

University of MN, Minnesota Nano Center  
Standard Operating Procedure

|                        |                                |                    |                      |
|------------------------|--------------------------------|--------------------|----------------------|
| <b>Equipment Name:</b> | <b>Atomic Force Microscope</b> | <b>Revisionist</b> | <b>Paul Kimani</b>   |
| <b>Badger name:</b>    | <b>afm DI5000 PAN</b>          | <b>Date:</b>       | <b>April 9, 2020</b> |
| <b>Model:</b>          | <b>Dimension 5000</b>          | <b>Revision:</b>   | <b>1</b>             |
| <b>Location:</b>       | <b>Bay 1 – PAN</b>             |                    |                      |

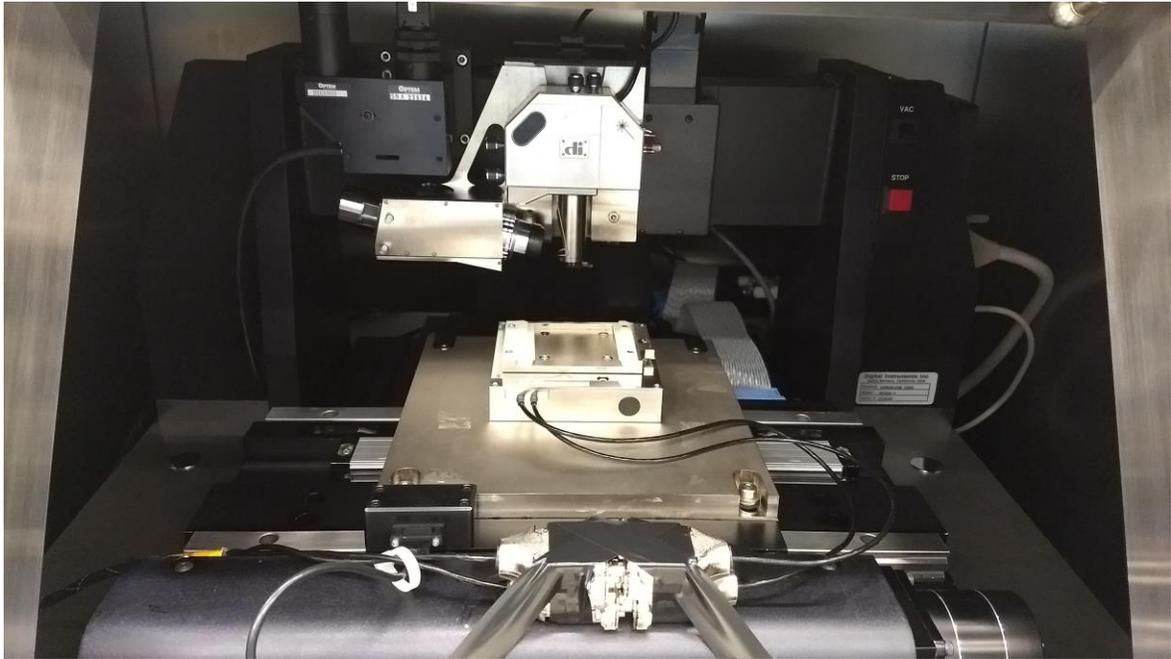
**A. Description**



- i. Enhanced Motorized Positioning Stage:**
  - Travel: X-axis 200 mm (7.87")
  - Y-axis 250 mm (9.84")
  - The Z-stage is driven by a motorized lead screw for coarse approach of the head to the sample
- ii. Integrated Dimension Controller**
  - The Dimension controller integrates the illuminator and power supply
- iii. Optical Microscope**
  - The optical microscope includes a computer-controlled illuminator for easier optical focusing and zooming.
- iv. Video Image Capture Capability**
  - Video image capture capability allows the user to easily incorporate video images into reports and publications.
- v. Computer System**
  - The Dimension 5000 ships with a high quality tower-style Pentium PCI computer system.

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### B. Safety:

Follow good “rules of engagement.”

“Engagement” refers to the process of bringing the tip and surface together. Some probes (especially single crystal silicon Tapping Mode™ probes) are prone to breakage if engaged too quickly or too hard. Ensure that engagement settings never exceed the limits of safety and never attempt to engage manually using coarse adjustment screws.

**Never move the head while imaging**

The head contains the tip holder, laser and photodiode array. An XY translation stage is provided for moving the head and tip several millimeters across the sample for coarse adjustment. Even for relatively smooth samples, the head should NEVER be moved with the tip engaged. This almost always results in tip breakage. Always disengage first before using the XY stage to move the tip.

**Never leave your controller ON while the computer is turned OFF**

Operators are advised to turn OFF their controller when finished the imaging. If the controller is left ON for an extended period without an energized computer, damage to the scanner may result.

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### C. Restrictions/Requirements

Tip changing will be done with assistance only. If you need to regularly change the tip, training can be arranged.

### D. Required Facilities

- i. Vacuum source (-25 in Hg)
- ii. Dedicated 115V, 15A Standard duplex outlet
- iii. 90 PSI, CFM

### E. Definitions

### F. Setup

- a. Log into Badger
- b. Open the hood
- c. If you intend to use a different type of probe other than the existing tapping mode probe or if the probe is not functional, load a new probe. Otherwise skip to step G.
- d. Using sharp tweezers, load a probe into the probe holder carefully
- e. Load the probe holder onto the scanner. There is only one way it will fit. Load it so that the tip at the end of the probe is facing outward.

*Please be very careful when loading the probe onto the four pins! Aggressive loading can snap the pins out. If you have problem loading probe, let MNC staff know*

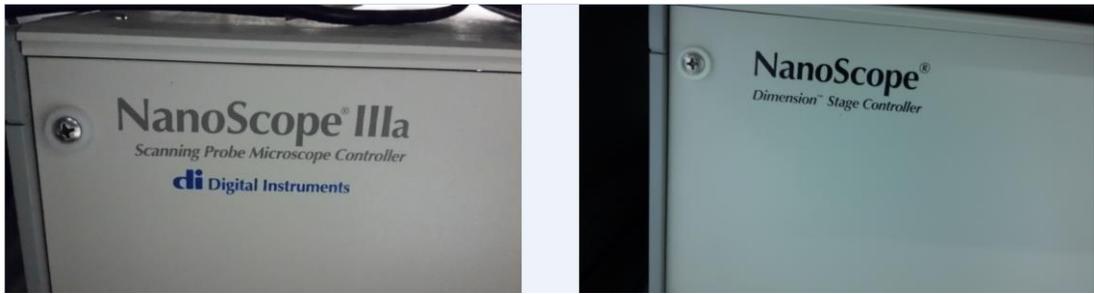
Load scanner: slide the scanner in place. **Loosen** the knob on the frame (until the thread is just loosened plus approximately 1½ turns) to *tighten* the scanner in place. Connect the head to the stage controller electronics by inserting the SPM microscope head's black 21 pin connector plug into the connector socket just behind the Z-stage.

### G. Power on in the following order:

- Turn on the computer using the push-button switch located on the front of the computer (in case it was off; it should always be left on)
- Turn on the NanoScope IIIa controller using the power switch located on the rear of the NanoScope controller (switch at the front of the work console).

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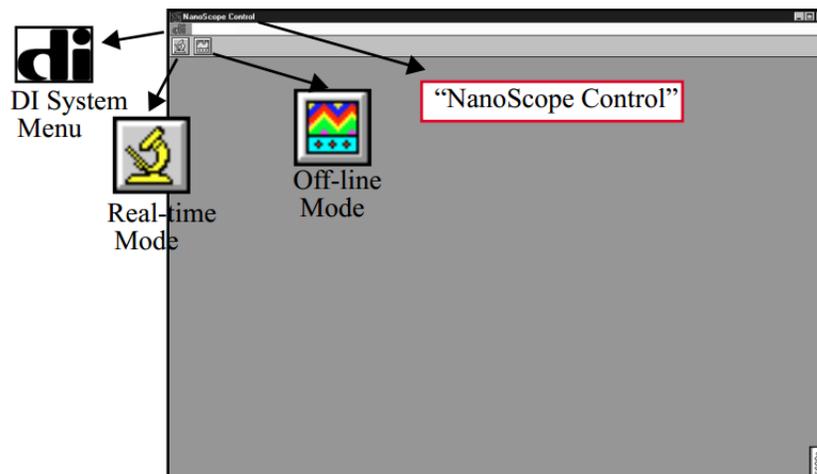


- Turn on the Dimension 5000 controller using the power switch on the rear of the Dimension stage controller (switch at the back of the work console).
- Turn on the Nanopoint system control box switch (on top of work console, if it has been turned off)
- Illumination can be computer or manually controlled. This box is on the right rear of the work console. The knob located on the front of the illumination box regulates manual illumination control. For computer control, there is an “illumination” parameter in the **Other Controls** panel on the monitor to control brightness. The illuminator control knob must be in the “off” position to allow software control.

### H. Starting the software

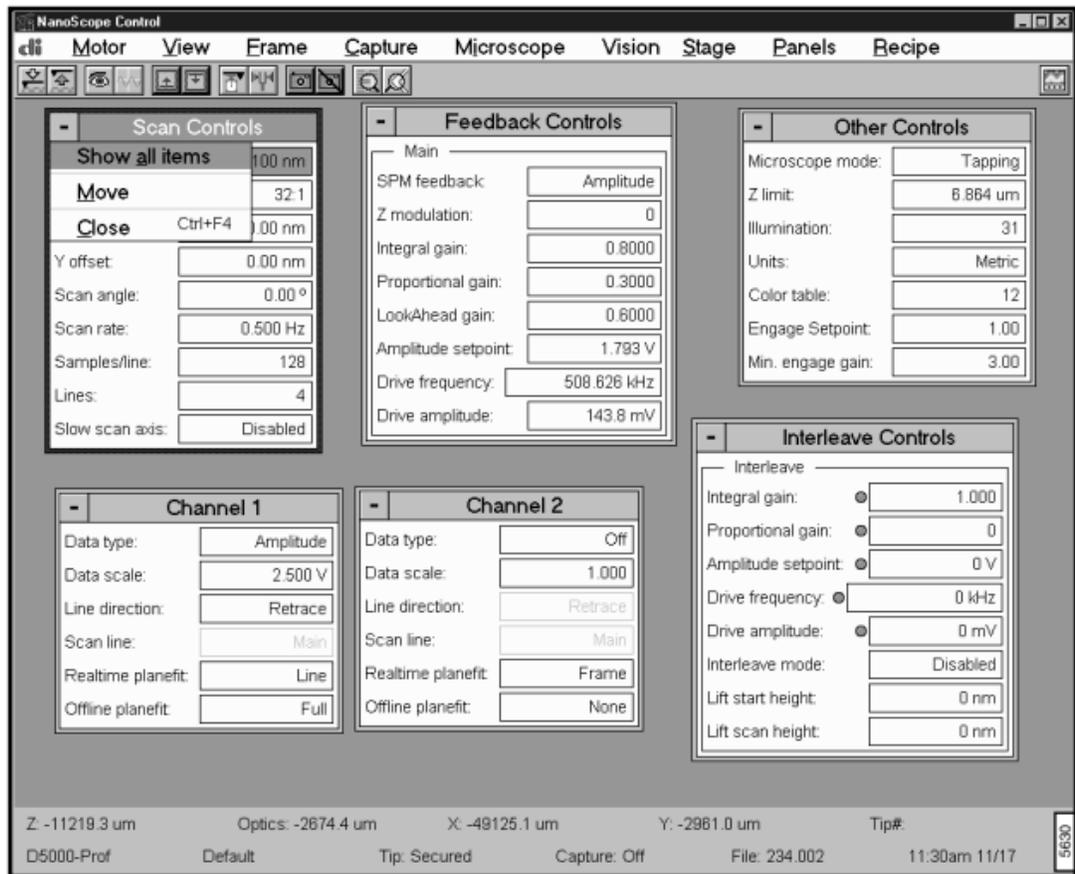
- a) Start the NanoScope software by double-clicking on the desktop shortcut labeled **NanoScope III v531r1**.
- b) Click on the **Real-Time** (yellow microscope) icon to begin using the AFM
- c) Note the default mode is **Tapping Mode**

The two monitors should display start-up screens



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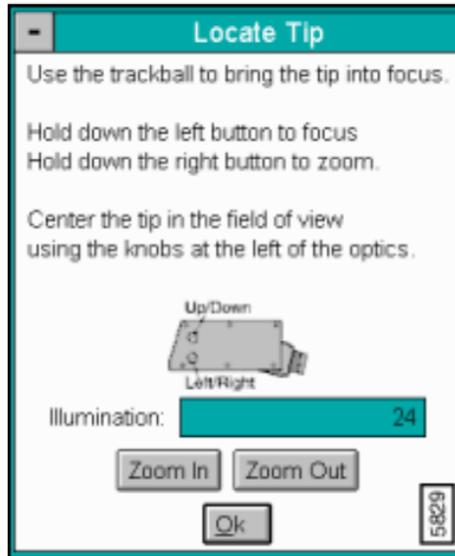
Real time control monitor

### I. Locate tip

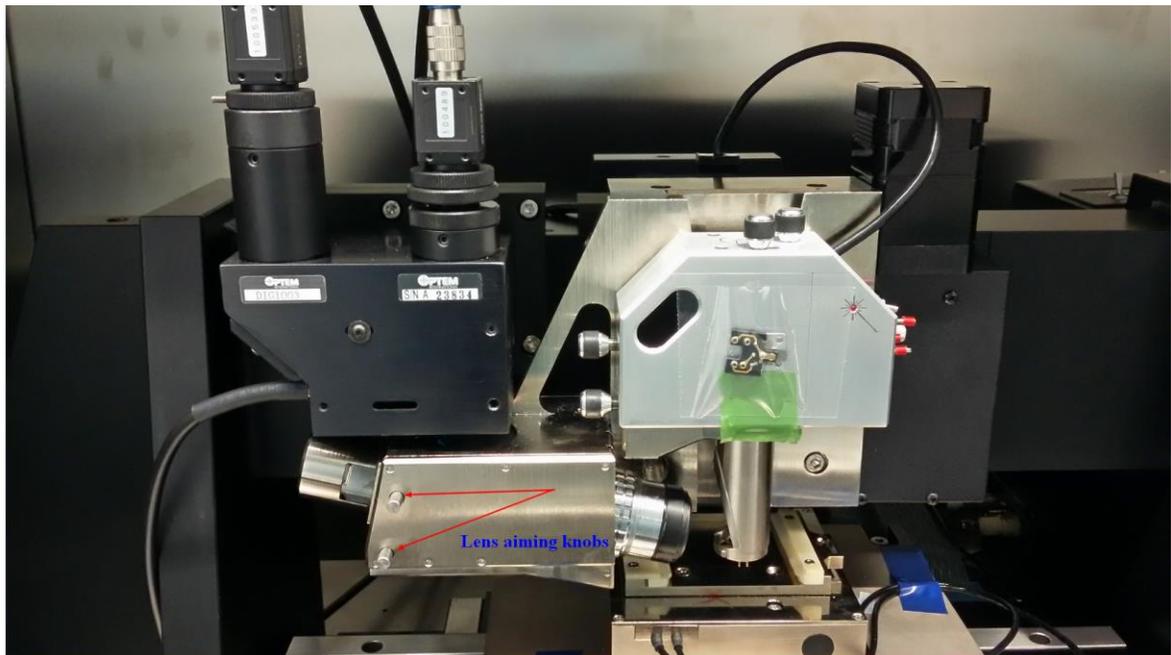
- a) Select **Stage > Locate Tip...** or click the **Locate Tip** icon.  (Magnifying glass on yellow tip). This command is used to locate the tip position (Z-height) using optical focal distance measurements.

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Locate Tip Instructions Panel



- b) Zooming out as far as possible may help in locating the tip.
- c) Once the tip has been located, use the optical objective's two lens aiming knobs (lower-left corner of the zoom optics assembly) to aim the optical microscope lens so the probe tip's cantilever beam is approximately centered in the field of view.



- d) Adjust the focus by pressing the focus button on the trackball, and sliding the trackball accordingly. If the entire cantilever cannot be in focus at the same time

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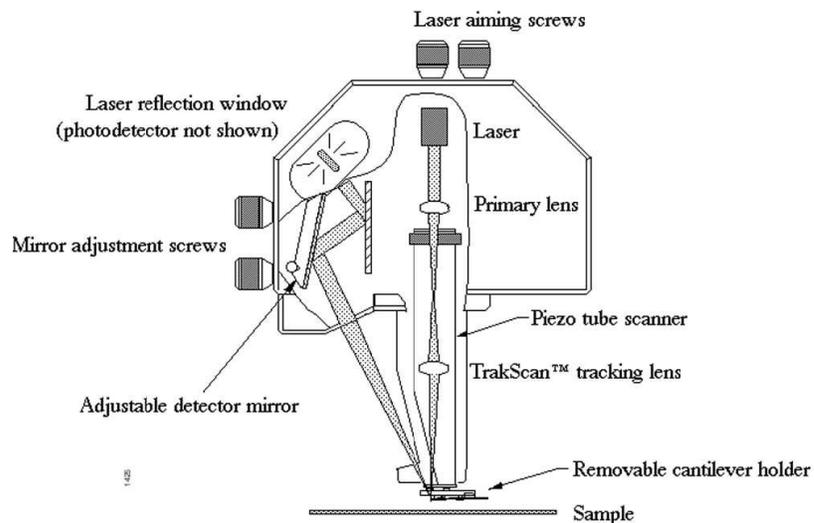
(remember it is held at a downward angle), make sure the tip of the cantilever is in focus. It may help to zoom in before adjusting the focus.

- e) Moving the optical focus in this command does not move the Z-stage.
- f) Quit the **Locate Tip** sequence by clicking on **Ok**.

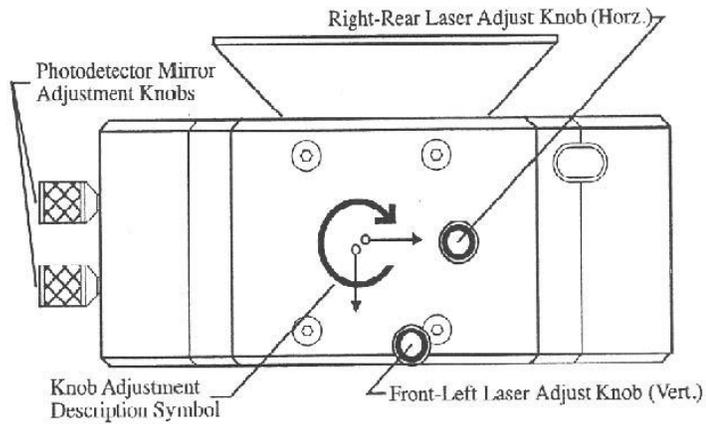
### J. Laser Alignment – Tapping mode

For both Tapping Mode and Contact Mode AFM, the user aligns the laser by moving the laser beam relative to the cantilever while observing the laser spot on the granite surface (a piece of white paper also works well) below the Dimension head. If the laser is not on the cantilever substrate, the laser appears as a bright red spot on the surface below. When the laser is aligned on the cantilever, a shadow appears on the surface below.

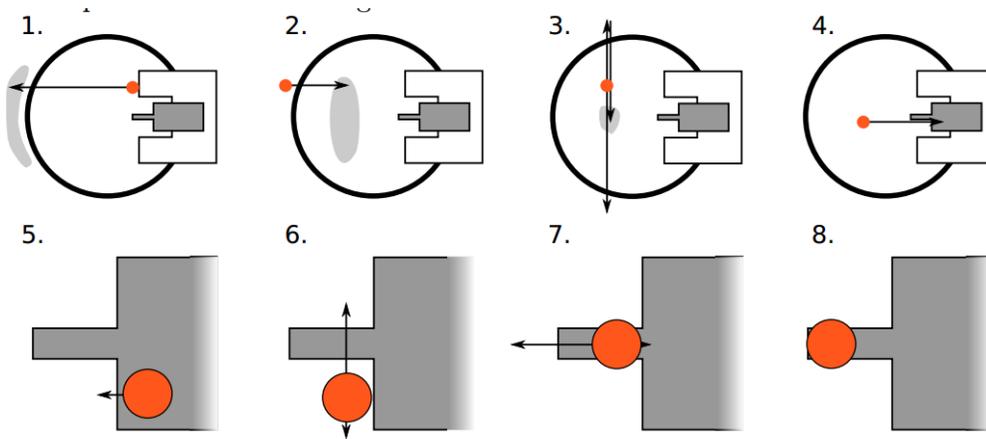
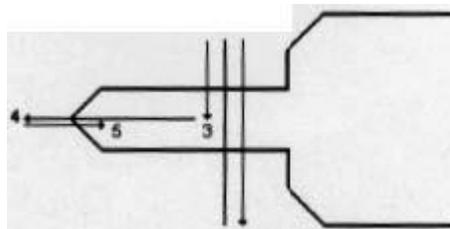
- a) For the following alignment procedure, the X direction runs along the major axis of the probe's substrate (parallel to the length of the cantilever beam) and the right-rear laser positioning screw on top of the SPM head controls the laser beam movement along the X-axis (and right-to-left on the photo-detector/image monitor). Turning this screw clockwise moves the laser spot to the right.



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- b) The front-left positioning screw atop the SPM head moves the beam along the Y direction perpendicular to the probe tip cantilever beam. It also moves the laser spot top-to-bottom (vertically) on the photodetector/image monitor. Turning this screw clockwise moves the laser spot to the bottom of the detector screen.
- c) Align Laser beam onto the tip of the cantilever using the two lasers adjust knobs located on the top of the AFM head.

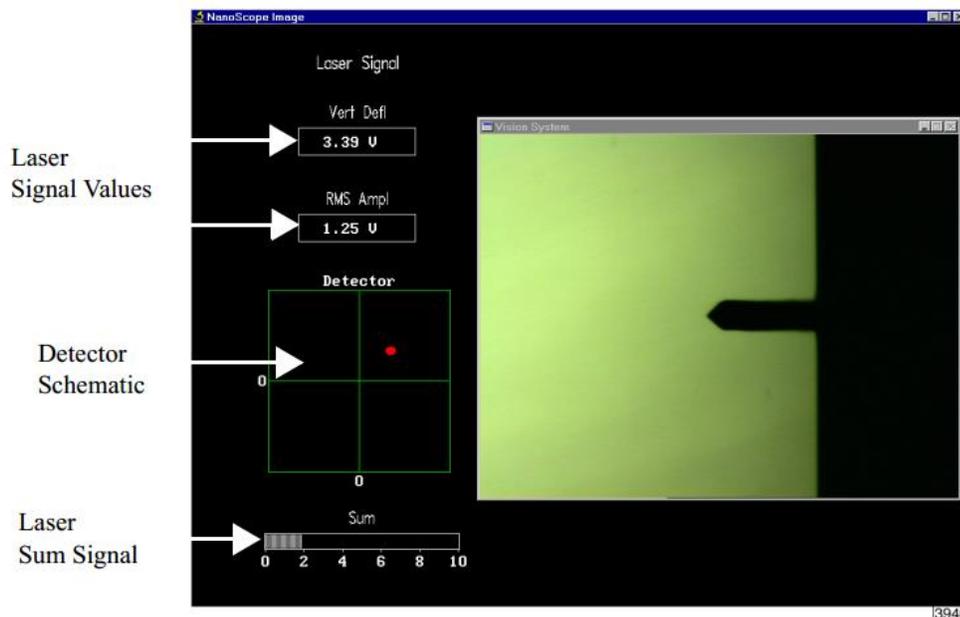


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1. Move the laser to the left until it is shadowed by the scanner ring.
  2. Move somewhat back to the right .
  3. Find the center of the scanner ring by moving the tip up and down.
  4. Move the laser to the right until it is shadowed by the chip (you may see the back reflection).
  5. Move the laser back to left until it is just over the edge of the chip.
  6. Move the laser up and down until it crosses the cantilever.
  7. Move the laser again to left until it is at the free end of the cantilever.
  8. That's it!
- d) Now that the laser is aligned on the cantilever, verify there is a spot visible on the Dimension head filter screen.
- e) Verify there is an appropriate laser sum signal displayed on the image monitor.
- Typical laser sum values:
- Contact Mode AFM: **4 - 6V**
  - TappingMode: **1.5 - 2.5V**

### Vision System Window



**Note:** If the laser sum signal is low, either the laser is not aligned or the

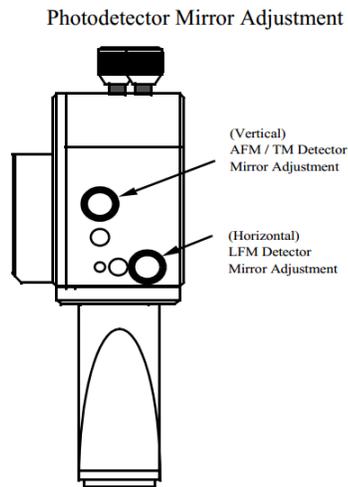
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photodetector

knob needs adjusting (described in [next step](#)).

- Center the laser detector signal using the photodetector adjustment knobs located on the left side of the Dimension head.



The image monitor displays the laser signal values and a schematic of the detector quadrants labeled **Detector**. The position of the laser is denoted by a red dot on the detector schematic.

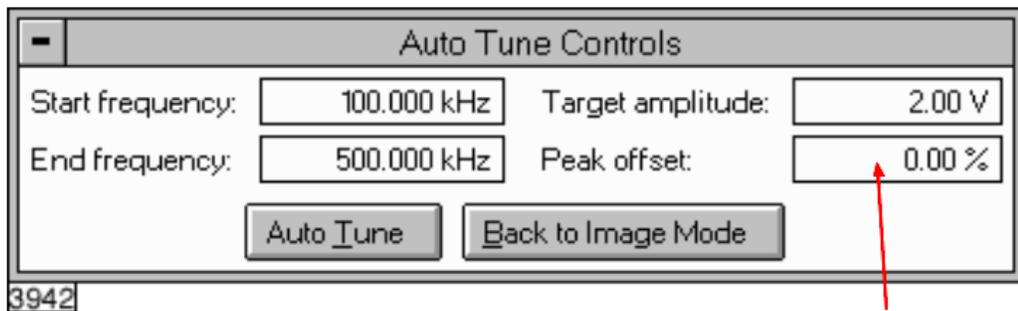
- The Vertical Deflection signal is the difference between the top and bottom photodetectors. For TappingMode, adjust this signal to 0. For Contact Mode, adjust this signal to -2.
- **Note:** In TappingMode, the **RMS Ampl** is an AC signal and does not have any real magnitude until the cantilever tune has been completed.
- **Note:** When the laser is positioned in the center of the detector schematic, the laser is also in the center of the screen on the front of the head. If the laser is severely out of alignment, it may help to first center the laser on the screen on the head using the photodetector adjustment knobs, and then use the detector schematic on the image monitor to finish positioning the laser.

### K. Tuning the Cantilever

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- a. A range of vibration frequencies is applied to the cantilever to determine the frequency which produces the largest response (the resonance frequency). In most instances, the resonance peak will have a sharp Gaussian distribution but sometimes the peak can be somewhat rugged. The system will tolerate some deviation in the shape of the peak.

Auto Tune Control Panel



set at 5 - 10%

- b. Click on view/cantilever tune or the Autotune icon (blue tuning fork) . Make sure **Start Frequency** is set at 100 kHz and **End Frequency** is at 500 kHz. Target amplitude should be 2 - 2.30V. Click on Autotune button.
- c. **AUTO TUNE** executes the automatic tuning procedure: the cantilever is excited through a range of frequencies beginning at the Start frequency and ending at the End frequency. A plot of the cantilever's response curve is shown on the Display Monitor
- d. **Peak offset**—Percentage of cantilever's free-air resonant frequency to be automatically offset. Peak offset is used to compensate for changes in resonance before engagement due to the tip's interaction with the surface after engagement.
- e. **BACK TO IMAGE MODE**—Returns the software to image mode.

**L. Controls – Tapping mode**

The **Scan Controls** panel includes parameters influencing piezo movement and data acquisition, as well as the ability to execute non-square scans.

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*Non-square* scans are set using the **Aspect ratio** parameter. *Square* scans have an aspect ratio of **1:1**; therefore, any other setting (**2:1**; **4:1**; **8:1**; **16:1**, etc.) will produce a non-square scan.

1. **Other Controls:** Set **Units** to **metric**, **Color Table** to **2**, **Offline Planefit** to **Full**, **AFM Mode** to **Tapping**. Set all filters to **Off**.
2. **Main Controls:** In the **Scan Controls** panel, set the following:
  - **Initial Scan Size** is set to **1 $\mu$ m** (or desired scan size). Bruker recommends you always initially engage with small scan sizes.
  - **X and Y Offsets** are set to **0**.
  - **Scan Angle** is set to **0**.
  - In **TappingMode**, under the **Feedback Controls** panel, set the following:
    - **Integral Gain** is set to **0.4**.
    - **Proportional Gain** is set to **0.8**.
    - **Scan Rate** is set to **1Hz**.

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Suggested Feedback Controls Settings

| Feedback Controls   |           |
|---------------------|-----------|
| SPM feedback:       | Amplitude |
| Z modulation:       | 0         |
| Integral gain:      | 0.4000    |
| Proportional gain:  | 0.6000    |
| Amplitude setpoint: | 1.000 V   |
| Drive frequency:    | XXX kHz   |
| Drive amplitude:    | XXX       |
| Analog 2:           | 0 V       |

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Suggested Scan Controls Settings

| Scan Controls   |                    |
|-----------------|--------------------|
| Scan size:      | 1.00 $\mu\text{m}$ |
| X offset:       | 0.00 nm            |
| Y offset:       | 0.00 nm            |
| Scan angle:     | 0.00 deg           |
| Scan rate:      | 2.00 Hz            |
| Samples/line:   | 256                |
| Slow scan axis: | Enabled            |

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Other Controls Panel (D5000/Tapping Mode)

| Other Controls     |                     |
|--------------------|---------------------|
| Microscope mode:   | Tapping             |
| Z limit:           | 6.864 $\mu\text{m}$ |
| Amplitude limit:   | 2.500 V             |
| Illumination:      | 24                  |
| Units:             | Metric              |
| Color table:       | 12                  |
| Engage Setpoint:   | 0.900               |
| Scan line shift:   | 0.00                |
| Tip serial number: |                     |
| Serial number:     | 1417GN              |
| Min. engage gain:  | 3.00                |

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**NB:** The **Drive frequency** and **Drive amplitude** values were determined during the Cantilever Tune procedure. It is not necessary to enter a value for the **Amplitude setpoint**; this will be determined automatically during the engage process.

- Set **Number of samples** to **256** (later this may be set to **512** for better image clarity).
  - Verify **Slow Scan Axis** is **Enabled**.
  - Choose an **Engage Setpoint** of **0.9**; this specifies the initial set point amplitude ratio (ratio of reduced to free amplitude).
  - **Z Limit** should be at its maximum, which is slightly less than  $6 \mu\text{m}$ .
3. **Images:** Under **First Image**, set **Data type** to **Height**. Set **Z-Range** to a reasonable value for your sample. **Line Direction** can be set to either **Trace** or **Retrace**. **Second Image** can be disabled by setting **Data Type** to **Off**

### M. Sample Loading

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If it is a small sample use a sample puck and then put the puck on the



magnetic holder. **DO NOT** put sticky tape between the puck and magnetic holder! Mount the magnetic holder on the sample block in the center. Wafers can be set on top of the sample holder and scanned with/without vacuum.

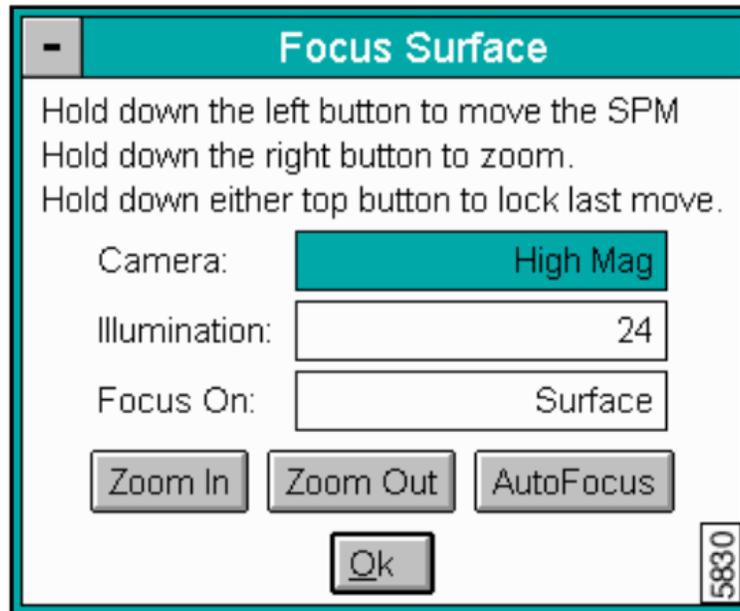
#### N. Focus Surface

The **Focus Surface** command accesses a panel to control the stage and view the surface. The purpose of this command is to optimize placement of the tip on the sample surface. **CAUTION:** Use caution when focusing on the sample surface. Moving the head too quickly while focusing can cause the tip to crash, which may damage the tip and/or sample.

1. Select **Stage > Focus Surface...** or click the **Focus Surface** icon.



### Focus Surface Panel



2. In the **Focus On** box, select **Tip Reflection** (or **Surface** if you are an experienced user). If the sample is very flat or reflective, or you are not sure of which option to use, choose **Tip Reflection**.
- a) If the scanner is too low, first pull up the scanner before moving the sample in. This can be done by keeping the focus knob next to the roller ball pressed while moving the roller ball away from you (hold both focus and lock for steady motion). Align the sample below the tip holder and focus on the **Tip** (or **Surface** if you've selected "**Focus on surface**") using the trackball with the focus button depressed and moving the roller ball towards you. To translate the stage only, simply slide the trackball, or press the lock button and slide the trackball once to maintain a steady translation.
- b) Lower the scanner (you are still in the **Focus Surface** mode) until the **TIP** is in good focus. This allows you to be in focus on the sample and not the bottom surface (if sample is transparent); (If you selected **Surface** in 2 above, look out for both the sample and tip to be in good simultaneous focus). Once you ask the



software to automatically engage , it goes **Only 200 microns**. If the sample is further away than that the AFM will be unable to engage and returns an error.

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Should that happen, Select **Motor > Withdraw** or click the **WITHDRAW** icon on



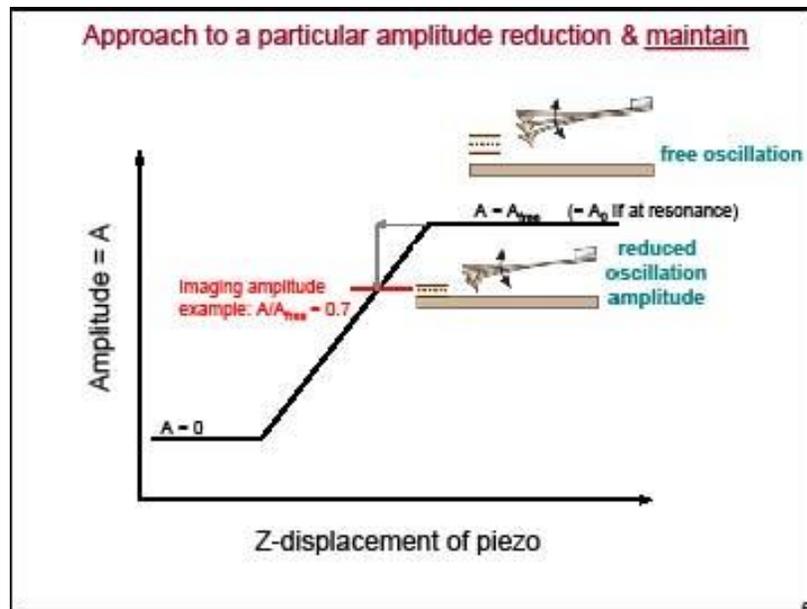
the toolbar **Withdraw** **once**, then click on **Focus Surface** icon and move the tip down a little further down than the previous engage point, then retry automatic engage



. Repeat as necessary.

### O. Engaging the microscope

- Move sample to the area of interest using the stage manipulator
- Click on Motor followed by Engage. A pre-engage check followed by Z-stage motor sound should be observed and the scanner housing and tip are lowered. This continues until amplitude voltage is reduced to the engage Setpoint times the free amplitude just prior to engagement.



At this point, the amplitude is reduced by 10% relative to its value just beyond the surface, due to onset of tip-sample interaction. Once the Setpoint is reached, the approach stops, the control box beeps, and imaging commences immediately. If engage aborts because the SPM head is still too far from the surface, go back to



Stage/Focus Surface (or ), click on the withdraw button once to bring the tip back to its pre-engagement height and use the trackball to re-locate the surface. Next lower your scanner a few more tens of micron more than your previous

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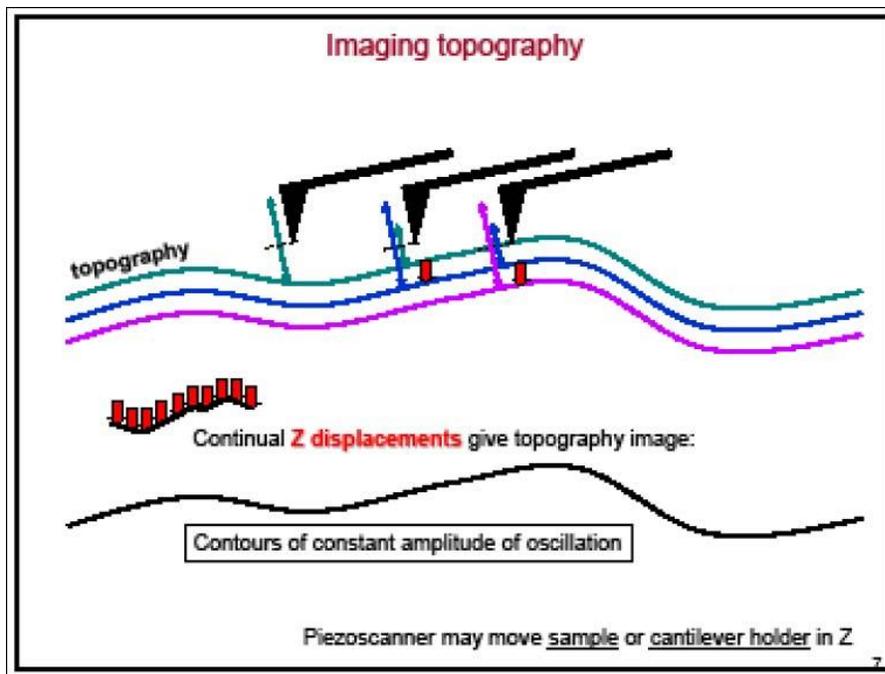
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attempt.

- c) False engagement: The 10% amplitude reduction is occasionally not genuine. In this case, reduce the engage set point to 0.8 and try engaging again. Increasing the drive amplitude may also help prevent false engagement. True engagement can usually be identified as a sudden (a) decrease of amplitude just before imaging initiates. This is because upon interaction of tip and sample, the amplitude changes rapidly versus decreasing distance (over a few nanometers of approach), whereas the air damping of the oscillating cantilever changes very slowly versus decreasing distance (many microns).
- d) If stage requires movement to get to another area of the sample, execute a **Withdraw** command first. Click **Withdraw** 1 times to ensure the tip does not run aground because of surface topography or tilt; otherwise tip damage can occur

### P. Upon Engagement

The amplitude Setpoint is computer selected according to **engage Setpoint** (fraction of Amplitude signal prior to engagement). The **Height** image is the vertical position **Z(x,y)**, needed to maintain a constant Amplitude. The Amplitude signal may be used to optimize topographic imaging as follows:



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**Q. Withdrawing the tip**



Select **Withdraw** from the **Motor** menu or click **Withdraw**. The SPM will cease scanning and ascend to the sample clearance height defined in the SPM parameter menu. Select **Stage/Load New Sample** to replace or remove the sample.

**R. Image Capture, saving and retrieval**

- Select a filename for your image. Click on the **Capture Menu**, and select the **Capture Filename** option. Type in the name of your file. The extension is automatically added as .000, and successive images will be given subsequent extensions (such as .001, .002, ...)
- Capture the image by clicking on the **Capture** icon (yellow camera). The image will not be saved until the full scan is complete. To cancel a capture at any time, click on the **Cancel Capture** icon (yellow camera with a red line through it)
- To expedite the capture process, the **Begin New Scan** icons (blue with arrow up or arrow down) may be used to initiate a new scan
- Withdraw the tip to stop the scan, if done capturing all desired images. If you need to engage again, re-position the surface for a new scan by clicking on the **Focus Surface** icon (magnifying glass on red bars), or load a new sample by clicking on the **Stage menu**, and selecting **Load New Sample**.
- To examine your captured images, click on the **Offline** icon (wavy rainbow all the way on the right). Note – if captured images need to be flattened, select an image, (Left/Center/Right) click on Modify/Flatten or the Flatten icon (rolling pin). Note images will only be viewable using DI AFM software, or other AFM specific programs like **Gwyddion**. To view using a graphics program, you must convert your file to a JPEG, or TIFF file. For this, select desired file select Utility menu – JPEG or Tiff Export.
- When done, power off in following order:
  - a) Unload sample
  - b) Exit AFM software.
  - c) Turn off the AFM controller
  - d) Turn off the Dimensions controller

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- e) Turn off the Nanopoint system controller
- f) Close the hood
- g) Log off the BADGER

**S. Back up data:**

On AFM PC, retrieve your files using the USB ports on the monitors or at the back of the computer.