Instructions for Dimension 3100 Atomic Force Microscope

- These instructions are to be used to begin to familiarize a new user with the microscope and the software interface. Training from the NCF staff is required before using the microscope independently.

- These instructions can also be used as a simple refresher in case a newly trained user has forgotten some of the steps.

- **NOTE**: Numerous reference manuals can be found on the Windows desktop:

DO NOT ATTEMPT ANYTHING YOU ARE UNSURE ABOUT. YOU DON'T WANT TO DAMAGE THE MICROSCOPE. ASK THE NCF STAFF FOR ASSISTANCE.

1. **Check the NCF administrative offices upstairs (DLC 1B49 or DLC 1B40)**
2. **Leave a voicemail with Kathy Schrader (303.492.0734)**
3. **E-mail ncf@colorado.edu**

There are two things to ALWAYS be aware of which can cause DAMAGE to the microscope.

1. **Handle the scanner head as if it was a newborn baby.**
   - **DO NOT drop the scanner head when handling it.**
   - **DO NOT bend the cantilever holder mounting pins at the base of the scanner head.**
   - **DO NOT handle the bottom half (the stem) of the scanner head.**

2. **Approach the sample surface at a reasonable speed.**
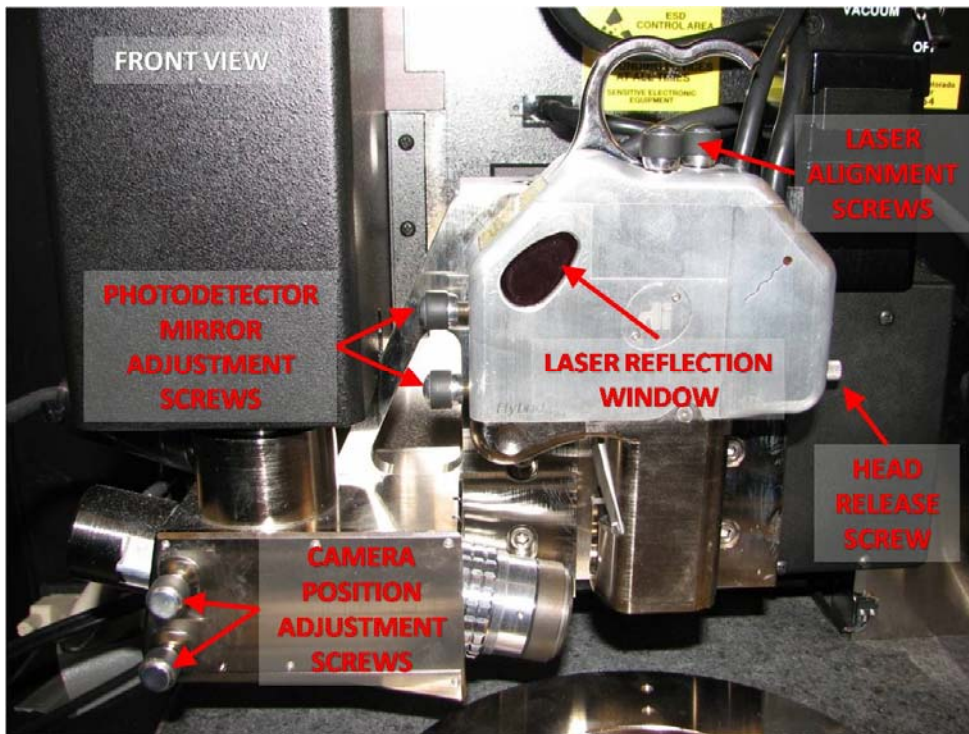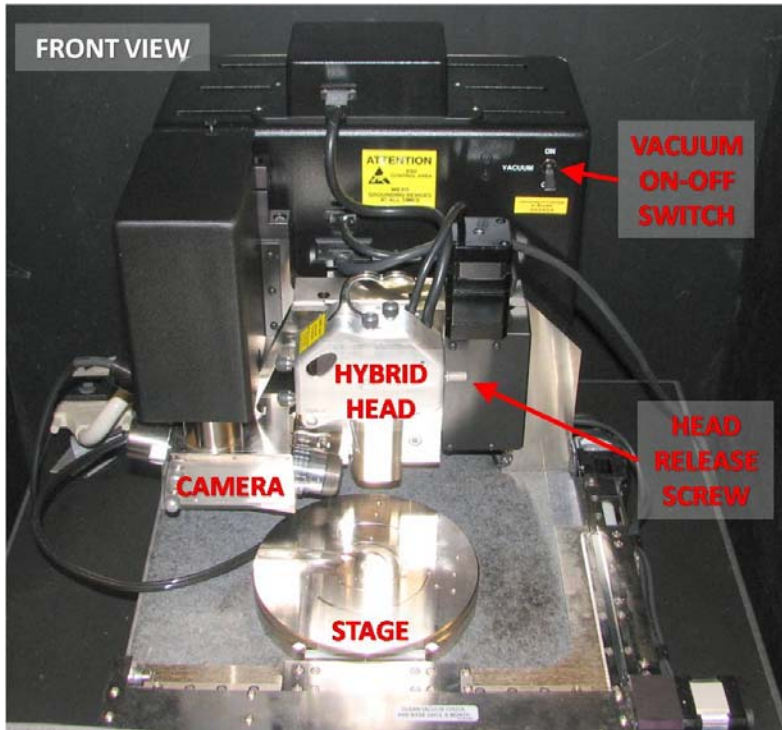   - **DO NOT run the Z-motor down in the fast setting.**

IMAGING IN AIR

Table of Contents

I. INTRODUCTION

1. HARDWARE

Controllers – turn both controllers on (on-off switches are located on the rear of the equipment on the right side)

2. SOFTWARE

i) Microsoft Windows – log into Windows using the NCF User account

ii) AFM Software – open Nanoscope v6 software

iii) Microscope – in the menu, Select **Realtime → Start Realtime** (or click on the **Microscope** icon)

- The *Add Views* dialog box will appear. Select *Navigate* and *Scan-Dual* views and Select *OK.*

iv) Workspace – using the mouse, select **Acquire → Scan-Dual** (or click on the **Scan-Dual** icon)
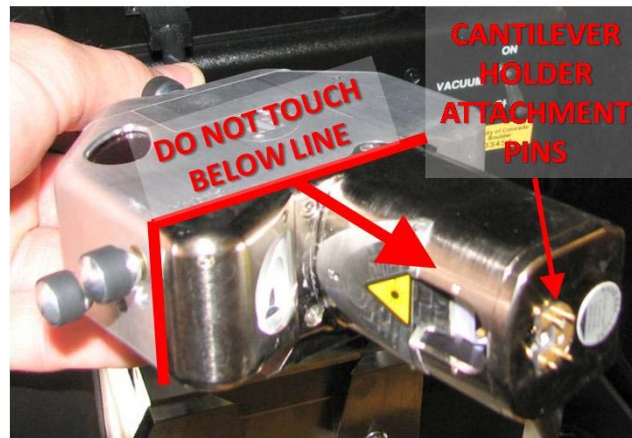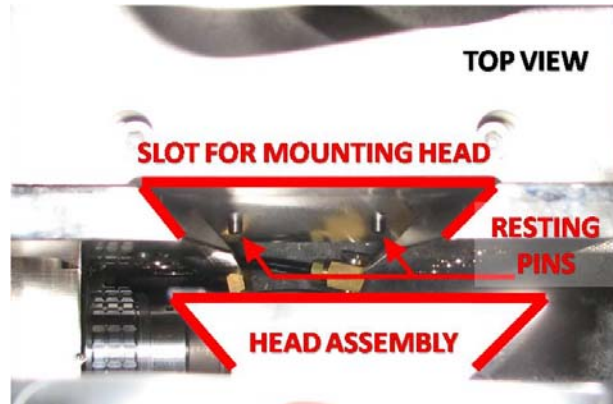

v) Microscope Mode Selection – in the Scan-Dual window under the **Other Controls** panel, set the **Microscope Mode** to Tapping or Contact.

II. OPERATING PROCEDURES

3. MOUNT THE TIP

DO NOT DROP THE HEAD – DO NOT TOUCH THE STEM OF THE HEAD

i)   Mount the probe into the cantilever holder. **Be sure that it is in firm contact with the groove.**

ii) Verify that you have not broken the tip during loading by using the optical microscope.

iii) Remove the Hybrid head from the AFM by tightening the screw (CW rotation) to the right of the head to loosen the head mounted in the dovetail groove.

iv) Mount the cantilever holder onto the end of the scanner head.

v)   Replace the head on the AFM making sure it is firmly seated in the dovetail groove before loosening the screw (CCW rotation) to the right of the head to lock it in place.

4. ALIGN THE LASER

Adjust the laser so that it is properly aligned on the tip using the two screws on the top of the scanner head. Refer to the method below for properly aligning the laser.

- Tapping Tip: The Sum signal should be about 2V.
- Contact Tip: The Sum signal should be 4-6V.

NOTE: The sum signal value is located in the realtime status window on the left side of the monitor screen under the photodetector alignment crosshairs.

Method for Properly Aligning Laser
Note: The laser reflection window on the front of the head should display a bright red reflection when laser is aligned properly
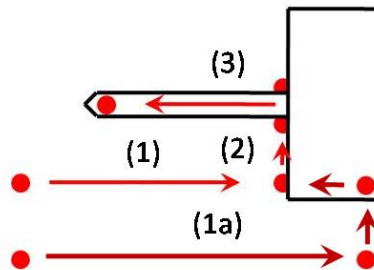
Path 1

(1) Go right until laser beam disappears due to substrate interference.

(2) Slightly back off surface and go up to locate the cantilever. Center beam on cantilever.

(3) Move the beam to the left to align the laser at the end of the cantilever.

Path 1a

(1a) If the laser does not encounter interference, stop and traverse up until the laser interferes with the substrate (substrate interference, in this approach, is determined by numerous turns of the knob), then traverse left until the laser returns. Continue with (2) above.

5. ADJUST THE PHOTODETECTOR

Tapping Mode

Contact Mode
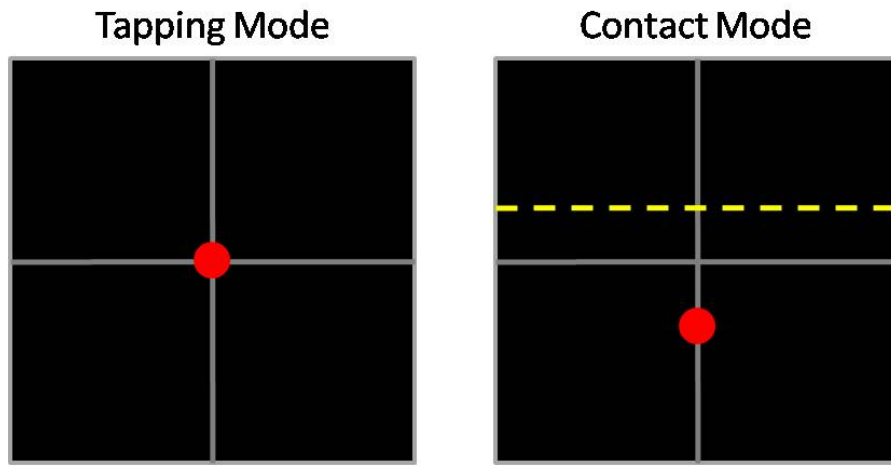
NOTE: The photodetector window is located on the left side of the monitor screen.

i)   Adjust the photodetector so that the red dot moves towards the center of the square
      (shown on the monitor), using the two screws on the left side of the head.

      NOTE: CW rotation of the *top screw* – red dot moves down
                   CW rotation of the *bottom screw* – red dot moves left

      - Tapping Mode: Center the red dot & set the vertical deflection (**Vert Defl**) to a value
        close to 0V.

      - Contact Mode: **Horiz Defl** should be about 0.0 volts, and **Vert Defl** should be about
        -2V.

6. LOCATE THE TIP
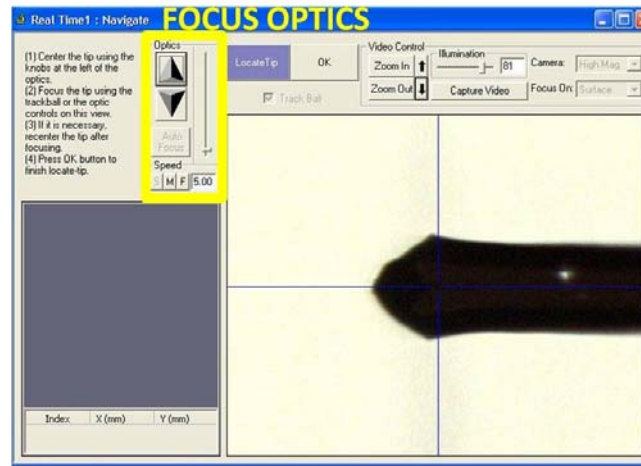
Crosshair Alignment

i) Select **Acquire → Navigate** and select the **Locate Tip** button in the Navigate window (or click on the **Locate Tip** icon).

ii) Select the **Zoom Out** button in the Navigate window and adjust the Illumination to determine the current crosshair location.

iii) Center the tip on the cantilever under the cross hairs using the two adjustment screws to the left of the optical objective (*camera*) on the microscope.

NOTE: CW rotation of the *top screw* – crosshairs move down
        CW rotation of the *bottom screw* – crosshairs move right

Focus Tip

i) Select the **Zoom In** button in the Navigate window. Focus on the tip end of the cantilever using the Optics control arrow buttons.

ii) Make minor adjustments to crosshair alignment if needed.

iii) When finished select the **OK** button.

7. LOAD THE SAMPLE

Stage Preparation

i)   If needed, using the switch near the top of the AFM, turn the **VACUUM** on.

ii) If a sticky tab or some other adhesive is used to mount the sample to the stage surface, please remove any residue leftover on the stage after the sample is removed.

Sample Alignment & Clearance

ENSURE YOUR SAMPLE DOES NOT INTERFERE WITH THE MOUNTED AFM PROBE

i)   Place the sample on the stage and **VERIFY THAT ENOUGH CLEARANCE EXISTS** between the sample surface and the tip before rotating the stage to properly align the sample scan region under the tip.

ii) If the stage needs to be moved to accommodate the scan region on the sample, select the proper arrows under the XY Stage heading in the Navigate window (Select **Acquire → Navigate** to open this window).
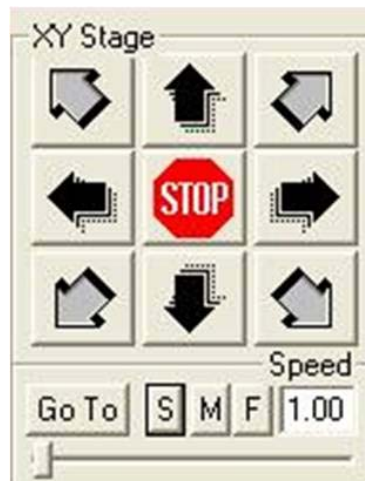
8. FOCUS SURFACE

CARE MUST BE TAKEN WHEN MAKING THIS ADJUSTMENT TO ENSURE THAT THE TIP DOES NOT HIT THE SAMPLE SURFACE. DO NOT APPROACH THE SURFACE IN THE FAST SETTING
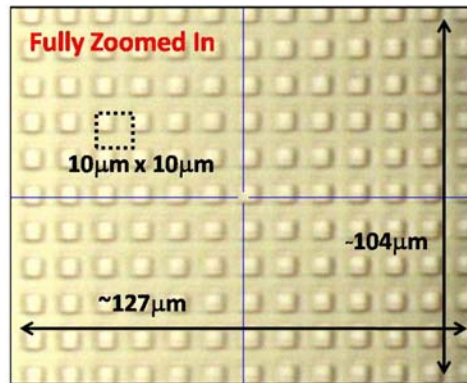
Optical Microscope View when Fully Zoomed In

i) In the Navigate window select Focus Surface.

ii) Select the **Zoom Out** button and adjust the Illumination accordingly.

iii) Determine if your sample surface is or is not highly reflective.

Non Highly Reflective Surface (or Topographical Landmarks Exist)

- The default **Focus On** selection is **Surface**.

- This method focuses on the topographical features present on the sample surface. When these features come into focus, the tip to sample clearance is set.

Highly Reflective Surface (or No Topographical Landmarks Exist)

- Change the **Focus On** selection to **Tip Reflection** in the Navigate window.

- This method focuses on the shape of the tip rather than the surface. Once the tip comes into focus the tip to sample clearance is set.

iv) Using the mouse, select a **MEDIUM** to **SLOW** speed under the Z Motor heading.

v) Approach the surface by selecting the **DOWN ARROW** under the Z Motor heading.

NOTE: Once the image comes into focus do the following:

- Reduce the approach speed to **SLOW** to decrease the chances of damaging the tip by ramming the surface of the sample.

- Select the **Zoom In** button and adjust the Illumination.

9. CANTILEVER TUNE (TAPPINGMODE ONLY)

i) Select **RealTime** → **Cantilever Tune** (or click on the **Tune** icon)

ii) For **Auto Tune** Controls set the following parameters

- Start Frequency: 100 kHz

- End Frequency: 500 kHz

- Target Amplitude: 2V

- Peak Offset: 2.5%

iii) Click **Auto Tune**. Once **Auto Tune** is done, exit.

10. SET INITIAL SCAN PARAMETERS

i) In the **Scan Controls** panel set the following parameters

- Initial **Scan Size:** 1µm

- **X** and **Y Offsets:** 0

- **Scan Angle:** 0

- **Scan Rate:** 1 Hz.

  Tapping Mode: In the **Feedback Controls** panel, set the **Integral Gain** to 0.4 and the **Proportional Gain** to 0.6.

  Contact Mode: In the **Feedback Controls** panel, set the **Setpoint** to 0V, the **Integral Gain** to 2.0 and the **Proportional Gain** to 3.0.

ii) In the **Other Controls** panel, ensure the **Z Limit** is at its maximum value using the right arrow key (reference discussion in §13 for more about this parameter).

iii) In the **Feedback Controls** panel, ensure the **Z Modulation** is disabled (*imaging in air only*).

11. ENGAGE

    i)   Close the AFM sound hood.

    ii)   Select **RealTime → Engage** (or click on the **Engage** icon)

    iii) After successfully engaging the surface, if the *Trace* and *Retrace* lines in the *Height* channel are not tracking each other, it is suggested to run the **Auto Tune** process again (tapping mode only).

          •   Use an offset value of 300nm when prompted.

12. ADJUST SCAN PARAMETERS

- This process is dependent on type of tip used, sample, ambient conditions and user experience.

- The goal of adjusting the scan parameters is to get the **Trace** and **Retrace** lines shown in the Height Channel to track each other to ensure a good image.

- Check to see if **Trace** and **Retrace** are tracking each other well. If they are tracking, the lines should look the same, but they will not necessarily overlap each other, either horizontally or vertically.

- Once the scan parameters are optimized, scan size and other features may be adjusted for capturing images for analysis.

NOTES: When changing the scan size, remember that the scan rate will need to be lowered for larger scan sizes.

If a large X or Y offset is needed to image a different region and the topographical features are unknown on the sample, withdraw and reengage after the stage has been repositioned using the Navigate window.

Tapping

(Amplitude) Setpoint

Adjust the **Setpoint** under the Feedback Controls so that the smallest amount of force is applied during scanning while still maintaining a stable engagement on the surface.

i)   Gradually increase the **Setpoint** using the right arrow key until the lines are completely different (unless they already are).

ii)   Gradually reduce the **Setpoint** using the left arrow key until they become similar. Further reduce one or two key strokes to guarantee stable engagement.

NOTE: In Tapping mode, increasing the **Setpoint** decreases the force between the tip and sample surface.

Integral Gain

Increase the **Integral Gain** under the Feedback Controls using the right arrow key. As you increase the **Integral Gain**, increase the **Proportional Gain** as well (Proportional Gain can usually be 30-100% more than Integral Gain).

i) Optimize the scan by increasing the **Integral Gain** using the right arrow key until noise begins to appear in the Trace and Retrace lines. Reduce the **Integral Gain** to achieve a good scan.

ii) Adjust the **Proportional Gain** accordingly.

Scan Rate

Adjust the **Scan Rate** under the Scan Controls to the lowest speed with which you feel comfortable.

i) Setting the **Scan Rate** too high will result in poor surface tracking. Suggested scan rates from Veeco[1].

- 1-3μm scan sizes → 2Hz

- 5-10μm scan sizes → 1Hz

- Larger scans → 1.0-0.5Hz

Contact

(Amplitude) Setpoint[1]

Adjust the **Setpoint** under the Feedback Controls so that the smallest amount of force is applied during scanning while still maintaining a stable engagement on the surface.

i) Increase the **Setpoint** using the right arrow key and observe the **ZCenter Position**.

- If the line in the Z-Center Position moves far to the extended end, the tip has false engaged. Increase the setpoint by +2 volts and execute the engage command again.

- If the Z-Center Position does not change greatly, you are probably on the sample surface

NOTE: In Contact mode, increasing the Setpoint increases the force on the sample.

Gains & Scan Rate

Follow methods described above in Tapping mode.

13. OTHER SCANNING PARAMETERS

Z-Limit

Adjusting the **Z-Limit** should only be done when you can make certain that your sample has a very smooth surface (< 5nm RMS).

i) Gradually decrease the **Z-Limit** using the left arrow key to decrease the vertical sampling size effectively increasing the Z resolution.

> **WARNING – DO NOT REDUCE THE Z-LIMIT IF THERE IS A CHANCE THAT A FEATURE WILL EXCEED THE REDUCED VERTICAL RANGE.**

Z-Center Position

The **Z-Center** scale bar is located on the far left of the screen. When making height measurements it is suggested that the **Z-Scale Position** is kept close to the base line voltage represented by the line without arrows.

i) To adjust the **Z-Center Position** use the step motor feature by selecting **RealTime → Step Motor** and reduce the step size to its minimum value of 0.139 m.

ii) To move the **Z-Center Position** up and down select **Tip Down** and **Tip Up**, respectively.

14. IMAGE CAPTURE

i)   Set the default image capture directory by selecting **RealTime → Capture Filename** and selecting the correct directory. Enter a **Filename** for the images you intend to capture. Click **OK**.

ii) Once the scan parameters have been optimized, select the **Frame Up** icon or **Frame Down** icon depending on where the current line is with respect to the bottom or top of the scan area, respectively, to begin the image capture process.

iii) The Capture controls are located under the **RealTime** menu (or using the Capture icons). Use the **Capture** icon to turn on the Capture. Use the **Capture Now** icon to take the current image. Use the **Abort Capture** at any time if needed.

NOTE: Changing scan parameters during a scan while capture is on may result in a partial image capture. It is best to adjust the scan parameters before attempting to capture and image.

15. WITHDRAW

i) When finished imaging the sample, select **RealTime → Withdraw** (or click on the **Withdraw** icon) to withdraw the tip from the sample surface.

ii) Access the Navigate window using the steps given in §7 of this manual. Select **Z Motor UP Arrow** icon to increase the clearance between the tip and the sample for safe removal of the sample.

iii) If imaging another sample, insert the new sample and restart from §7 above. If a new tip is needed restart from §3 above.

iv) Otherwise, save your work and close the Nanoscope v6 software, log out of Windows and turn off both controllers. Remember to turn **OFF** the vacuum chuck when finished.

16. IMAGE POST-PROCESSING

Please refer to the manuals in the lab or the PDFs located in the manuals link on the Windows desktop.

17. REFERENCES

i) Veeco SPM Training Notebook revision E, 2003 (manual can be found using the Nanoscope manuals link on the Windows desktop).

III. FREQUENTLY ASKED QUESTIONS

<u>Pre-Engage Issues</u>

MOST PROBLEMS THAT OCCUR BEFORE ENGAGING THE SURFAFCE ARE DIRECTLY RELATED TO THE SETUP PROCESS, I.E. TIP MOUNTING & LASER ALIGNMENT.

i) During the laser alignment in §4 the signal sum is always less than the nominal value (which is tip dependent). How can this be fixed?

This can result from many things: damaged tip/cantilever, improperly mounted tip or laser incorrectly aligned.

- Before starting over from §3 verify the laser position. Back the laser off of the tip and realign the laser once or twice.

- If the signal sum is still not above the nominal value, remove the tip holder from the scanner head and verify the tip is mounted correctly in the holder, i.e. be sure the tip is in firm contact with the groove (one side must be flush with the corresponding side of the groove). After remounting the tip attempt aligning the laser again.

- If the problem persists and it is certain that both the tip mounting and laser alignment steps were done correctly, change your tip to a new one and start over.

ii) During the Auto Tune process, an error occurs not allowing the Auto Tune to finish. Verify the initial Auto Tune controls have been set as described in §9 above before investigating further.

This issue can be related to the any of the problems listed in (i) above including improper photodetector alignment (verify this before attempting any of the above suggestions).

- If the photodetector is aligned properly and the Auto Tune will not work, work through the above bullet points in (i) above.

iii) When engaging the surface, nothing happens. It appears to be engaged because the Engage icon is depressed but nothing is happening.

As with the other problems, this issue can be related to the setup process with the tip and laser alignments, but more frequently it is due to improper focusing on the surface.

- First click the Withdraw icon to disengage the surface.

- From here, go back to the Navigate window and verify the surface has been focused properly. Sometimes it helps to cycle between the two **Focus On** options (Surface and Tip Reflection).

- Usually approaching the surface a little more and reengaging the surface will solve this problem

Imaging Questions

i) What controls the imaging resolution?

- The image resolution is dependent on two things: the tip geometry and the scan size and number of samples per line.

- The tip geometry can affect the image resolution. Usually, if a feature is smaller than the tip diameter then the image will exhibit the shape of the tip.

- The scan size and samples per line can be used to determine the image resolution by dividing the scan size by the number of samples per line, e.g. if the scan size is 10 m and the number of samples per line is 512 then the minimum feature dimension that can be resolved is approximately 19.5nm.