

Hysitron Ubi 1 Operating Procedure

Start-up

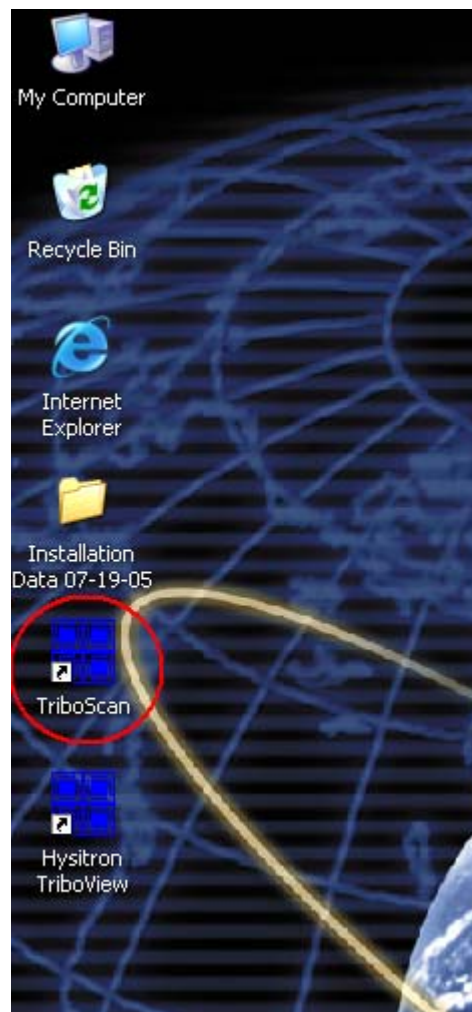
- Turn on the Computer and monitor
- Turn on the Z-axis motor controller(1), Piezo controller(2), and Transducer controller(3) in that order. The reverse order is used during shutdown.



- After the Transducer controller has been turned on, turn the display gain to 1 and check the LOAD value. It should be around 320 depending on the tip being used. If the value is close to 320 then press the auto adjust button and turn the display gain knob to 100. See picture on next page.



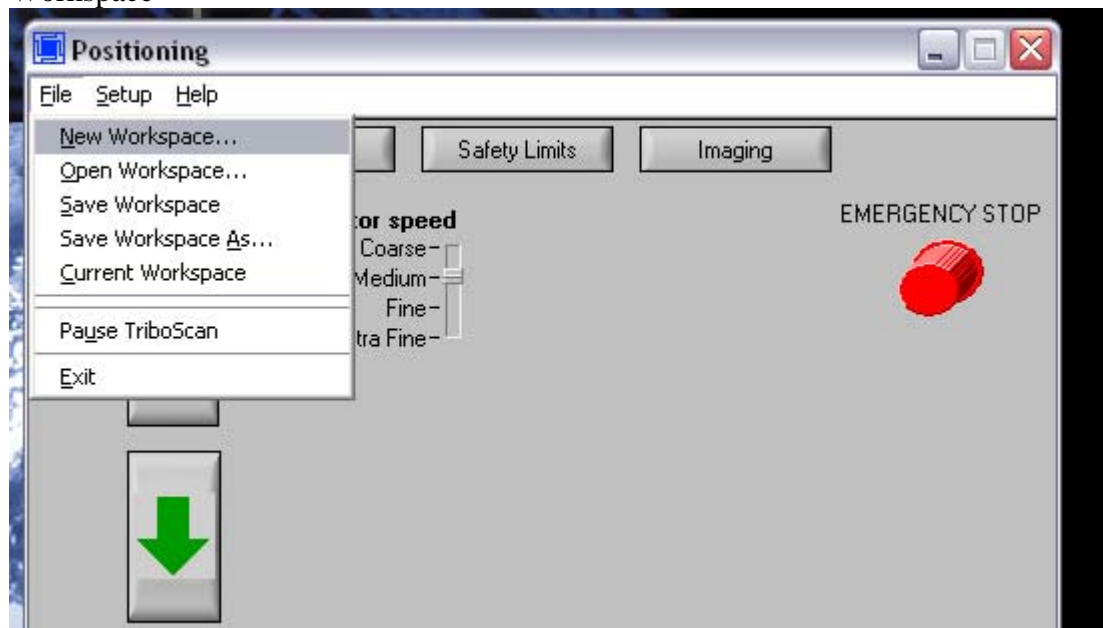
- Once the computer boots, you are ready to access the TurboScan program. Double click on the TurboScan Icon in the middle left of the screen.



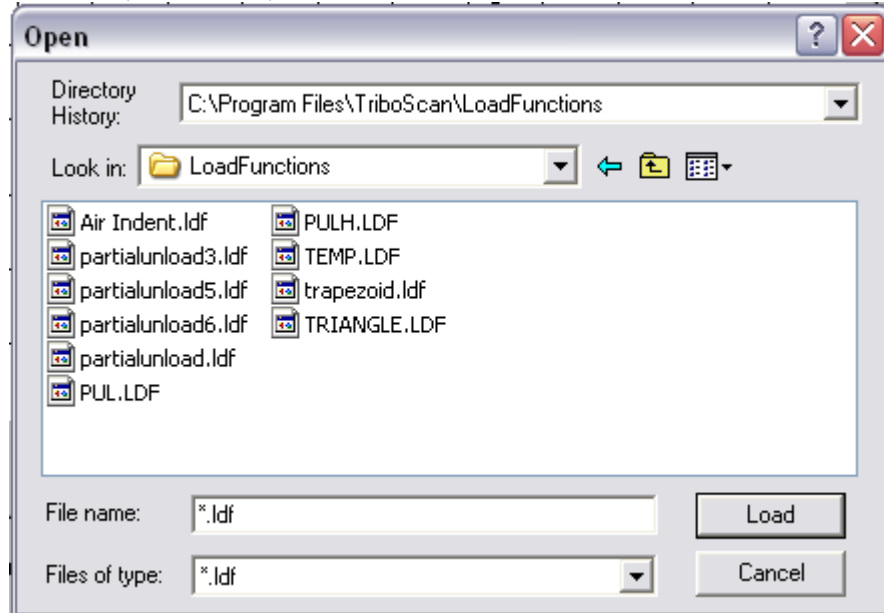
- At this point a dialog box will appear in the center of the screen, you can either click cancel or allow the clock to expire to start the program, or you can click Analysis Only to use the analysis program without starting the machine.



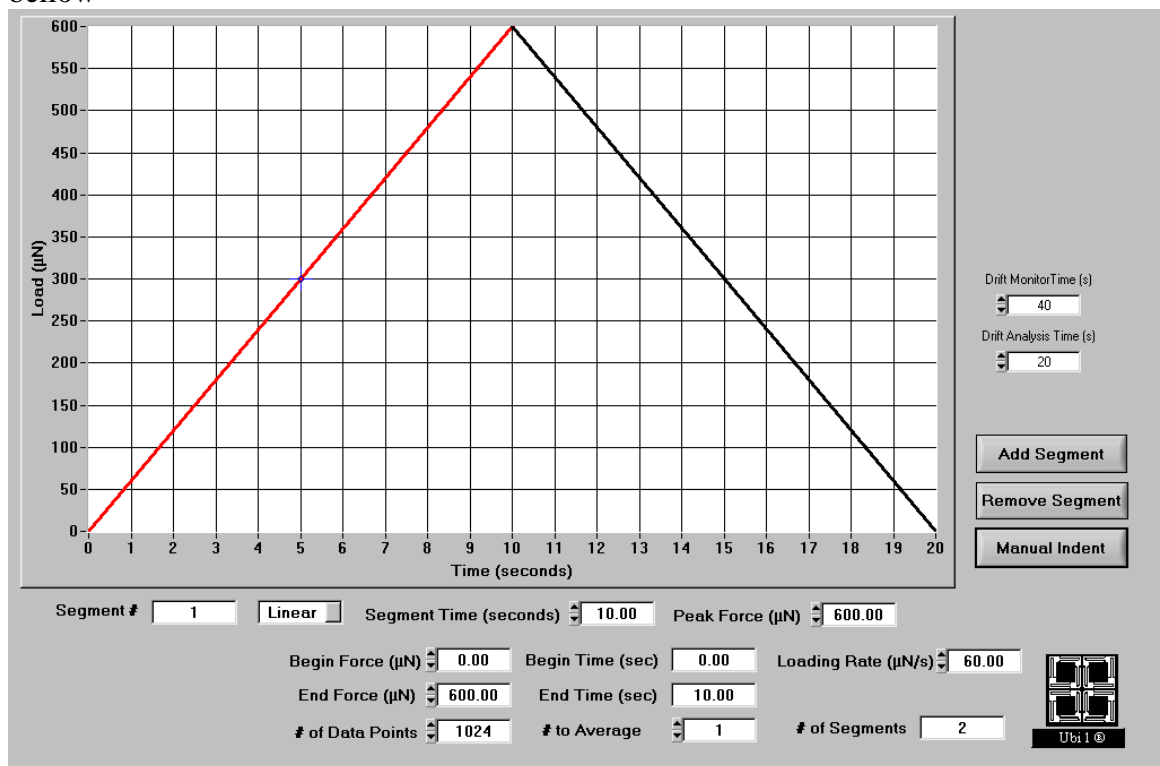
- Once the clock expires or you click cancel the machine will proceed to the startup procedure. Once it has finished this process it will ask you to re-zero the front display, once you have done this click “OK”
- If this is your first time to access the program than you need to set up your workspace. In the Positioning window click on file and then on New Workspace



- When the file menu opens, open the C:\Program Files\TriboScan\LoadFunctions file and select Air Indent.ldf

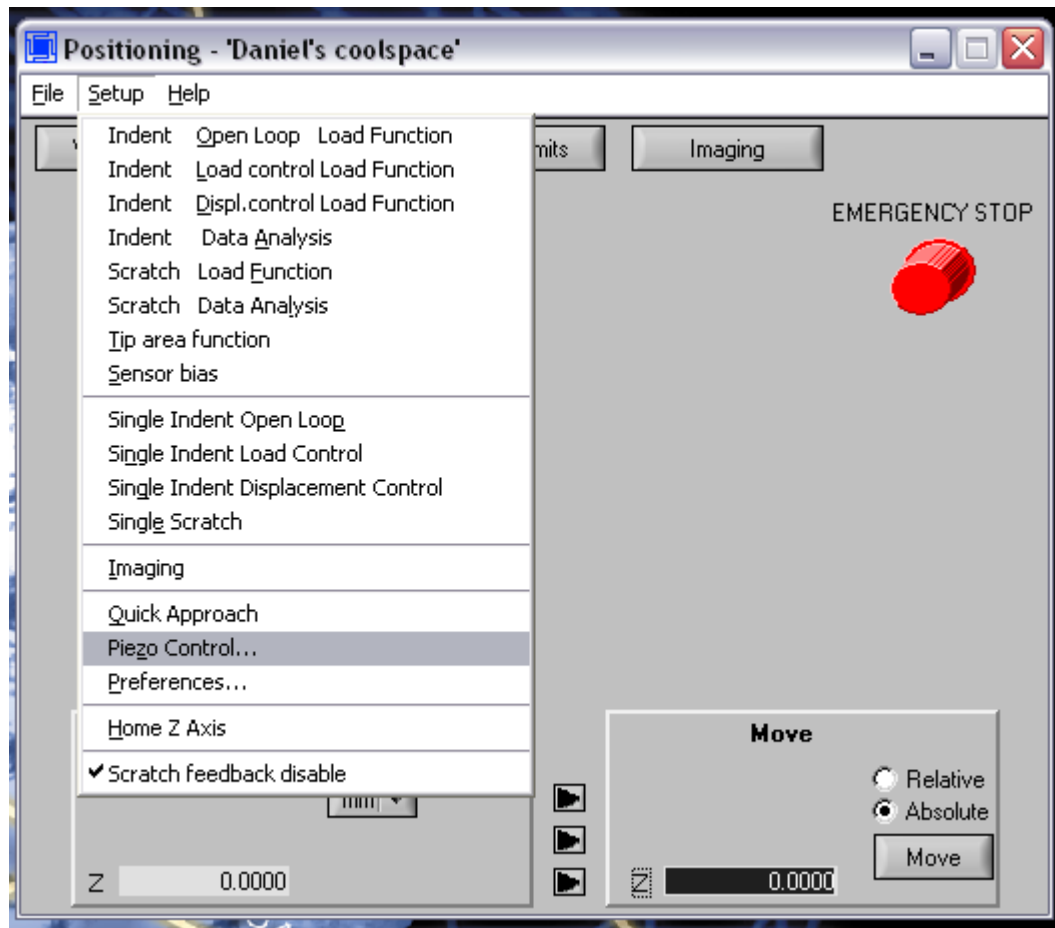


- Double check the parameters of the air indent load function so they match the bellow

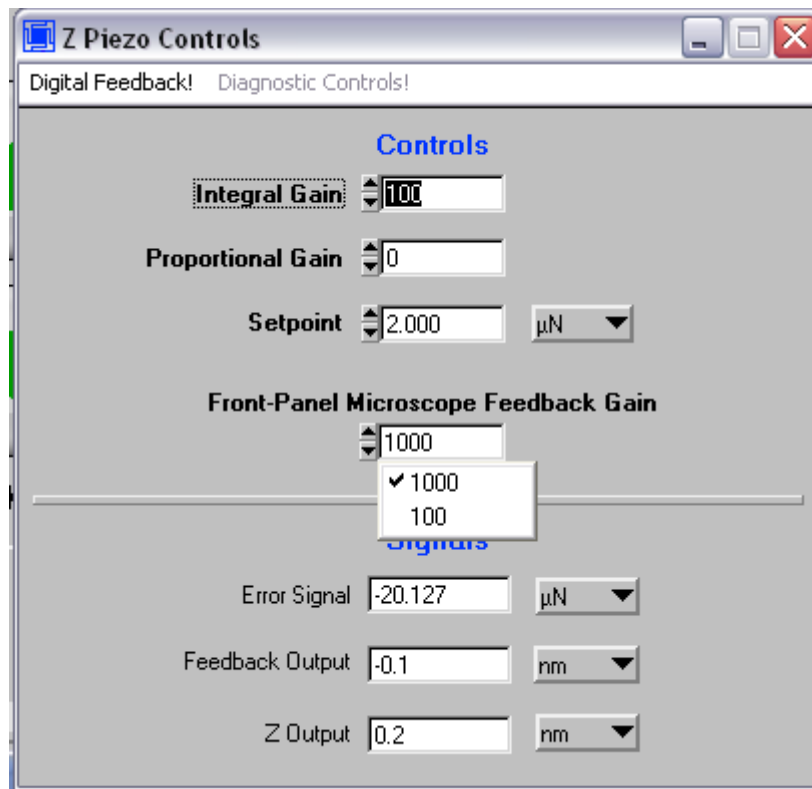


- Ensure that both segments are 10 seconds in duration and the peak force is 600µN
- DO NOT SAVE OVER THE AIR INDENT FILE

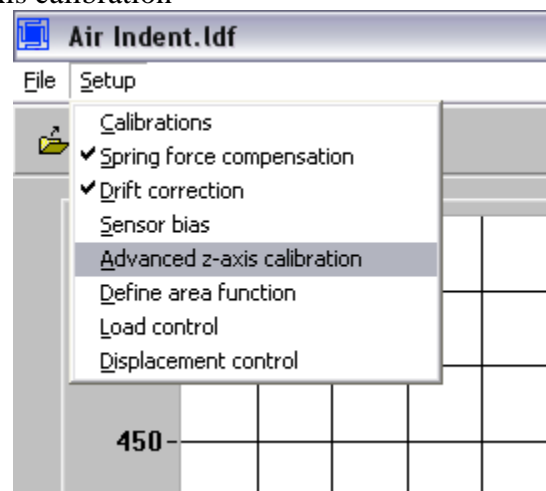
- Next go back to the position window, and under setup click on “Piezo Control”



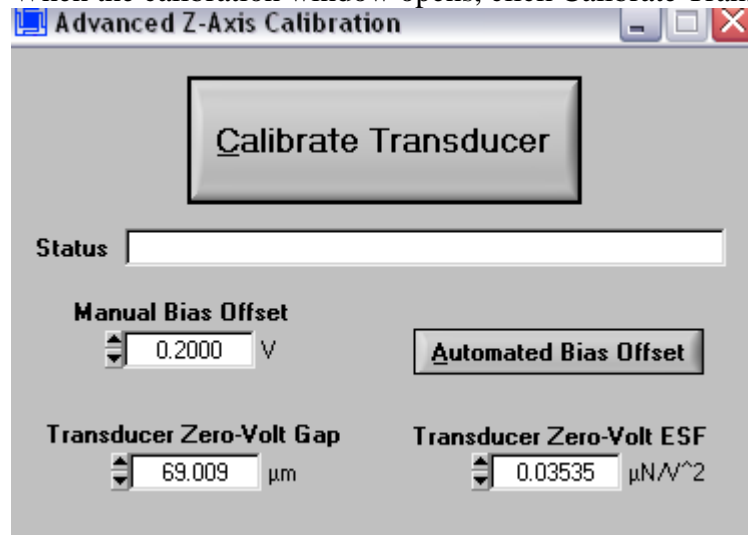
- Here we need to adjust the Microscope Feedback Gain to 100, this MUST be done BOTH in the computer and on the transducer controls! See pictures on the next page



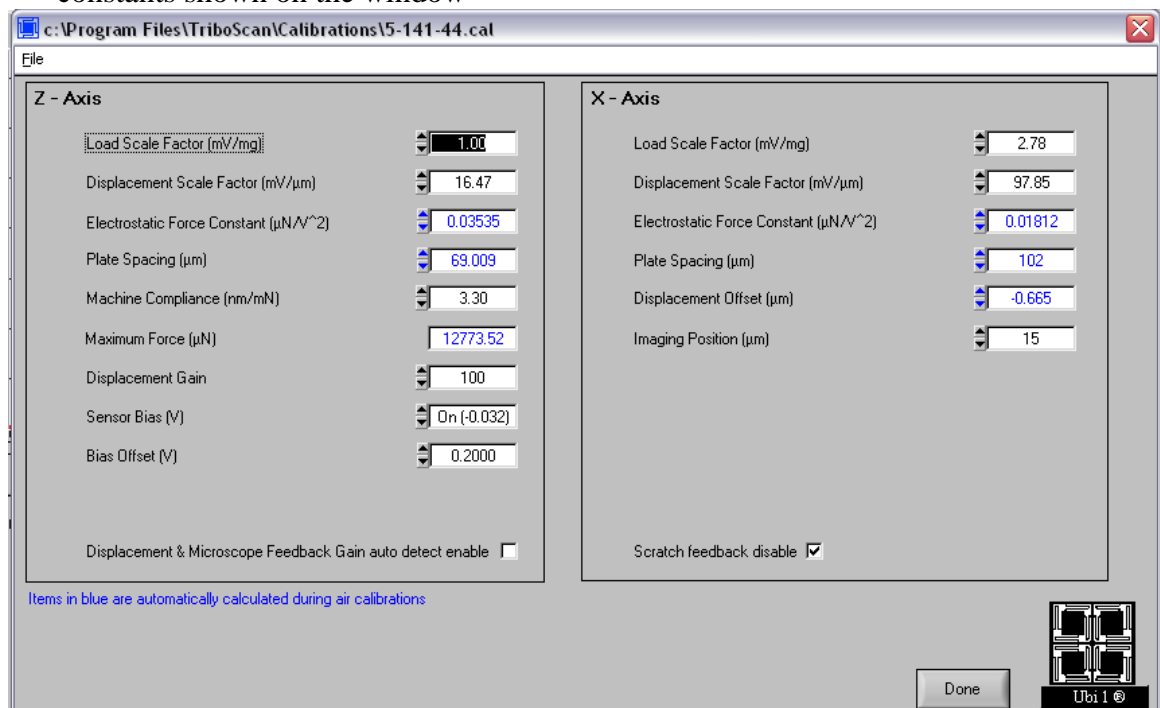
- Once you have adjusted the microscope feedback gain, you are ready to calibrate
- Go to the air indent load function window and under the setup menu click Advanced z-axis calibration



- When the calibration window opens, click Calibrate Transducer



- Once you have checked that there is at least 5μm between the tip and any surface, click yes
- When the next window opens you will need to retrieve the Transducer Calibration Constants sheet, double check the Z Axis constants with the constants shown on the window



- The numbers in black are constants, and the numbers in blue are calculated. Any numbers in black should match up exactly with the numbers on the sheet, and the blue numbers should be close

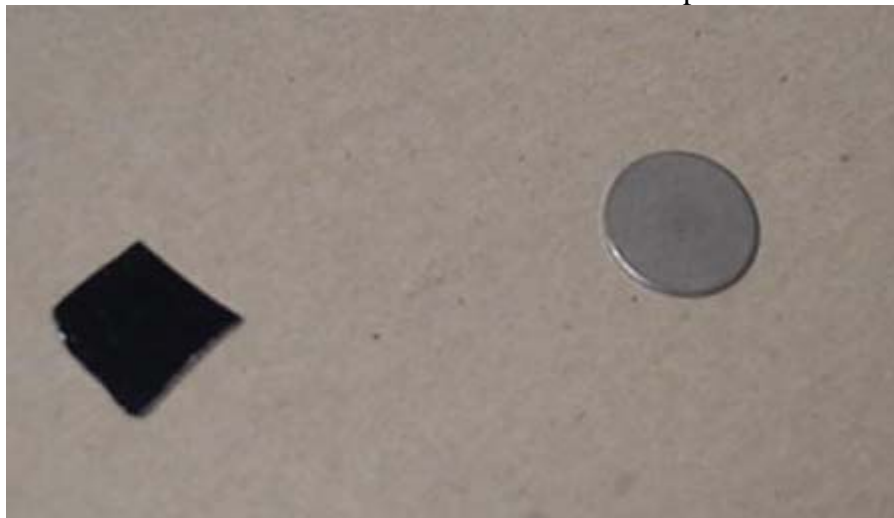
- Also check that the displacement gain is set to 100 BOTH in the window and on the transducer control panel. Hit done once you have checked the constants
- All that is left is to follow the dialog boxes
- When the program prompts you to set the microscope feedback gain to 1000, go ahead and re-zero the front panel and then click ok. Now you will need to go back into the piezo control menu under setup in the positioning window to reset the microscope feedback gain to 1000 BOTH in the computer and on the transducer controls
- The program is now ready to approach the sample, this ends the startup procedure

Shutdown

- To shut down the system you must first remove the tip from the sample, and close the imaging window
- Close the positioning window, once the program has completely closed you can power down the transducer control, the piezo control and then the Z axis motor control, in that order.
- Shut down the computer and turn off the monitor.

Sample Preparation and Approach

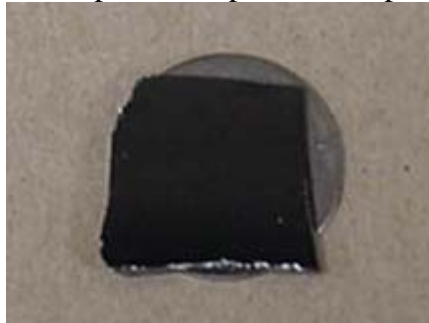
- The nano-indenter can handle sample sizes of about 2cm^2 , if your sample is larger than this it will need to be cleaved or cut to fit on the puck.



- Ensure that the puck is face up, there will be a slight rounding of the top edge of the puck due to the manufacturing style (stamped). If this edge is not facing the sample it will cause a very slight “rocking” of the sample during testing
- Use a small amount of super glue on the puck



- Place the sample on the puck, face up

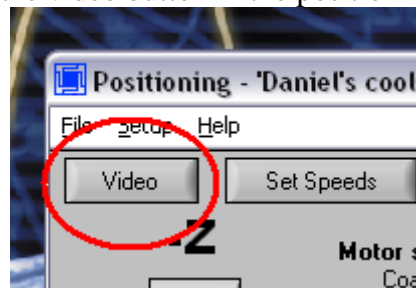


- Press the sample down on the puck using a Kimwipe for thirty (30) seconds, then allow the glue to dry for several minutes.
- Place the puck/sample on the stage near an edge. There is a strong magnet at the center of the stage that holds the puck in place, trying to place the sample directly on the magnet could rip the sample from the puck. Slide the puck towards the middle of the stage and allow the magnet to pull it to center.
- Use the micrometers on the stage to roughly align the sample under the tip, the Z motor should still be at the origin with the well away from any possible contact. Be very careful not to accidentally touch the tip, as the slightest contact could damage the tip or dislodge it, replacement cost is about \$2000.00

- Once the sample is in position under the tip, you need to make sure that the floating palate is properly adjusted. Open the bottom door on the Ubi 1 and turn the load adjust knob until the positioning bar is aligned in the center

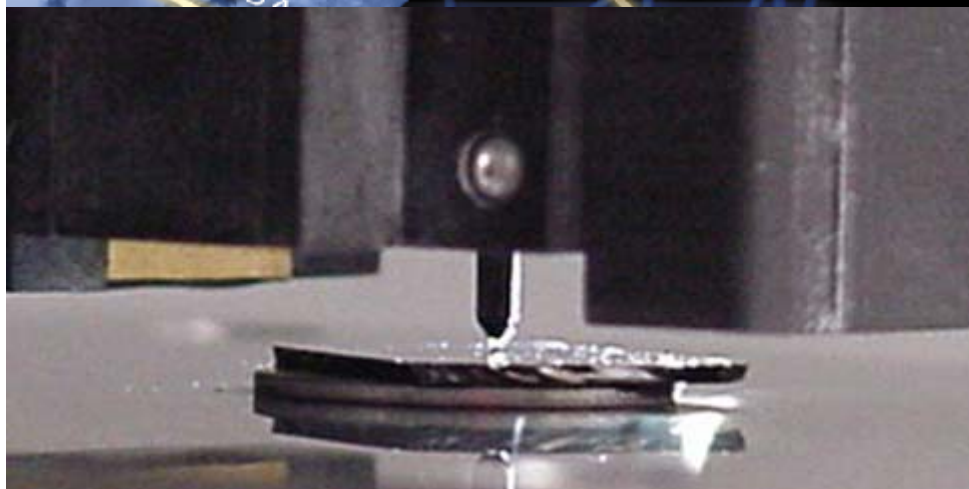
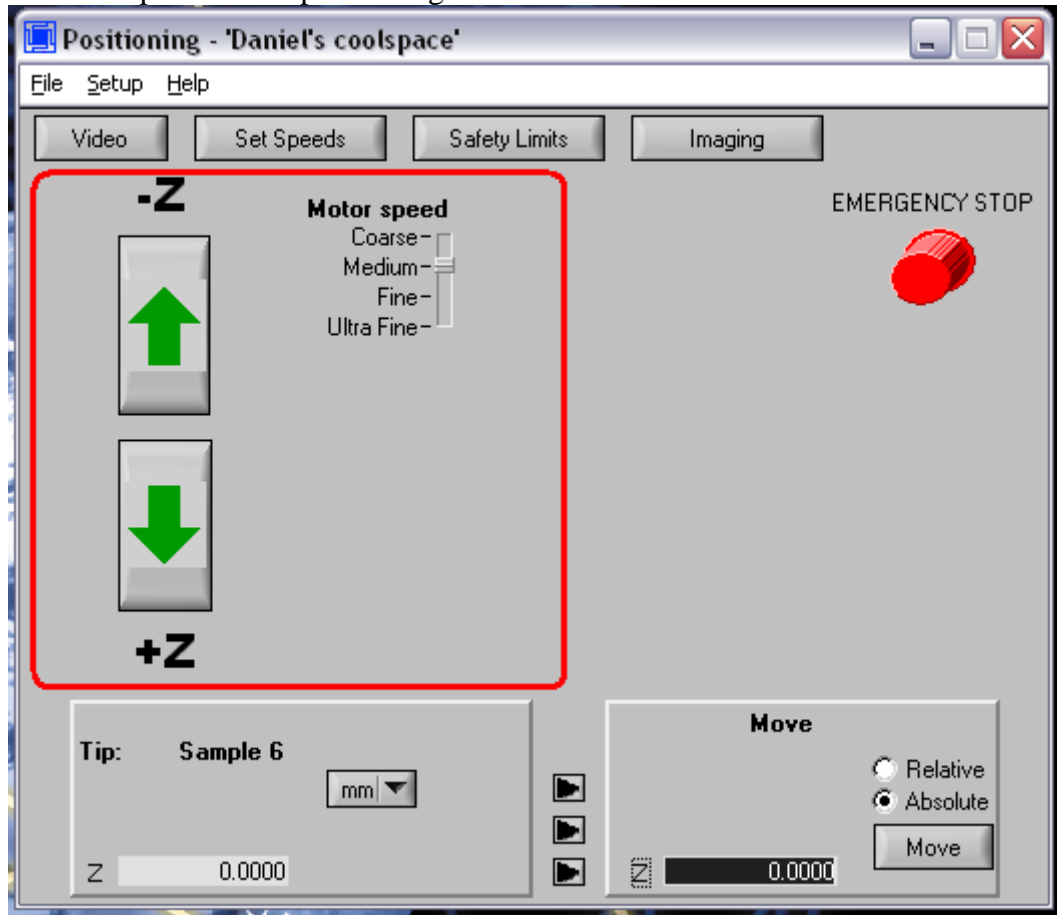


- Click on the video button in the positioning window



- Once the video window is up, make sure the tip backlight is on, the toggle switch is on the left side of the Ubi 1, when the green light is lit the backlight is on

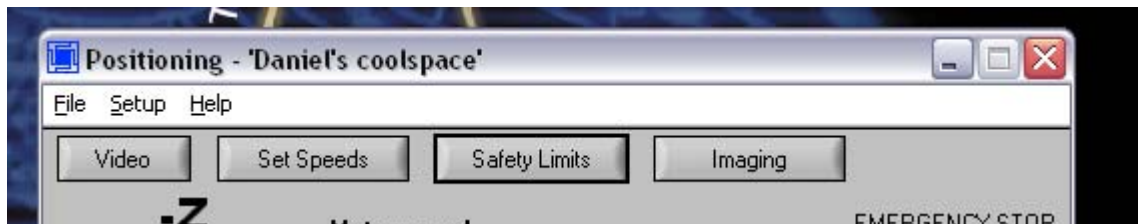
- From the positioning window, manually lower the tip until it is visibly close to the sample, be very careful not to accidentally run the tip into the sample, use the different speeds in the positioning window



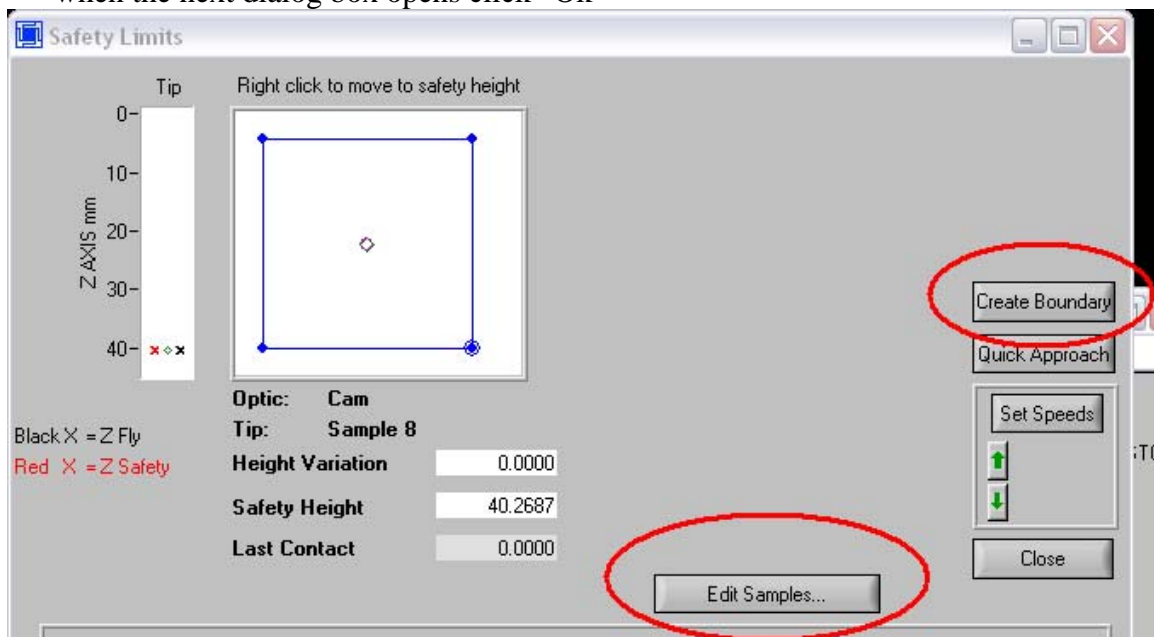
- At this point you should be able to see the tip approaching the sample in the video window, continue to approach the sample until the tip is close to sample

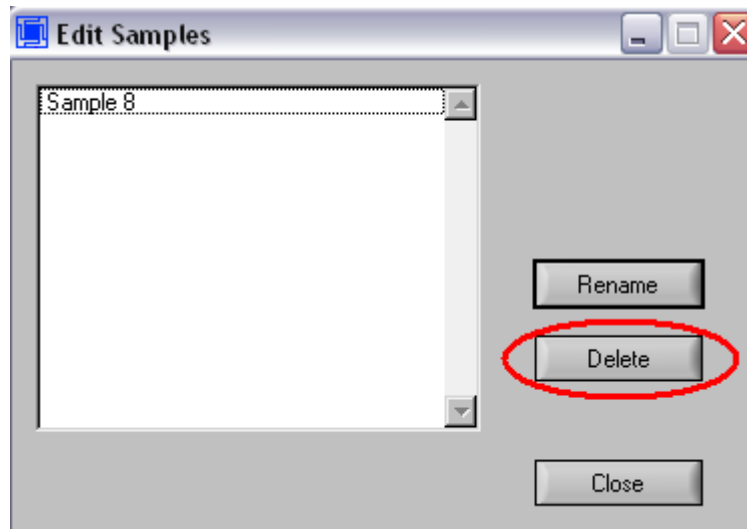


- This should be made the safety boundary. Click on the “Safety Limits” button in the positioning window

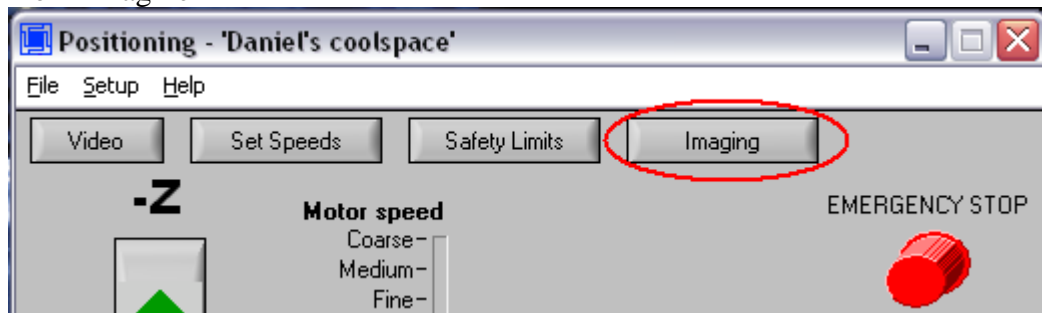


- Once in the safety limits window, click “Create Boundary, if you have previously done this you will need to erase the old boundary before you can make a new one. To do so click on the “Edit Samples” button, then click delete, and when the dialog box opens click yes. After this you can Click “Create Boundary,” And when the next dialog box opens click “Ok”

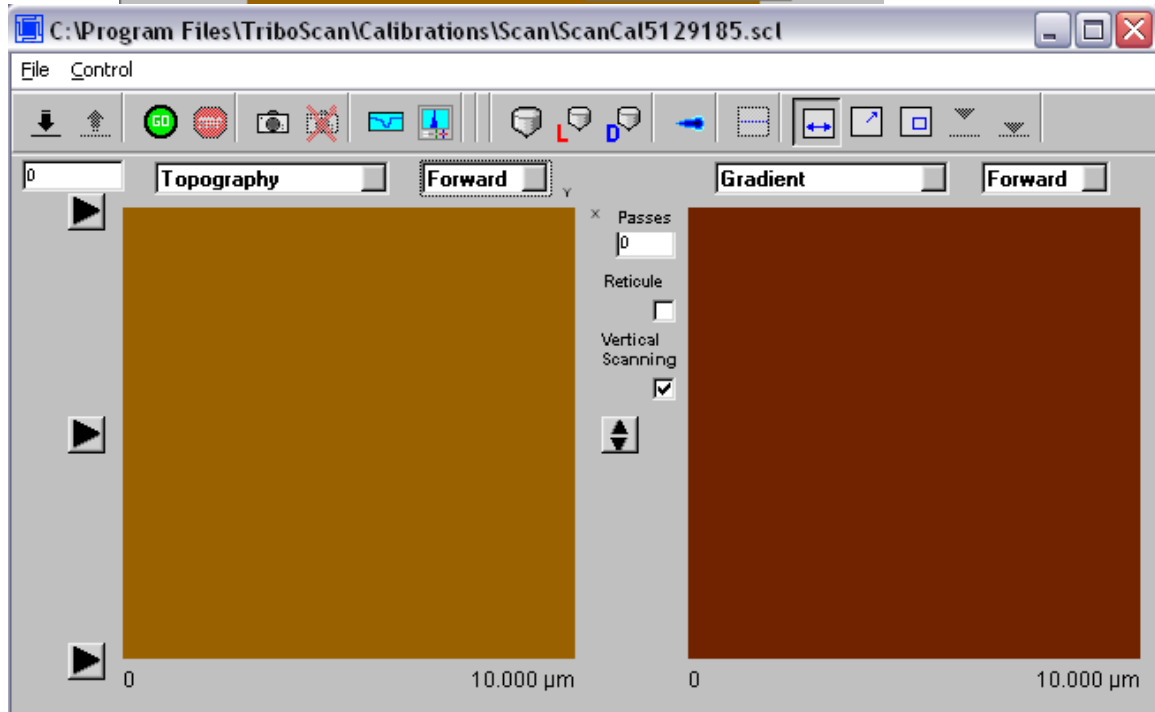
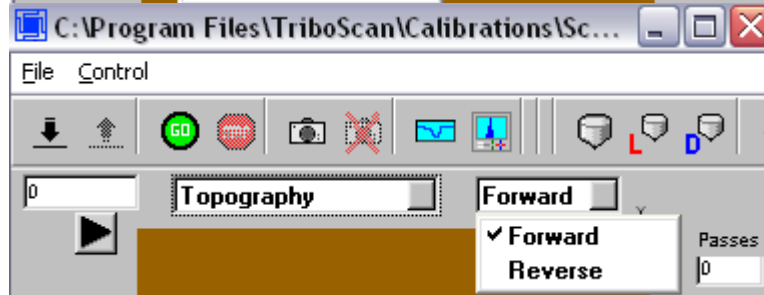
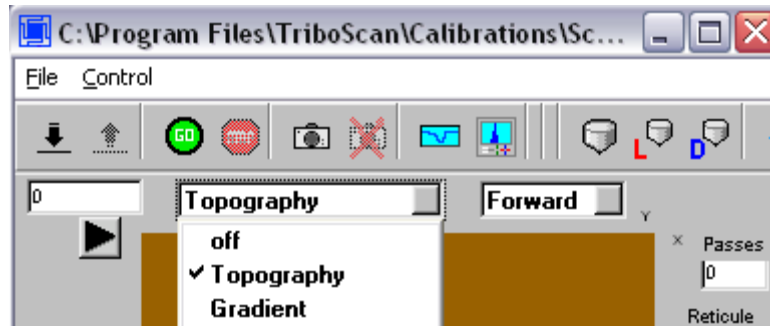


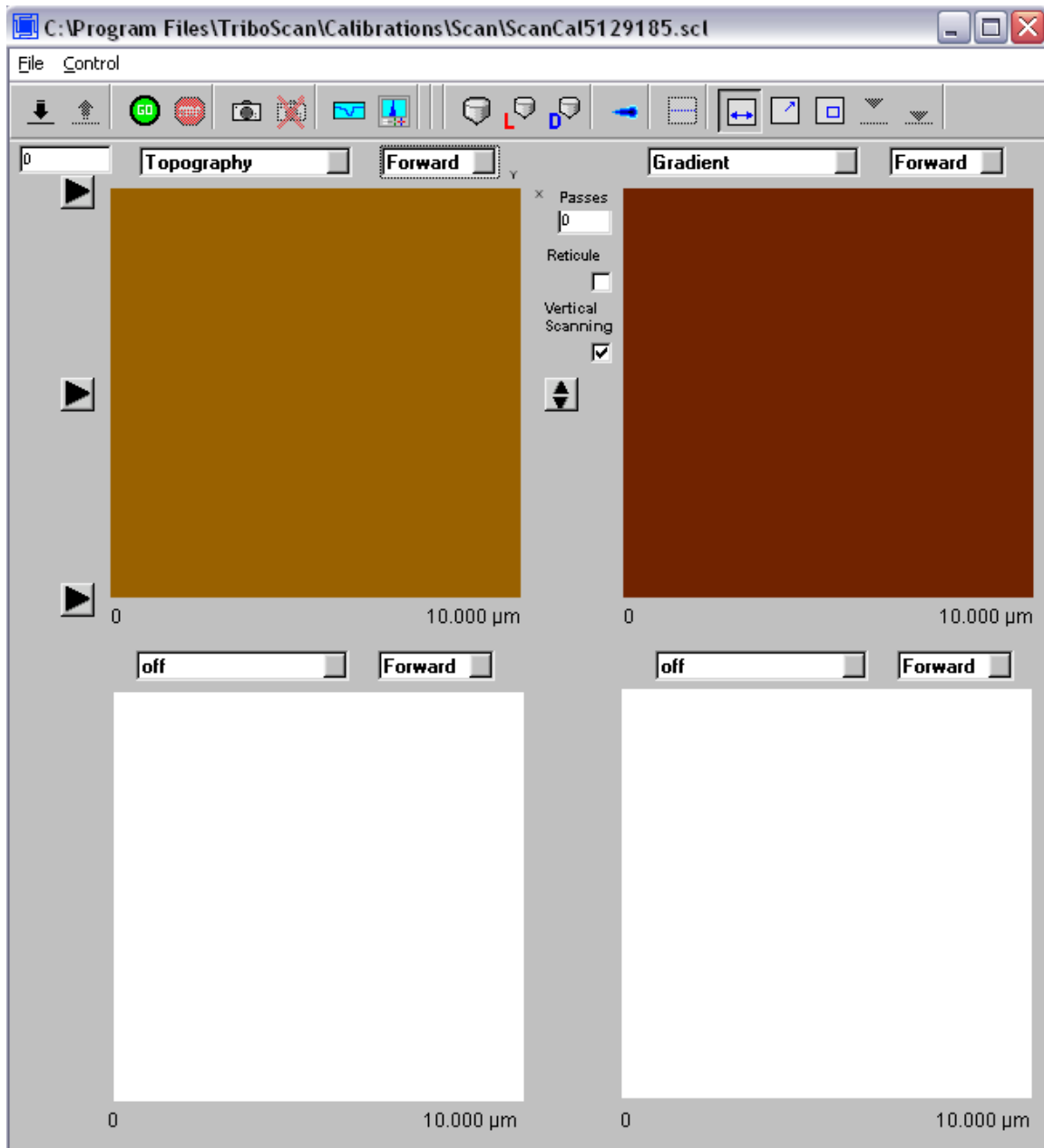


- Once you have created the boundary you are ready to make the first approach to the sample. In the Safety Limits window click on “Quick Approach”, it is directly under the “Create Boundary” button
- The quick approach option takes the tip into contact with the sample more quickly than that of normal approach. The reason for doing this is that the first time the tip comes into contact with the sample the computer remembers the z height at which it happened, so when you next approach the sample, using the normal approach, it will quickly approach a point just above the sample and then proceed to do the normal slow approach from that point. The quick approach will also leave a small mark on the sample, so it is advised to perform your indents on a different location
- The quick approach touches the sample and then goes back to the boundary height, so now we must do the normal approach. In the positioning window, click on “Imagine”

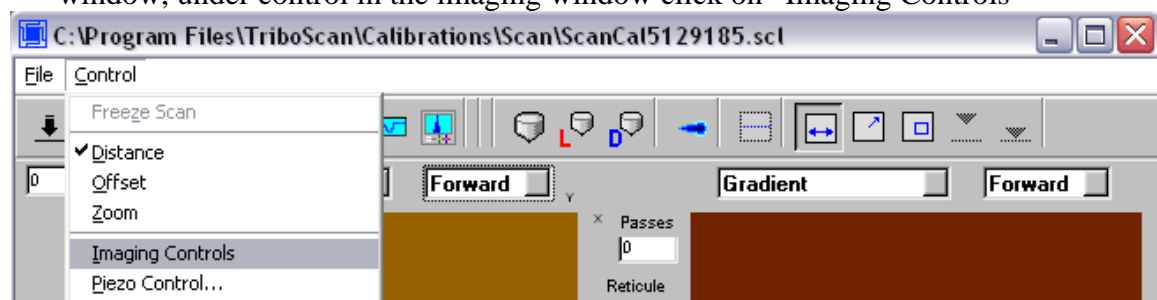


- The Imaging window is the main window from which you will conduct all of the your tests, and it is also the topographical and gradient scanning window. You may monitor up to four different sets of scanning data at once. You can monitor both topographical and gradient data from the scan, and you can monitor each in either forwards or backwards scanning. To add or remove any of the scanning regions adjust the size of the imaging window, see the pictures on the next two pages

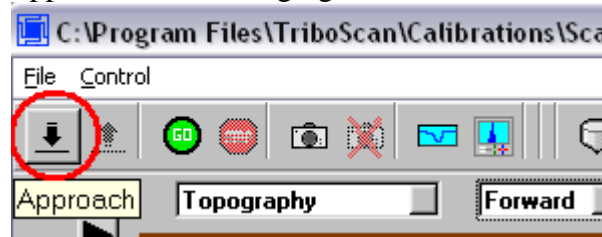




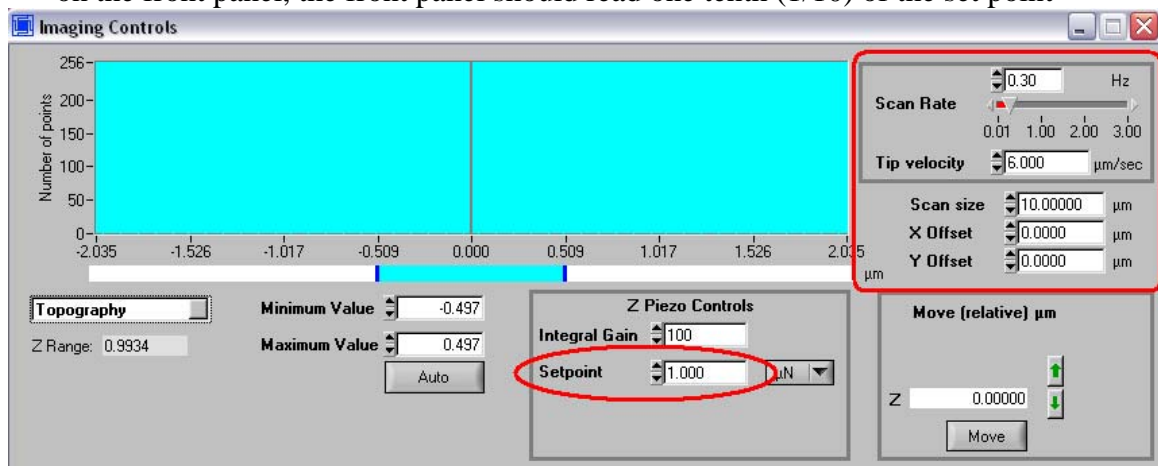
- Before we approach the sample there is one last thing, the imaging controls window, under control in the imaging window click on “Imaging Controls”



- This window is the “brains” of the imaging system, but we will come back to that later.
- Now is a good time to re-zero the front panel, after first startup the front panel has a tendency to “wander” a bit, and re-zeroing now allows for the best possible scan. You are now ready to approach the sample, simply click on the Approach button in the upper-left of the imaging window

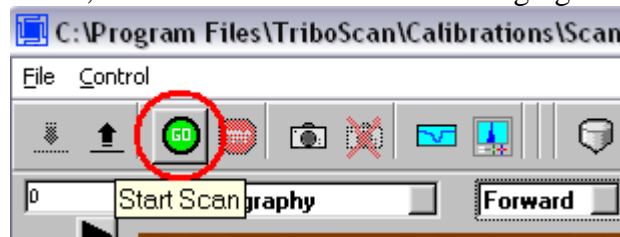


- After a few short moments you should be on the surface of your sample, congratulations, it only took 17 pages to get here!! To ensure you are actually on the surface check the set point on the Imagine Controls window and the reading on the front panel, the front panel should read one tenth (1/10) of the set point

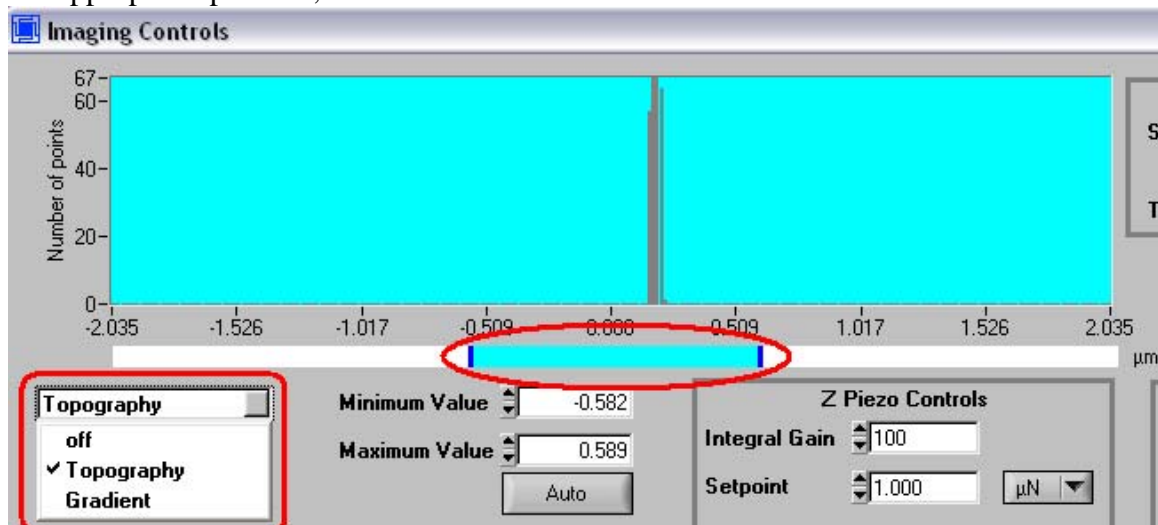


- While we're here, I will explain the purpose of the Imagine Controls window. First, the set point is the normal force applied to the sample, meaning the higher the set point the harder you are pushing on the sample. Lower set points require lower scan speeds for good image, and softer samples require lower set points.
- The scan size is the size of one side of the scan in micrometers; since the scans are square the scan area is the square of the scan size. In this case, the scan size is 10μm, so each side of the scan is 10μm and the scan area is 100μm²
- The X and Y offsets simply offset the piezo the amount desired. The Piezo is limited to 40 microns in each direction, and scanning at the limit will not produce repeatable results

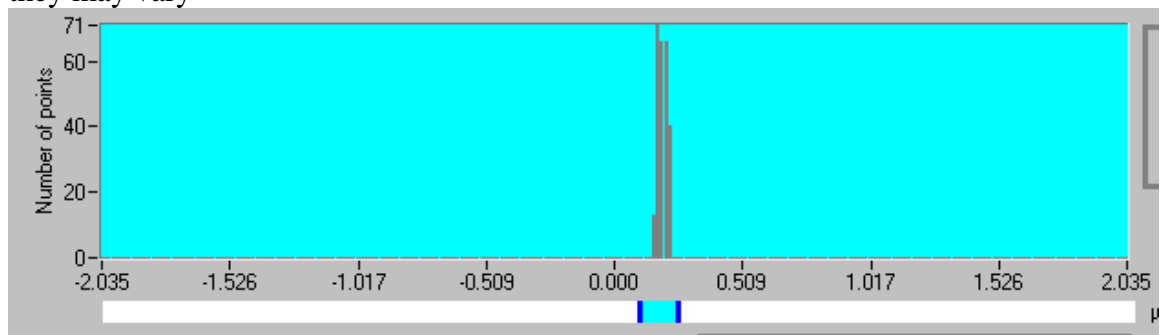
- To start the scan, click the “Go” button in the Imaging window



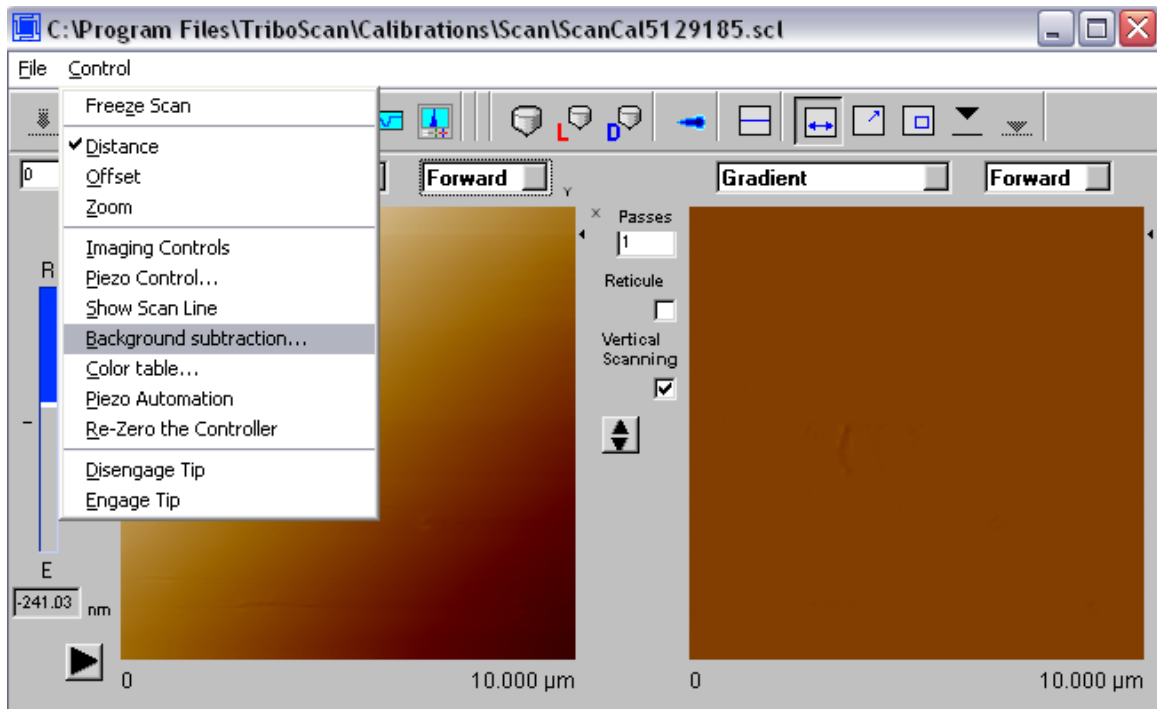
- To get a good image you must adjust the range of the scan. Essentially we need to tell the computer to focus in on a certain Z range, if the range is too large the details of the scan get washed out, and if it is too small any details outside of the range will be truncated. To adjust the range slide the range bars under the main graph in the Imagine Controls window to just on either side of the gray bars in the graph. To do this click and hold on the dark blue bars and slide them to the appropriate position, see bellow



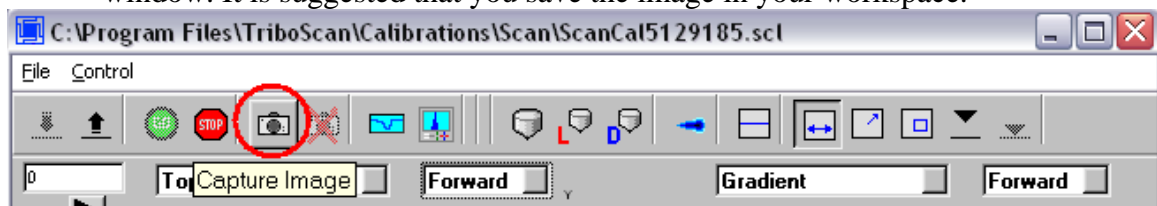
- Also, notice that you must adjust topography and gradient scans independently, as they may vary



- Due to the fact that it is virtually impossible to make a sample that does not have some slight slope to the surface, there will be a sort of fading to the topographical image. To account for this we will turn on background subtraction. Go to control in the Imagine window and click on “Background Subtraction,” see next page



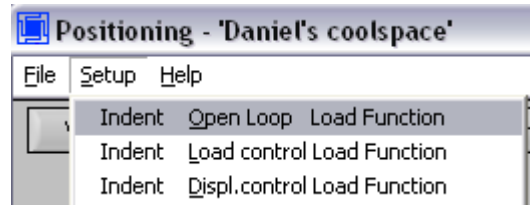
- In the Background Subtraction window, chose the desired type of background subtraction, linear regression in this circumstance normally works best. You must now re-adjust the range.
- If you wish to save a scan image(s), click on the camera icon in the imaging window. It is suggested that you save the image in your workspace.



- This concludes the sample preparation and approach

Open Loop Indent

- The open loop indent is the simplest and easiest indent to perform. It is called open loop because it does not use feedback during the indent.
- In the positioning window go to setup and click on “Indent Open Loop Load Function”

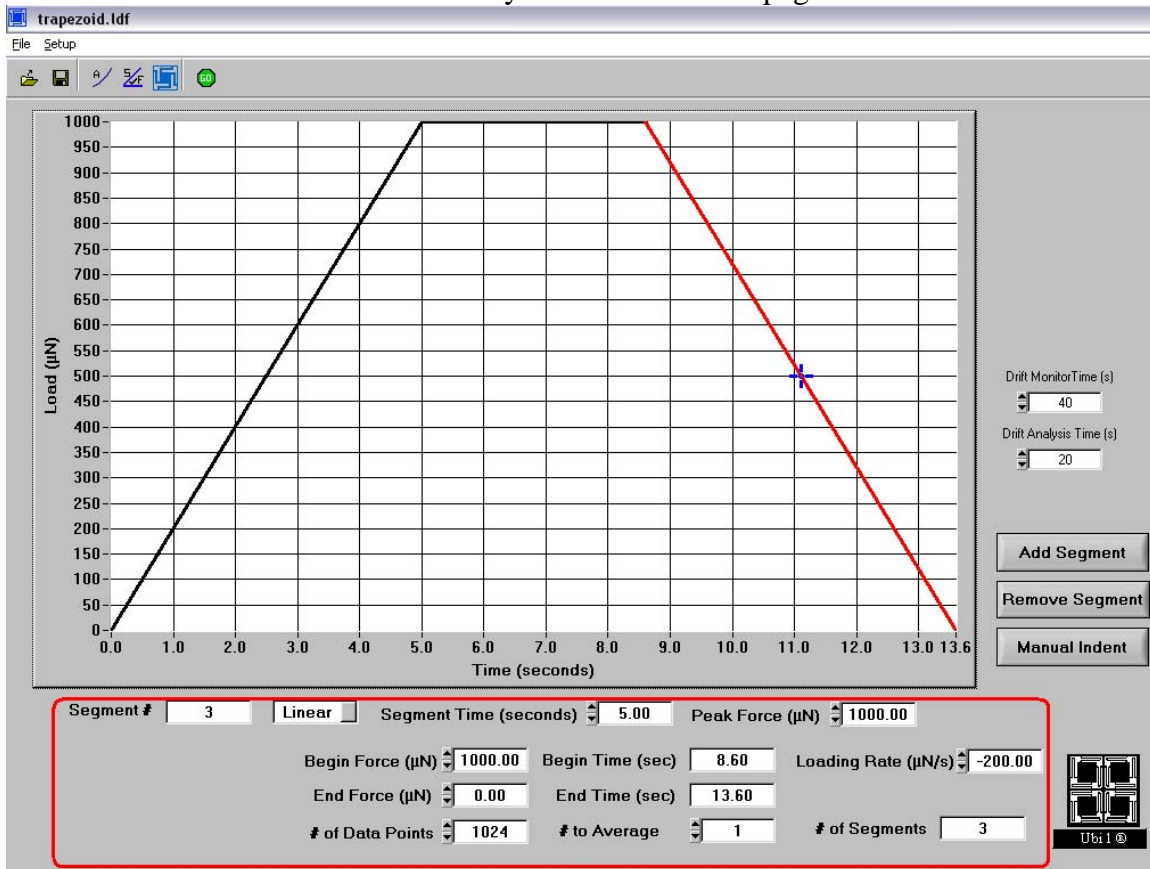


- The window that opens should look familiar, as we were using it at the beginning of this OP. Adjust the load function as desired and save is, DO NOT SAVE OVER THE AIR INDENT LOAD FUNCTION!
- A typical indent will be a trapezoid, having a loading segment, and holding segment and an unloading segment, see bellow

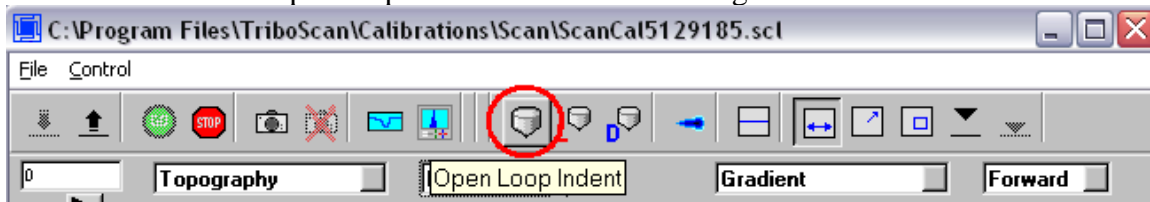


- You can also use “saw tooth” functions, which will do a series of loading and unloading segments. Or a triangle load function like the air indent load function

- You can adjust the number of segments, the segment time, and the beginning and end force of each segment as desired. It is suggested that you do not alter the Drift Monitor Time or the Drift Analysis Time. See next page



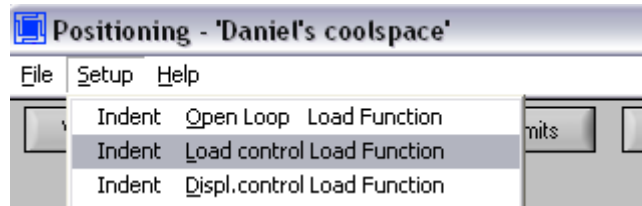
- Once you have the load function set up, you are ready to indent. All that is left is to click the "Open loop Indent" button in the Imagine window



- Once the dialog box appears, confirm that you have the load function setup properly, and the indent will start. Once the indent has started it will take several minutes to complete. After the indent the Ubi 1 will resume scanning. This concludes the open loop indent procedure

Load Control Indent

- The load control indent is a feedback mode indent. This indent uses feedback to monitor the load place on the sample. It is typically more accurate than an open loop indent we deeper indents are necessary.
- In the Positioning window, go to setup and click on “Indent Load Control Load Function”

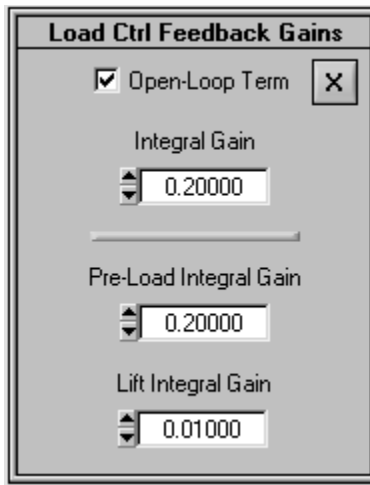


- The load control load function window is similar to the open loop function window, but the load function differs in a few specific ways
- This load function lifts the tip from the sample before the indent, and you must specify this lift height. The lift height should be at least several times the roughness of the sample
- Also, this load control can only do trapezoid and triangle load functions. You must specify the maximum load, loading rate, pause time, and unloading rate. You chose a pause time of zero you will have a triangle load function
- The preload should be at least the set point of your scan, if not higher, the preload should not be so high that it “pre-indents” the sample
- Again, it is suggested that you do not adjust the Drift Monitor Time or Drift Analysis Time

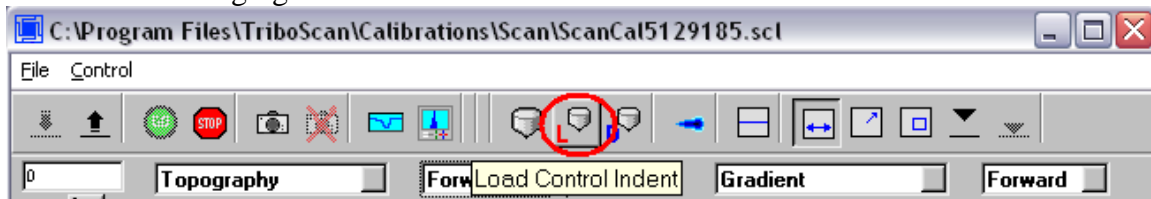
Parameter	Value
Lift Height (nm)	10.000
Maximum Load (μN)	50.000
Loading Rate (μN/s)	5.000
Hold Time (s)	5.000
Unloading Rate (μN/s)	5.000
Total Time (s)	25.000
Pre-Load (μN)	1.000
Drift Monitor Time (s)	40
Drift Analysis Time (s)	20

- Nothing will appear in the graph until after you run the indent

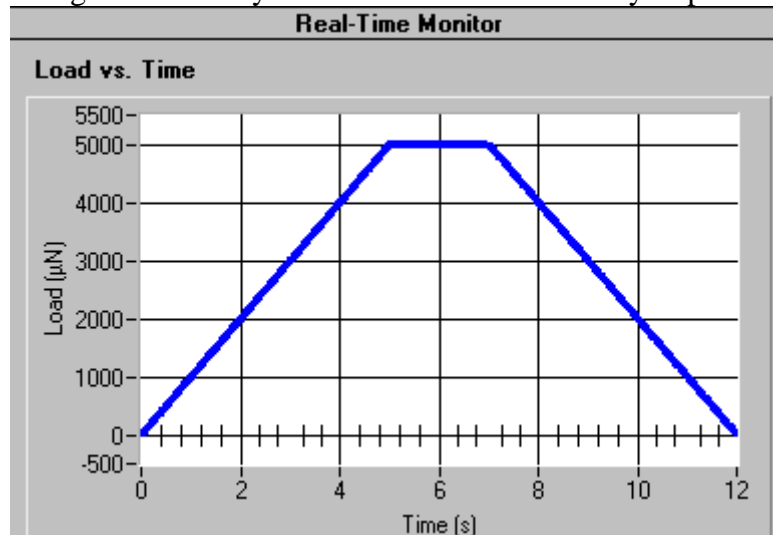
- The next part of the indent is the trickiest part. Because this is a feedback load function, you must adjust the load control feedback gains, but we will come back to this in a moment



- Once you have the load function set up, click on the Load Control Indent button in the Imaging window



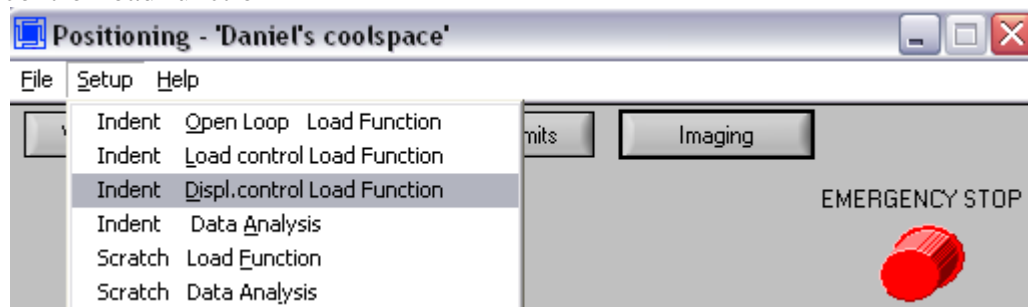
- Once you have confirmed that the load function is set up properly, click “start”
- While the indent is running a real time monitor window show you the progress of the indent. This will be in the form of a red line “traced” blue dots. The blue dots are real time data coming back, and the red line is the intended load function. If the blue dots do not follow the red line, than you need to adjust the load control feedback gains until they follow the red line as closely as possible



- Once the indent is finished, it will continue to scan. This ends the Load control procedure

Displacement Control

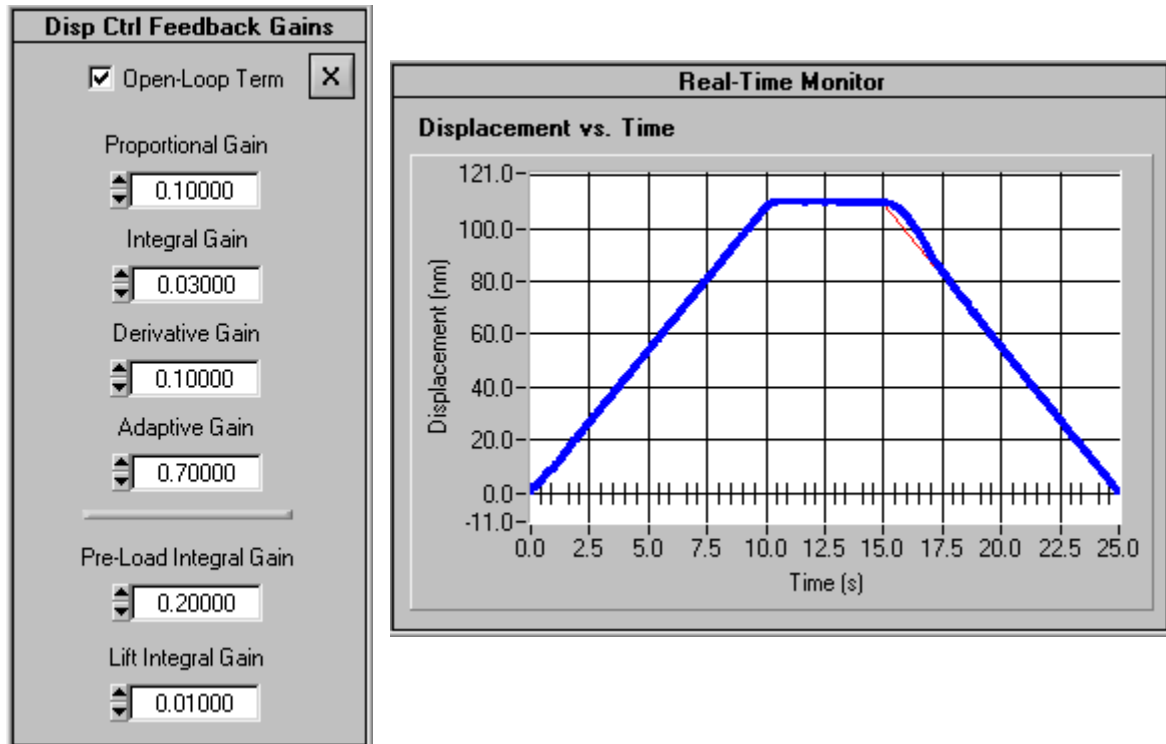
- Displacement control is very similar to Load control, except that instead of going to a specific load you go to a specific depth. This type of control is very useful for indenting into thinner films where accurate depth is key to getting good data. The downside, however, is that it is more difficult to properly adjust the feedback gains
- To start, go to setup in the positioning window and click on Indent displacement control load function



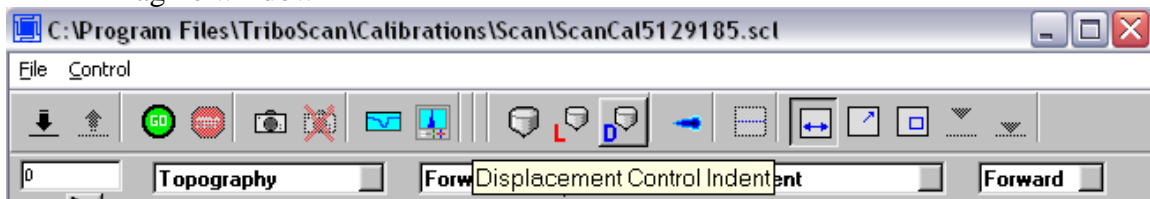
- This will open a window similar to the load control window. Again, set the lift height appropriately for the given roughness of the film. Then adjust the forward displacement, approach rate, hold time, and withdraw rate for the desired indent
- The preload should be set to at least that of the set point for the topographical scan, but not so high as to pre-indent the sample. Again, it is suggested that you do not adjust the Drift Monitor Time or Drift Analysis Time

Lift Height (nm)	10.000
Forward Disp (nm)	110.000
Approach Rate (nm/s)	11.000
Hold Time (s)	5.000
Withdraw Rate (nm/s)	11.000
Total Time (s)	25.000
Pre-Load (μN)	1.000
Drift MonitorTime (s)	40
Drift Analysis Time (s)	20

- On the right side of the window you will see the feedback gain controls, and there are quite a few more of them for this type of indent. Adjust them as necessary to have the indent follow the trace as with the load control indent



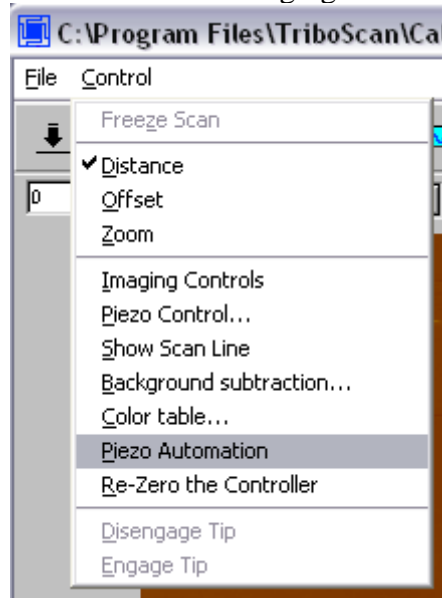
- Notice how in the above trace the blue line does not quite follow the red line. This can affect the results, as the unloading curve is not linear. Such discrepancies should be minimized
- To start the indent, click on the “Displacement Control Indent” button in the imagine window



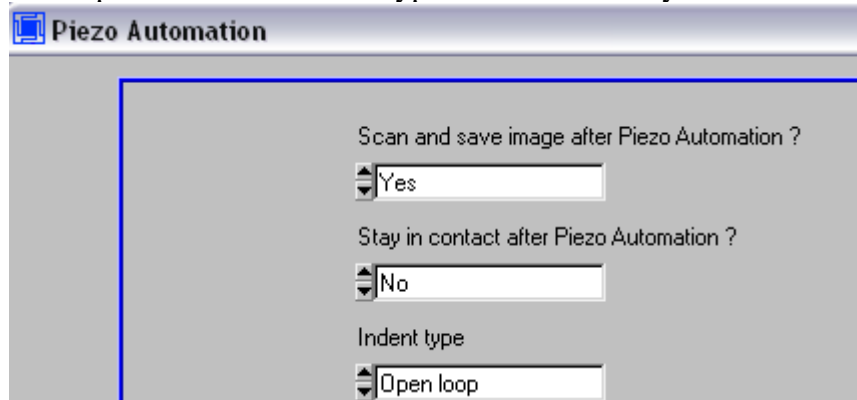
- Once you confirm the load function is correct, the indent will begin. Once the indent is done scanning will resume. This ends the Displacement Control procedure

Peizo Automation

- Peizo automation is an automated set of indents, or an indent array. This should be used for final analysis of a sample. Once you have set up the desired load function, go to Control in the imaging window and click on “Piezo Automation”



- Once the piezo automation window opens there are several selections to make. First of which is if you would like to scan and save the image after the indent array is complete, this is suggested. Next, would you like to have the tip stay in contact with the surface after the array is complete, it is highly suggested that you do not do this unless you will be working with the sample immediately after the array is complete. And then which type of load function you would like to use



- Next, you must chose the number of indents and spacing between the indents in both the x and y directions. It is suggested that you make the spacing between the indents several times the size of the indent, this will help prevent any interference caused by the disruption of the film. And next would be to pick the type of pattern run, either constant direction or serpentine. Serpentine is suggest to reduce piezo drift. See image on the next page

Indentation Array Parameters

Number of Indents in X	<input type="text" value="2"/>	
Spacing Between Indents in X	<input type="text" value="10.0000"/>	μm
Total Distance in X	<input type="text" value="10.0000"/>	μm
Number of Indents in Y	<input type="text" value="2"/>	
Spacing Between Indents in Y	<input type="text" value="10.0000"/>	μm
Total Distance in Y	<input type="text" value="10.0000"/>	μm

Piezo Translation Protocol

Constant Direction (Always Left-to-Right) ▼

- It is suggested that you do not adjust the delay times. At this point you can click on “Run Piezo Automation” to start the indent array. You will then be asked if the load function is set up properly. When you are selecting a directory to save the data, it is suggested that you save the data in a file in your workspace. You will then be asked to name the indent files, and when you are asked if you wish to run the automation from a script file click no
- The final menu allows you to vary either the load or displacement, depending on which type of load function you are using, through the indents. The top box shows the number of indents in the array. The left box allows you to choose the maximum load/displacement of the first indent, and the right box allows you to choose the maximum load/displacement of the last indent. The bottom box shows you the change in maximum load/displacement between each indent. Thus, if your start load is 9000 and you end in 1000 and you 9 indents, the load increment is -1000. So the first indent would be 9000, the second 8000, the third 7000, and so on until the last which would be 1000. See the pictures on the next page

Vary Peak Forward Disp, Constant Segment Time

Number of Indents
4

Start Forward Disp (nm)
100.00

End Forward Disp (nm)
1000.00

Forward Disp Increment (nm)
300.00

Continue **Cancel**

Vary Peak Load, Constant Segment Time

Number of Indents
4

Start Load
100.00 μN

End Load
1000.00 μN

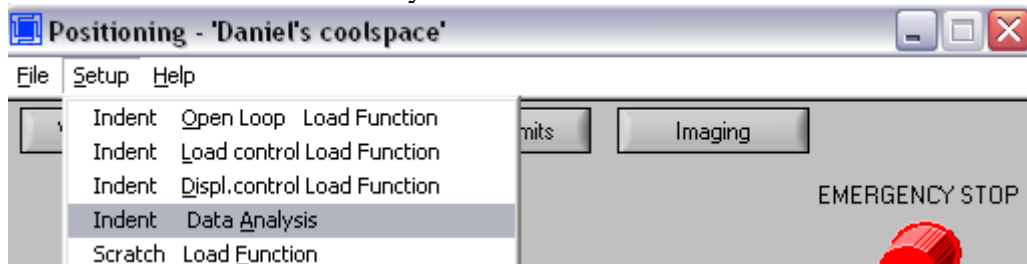
Load Increment
300.00 μN

Continue **Cancel**

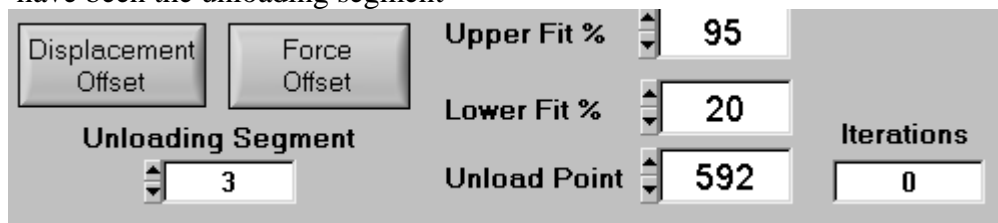
- After you have chosen the appropriate values click continue to start the array. This concludes the Piezo Automation procedure

Indent Analysis

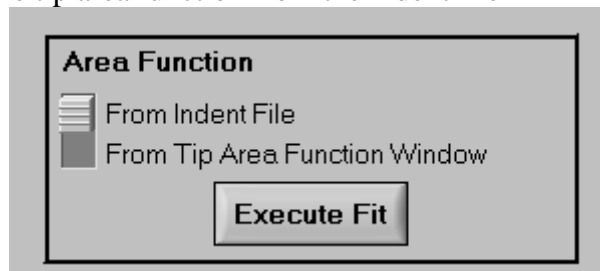
- Indent Analysis is the final step in taking an indent. The indent Analysis window will open at the end of an indent, or if you go to setup in the positioning window and click on “Indent Data Analysis”



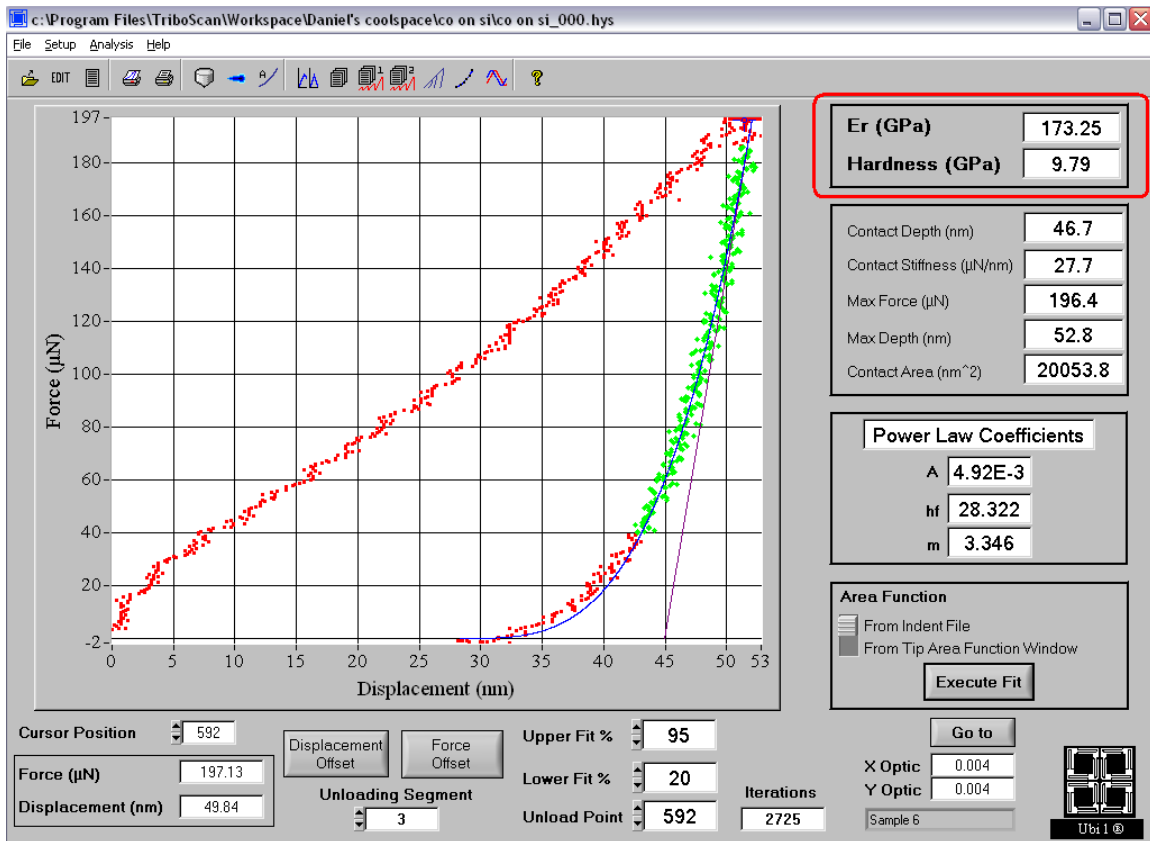
- Ensure that the “Unloading Segment,” at the bottom of the of the screen, is correct. I.E. if your indent was a trapezoid than the unloading segment would be the third segment, if your indent was a triangle than the second segment would have been the unloading segment



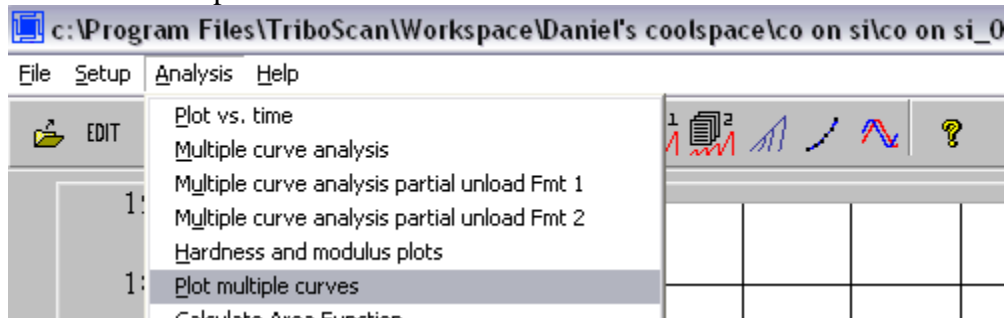
- Next you must chose if you wish to use the current tip area function of the area function from the indent file. For the most part this will not make a difference, but in some cases if the indent file is older than the tip area function than you will want to use the tip area function from the indent file



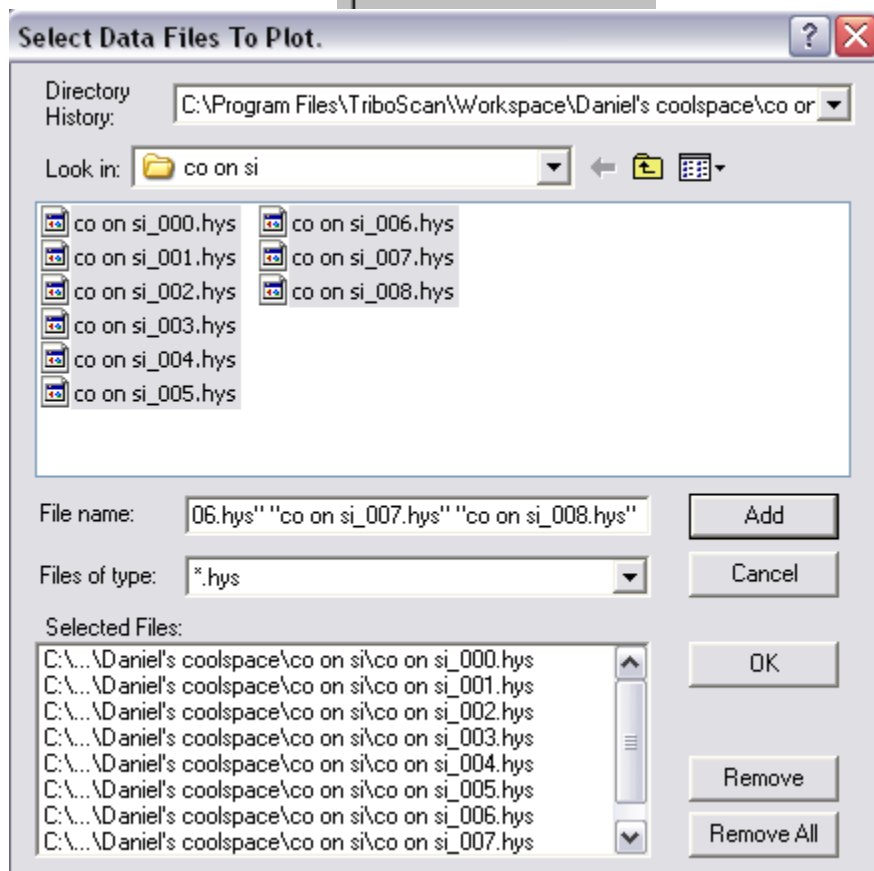
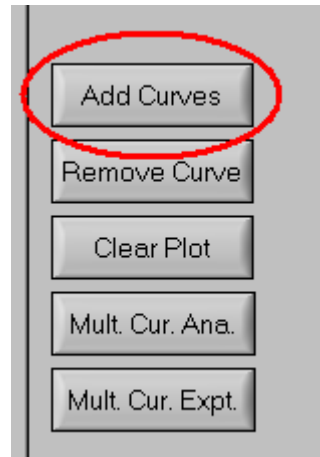
- All that's left is to click “Execute Fit” and the hardness and modulus numbers will be generated and displayed at the top right of the screen, see the picture on the next page



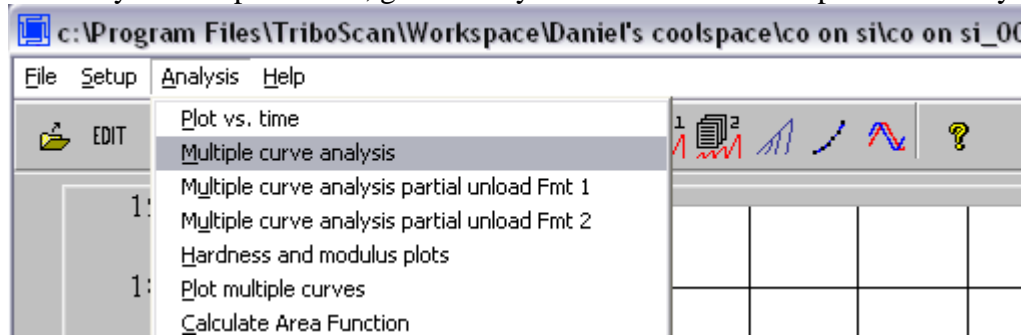
- To plot multiple curves at once, go to the “Analysis” drop down menu and click on “Plot multiple curves”



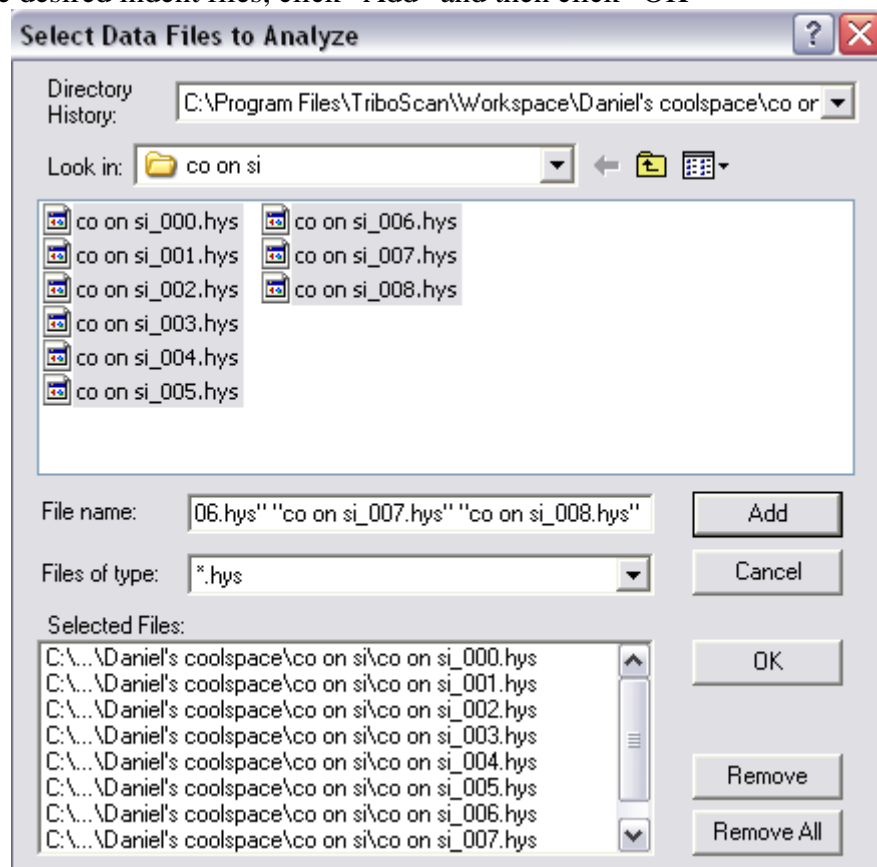
- When the next window comes up, click on “Add Curves.” When the “Select Data Files to Plot” window appears, highlight the desired curves, click “Add”, and then click “OK”



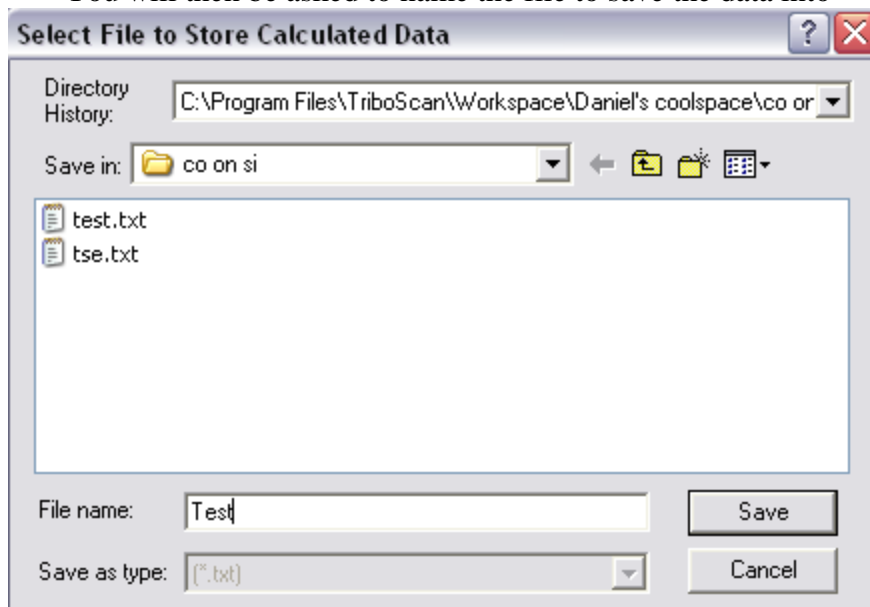
- To analyze multiple curves, go to Analyze and click on “Multiple curve analysis”



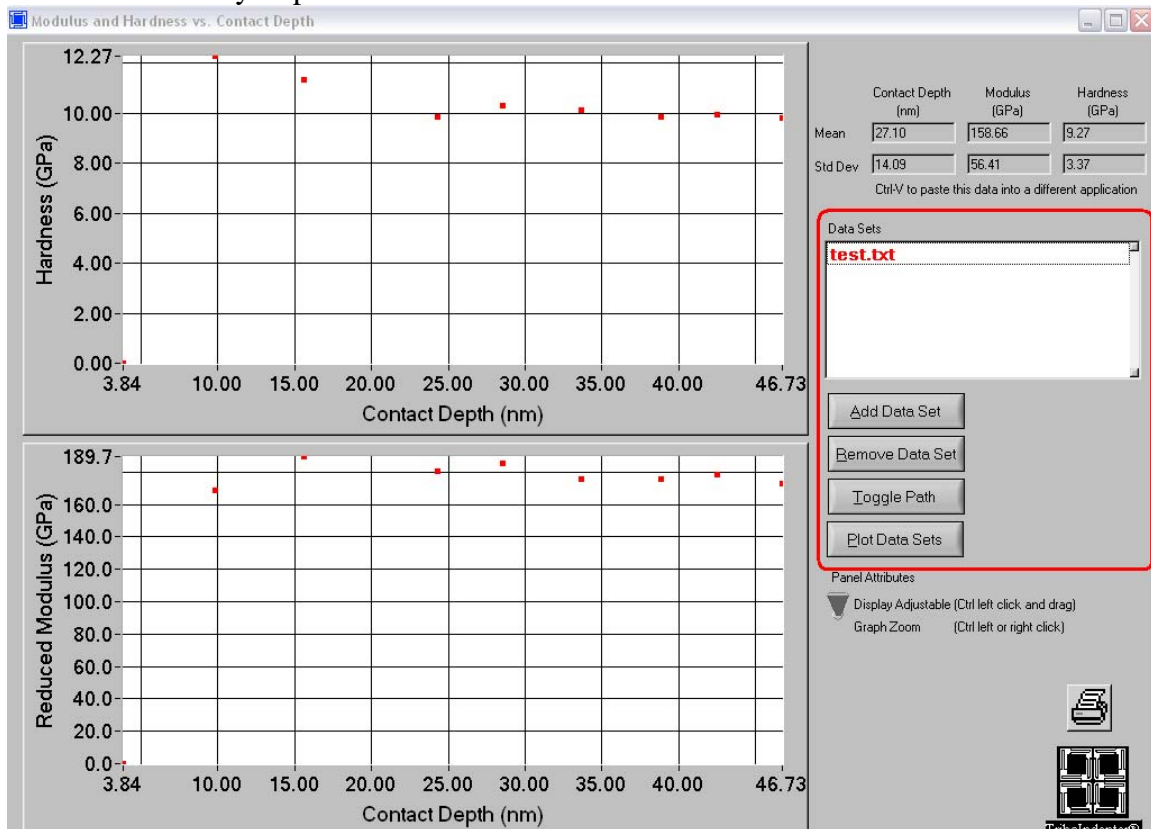
- This time you will be asked to select the curves to analyze first. Again, highlight the desired indent files, click “Add” and then click “OK”



- You will then be asked to name the file to save the data into

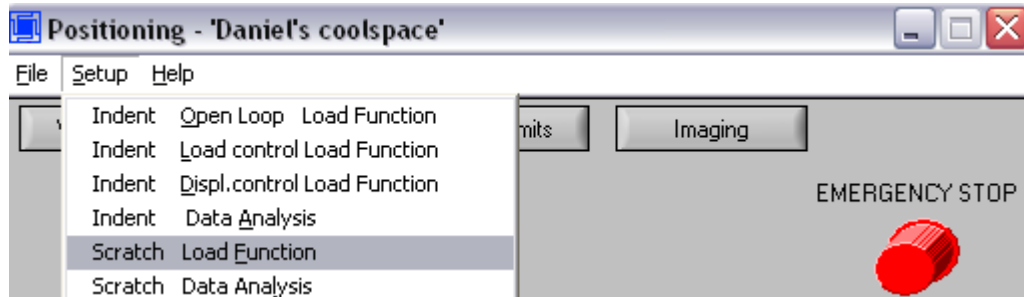


- When the multiple curve analysis window opens, you will have the option of loading other data sets to compare, see picture below. This concludes the multiple curve analysis procedure

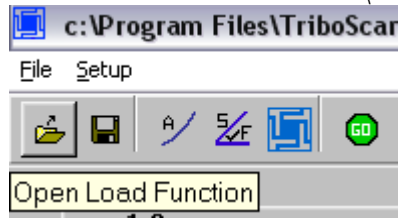


Scratch Test

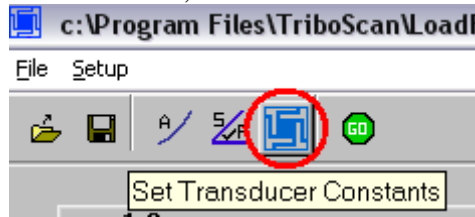
- The scratch test is performed similar to any indent. First, you must load the load function. Go to setup in the positioning window and click on “Scratch Load Function”



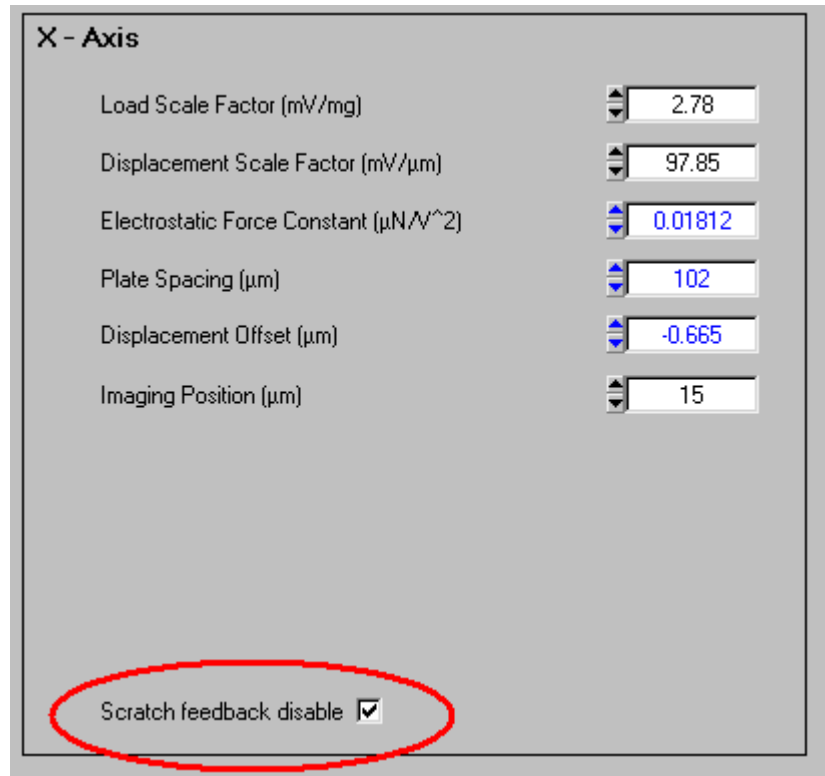
- Now we must do an x-axis calibration. Similar to the z-axis calibration, we will do an air scratch, and calibrate the transducers from this. In the scratch load function window, click on the open icon and load the x_axis_calibration_scratch file. The file is located under C:\Program Files\TriboScan\LoadFunctions



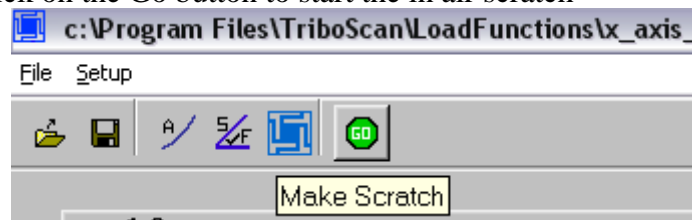
- Once this is done, click on the “Set Transducer Constants button”



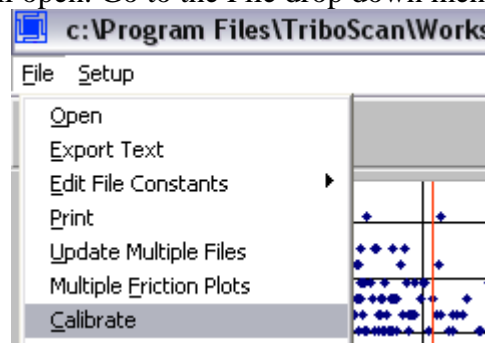
- When the transducer constants window comes up, uncheck the “Scratch Feedback Disable” box in the X-Axis section. To be clear, you must have this box **UNCHECKED** to run a scratch test. Once this is done, check the x-axis constants with the Transducer Calibration Constants sheet supplied. You may now close this window



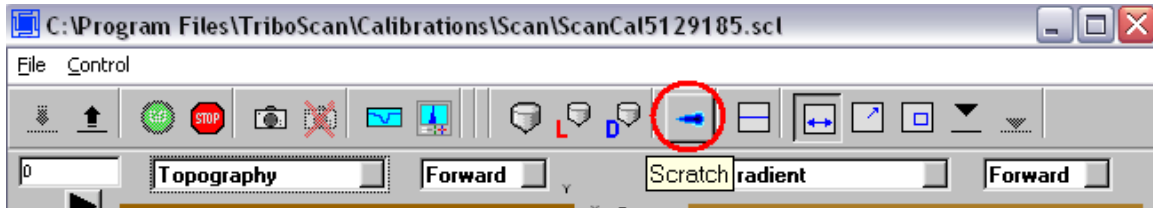
- Click on the Go button to start the in air scratch



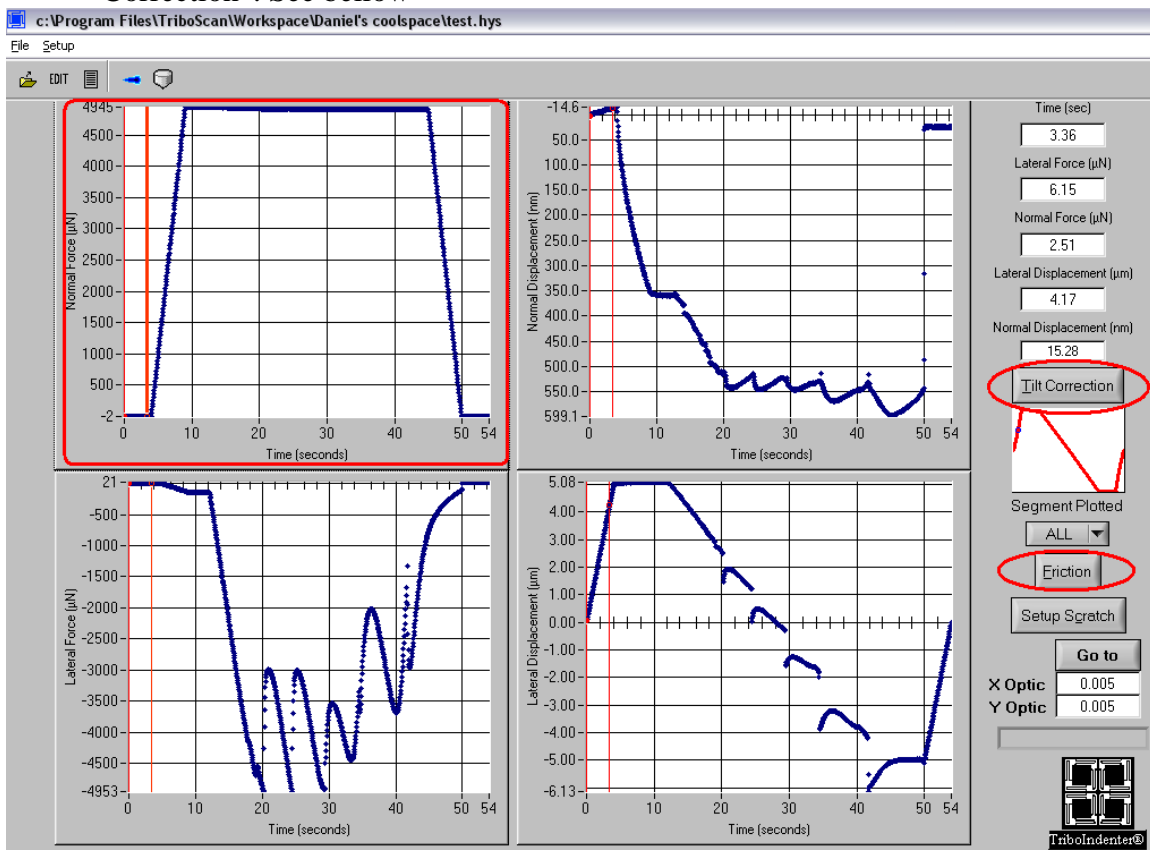
- Once the test is complete and you have saved the file, the scratch analysis window will open. Go to the File drop down menu in this window and click on “Calibrate”



- Now you are ready to create your scratch load function. There are two basic types of scratch load functions, constant force and ramp force, the ramp force test will either linearly increase or decrease the applied force during the scratch. You can also scratch in either the negative or positive direction.
- Once you have the desired load function, click on the scratch icon in the imaging window



- Once you have confirmed the load function the test will start. Once the test is finished you will be asked to save the test. After you save the test, the scratch analysis window will open. The first thing to do in this screen is to account for the gradient or tilt of the sample. In the upper left graph click and drag the two red lines so that they sit on the first level part of the graph, then click "Tilt Correction". See below



- Once you have corrected for the tilt you can find the “friction” from the sample by click on the friction button, see the picture on the previous page. Note that the friction value is not truly the coefficient of friction of the sample, but also includes the “plowing” resistance. Thus, the deeper the indent the high friction value you will see. For the most accurate friction values use shallow scratches. This concludes the Operating procedure