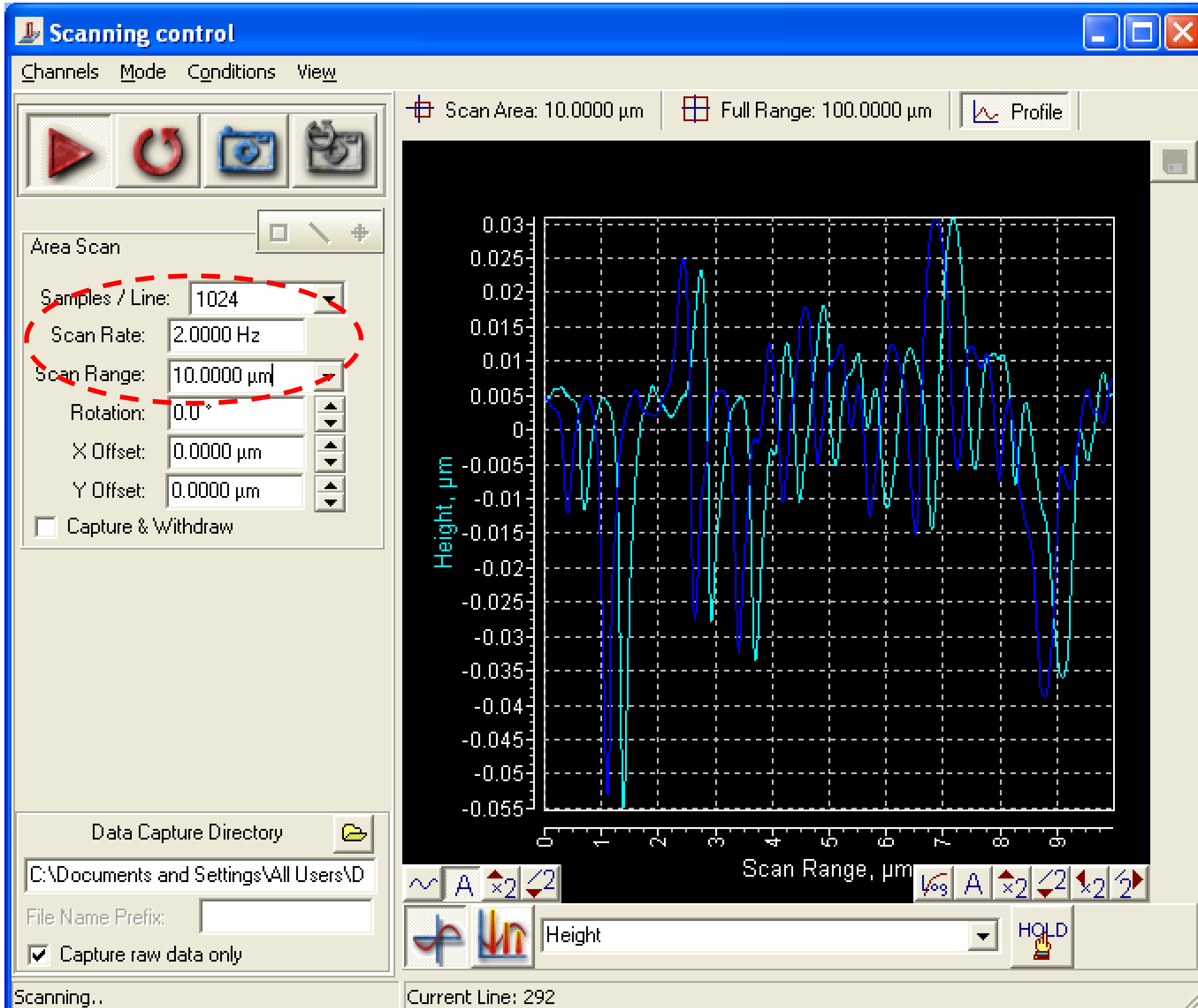
<u>A-B Signals</u>
Comparison of signal coming from halves of detector. Top – Bottom & Left – Right. Closer to zero is more centered.

<u>Total signal</u>
Should be >2 V

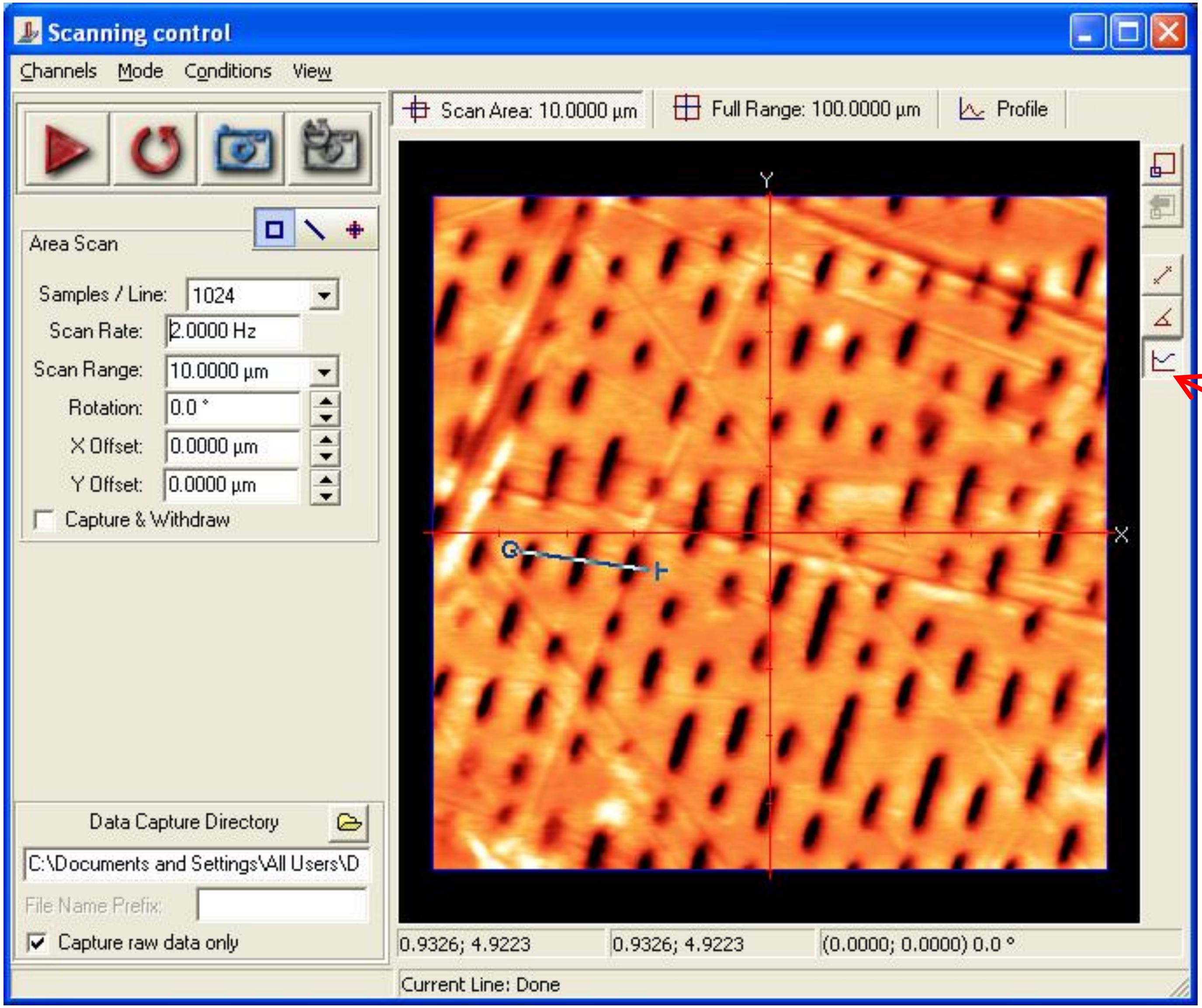
Acquiring an Image

- Adjust the feedback controls and scanning parameters to optimize the scanning process.





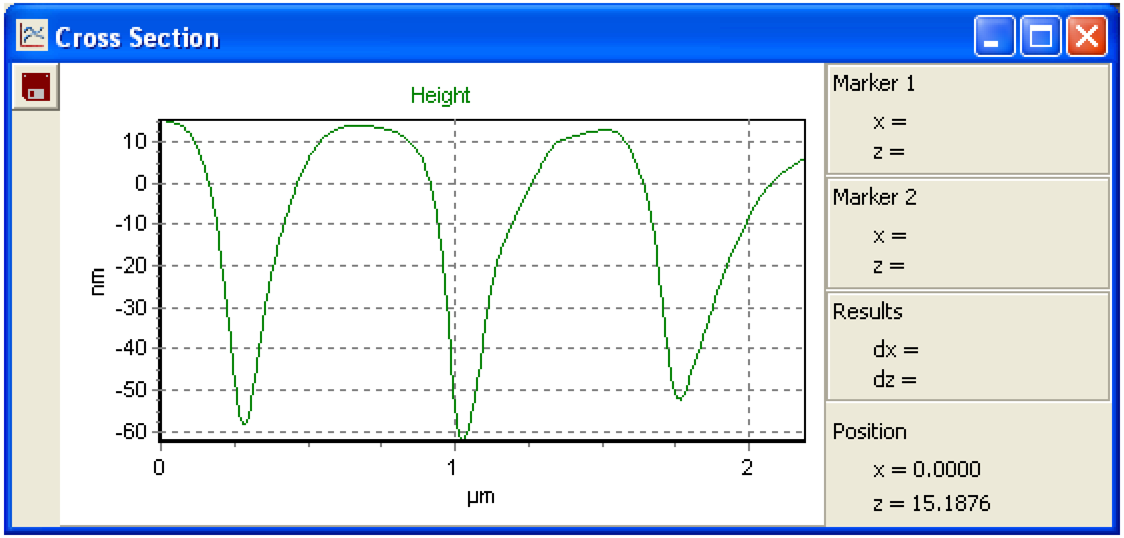
Analyzing Data: Measurements

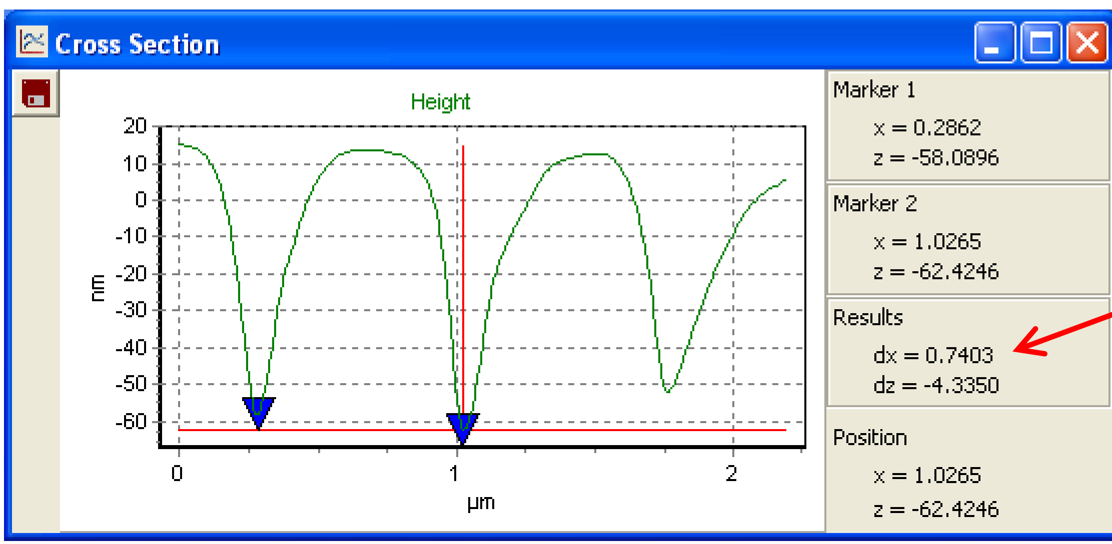


Cross-Section Measurement Tool

Data is gathered line-by-line and can be reconstructed to form a topographical map of the sample surface.

Example: Distance Measurement





The cross-section measurement tool can be used to make measurements on the data set.

Clicking with the mouse drops markers (triangles in the figure).

The distance (x and y between markers is displayed.

740 nm in x-direction

Summary

- Scanning probe techniques enable nano-scale characterization
- STM measures tunneling current between a metal tip and a conductive sample
- AFM is suitable for conducting and insulating samples
- A variety of AFM scanning techniques are available (contact, tapping, etc.)
- Each technique offers its own benefits and needs to be chosen based upon the properties of the sample

References

Ron Reifenberger; Arvind Raman (2009), "ME 597/PHYS 570: Fundamentals of Atomic Force Microscopy (Fall 2009)"

http://nanohub.org/resources/7320.