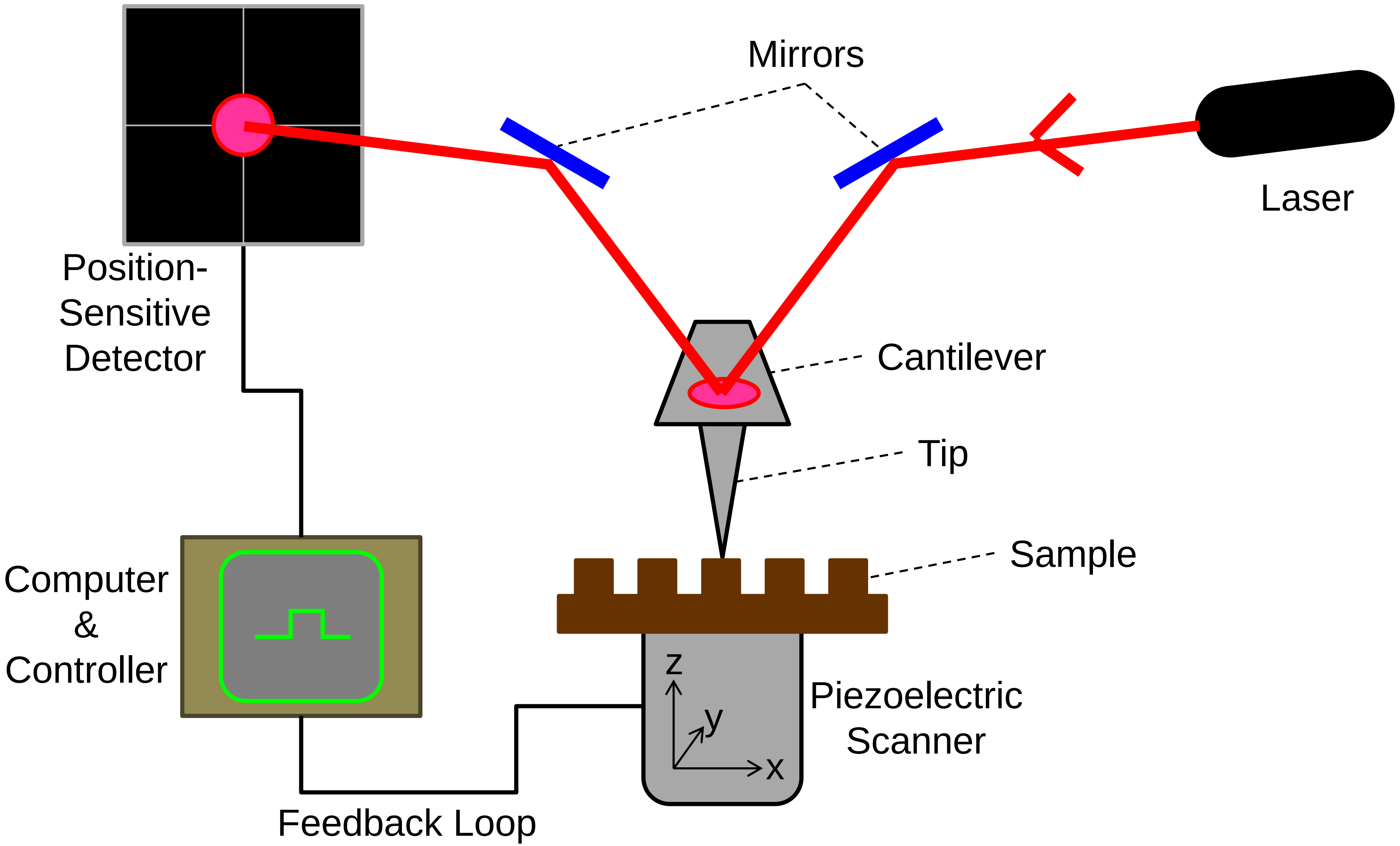
Building College-University Partnerships for Nanotechnology Workforce Development

Advanced Scanning Probe Microscopy II

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

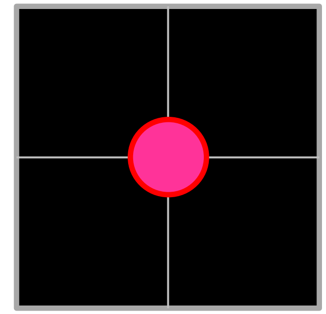
- An AFM can be operated in many different modes
- 2 modes will be overviewed:
  - contact
  - tapping mode
- Each mode has advantages and trade-offs.

Contact Mode

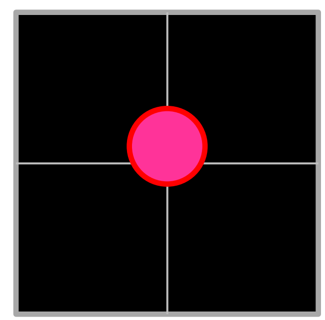
- In contact mode, the sample topography is measured by scanning the tip, which contacts the surface, across the sample.
- This is a popular mode of operation for durable samples.
- Since the tip is in contact with the surface, damage to tip and sample may occur as scanning proceeds.
- Frictional forces 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)

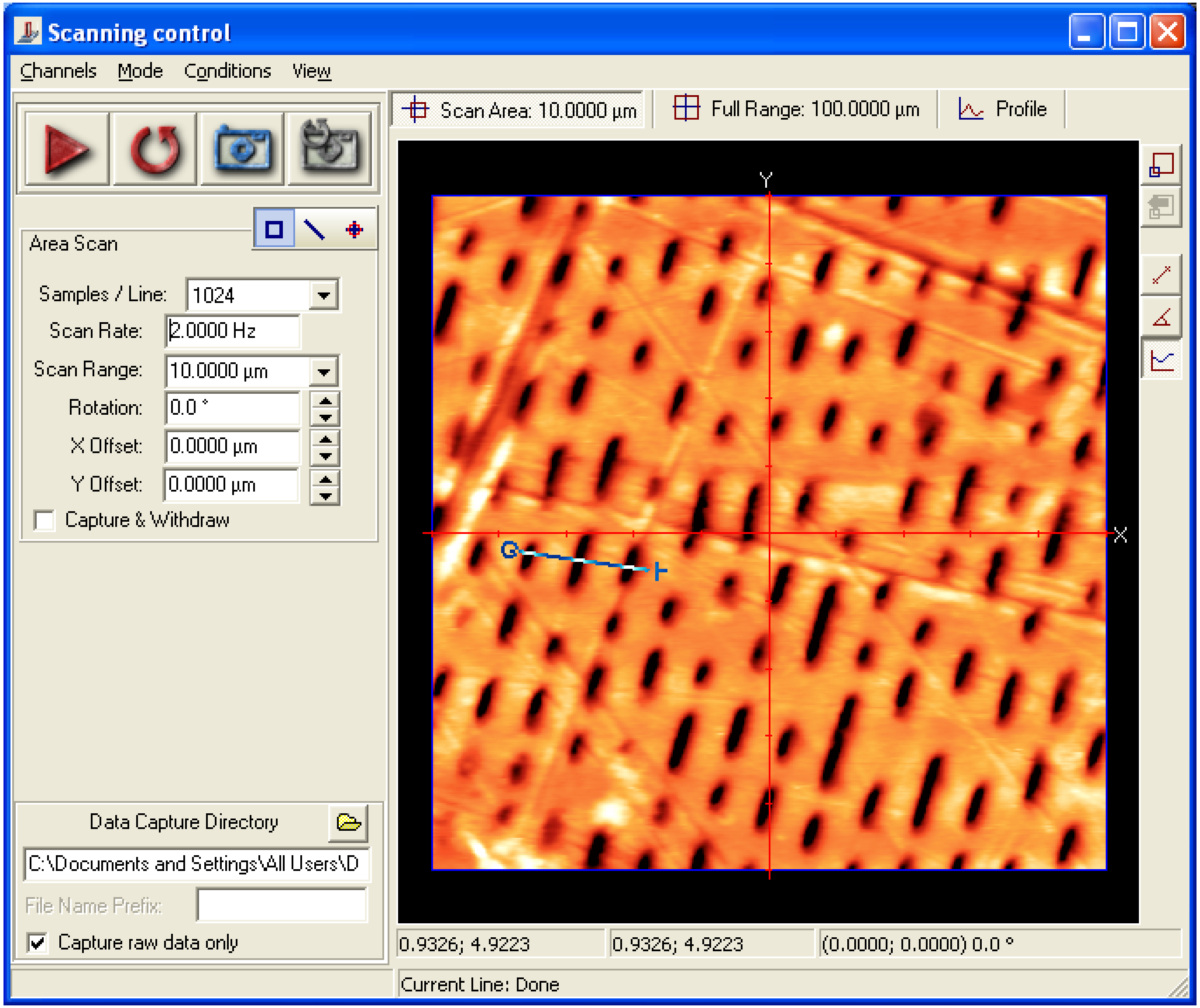


Top – Bottom = Small V



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

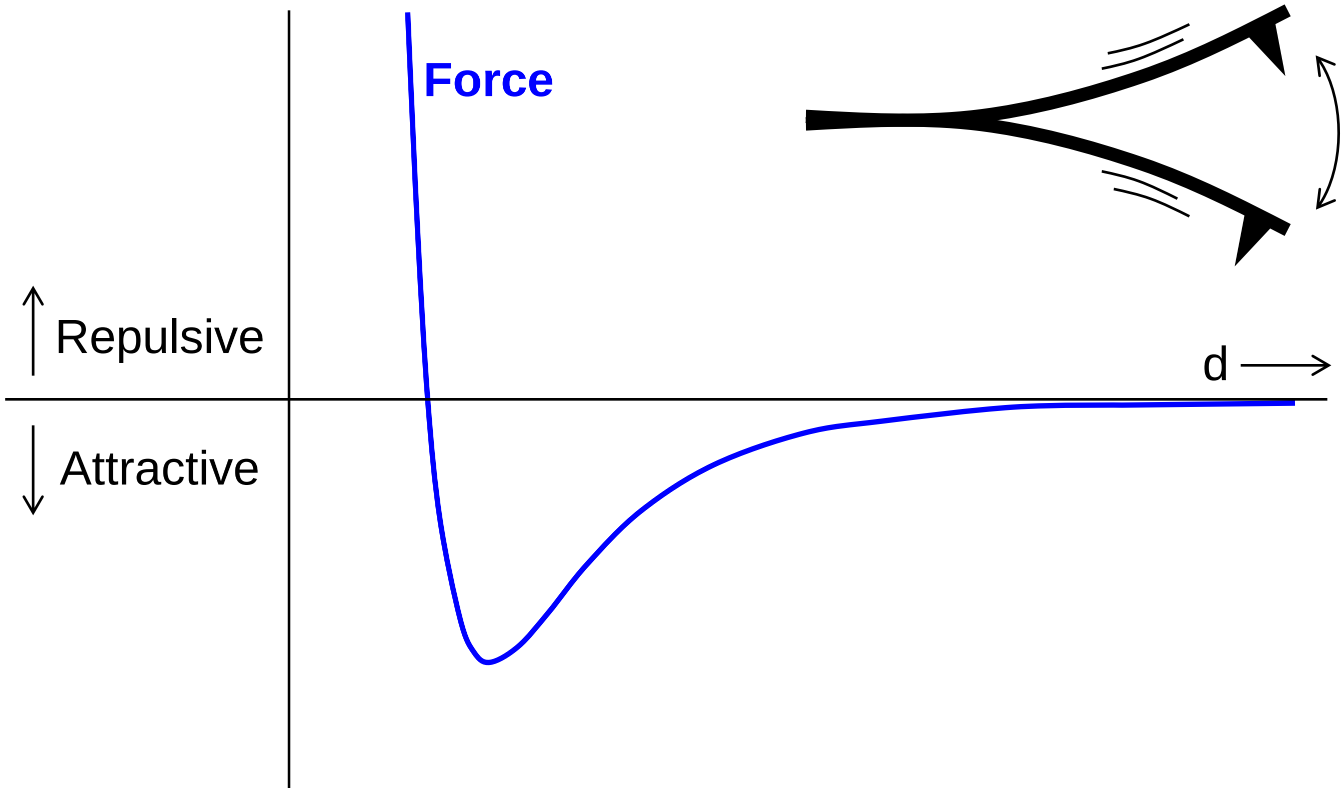
- Also called intermittent contact mode.
- In this mode the cantilever is excited close to its resonant frequency by an external piezoelectric ceramic attachment.
- Resonant frequencies are typically from 15 kHz to 500 kHz. (Compare to rate of scanning.)
- The amplitude of cantilever vibration is usually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

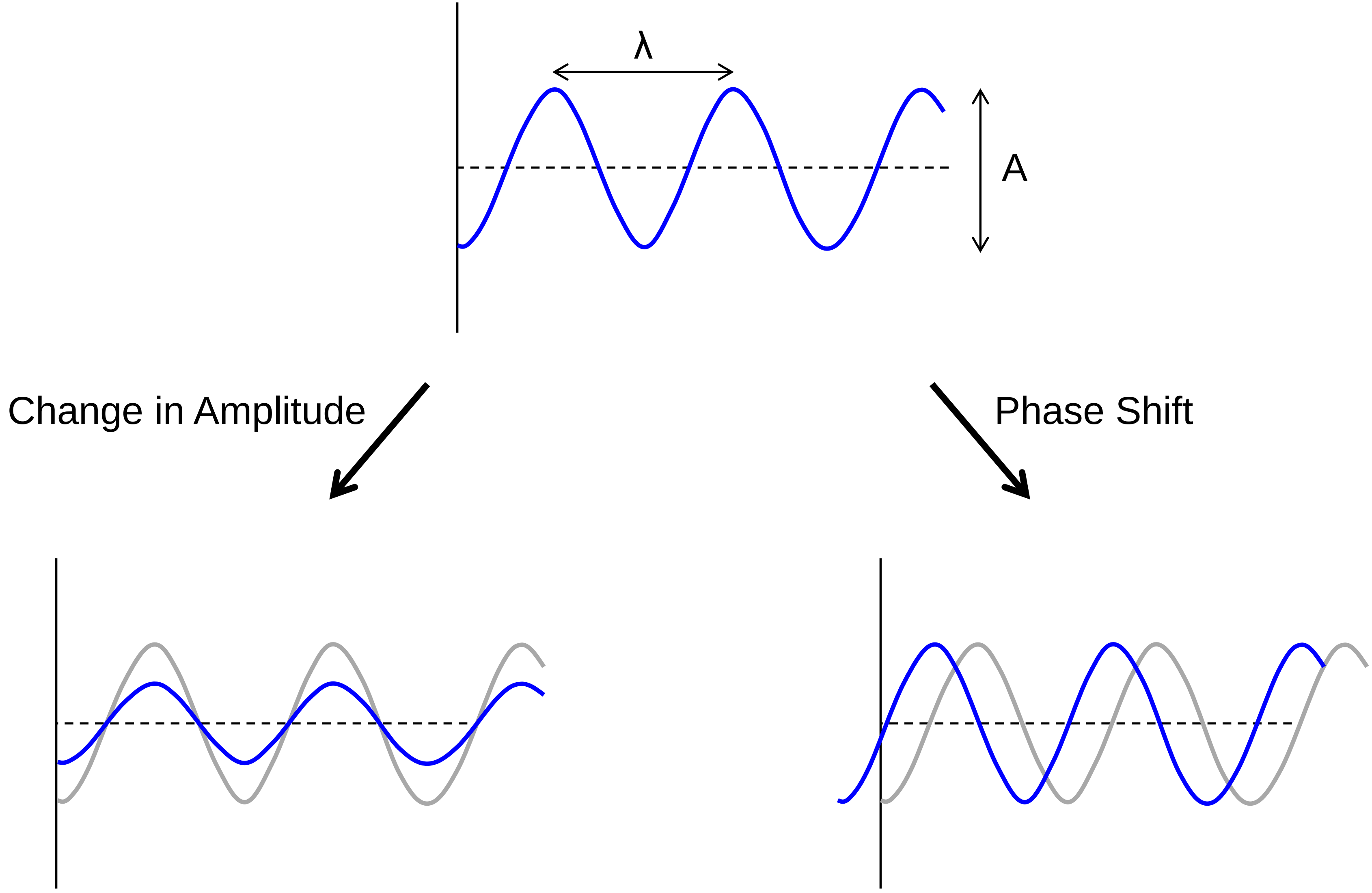
- At the bottom of each vibration, the tip comes close to the surface and may contact it briefly.
- Therefore, there is an abrupt change from weak attraction to strong repulsion.



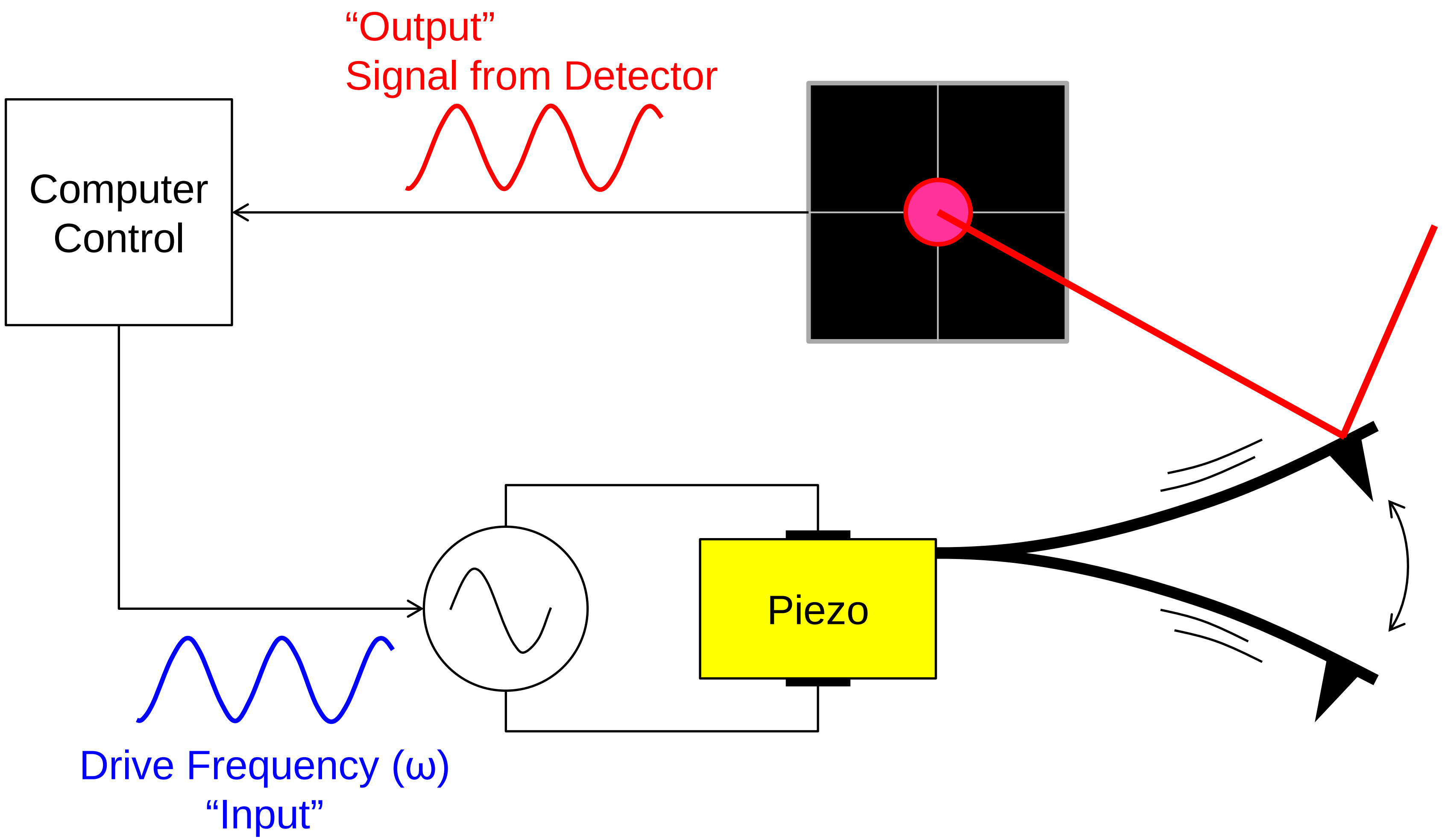
Tapping Mode

- 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.

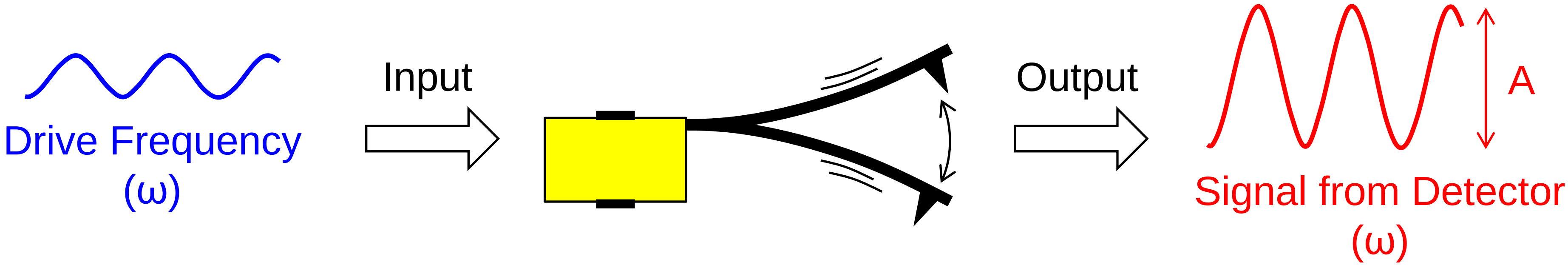
Amplitude and Phase Shift

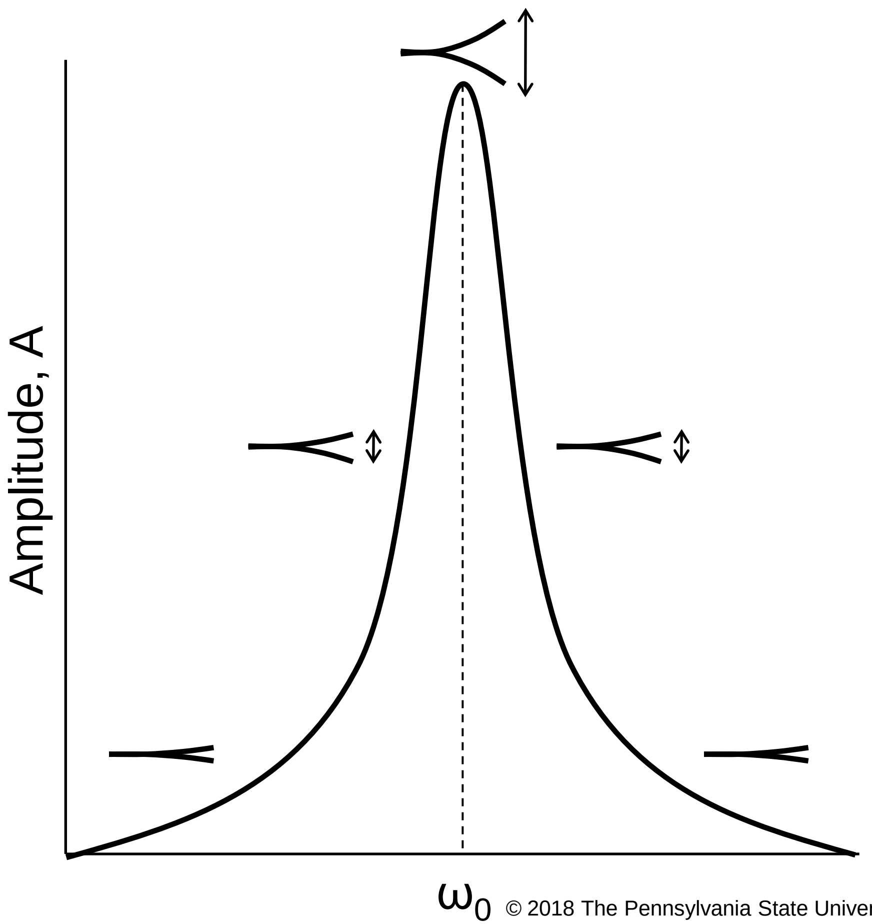


Tapping Mode



Tuning the Cantilever

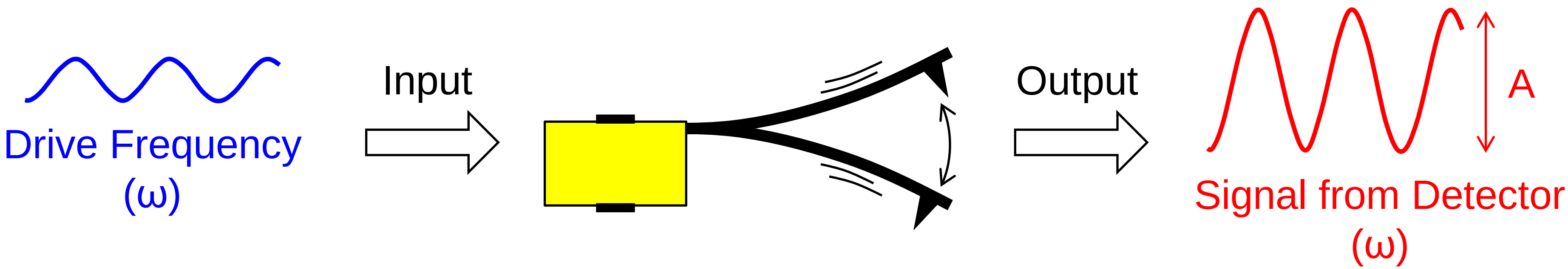


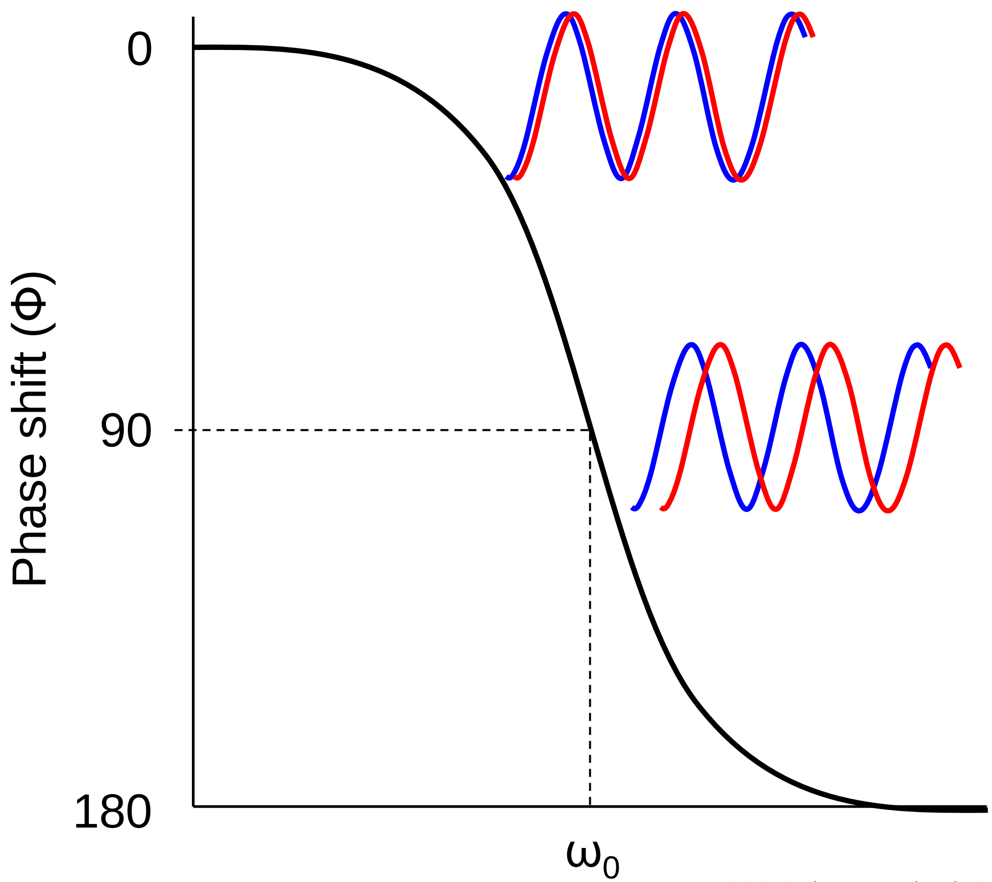


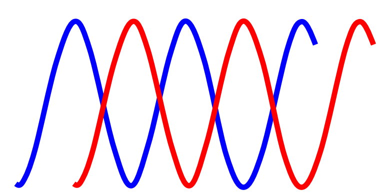
Sweep drive frequency and measure amplitude of cantilever vibration.

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

Tuning the Cantilever





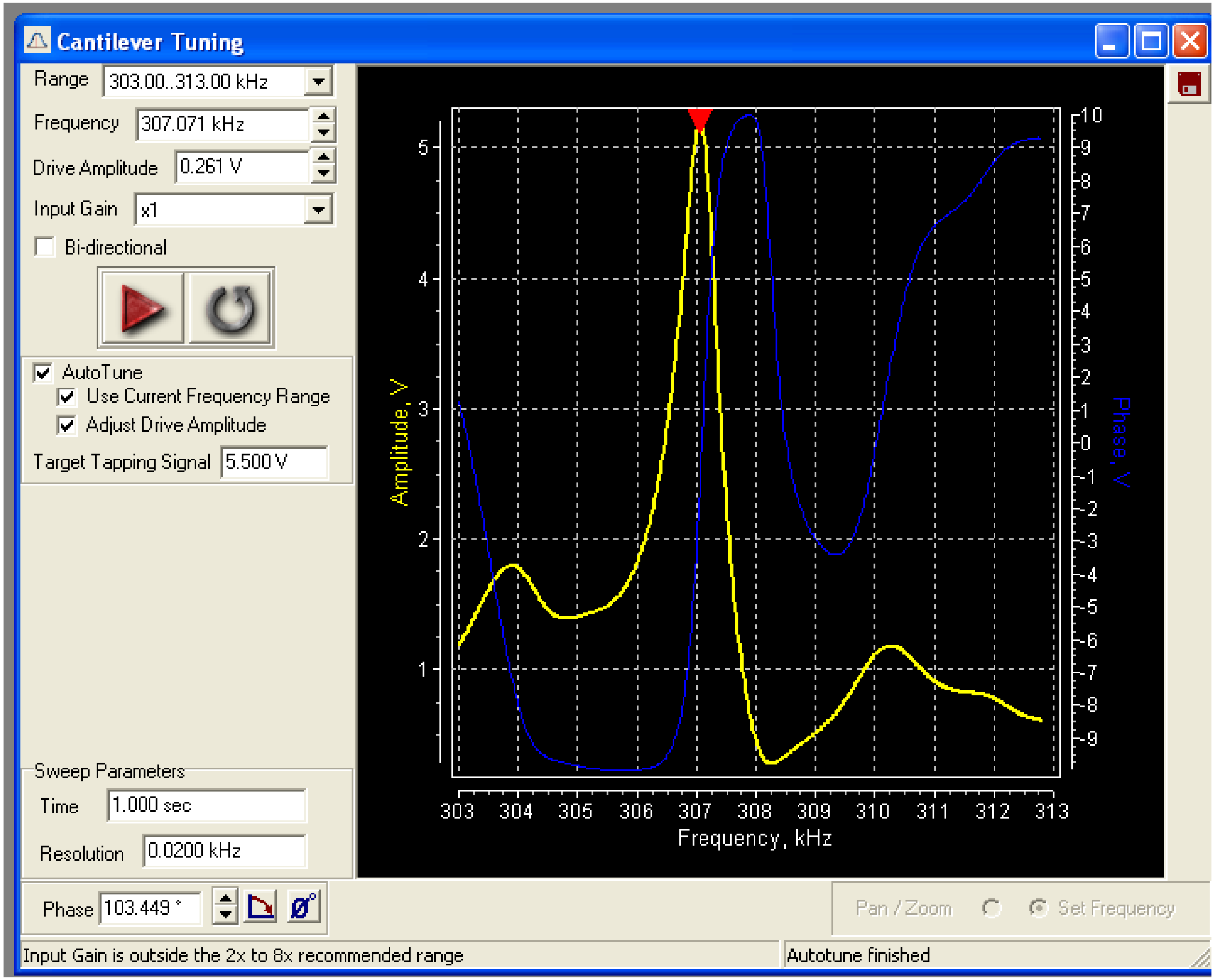


Phase shift (Φ) is an additional signal that contains information about the sample surface.

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.

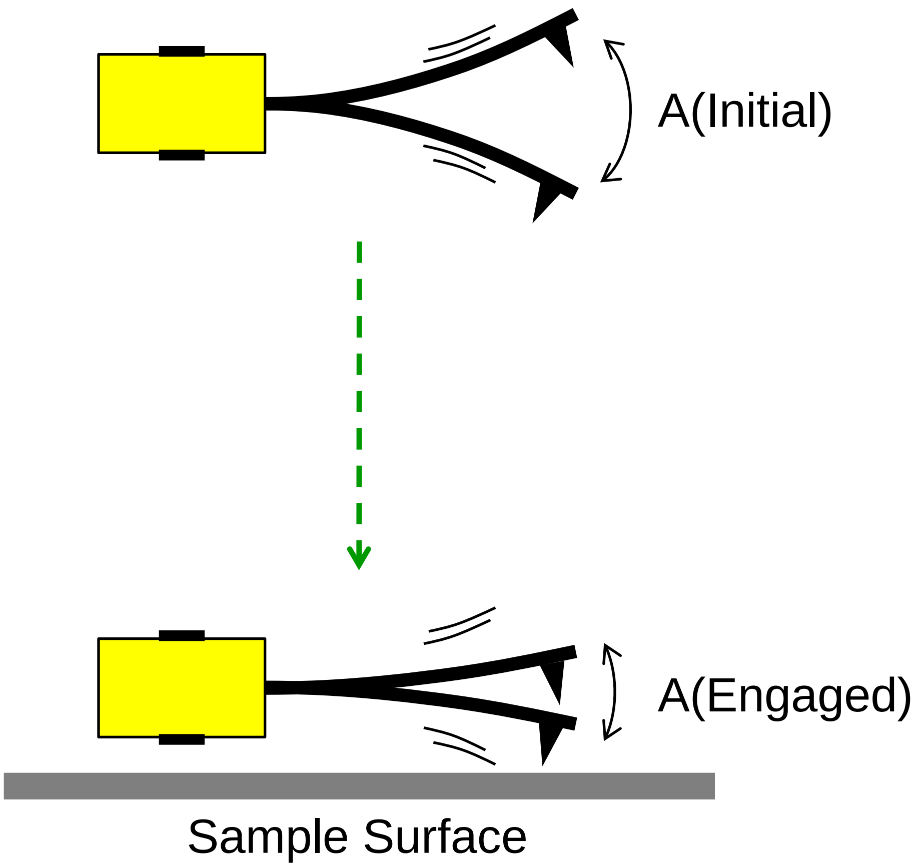
Interactions with the surface dampen the oscillation of the cantilever.

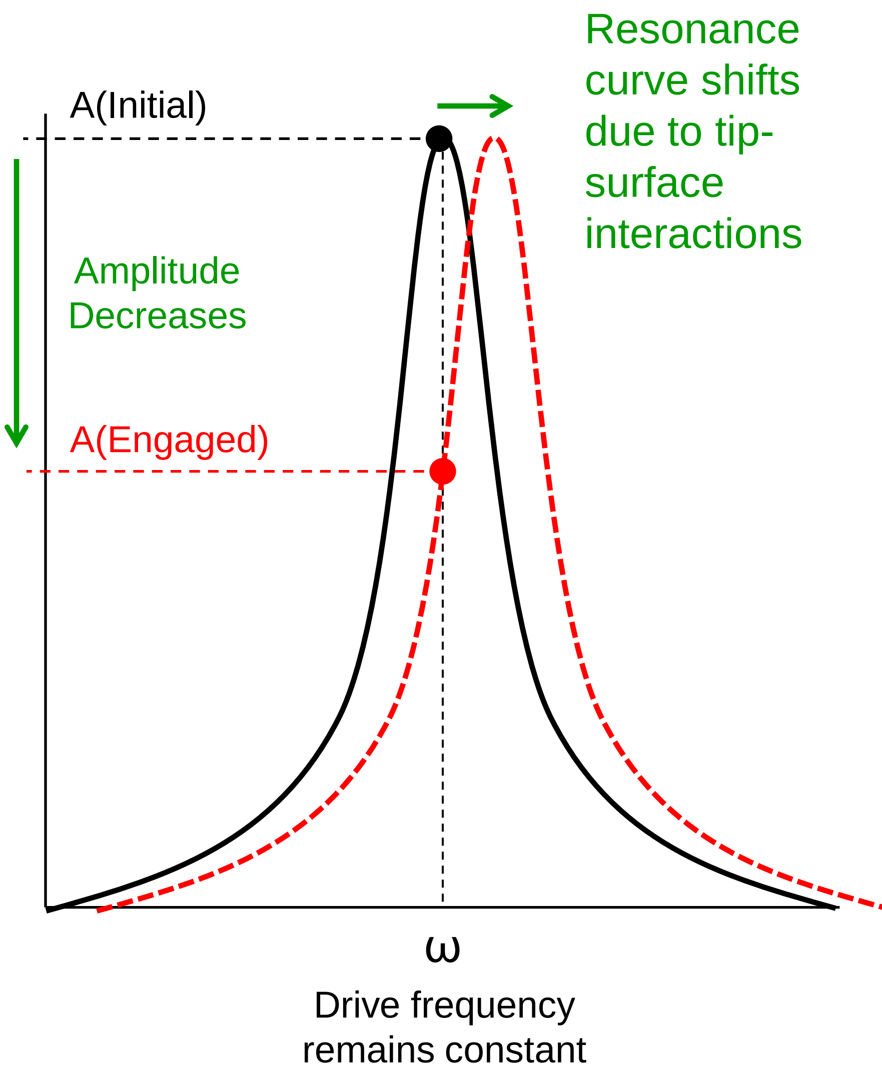
Tip-sample forces change (ω_0) such that the drive frequency $\omega \neq \omega_0$ when the tip approaches the surface.

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

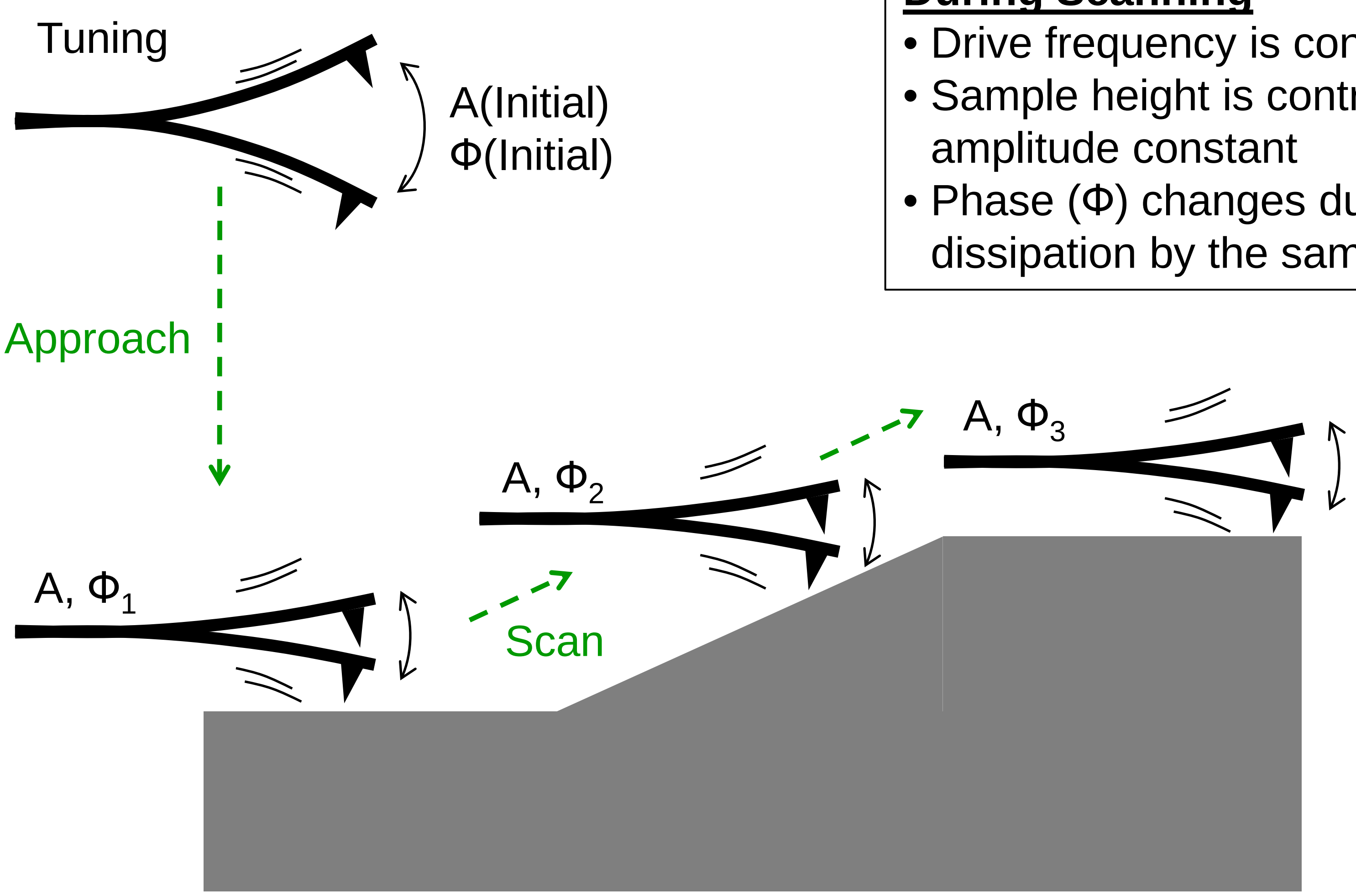
The system detects the lowered amplitude as a decreased voltage produced by the detector (compared to the higher voltage produced during tuning).

Approaching the Sample





Scanning the Surface



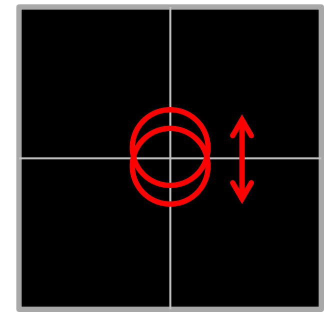
During Scanning

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

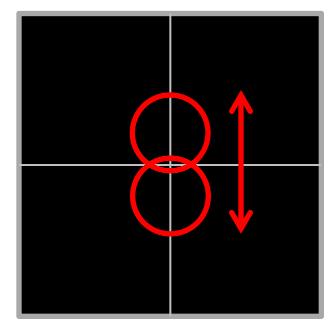
Note: A < A(Initial) and A is chosen by adjusting the Voltage Set Point.

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

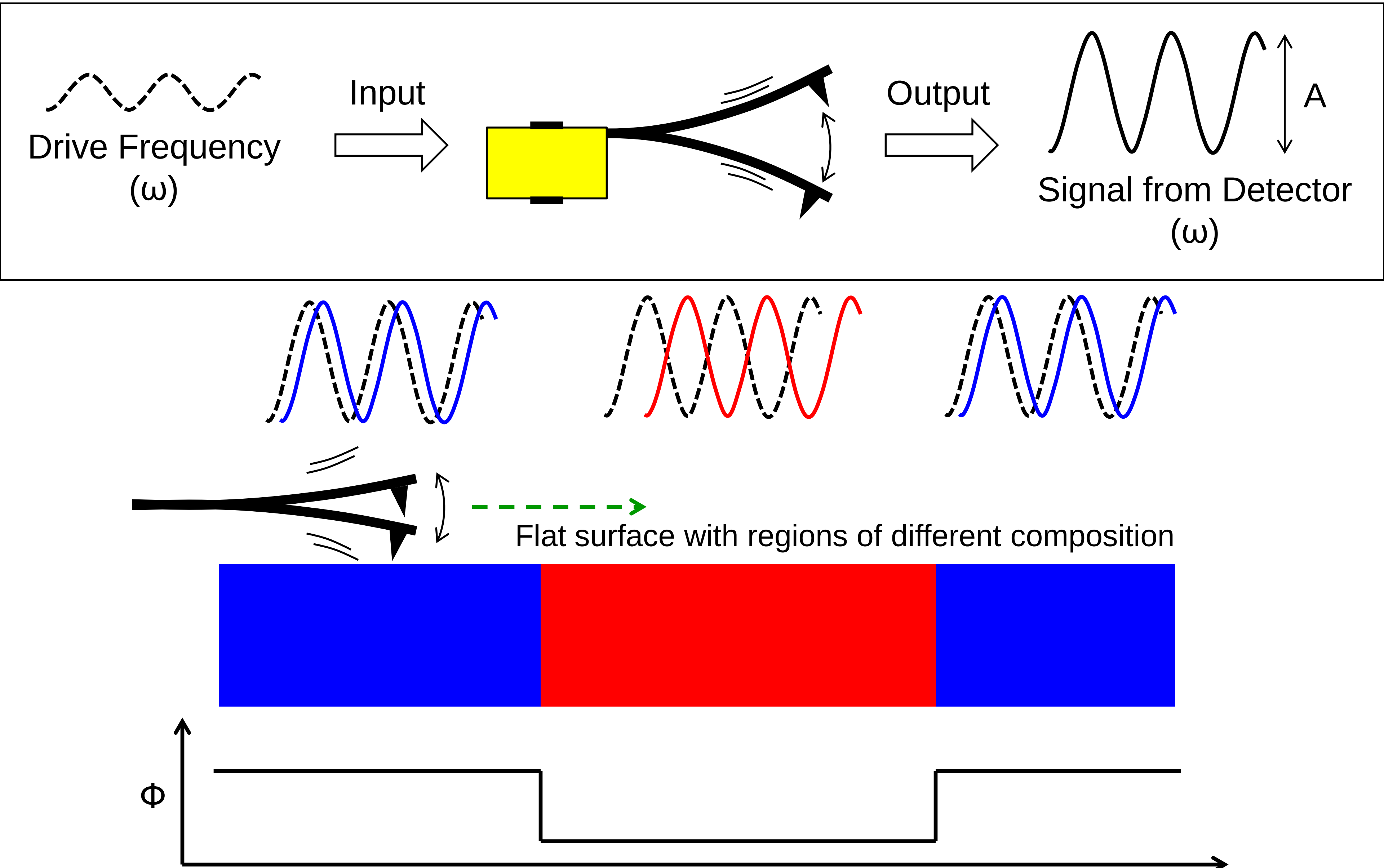


Top – Bottom = Large V
Higher Amplitude
Softer Tapping

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

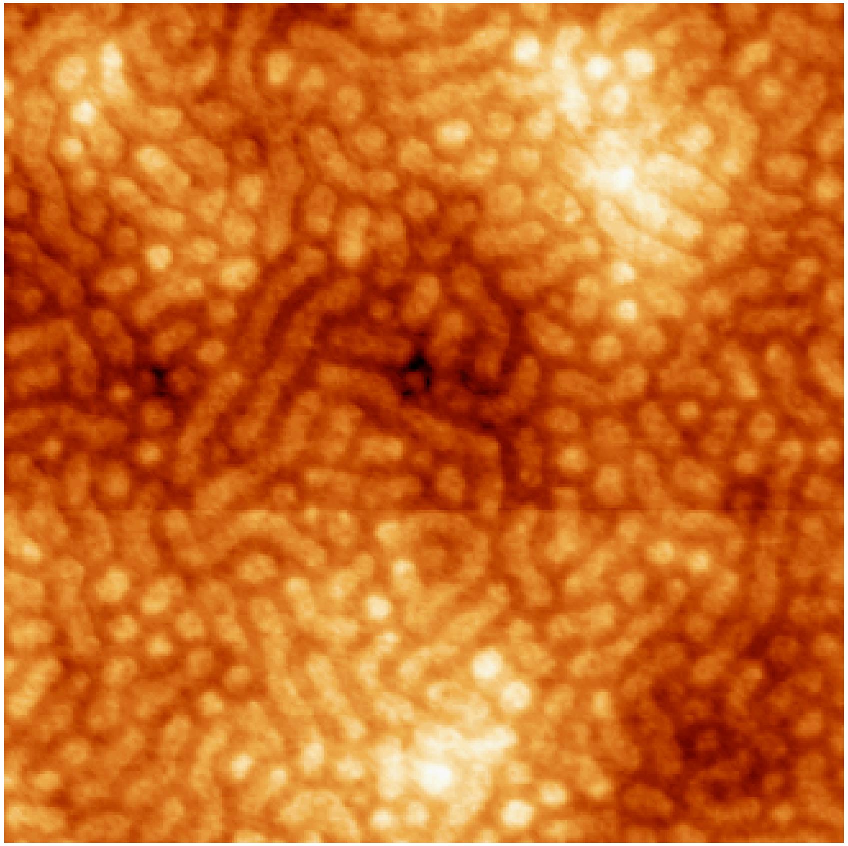
Phase Contrast



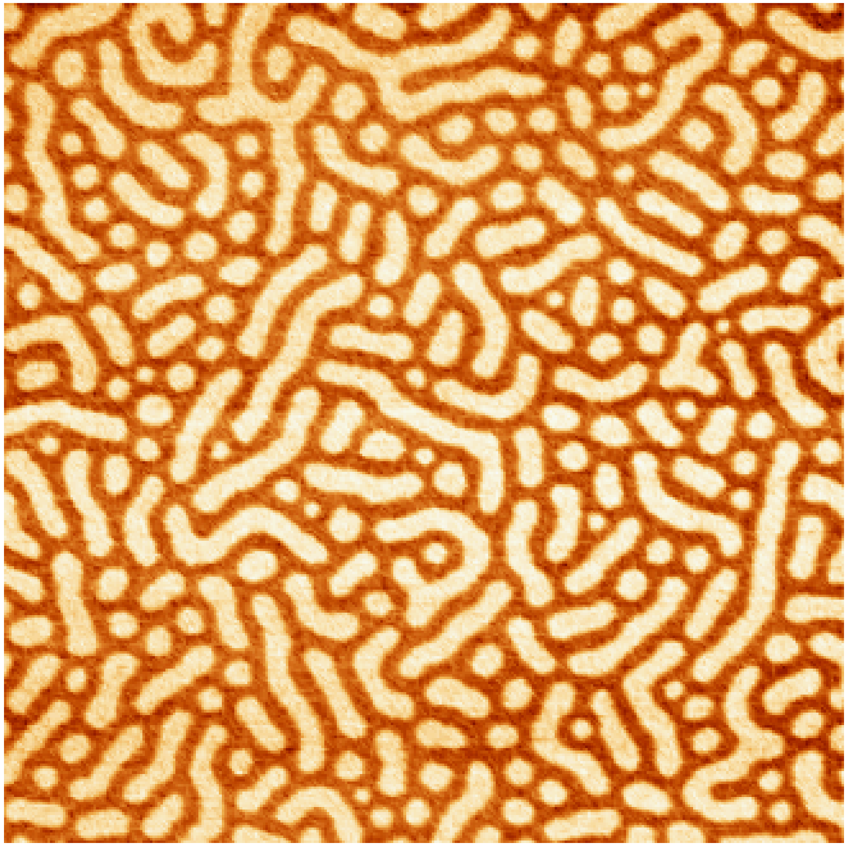
Example Images: Tapping Mode

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



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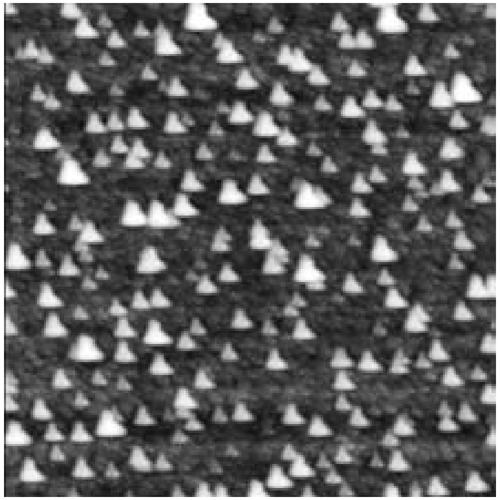
Common Problems

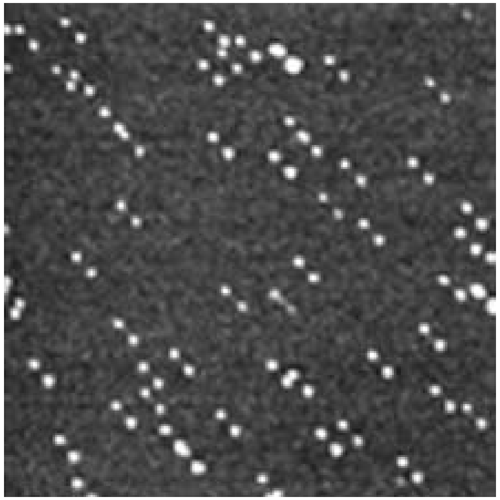
- If sample is not clean, dust particles will interfere with scanning.
- Improperly aligned laser and/or detector gives low signal (want > 2.00 V on detector).
- Image artifacts due to damaged or dirty tip
- Feedback Loop not tuned properly. This causes overshoot and “ringing.”

Image Artifacts

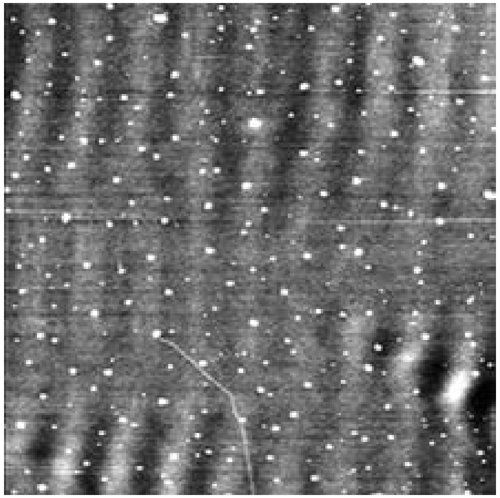
Dull or Dirty Tip



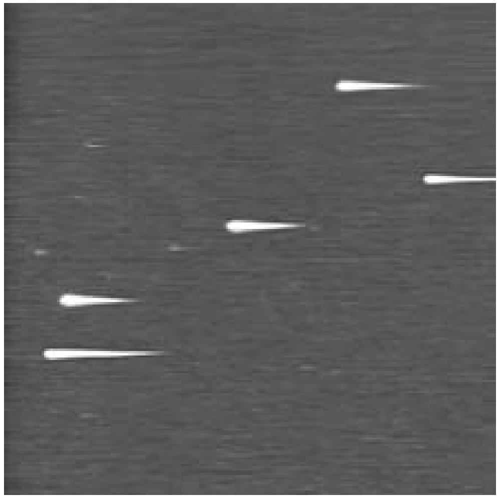
Multiple Tips



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

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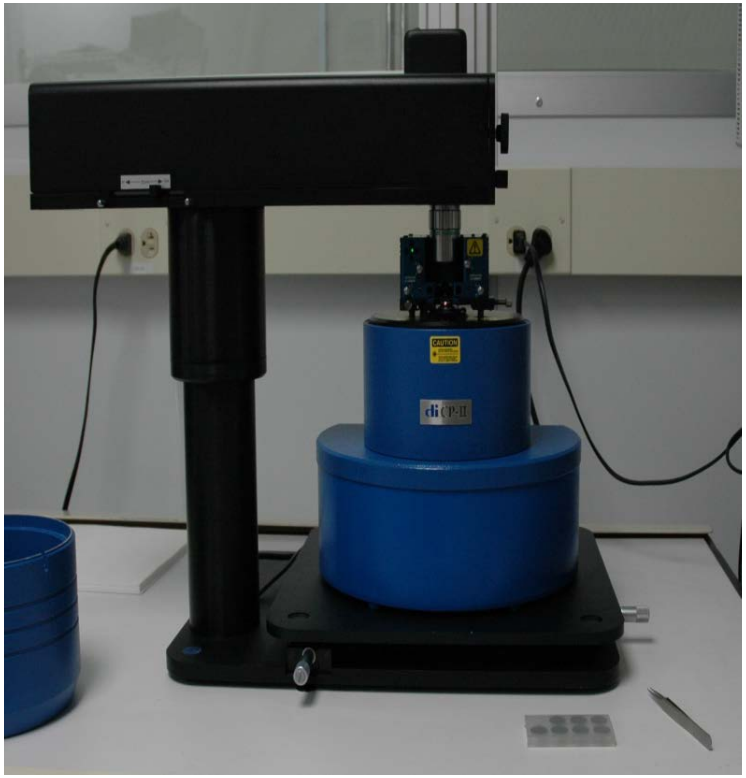
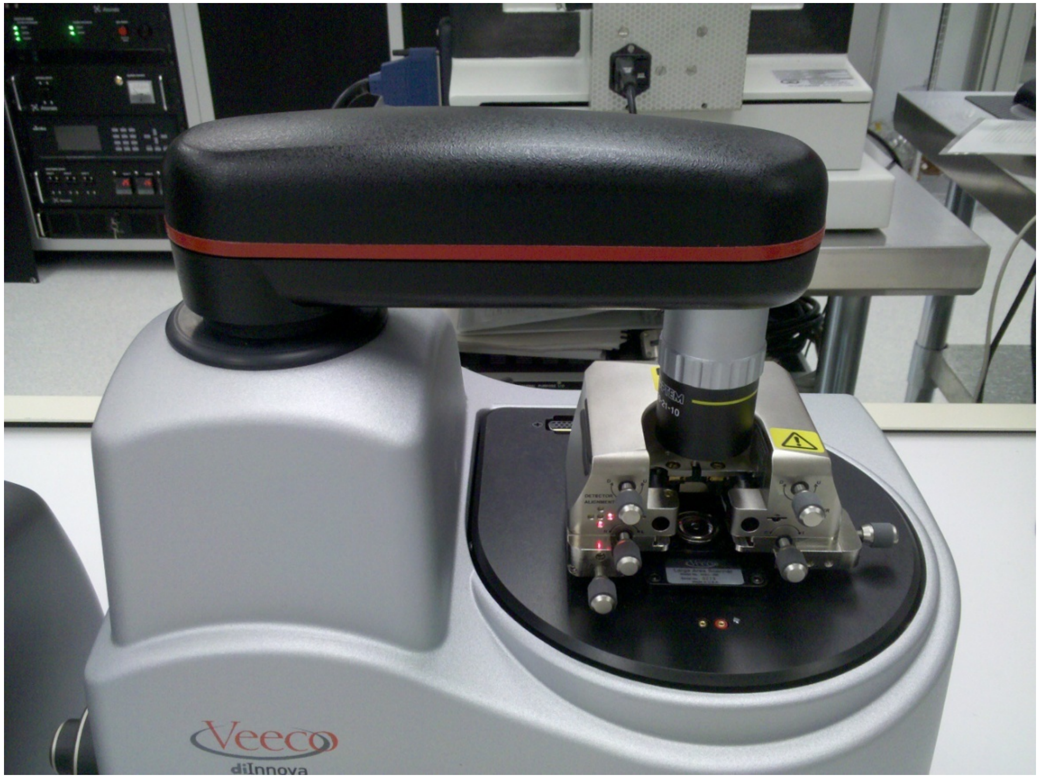
SPMs in The Teaching Cleanroom

Veeco CP-II



Veeco Innova



Penn Sate Center for Nanotechnology Education and Utilization

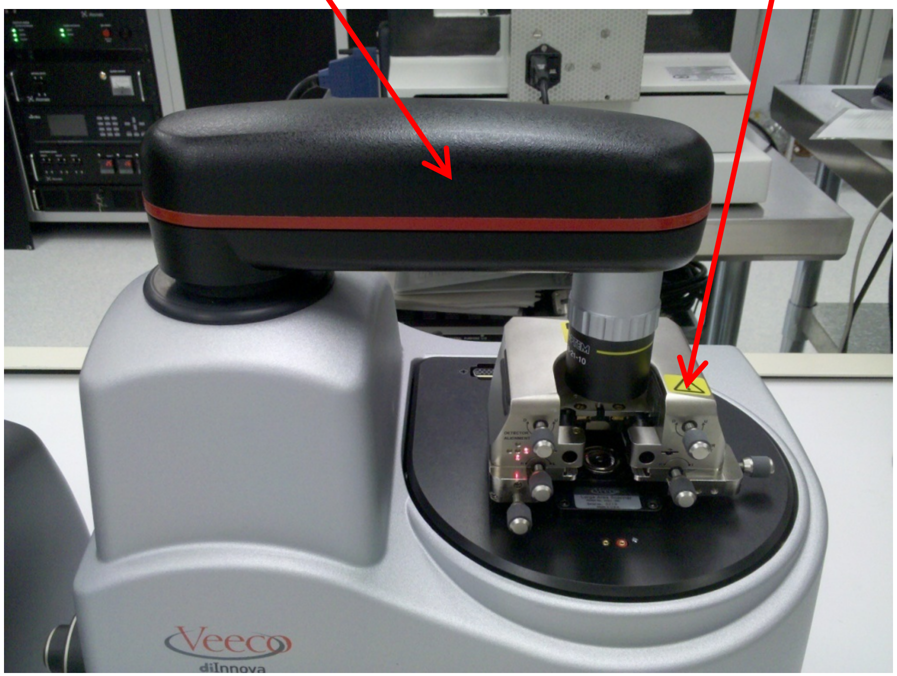
Example Specifications Veeco Innova SPM

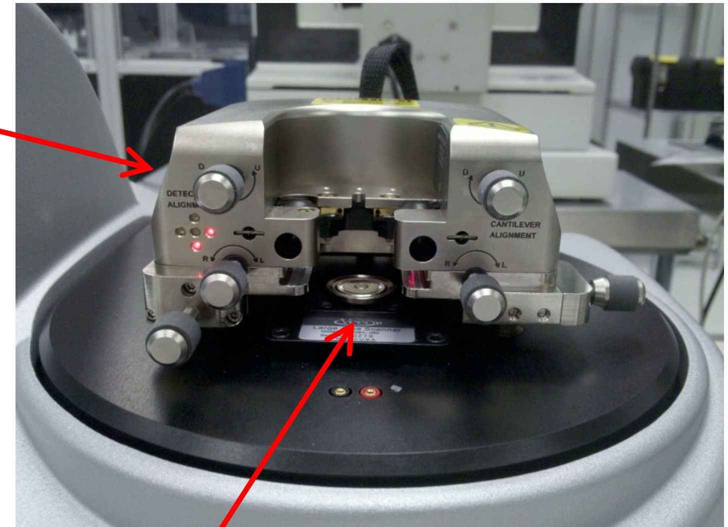
- **Closed-Loop Scanner:** X,Y > 90 µm, Z > 7.5 µm
- **Open-Loop Scanner:** X,Y > 5 µm, Z > 1.5 µm
- **Sample size:** 45 mm x 45 mm x 18 mm (X, Y, Z)
- **Motorized Z Axis Stage:** Z Travel: 18 mm
- **Optics:**
  - Camera: on-axis color CCD with motorized zoom
  - Field of view: 1.25 mm – 0.25 mm (motorized 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

Probe Head

Optical Microscope

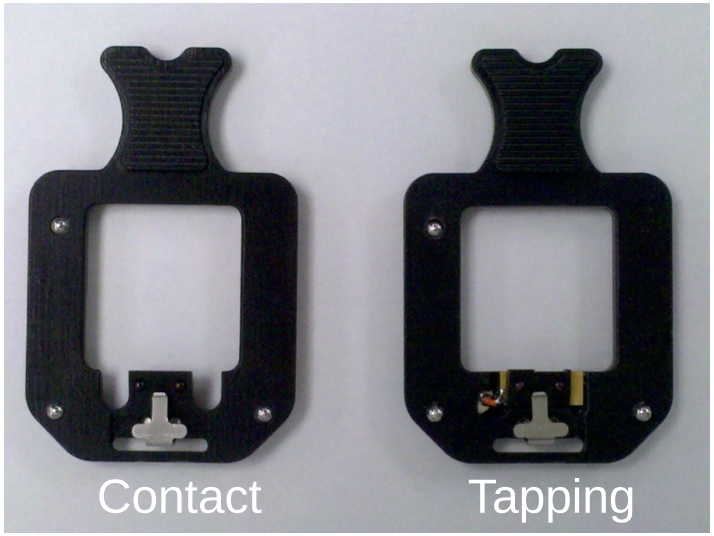




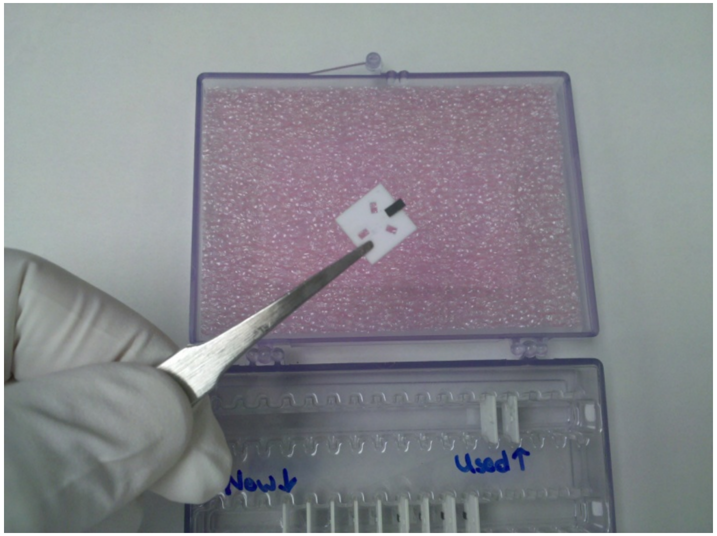
Sample Holder & Scanner

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.

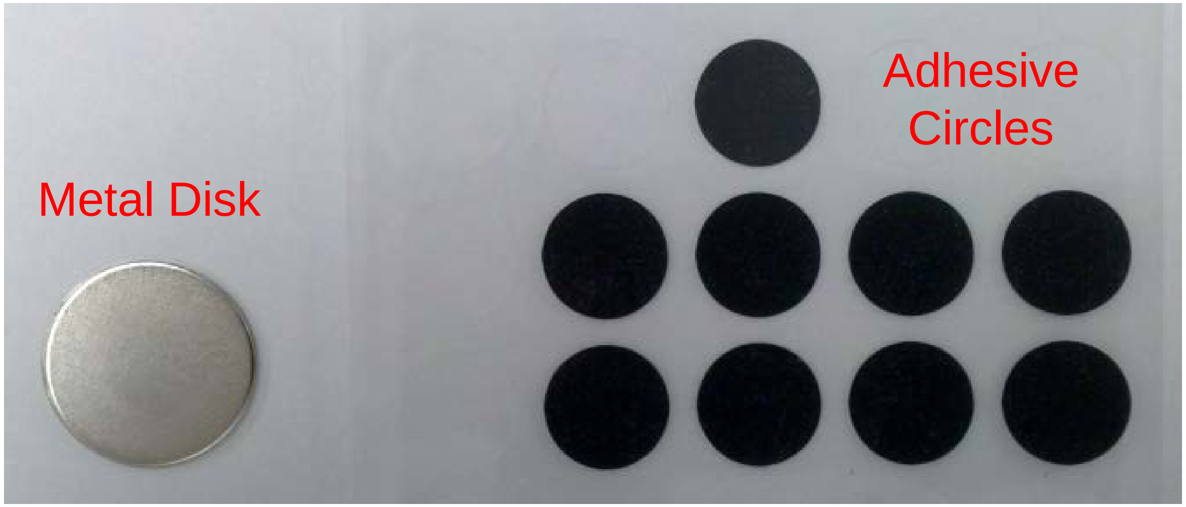
2. Gently apply pressure to open the clip.

3. Insert pre-mounted tip.

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

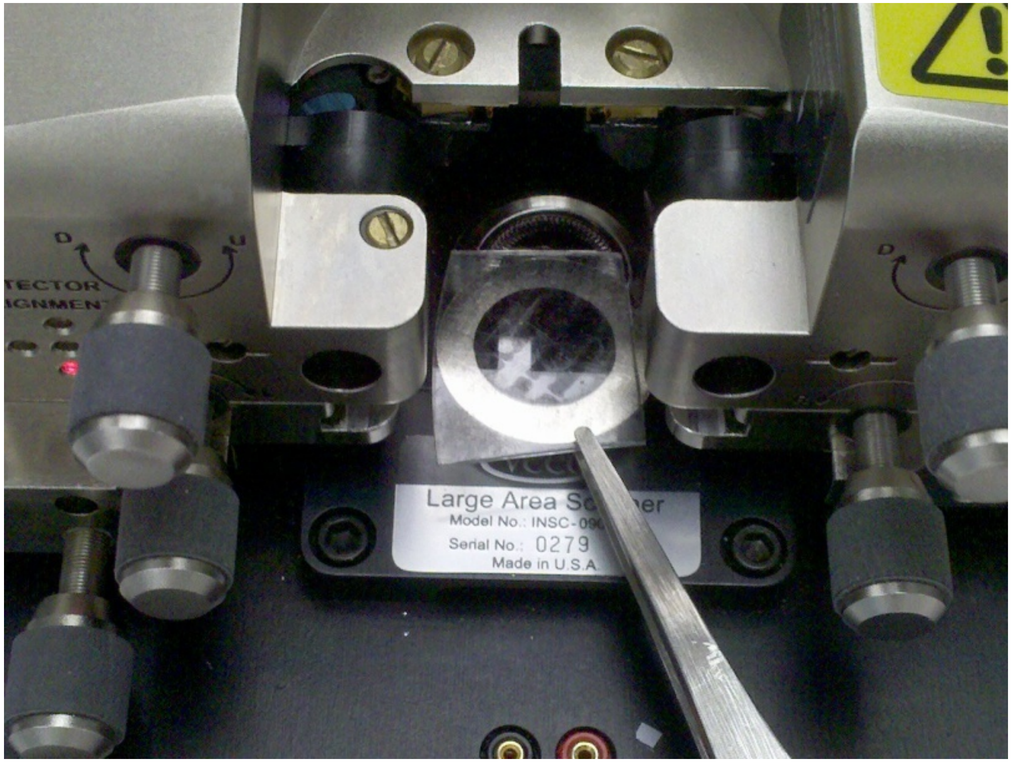
Sample Preparation and Mounting

- 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

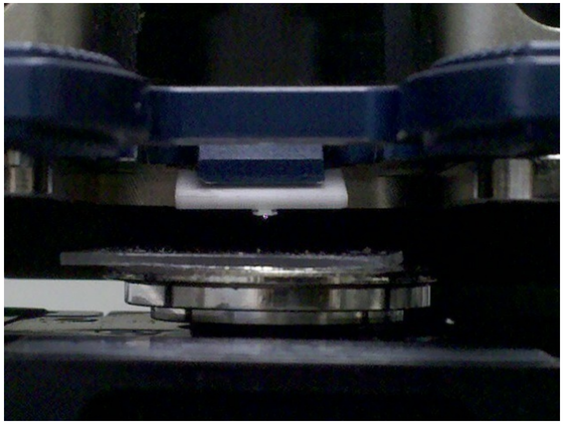
Loading the Sample



- 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.

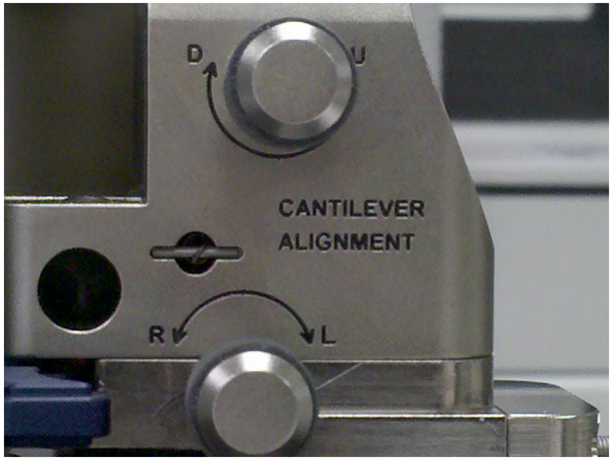
Preparing the Instrument

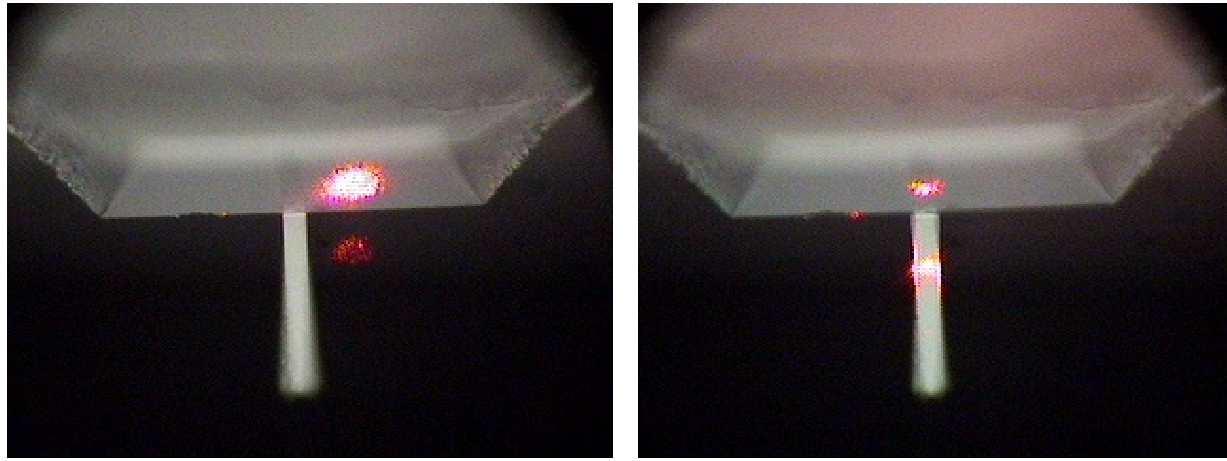
- Check probe head height to make sure the tip won't collide with the sample.
- Slide the probe cartridge into the probe head.
- Swing the optical microscope back into place over the sample.



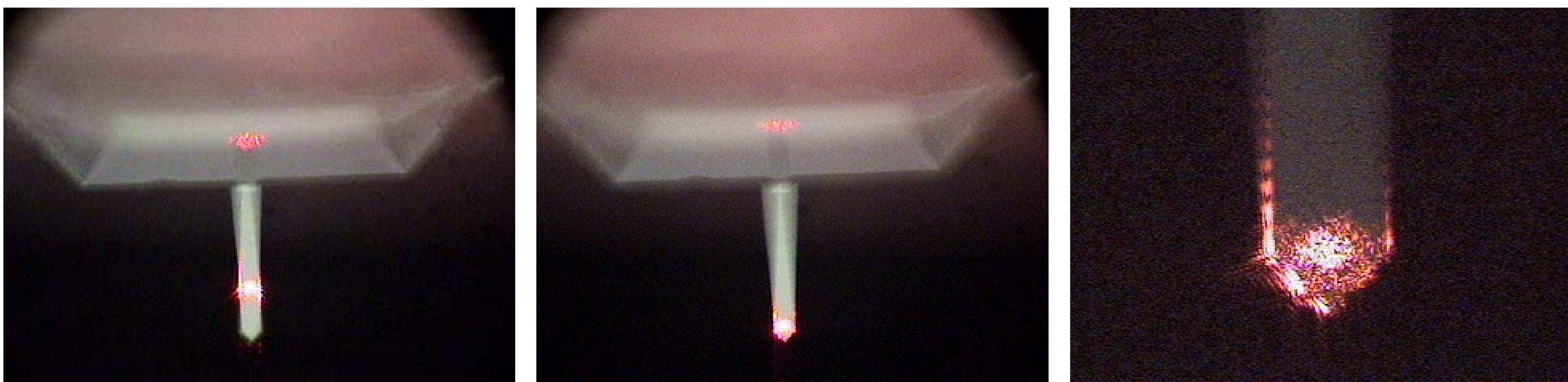
Laser Alignment on Cantilever

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





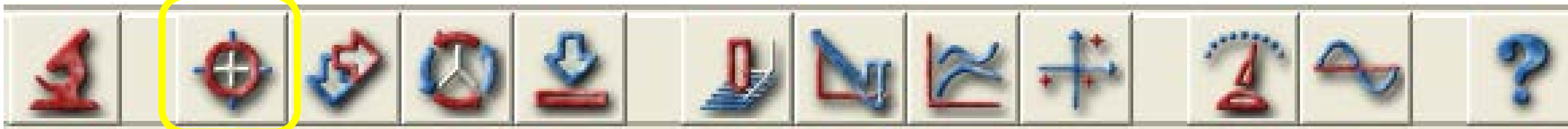
- Roughly position the laser spot, then walk it out to the correct position



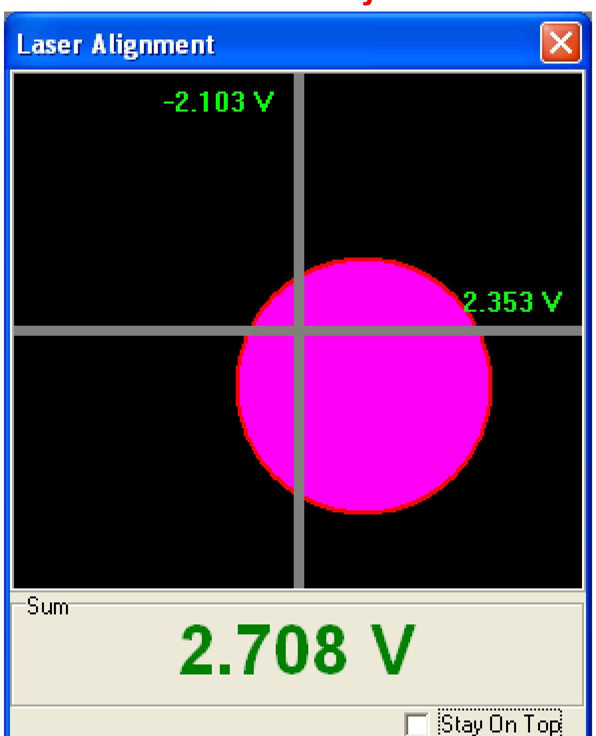
- Zoom in and refocus as necessary.

Detector Alignment

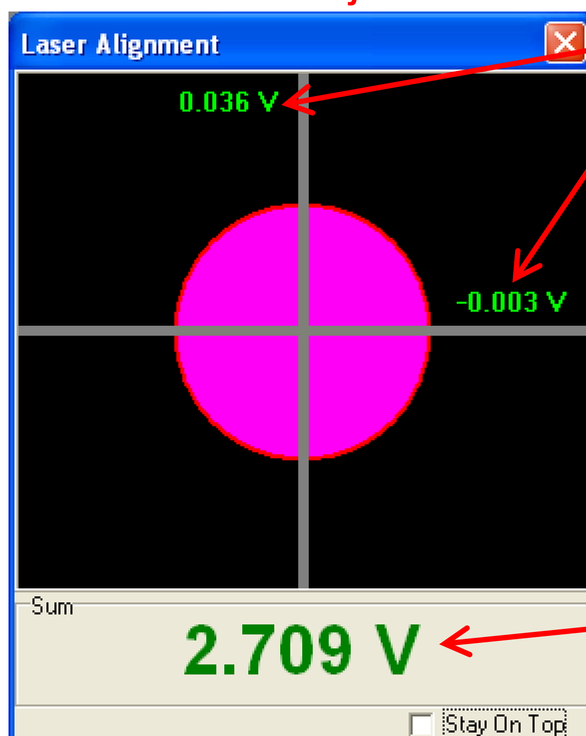
- Click on the 4-quadrant detector icon.
- A new window will appear which shows the position of the laser on the detector.
- Adjust the detector alignment knobs to center the spot.



Before Fine Adjustment



After Fine Adjustment

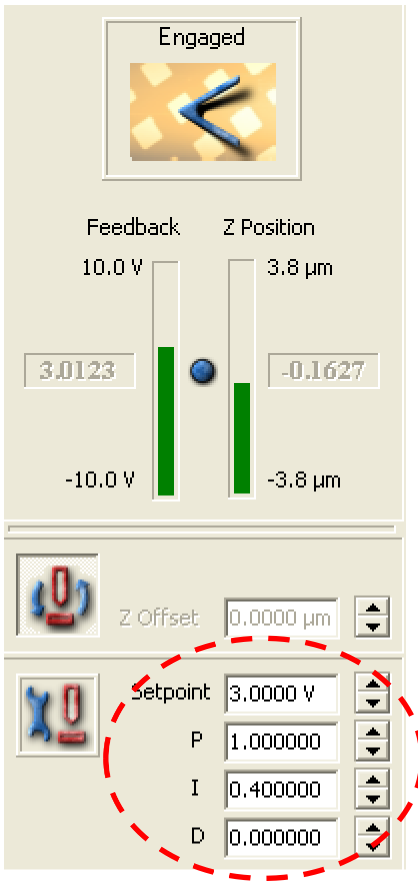


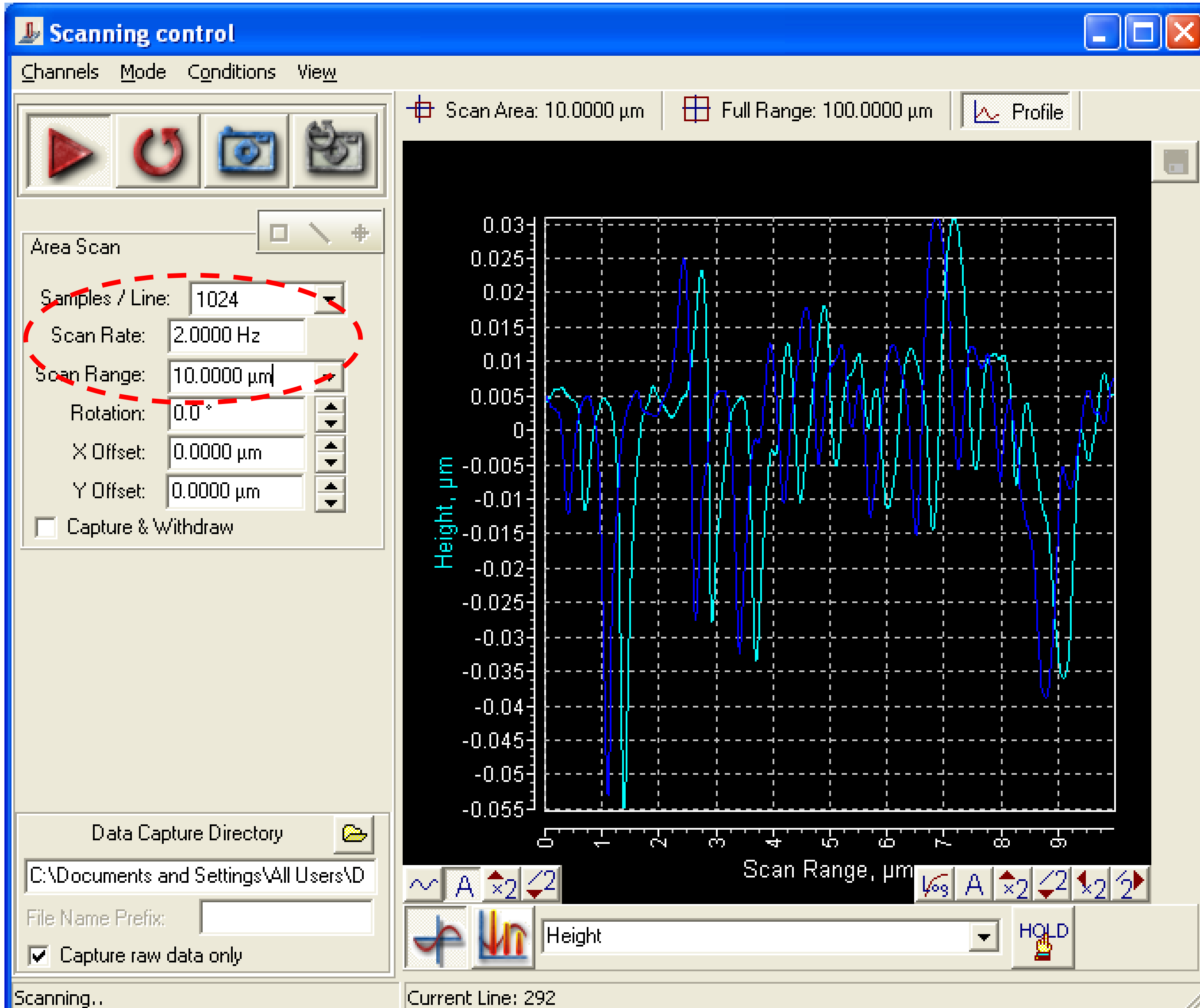
<u>A-B Signals</u>
Comparison of signal coming from halves of detector. Top – Bottom & Left – Right. Closer to zero is more centered.

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

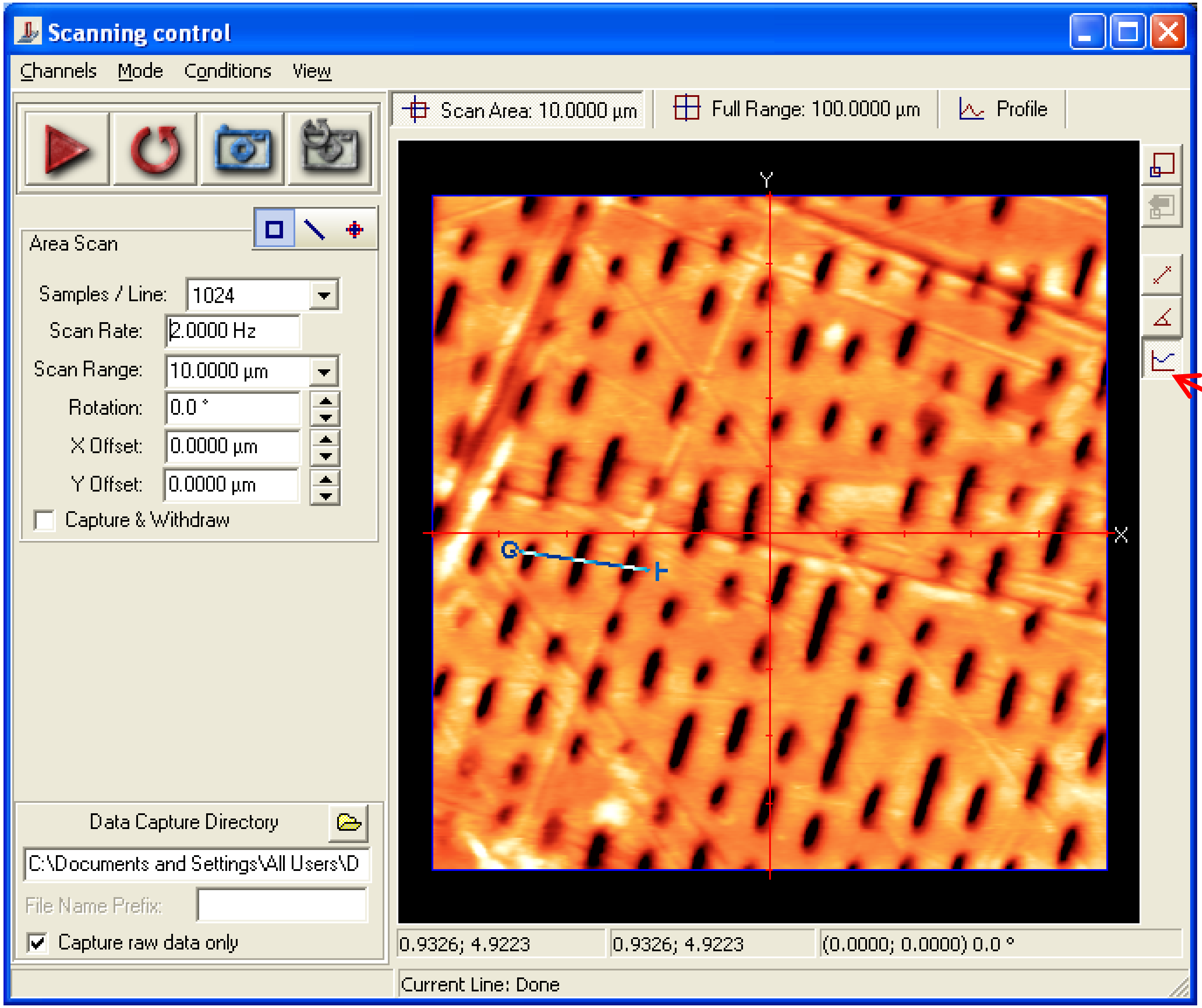
Acquiring an Image

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





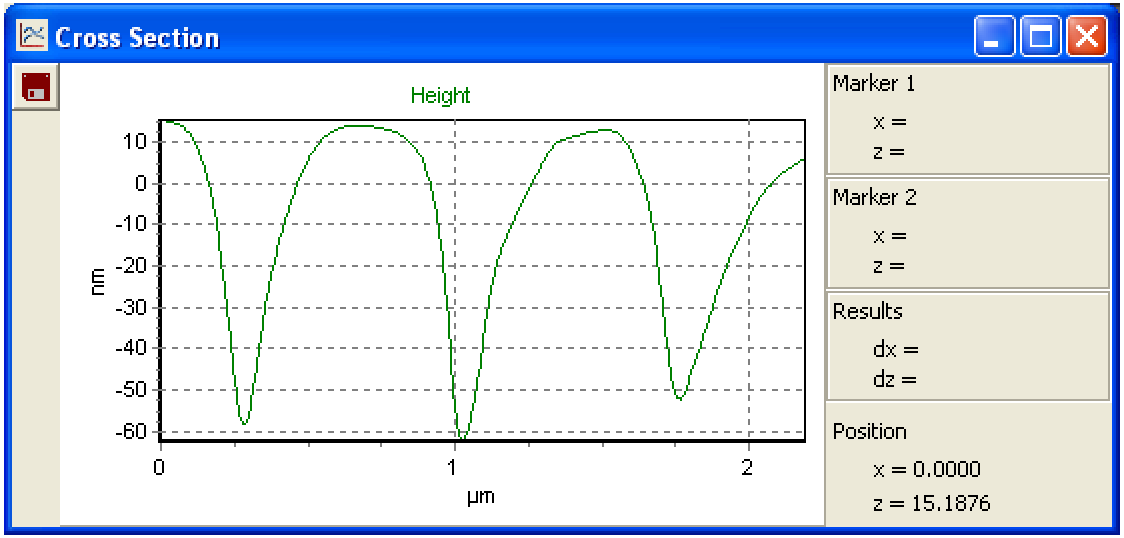
Analyzing Data: Measurements

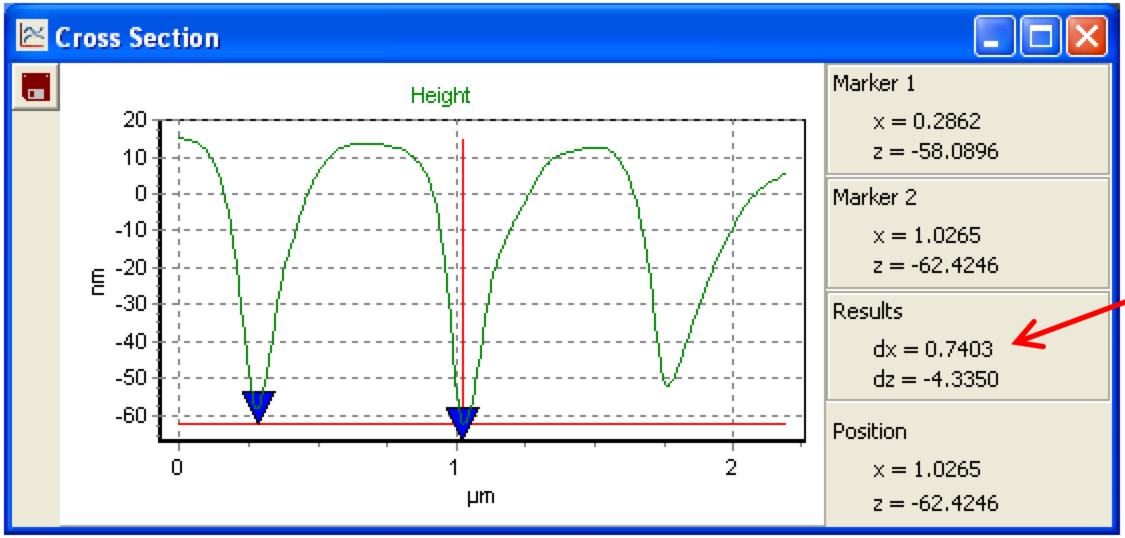


Cross-Section Measurement Tool

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.