

**University of Saskatchewan
Department of Mechanical Engineering
Standard Operating Procedure # ME0016**

Procedure Title: Nano Indentation with the NH-type Nanohead

Minimum Review Requirements: **Annually**

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Administrator of Procedure: **Robert Peace, P. Eng.**

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1. Version History

Version #: .1
Supersedes: n/a

The signatures below indicate that the person(s) responsible to administer and supervise this procedure have read and agree to abide by this SOP	Date

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

Section	Changes made to official copy	Date	Initials
X.X, 6.7	Software updated resulted in changes to approach	June 25, 2010	R.P.
All	Extensive updates due to new control system	Sept. 2012	R.P.

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2. Introduction

This procedure details the proper operation of the NH-1 Nanohead for Nano-indentation testing.

1. **The CETR NH TYPE NANOHEAD MANUAL** should be consulted for clarification, if further questions or clarifications are required please contact Robert Peace at 966-5461 or via email: rob.peace@usask.ca .
2. **ONLY AUTHORIZED TRAINED USER ARE PERMITTED TO USE THIS EQUIPMENT.**
3. The below guide assumes that the sample **tilt check** and proper leveling has occurred (see the Nanohead manual for details).

3. Definition

- NH – Nanohead
- UMT/UMNT – Universal Materials Tester
- FS – Fused Silica (reference sample)

4. Personnel

SOP issued under authority of: Prof. Qiaoqin Yang, Mechanical Engineering

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS's and that I will conduct myself in accordance with this SOP and the general laboratory rules.

NOTE: ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 2C26 – Mechanical Engineering Materials Lab, digital copies of SOP's are made available for convenience only. Printed SOP's are valid for 24 hours only, after that time their accuracy must be verified with the **OFFICIAL HARDCOPY VERSION.**

Name (Print)	NSID	Dep't	Signature	Date
Robert Peace	Staff	ME		

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5. Safety

- 1) When Mounting a specimen to the metal stub there is a burn hazard due to the high temperature, tongs should be used to handle the hot mounting puck and gloves should be worn to avoid contact with the hot glue.
- 2) The bonding material used is Crystal Bond 509 Amber, and its MSDS is located on the Mechanical Engineering Safety page.
- 3) Acetone is used to clean the metal stubs and possibly the samples of the Crystal Bond, the MSDS for Acetone can be found on the Mechanical Engineering Safety page.
 - a. Acetone **MUST** be used in a fume hood, with Latex gloves, NOTE: Nitrile gloves are not compatible with Acetone.
 - b. All general lab procedures for chemical handling, labeling and disposal **MUST** be followed for the Acetone and Crystal Bond used.

6. Procedure

Prior to Sample Insertion:

- Power to the UMT is generally left on permanently, if for some reason it has been powered off, it must be on for a minimum of **36 hours** prior to testing.
- The NH Nanohead controller must be on for a minimum of **2 hours** prior to testing.
- Any major re-arrangements, such as switching between scratch and nano-indentation require a minimum of **12 hours** for equilibration after the re-arrangement.

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6.1 Specimen Mounting Procedure

For Nanoindentation tests the specimens must be mounted on a metal stub (puck) using a high rigidity adhesive such as Crystal Bond. In this lab we typically use Crystal Bond 509 Amber.

- Crystal Bond 509 Amber Information:
 - Flow Point 121°C
 - Solvent: Acetone
- 1) Ensure that your specimen is clean, dry and free of grease or oils.
 - 2) Place a mounting stub onto the hot plate and turn the hot plate to an appropriate temperature (>121°C).
 - 3) Allow the metal stub to heat up, you can check the temperature of the top surface with a radiation thermometer (laser pointer kind).
 - 4) Once the top surface is a suitable temperature put a small amount of Crystal bond on the surface and wait until it flows (It will not flow like water, more like a thick oil).
 - 5) Place your sample on the Crystal Bond using tweezers and push down gently to secure the specimen – ensure that you do not touch the region that you wish to indent on.
 - 6) Remove the metal stub from the hot plate using metal tongs and place on a metal or heat resistant surface to allow air cooling.
 - 7) Turn off the hot plate.
 - 8) Wait for the specimen to cool to room temperature before inserting into the UMT for testing – if you do not wait it will affect your results.

6.2 Specimen Removal Procedure

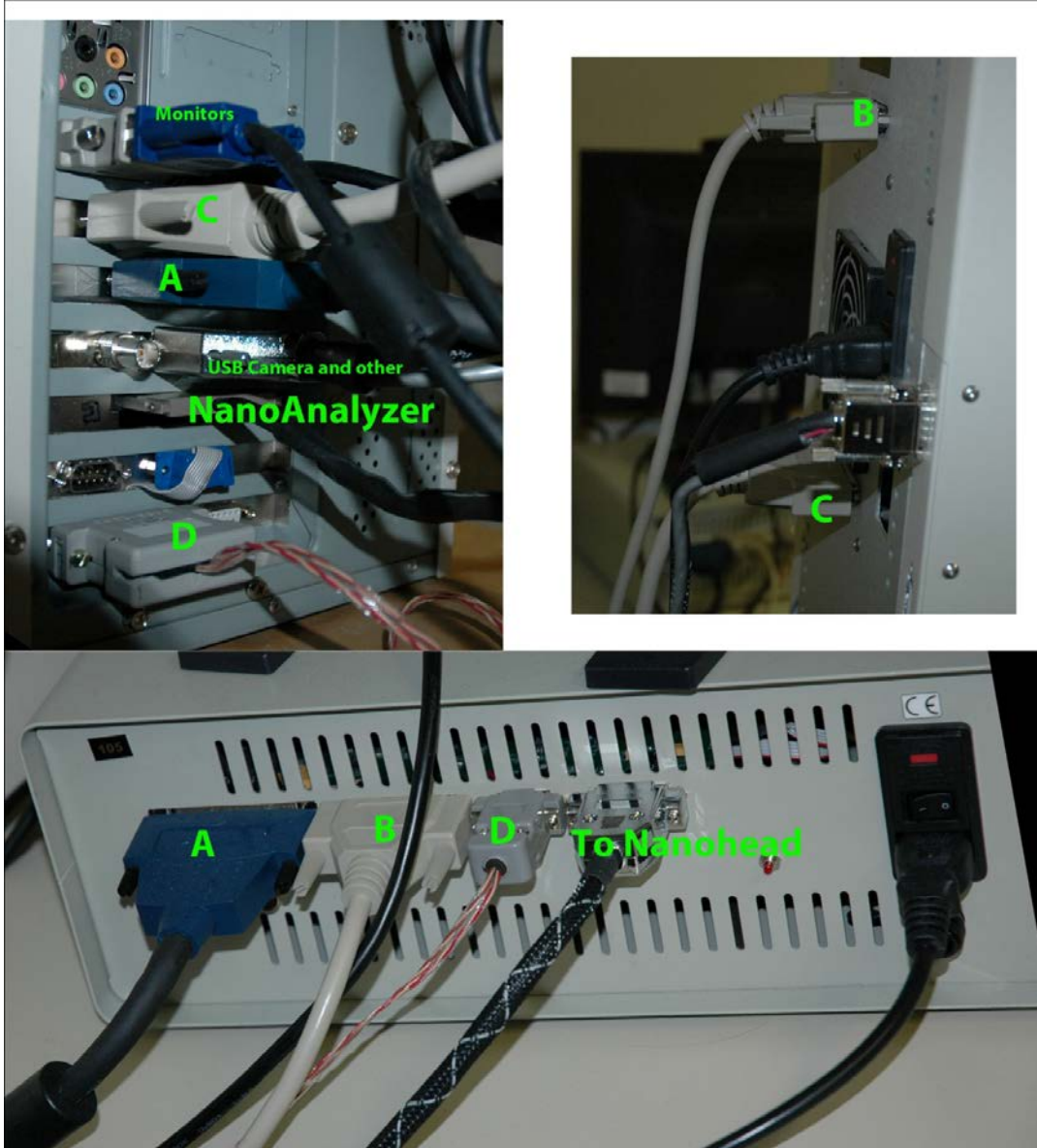
After testing the metal stubs must be cleaned to be 100% free of Crystal Bond.

- 1) Re-heat the stub using the hotplate, with the sample attached until the sample can be removed (using tweezers).
- 2) Turn off the hot plate and place the metal stub on a heat resistant surface and allow it to cool to room temperature (handle the hot components with tongs or tweezers).
- 3) Once the metal stub is cool, place it in a beaker containing Acetone (in a fume hood, while wearing Latex gloves and using tongs), allow the Acetone to dissolve the crystal bond.
- 4) Remove the Metal Stub, rinse with ethanol and dry, then return to the UMT room.

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- 5) Collect the Acetone and ethanol waste in an appropriate labeled waste container.
- 6) Wash the beaker and utensils used and collect the waste according to the Laboratory Procedures provided in Mat0001 and Mat0002.

6.3 Cable Connections between UMT, Nanohead Controller and PC



6.4 Tip Cleaning and Installation – **Only by a Trained Individual**

- Detailed instructions on proper tip cleaning **PRIOR TO INSTALLING ON THE NANOHEAD** can be found in the NH-1 manual in section 3.1.

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- **Never clean a tip when installed in the Nanohead, permanent damage can result.**

Detailed instructions on proper tip mounting onto the Nanohead are shown in section 3.XX of the NH-1 manual.

6.5 Nanohead Installation

- Detailed installation instructions can be found in the NH-1 manual in section 3.4, this should only be done by a trained individual.

6.6 Sample Insertion

- Samples should be inserted into the UMT chamber a minimum of **2 hours** prior to testing.
- The **2 hour** timer begins when the UMT doors are shut, and should they be opened the timer is reset.
- For additional protection from thermal air currents a cardboard box should be placed over the entire UMT after sample insertion.
- Only 1 sample should be on the granite block at a time, others can be placed next to the block in the enclosure to allow them to acclimate.

6.7 Initial set-up

- **Start the UNMT Software.** Icon CETR titled UMT.
- **Load the Options File.** Options>Load (C:/TRIB/Option files/Nanohead/NanoHead.opt) then click open
- **Open the Graphs** (Also called “real-time” in the Frequently used test procedures provided by Bruker)
 - i. This opens the Nanoposition (nP) graph
 - ii. Click the graphs icon again and select NanoForce (nF) and then “OK” to open the nF graph
 - iii. Right click the nP graph and select “graph properties”, deselect “Auto-center”, select “Manual” and change the Minimum to -100 and Maximum to +100, then click Apply
- **Open Semi-automatic Panel** (click on the wrench icon)
- **Open Position Adjustment Panel** (click on the box with arrows icon)
- **Open the Microscope Panel** (Optical image) and turn on the Vertical Lamp (Also called “top-down-view” in the Frequently used test procedures provided by Bruker)
 - i. **Start the Frame grabber of the Microscope** by clicking the green arrow icon.

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6.8 Initial Manual Approach

The Nanohead should not be moved very far from the starting position for the scripts (only a few mm left to right, forward to backward or up/down should be required).

Use the positioning controls detailed in section 6.7 to position the Nanohead as shown in Figure 1, with the camera prism directly over the sample at a height of approximately 3.0 cm from the specimen surface.

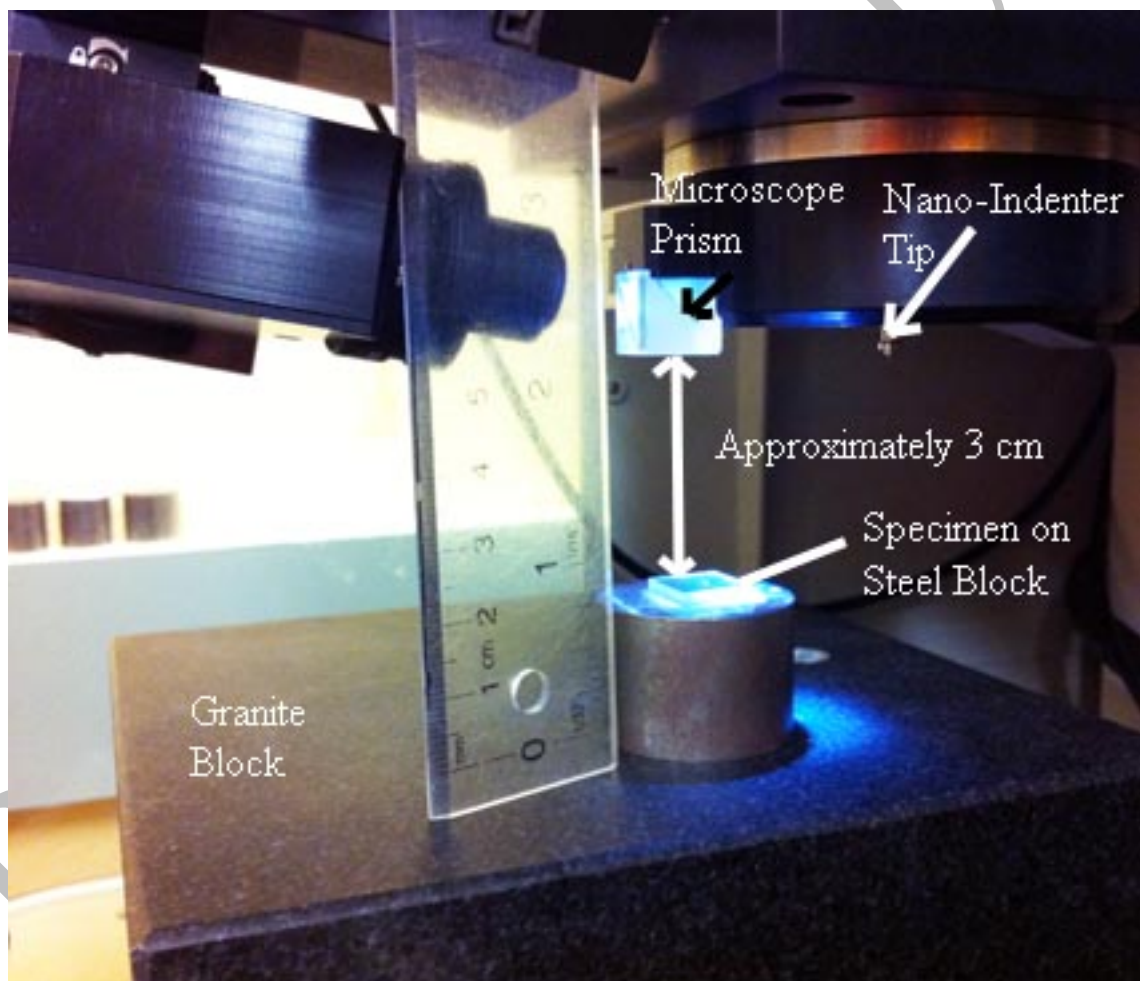


Figure 1: Approximate Starting Position for Nano-indentation Scripts.

Use the optical microscope image to focus the TOP of your sample, as shown in Figure 2 (a), Figure 2 (b) shows that you can focus through a translucent material (such as the reference material Fused Silica (FS)), this is an unsafe distance as

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when the Nanohead moves for indentation it will assume the surface is the thickness of the sample lower than it actually is, this could potentially result in a collision (and the Nanohead can-not be repaired except by the manufacturer at a very high cost).

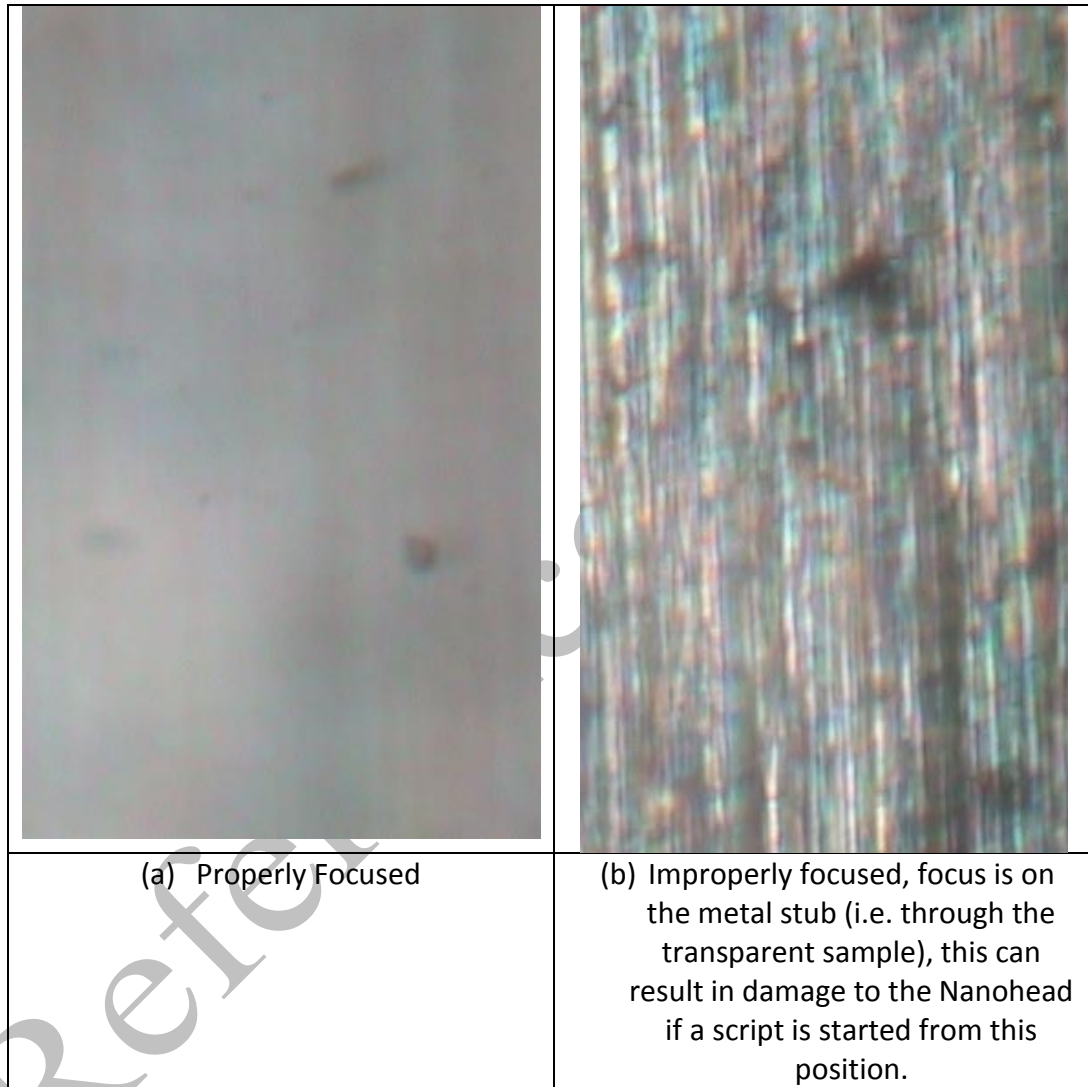


Figure 2: Properly focused specimen (a), focus through a transparent specimen (b), with the metal mounting stub in focus.

Figure 3 shows the edge of the FS reference sample in focus, which can be used as a focusing aid.



Figure 3: In focus edge of FS sample.

6.9 Movement Control of the Nanohead

Up/Down (Carriage)

1. Semi-Automatic Panel

- a. Carriage Tab
 - i. **Only For Long Movements and Only With A Large Margin For Safety.** Input desired direction and hit the ► icon, can be stopped at anytime by hitting the ■ icon.
- b. Up with Keyboard
 - i. **Fast Movement:** Alt + ↑ key
 - ii. **Slow Movement:** Ctrl + ↑ key
- c. Down with Keyboard
 - i. **Fast Movement:** Alt + ↓ key
 1. **DO NOT USE when you are anywhere near the surface (under 2cm) with the Nano Indenter**
 - ii. **Slow Movement:** Ctrl + ↓ key

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Front/Back (Linear)

Forward: Bottom stage moves away from UMT doors.

Backwards: Bottom stage moves towards UMT doors.

1. Semi-Automatic Panel

- a. Linear Tab
 - i. Input desired direction and hit the ► icon, can be stopped at anytime by hitting the ■ icon.
- b. Forward with Keyboard
 - i. **Fast Movement:** Alt + > key
 - ii. **Slow Movement:** Ctrl + > key
- c. Backward with Keyboard
 - i. **Fast Movement:** Alt + < key
 - ii. **Slow Movement:** Ctrl + < key

Left/Right (Slider)

Right: Nanohead moves towards computer.

Left: Nanohead moves towards door of room.

1. Semi-Automatic Panel

- a. Slider Tab
 - i. Input desired direction and hit the ► icon, can be stopped at anytime by hitting the ■ icon.
- b. Left with Keyboard
 - i. **Fast Movement:** Alt + ← key
 - ii. **Slow Movement:** Ctrl + ← key
- c. Right with Keyboard
 - i. **Fast Movement:** Alt + → key
 - ii. **Slow Movement:** Ctrl + → key

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6.10 Frequently Used Test Procedures

The most commonly used test procedures for nano-indentation are provided as pre-made scripts located in the document "Frequently Used Test Procedures" provided by Bruker, the instruction for their use follows, the instructions provided by Bruker assume that the Nanohead has to be moved into position, however this is not necessary if it is already in the approximate position, which is why the INSTALL-Module.cts script is not referenced below.

1. Single_Indent_Trapezoidal_Vex_OM
 - i. This script does a single trapezoidal indent using a 10mN load.
 - 1) Ensure that the sample position is in focus as indicated in section 6.6.
 - 2) Position the specimen using the motor drives to an optically clean position.
 - 3) Load and run the script:
C:\Trib\Scripts\Nanohead\System\Settle_VEX.cts
 - a. This will account for motor backlash.
 - 4) Re-check the specimen position and if necessary repeat steps 2-3
 - 5) Load and run the script:
C:\Trib\Scripts\Nanohead\System\Single_Indent_Trapezoidal_VEX_OM.cts
 - 6) Once the script is finished the specimen will be under the microscope and nearly in focus, use the fine Z (carriage) adjustment to focus the image.

Notes:

- The first sequence can be used to add a delay before starting the procedure (which is recommended)
- The second sequence is movement from the optical location to the indenter location and MUST never be altered in anyway.
- The third sequence is a surface approach, which extends the Nanohead to its maximum then retracts it and moves the carriage down, this repeats until the surface is found. This step results in a saw-tooth shaped graph on the nP graph if the minimum and maximum nP values are -100 and +100.
- The fourth sequence is the actual indent
 - Step 1: slow surface approach
 - Step 2: actual indent – NOTE: this is the step where the 10mN load is entered.
 - Step 3: 10s hold
 - Step 4: unloading
 - Step 5: hold for thermal drift
 - Step 6: retract tip
 - Step 7: retract tip by 5 um

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- The fifth sequence returns the Nanohead to the starting position.

- 2. Multiple_Indent_Trapezodial_VEX_OM
 - i. This script does a grid of 9 indents (3x3) with a spacing of 50um between each indent
 - 1) Ensure that the sample position is in focus as indicated in section 6.6.
 - 2) Position the specimen using the motor drives to an optically clean position.
 - 3) Load and run the script:
C:\Trib\Scripts\Nanohead\System\Settle_VEX.cts
 - a. This will account for motor backlash.
 - 4) Re-check the specimen position and if necessary repeat steps 2-3
 - 5) Load and run the script:
C:\Trib\Scripts\Nanohead\System\Multiple_Indent_Trapezodial_VEX_OM.cts
 - 6) Once the script is finished the specimen will be under the microscope and nearly in focus, use the fine Z (carriage) adjustment to focus the image.

Notes:

- This is the same as single indent trapezoidal, except it does a 3x3 grid of indents.
- The first sequence can be used to add a delay before starting the procedure (which is recommended)
- The second sequence is movement from the optical location to the indenter location and MUST never be altered in anyway.
- The third sequence is a surface approach, which extends the Nanohead to its maximum then retracts it and moves the carriage down, this repeats until the surface is found. This step results in a saw-tooth shaped graph on the nP graph if the minimum and maximum nP values are -100 and +100.
- The fourth sequence is the actual indent and movement to the indent location
 - Step 1: slow surface approach
 - Step 2: actual indent – NOTE: this is the step where the 10mN load is entered.
 - Step 3: 10s hold
 - Step 4: unloading
 - Step 5: hold for thermal drift
 - Step 6: retract tip
 - Step 7: retract tip by 5 um
- To do the grid pattern sequence 4 has movements built into it at 50um spacings.
- The fifth sequence returns the Nanohead to the starting position.

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6.11 Script Configuration and Customization

The pre-created scripts MUST NOT BE ALTERED in anyway, but they are the best basis for creating custom scripts. Two examples are provided for creating custom scripts, the first is a single indent trapezoidal with a different (than 10mN) load. The second is a Multiple indent trapezoidal script where the loads vary between indents and the spacing between indents is also varied.

Example 1: Single Indent Trapezoidal with a different load:

To create a single indent Trapezoidal script with a different load do the following:

- 1) Open the script: Single_Indent_Trapezoidal_VEX_OM.cts
- 2) Navigate to the 4th sequence and the second step called “Loading_LinearForcebyTime_FixedTime” and select the “Carriage” tab as shown in Figure 4.

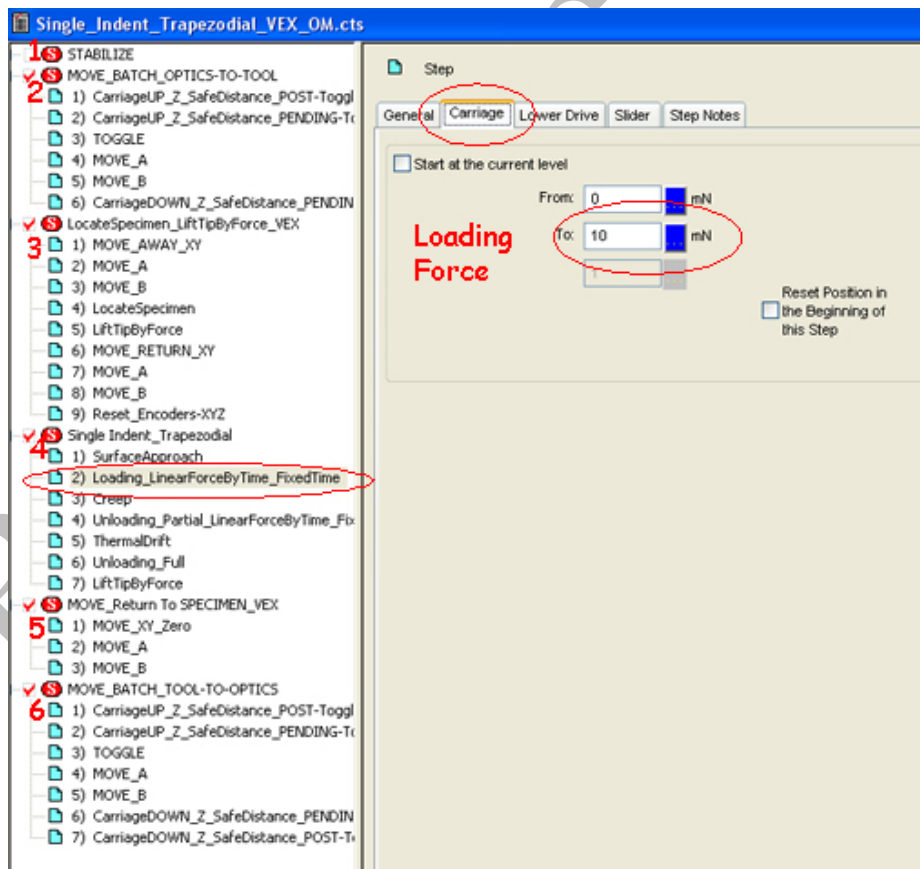


Figure 4: Loading Force in the Single_Indent_Trapezoidal_Vex_OM.cts script.

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- 3) Change the 5mN load to the desired load i.e. 5mN as shown in Figure 5.

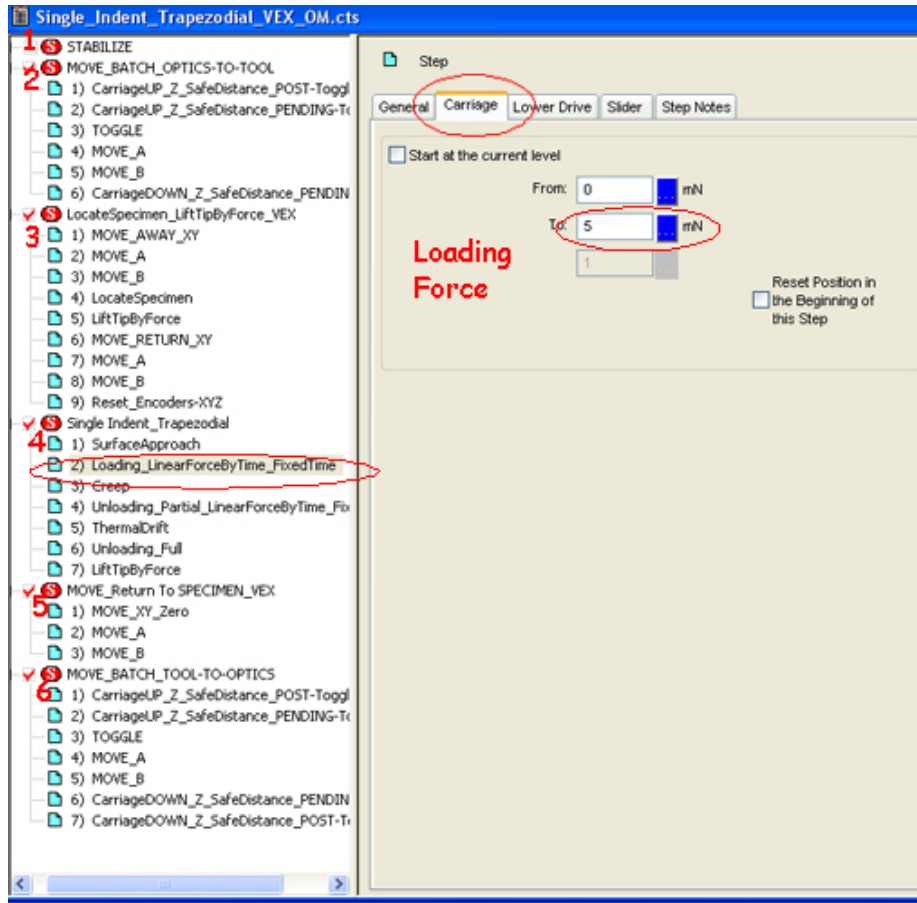


Figure 5: Loading Force changed to 5mN.

- 4) The new script now has to be Saved As a NEW SCRIPT.
 - a. **DO NOT SAVE a modified script over an original one!**
- 5) Save the new script (you can create your own script folder if you like, but use a different name than the pre-built scripts) Figure 6 shows a new script being saved into a user's folder:
 - a. Single_Indent_Trapezodial_Vex_OM_5mN.cts

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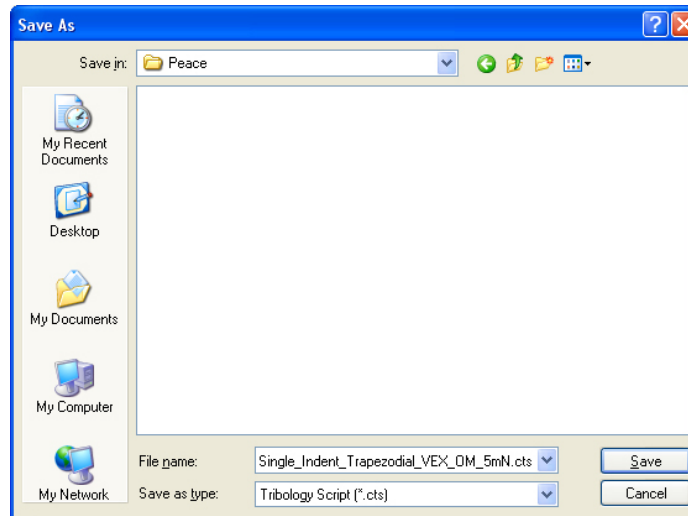


Figure 6: Newly created script being saved to a user's folder.

6) This new script can now be used.

Example 2: Multiple Indent Trapezodial with different loads and spacing

- In this script the Loads and indent spacings are controlled by external files which can be altered to change the loading and/or spacing of the indents.
- If different loads are desired or some are to be eliminated a new layout file must be created and the script saved as a different name, **DO NOT MODIFY OR RE-NAME THE SUPPLIED TEST SCRIPTS.**

To create a multiple indent Trapezodial script with different load(s) and spacing(s) do the following:

1) Open the script: Multiple_Indent_Trapezodial_VEX_OM.cts

This script creates a 3x3 grid of indents, using the movement pattern shown in Figure 7.

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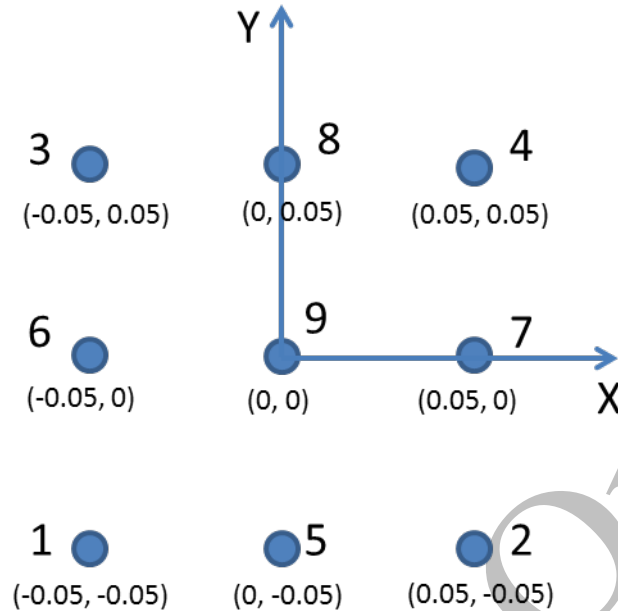


Figure 7: Movement pattern for Multiple_Indent_Trapezoidal_VEX_OM.cts
Coordinates are in mm, 0.05mm=50 μ m

- 2) Navigate to the 4th sequence and the sixth step called "Loading_LinearForcebyTime_FixedTime" and select the "Carriage" tab as shown in Figure 8.

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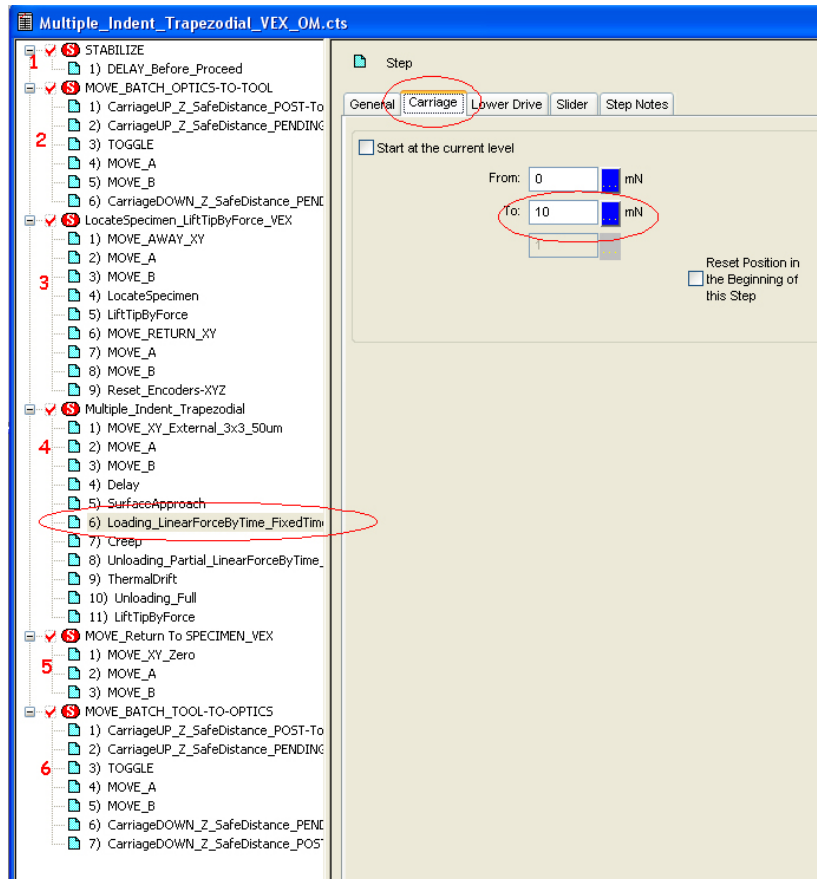


Figure 8: Location of Indentation Force.

- 3) The load for this script is a single number with a white background, which indicates an explicit load, in this case the load will be the same for each of the 9 indents.
 - a. To change the load for all of the indents, simple change the load number from 10mN to a different number such as 20mN as shown in Figure 9.

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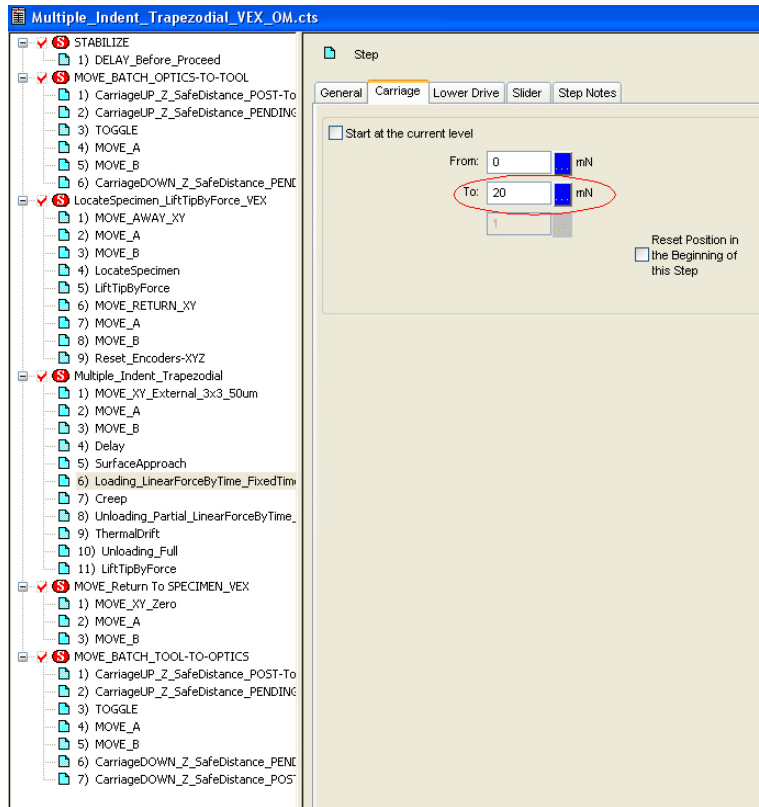


Figure 9: Changed Indentation force to 20mN.

- b. To change the load and vary it from indent to indent, the load has to be changed from “explicit” to “external file”
 - i. Click the blue box next to the load as shown in Figure 10.

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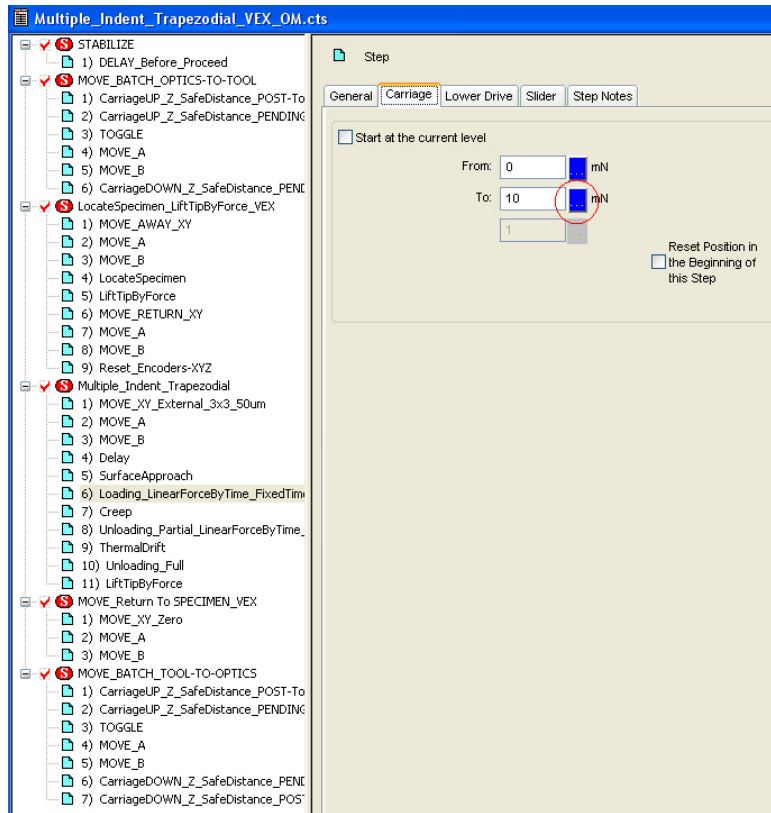


Figure 10: Button to select External Force Control.

ii. A new window will appear as shown in Figure 11.



Figure 11: Explicit Option means the number inputted as in Figure 9.

iii. Select the dropdown menu which has “Explicit” in it and select “External” as shown in Figure 12.



Figure 12: External means the load is controlled by an external file.

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- iv. Check the “External File” checkbox and an “External Contents” bar will appear as shown in Figure 13.

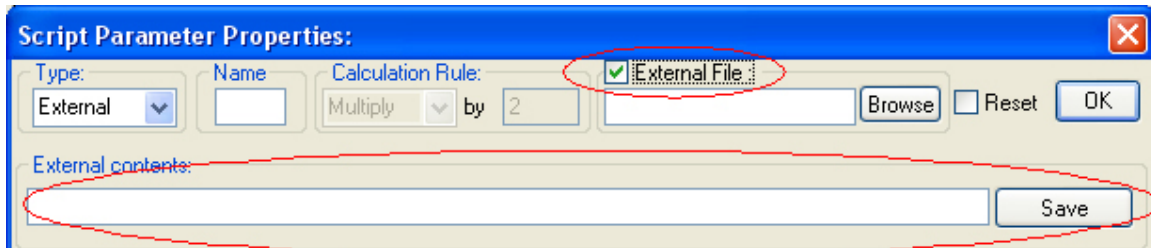


Figure 13: The External File check box must be selected to open the External Controls Tool bar.

- v. Enter the desired loads in the toolbar as shown in Figure 14.

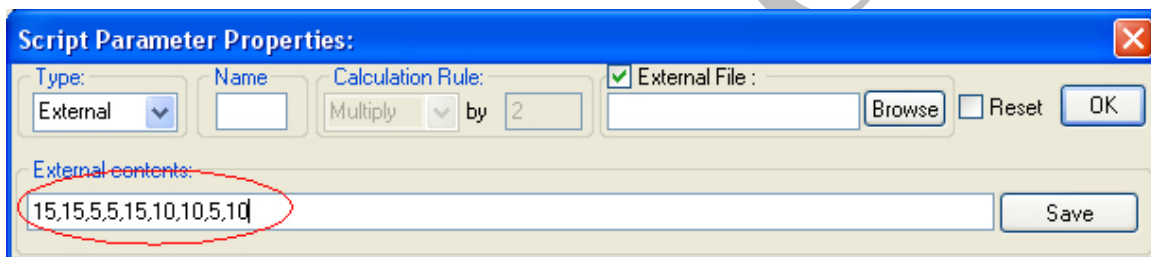


Figure 14: The Loads that are desired for this example to generate the grid shown in Figure 15.

- vi. Save the file to the appropriate location with an appropriate name such as:
C:\Trib\Scripts\NanoHead\Peace\External_Files\Multi_15_10_5.txt where the **Peace** and **_15_10_5** are your name and a designation of your choosing. **DO NOT overwrite** any other external files! In this example the loads will be 15mN, 15mN, 5mN, 5mN, 15mN, 10mN, 10mN, 5mN, 10mN
1. This will make a 3x3 grid with the top row having 5mN indents, the middle row 10mN indents and the bottom row having 15mN indents (see Figure 7 for explanation and Figure 15 for the new load settings).

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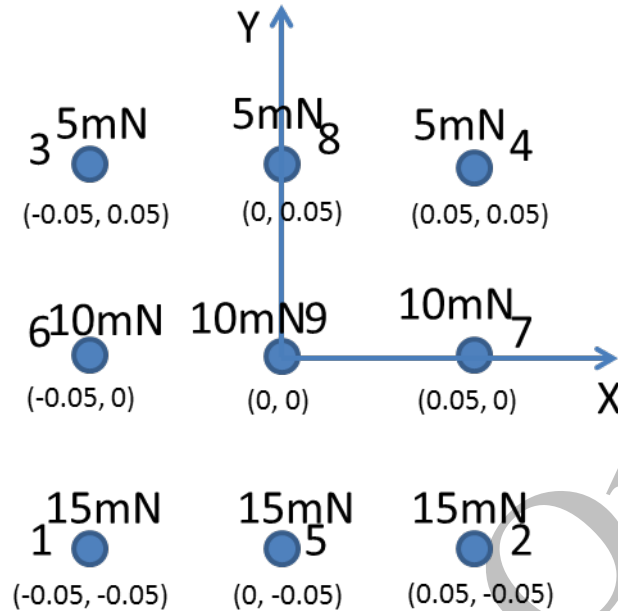


Figure 15: Indentation grid with the new loads programmed above.

- 4) The movements in x (slider) and y (linear) for this script are controlled by external files using coordinates (the origin is set during initial surface approach)
- 5) To change the spacing of the indent, the coordinates must be changed and then the external file saved, **DO NOT save over** the original external file, re-name it to a different file name.
- 6) In this example we will change the spacing as shown in Figure 16 and Table 1.

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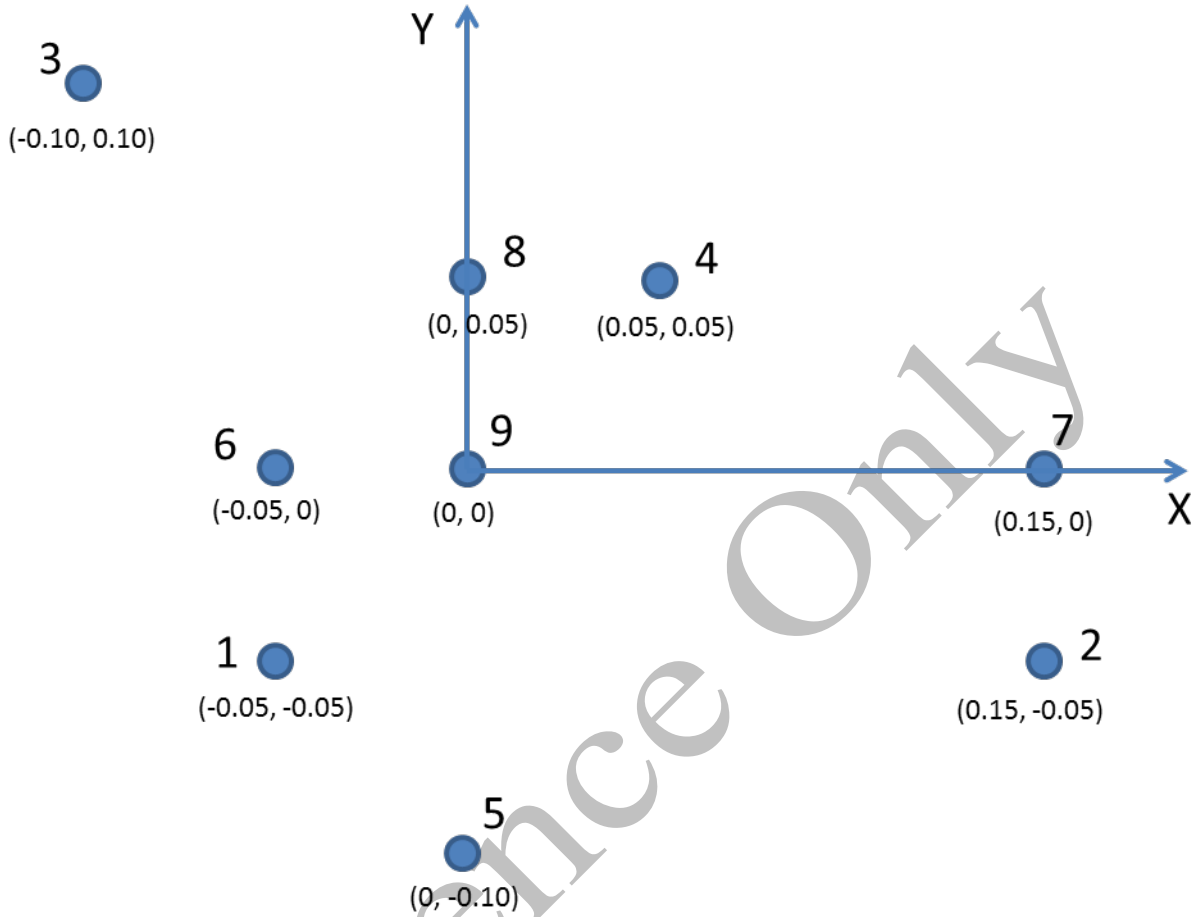


Figure 16: Custom “grid” spacing for this example.

Table 1: Coordinates for the spacing shown in Figure 15 (mm).

Point	1	2	3	4	5	6	7	8	9
X (slider)	-0.05	0.15	-0.10	0.05	0.00	-0.05	0.15	0.00	0.00
Y (linear)	-0.05	-0.05	0.10	0.05	-0.10	0.00	0.00	0.05	0.00

- 7) To change the x coordinates we need to edit and re-save the External file for the slider movement to match the desired coordinates.
 - a. Navigate to the 4th Sequence and the 1st step “Move_xy_External_3x3_50um”, then select the “slider” tab as shown in Figure 17.

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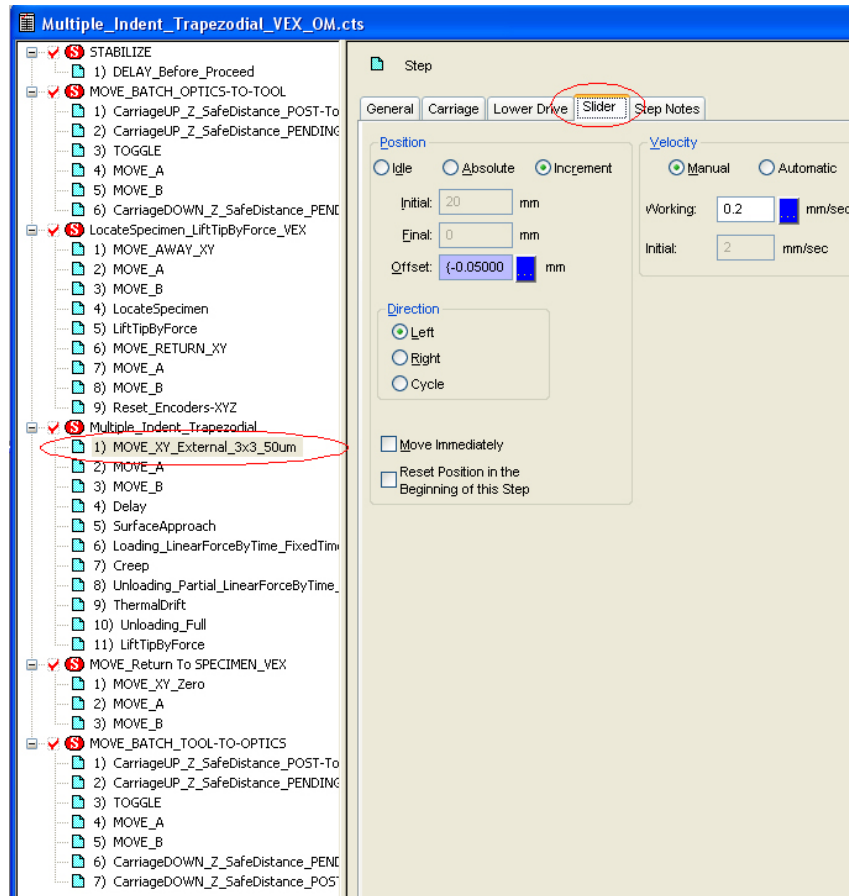


Figure 17: Location of the Slider (x) movement for the grid spacing.

- b. The “Offset” parameter is shown with a blue background, click the blue box next to this parameter to open a new window to edit the coordinates, as shown in Figure 18.

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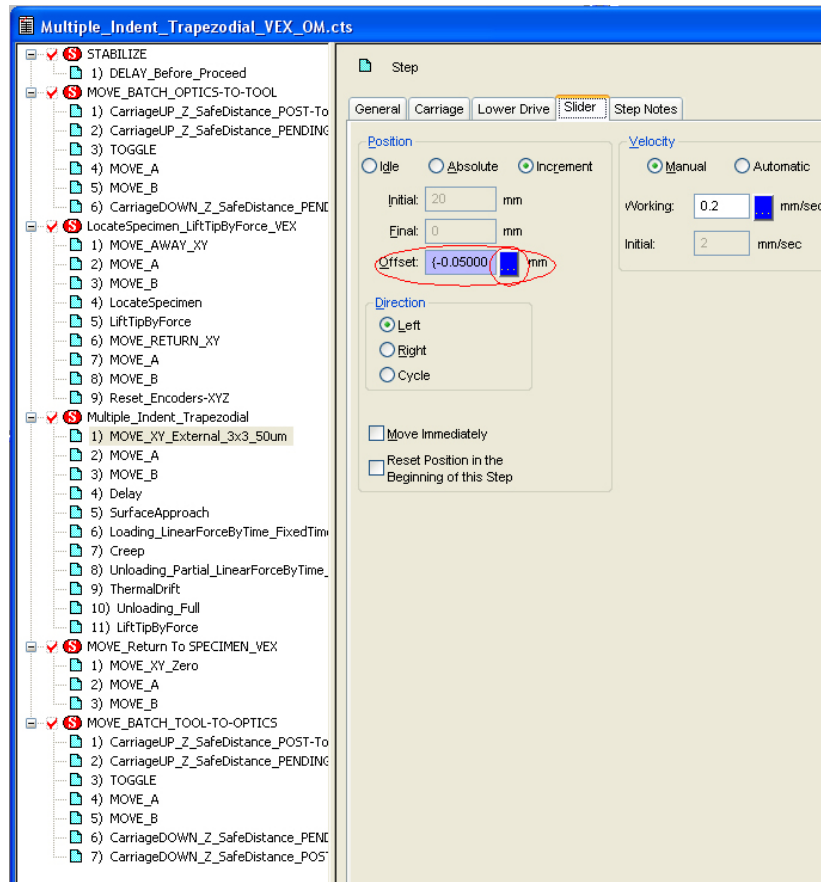
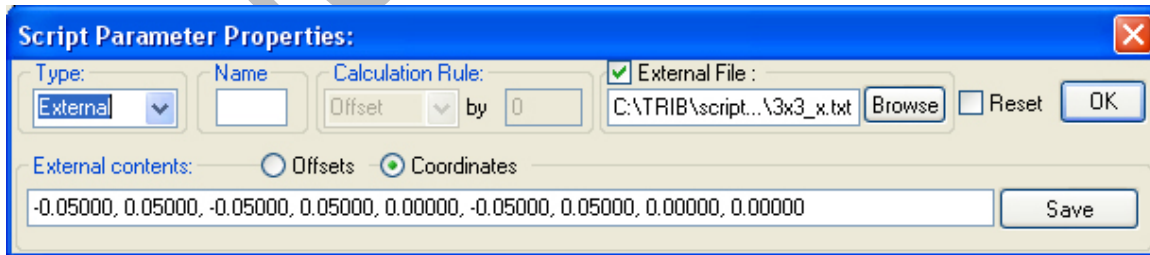


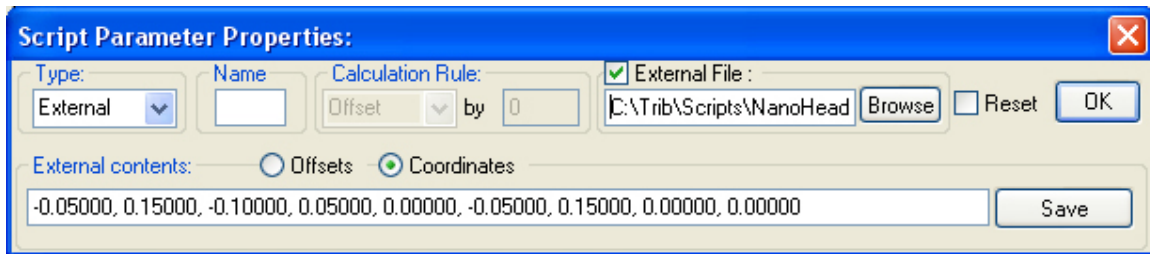
Figure 18: Button to changed external file movement for the Slider (X).

- c. Change the coordinates to those in Table 1 for “X” as shown in Figure 19.



(a) Original Slider coordinates

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(b) New Slider coordinates

Figure 19: (a) Original x (slider) coordinates, (b) new x (slider) coordinates.

d. Save the File:

C:\Trib\Scripts\NanoHead\Peace\External_Files\3x3_x_demo.txt in the appropriate folder, where **demo** is a designator of your choosing, and **Peace** would be your name. **DO NOT SAVE OVER THE ORIGINAL EXTERNAL FILE!**

Click OK

- 8) To change the y coordinates we need to edit and re-save the External file for the linear movement to match the desired coordinates. This is done in a similar manner to the x coordinates, only it begins on the linear tab.
 - a. Navigate to the 4th Sequence and the 1st step, then select the "Lower Drive" tab as shown in Figure 20.

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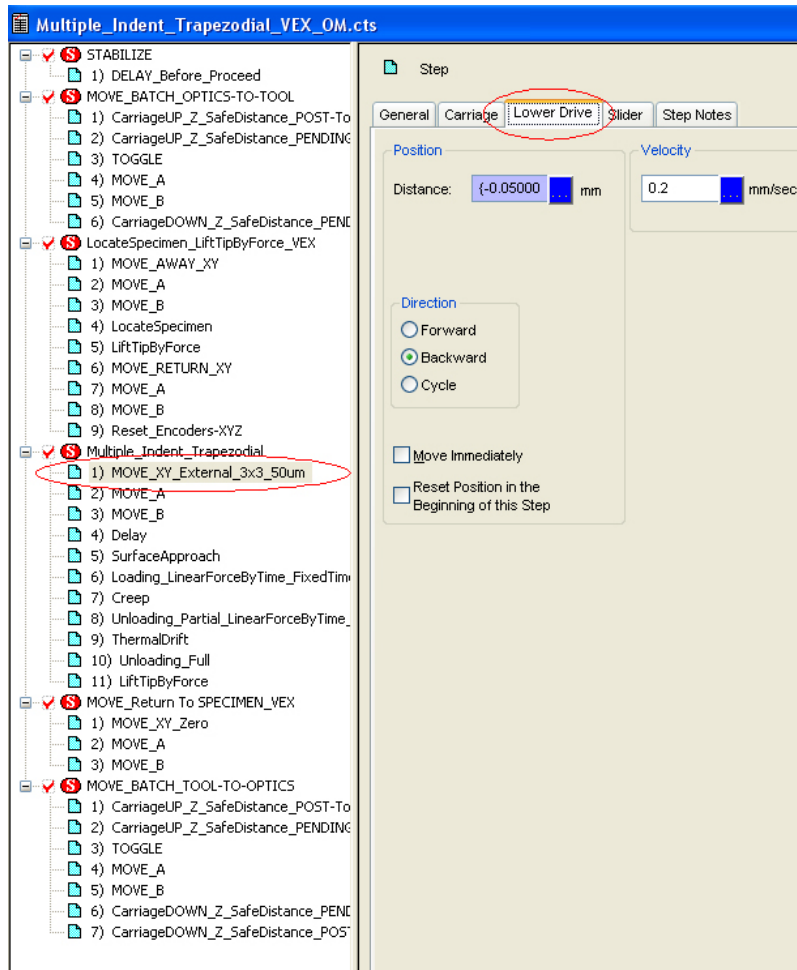


Figure 20: Location of the lower drive (Y) movement control for the grid spacing.

- b. The “Distance” parameter is shown with a blue background, click the blue box next to this parameter to open a new window to edit the coordinates, as shown in Figure 21.

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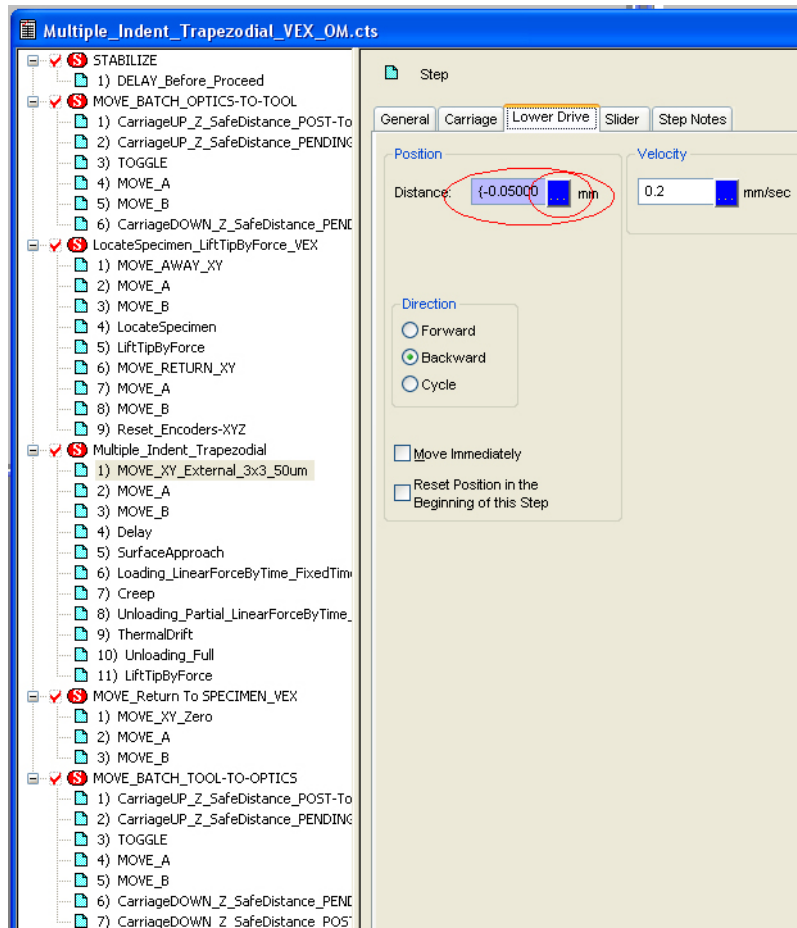
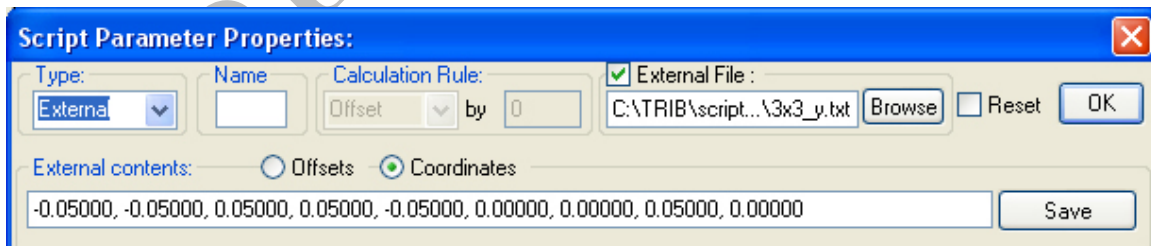


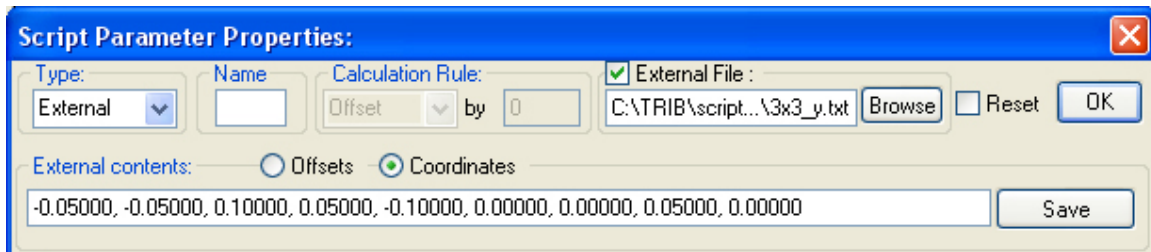
Figure 21: Location to change the Lower Drive (Y) grid spacing external file.

- c. Change the coordinates to those shown for Y in Table 1 as shown in Figure 22.



(a) Original Lower Drive (Y) coordinates

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(b) New Lower Drive (Y) coordinates

Figure 22: (a) Original y (linear) coordinates, (b) new y linear) coordinates.

- d. Save the File As
C:\Trib\Scripts\NanoHead\Peace\External_Files\3x3_y_demo.txt in the appropriate folder, where **demo** is a designator of your choosing, and **Peace** would be your name. **DO NOT SAVE OVER THE ORIGINAL EXTERNAL FILE!**
- 9) The new script now has to be Saved As a NEW SCRIPT.
 - a. **DO NOT SAVE a modified script over an original one!**
- 10) Save the new script (you can create your own script folder if you like, but use a different name than the pre-built scripts) Figure 23 shows a new script being saved into a user's folder:
 - a. Multiple_Indent_Trapezodial_Vex_OM_5_10_15mN.cts

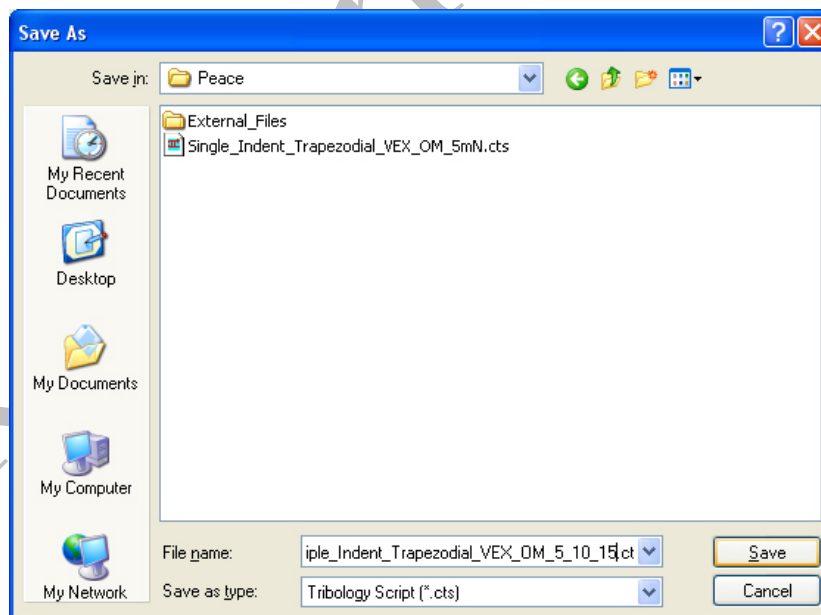


Figure 23: Saving the newly created script to a separate file.

- 11) This script is now ready to use.

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6.12 Other Types of Tests:

Frame Stiffness:

The frame stiffness calculation is covered in section 5.3 of the NH-1 manual, it is completed in a similar manner to the Indent scripts and can be found in the "Frequently Used Test Procedures" Manual.

An accurate frame stiffness is essential for repeatable and accurate results.

A typical value for this system is: 2.76N/um.

Tip Area Function:

The Tip Area Function calculation is covered in section 5.4 of the NH-1 manual, it is completed in a similar manner to the Indent scripts and can be found in the "Frequently Used Test Procedures" Manual.

An accurate Tip Area Function is essential for repeatable and accurate results.

The tip will wear over time resulting in a slightly altered Tip Area Function, and therefore this should be repeated periodically.

6.13 Data Analysis

Data analysis is completed using Viewer.exe icon on the desktop. A manual for the viewer software is provided in the lab.

Supplement to Viewer Manual:

Basic Data Processing for Indentations:

- 1) Open the data file in the viewer software.
- 2) Push the indentation process button as shown in Figure 24.

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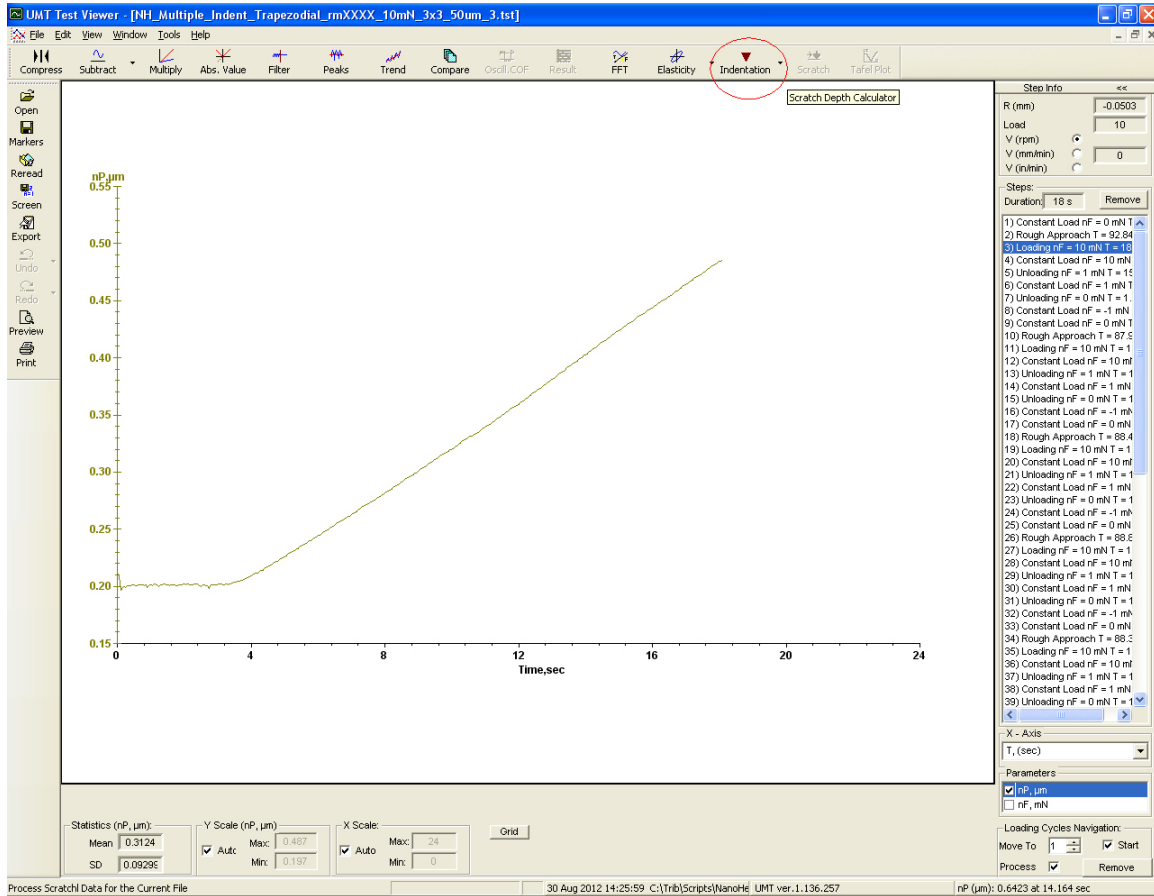


Figure 24: Indentation Process Button.

- 3) After processing a new window with 2 curves (Load vs. displacement (depth), and hardness and Young's Modulus vs. contact depth. As shown in Figure 25.

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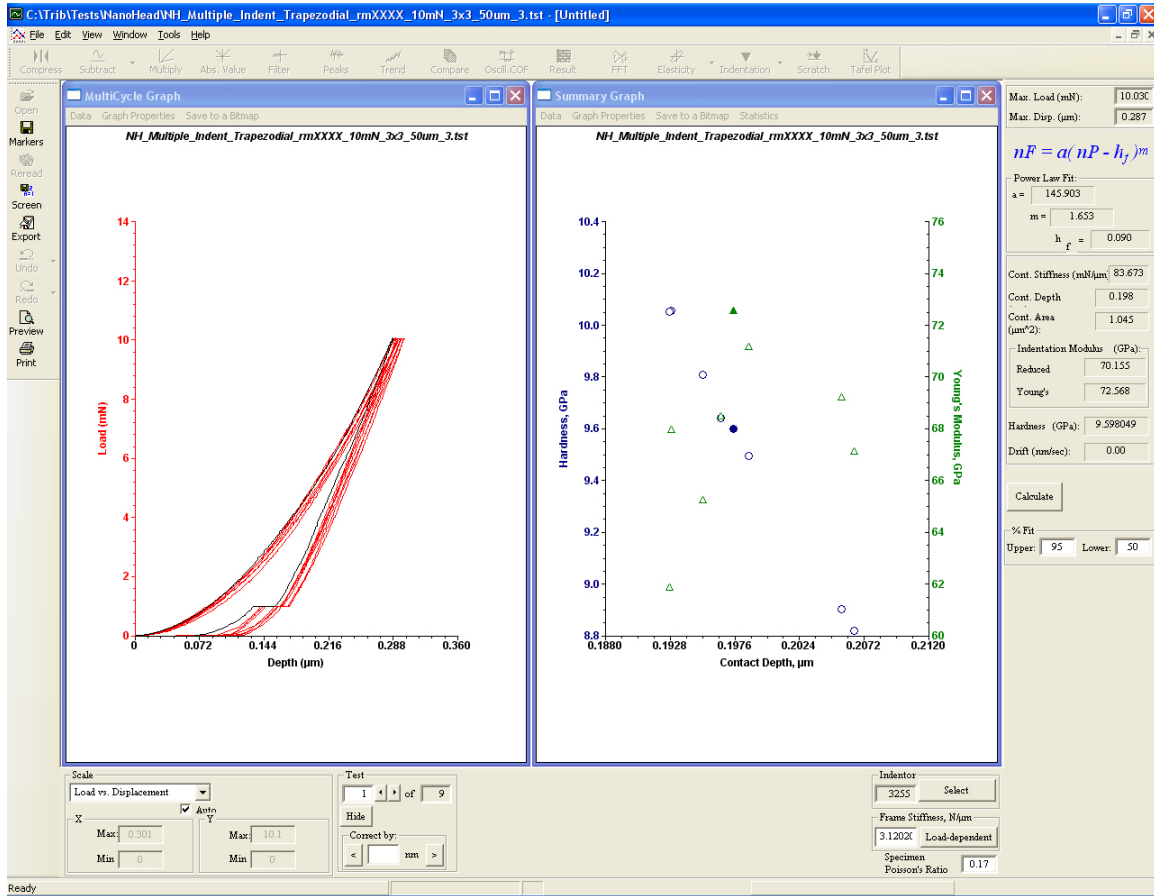


Figure 25: Dual Curve Window.

- 4) To adjust the detected contact point select the indentation test and use the “correct by” to shift the curve the entered amount, as shown in Figure 26.

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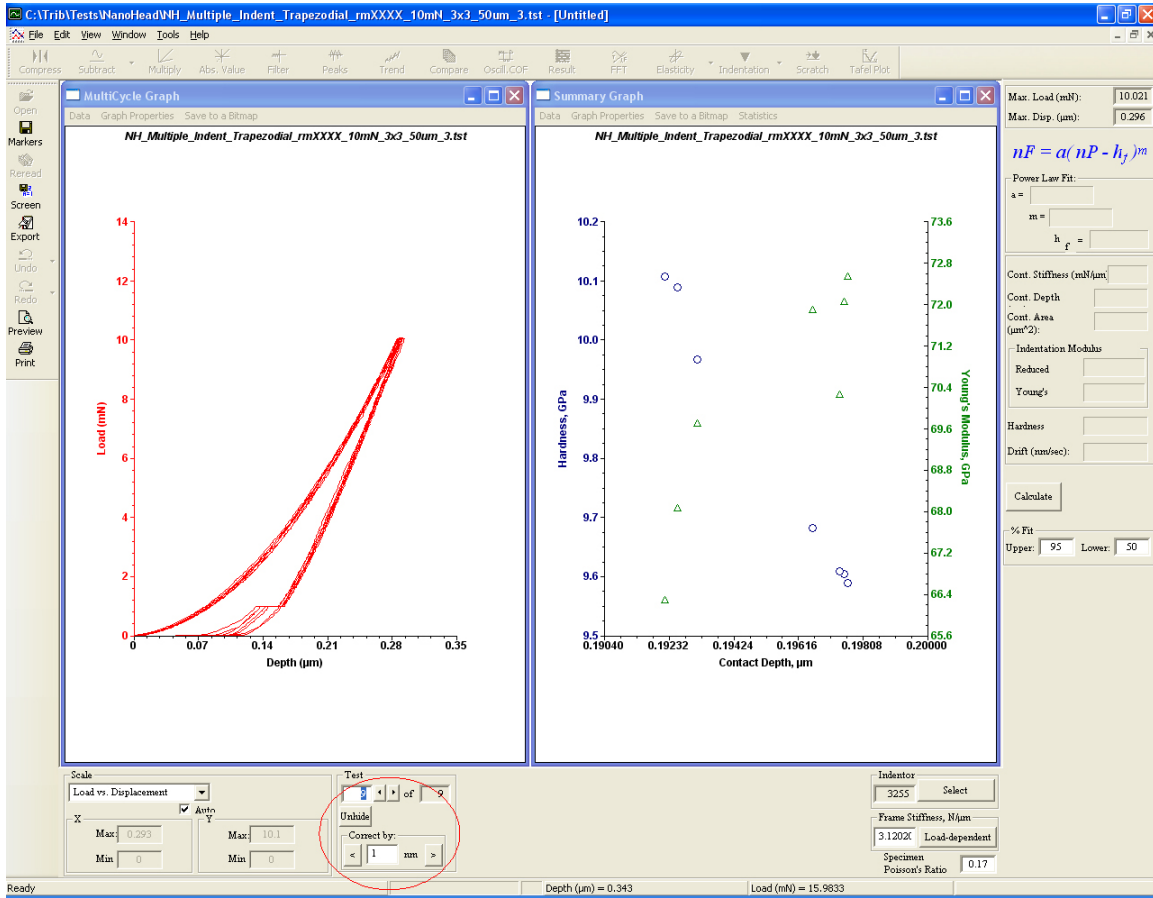


Figure 26: Adjusting the contact point of an indentation cycle.

- 5) Figure 27 shows an indent that is shifted by 100nm to the right and once corrected by the 100nm lies more in line with the other data points.

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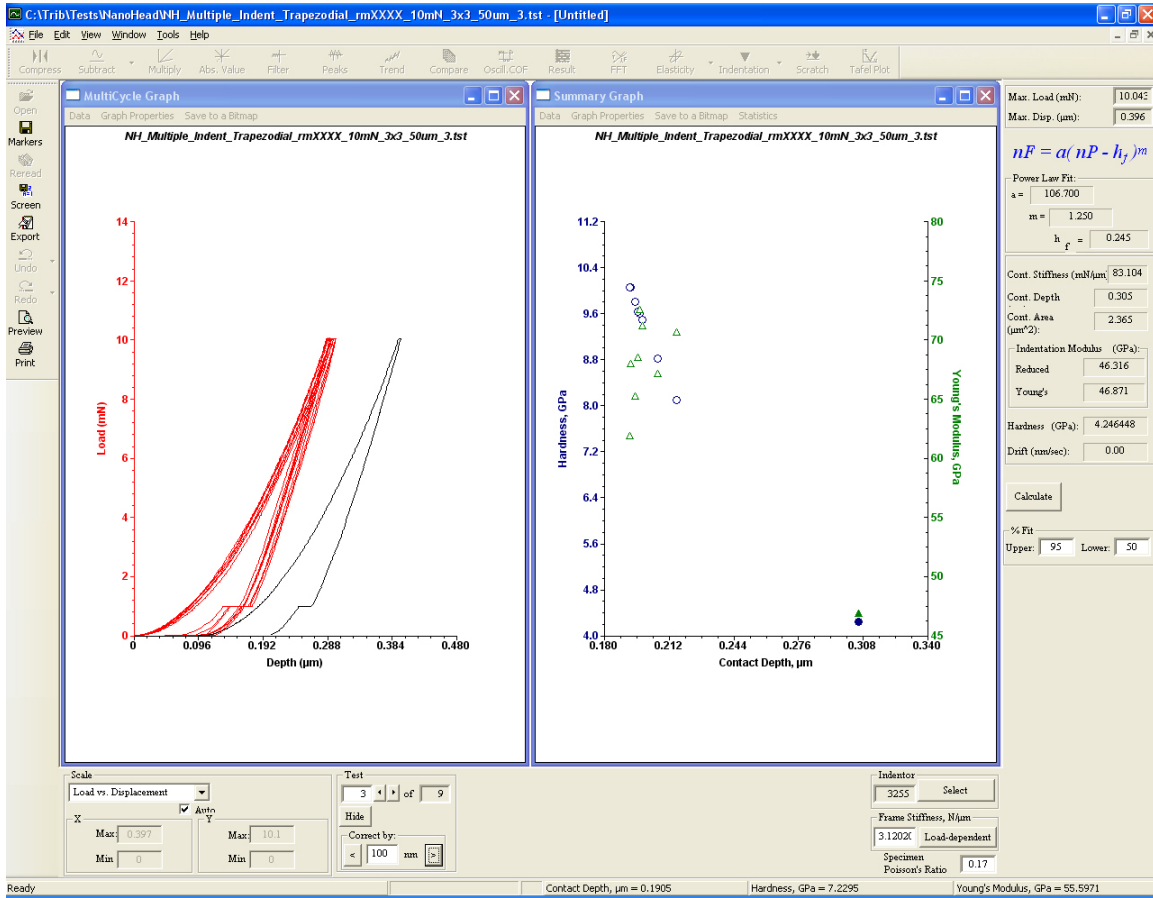


Figure 27: Improper contact point.

- 6) Figure 28 shows an indent that is shifted too far to the left and has to be corrected to be more in line with the other data points.

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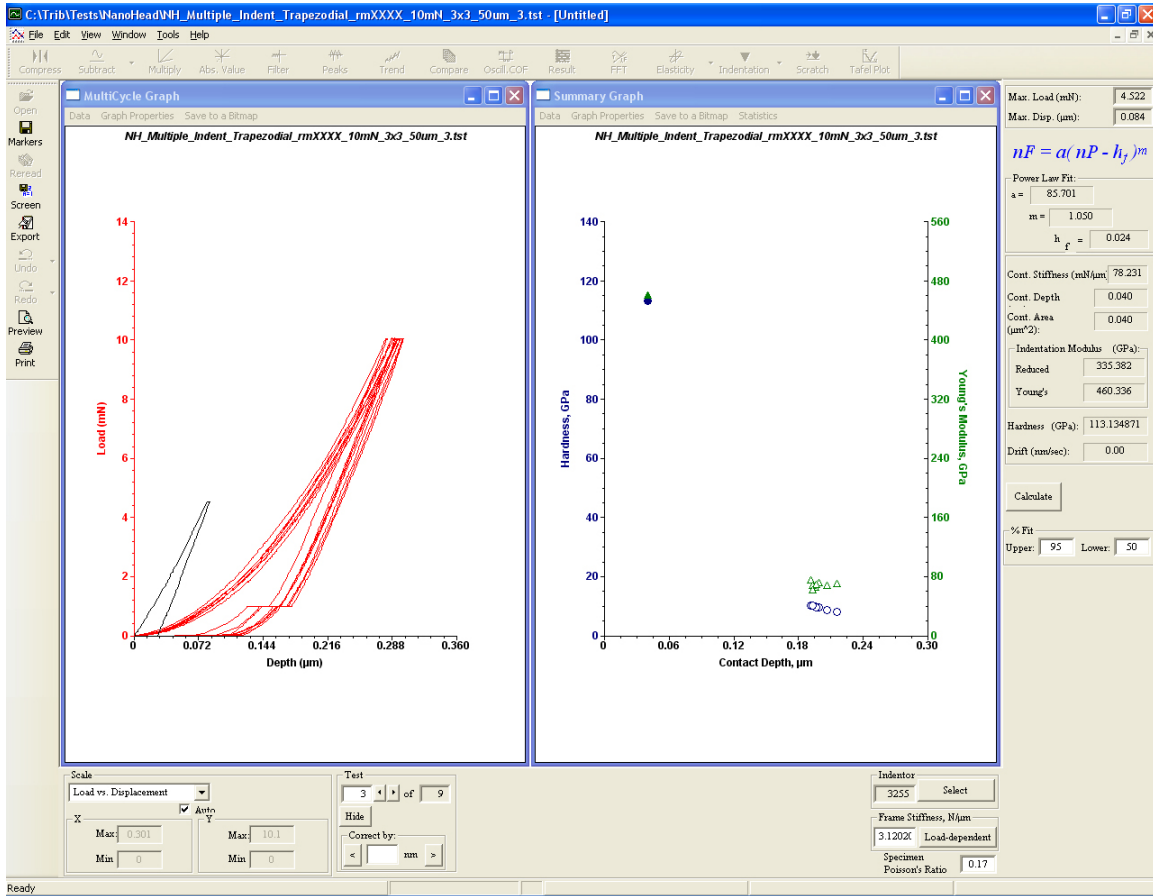


Figure 28: Improper contact point.

- 7) Figure 29 shows an individual indent's nP vs. time graph after data processing, the red line corresponds to the touch point (where the nP starts to rise).

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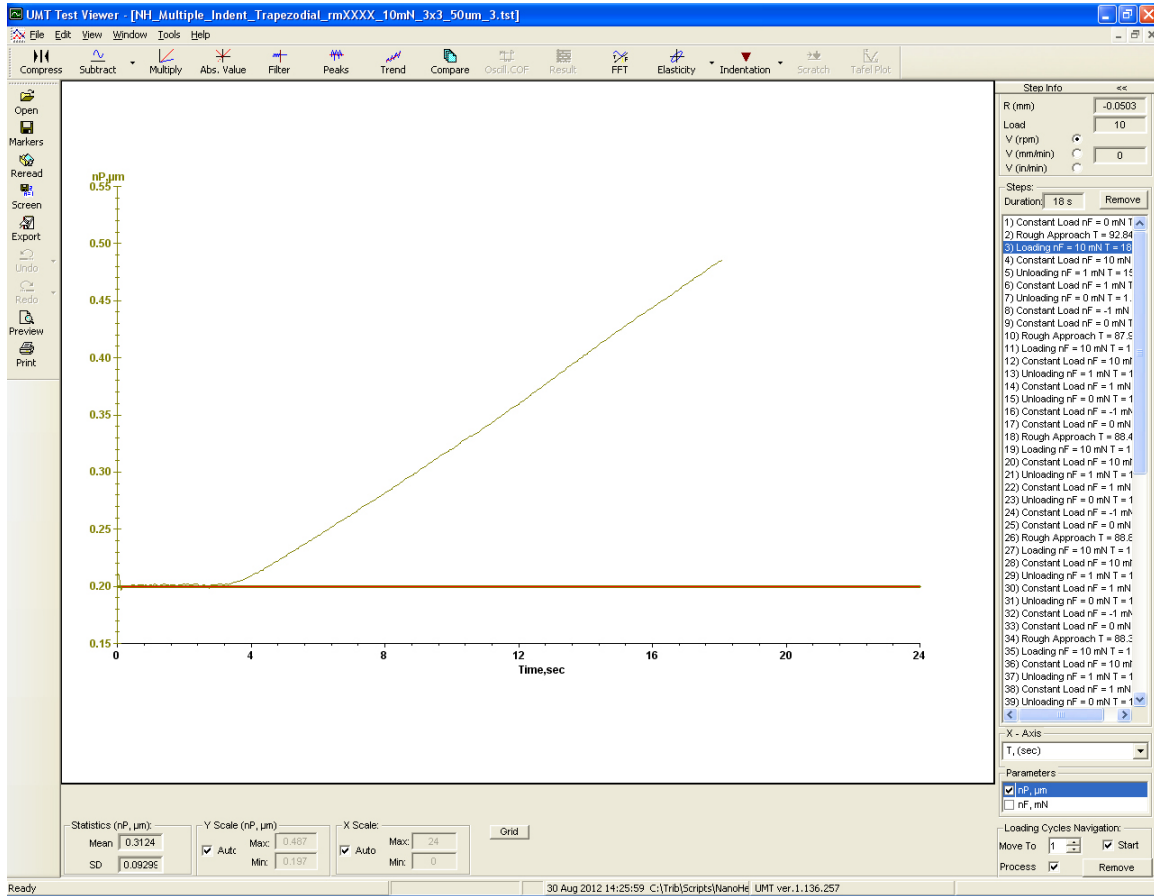


Figure 29: Properly set touch point.

8) Figure 30 shows an improperly set touch point (the same point as shown in Figure 28).

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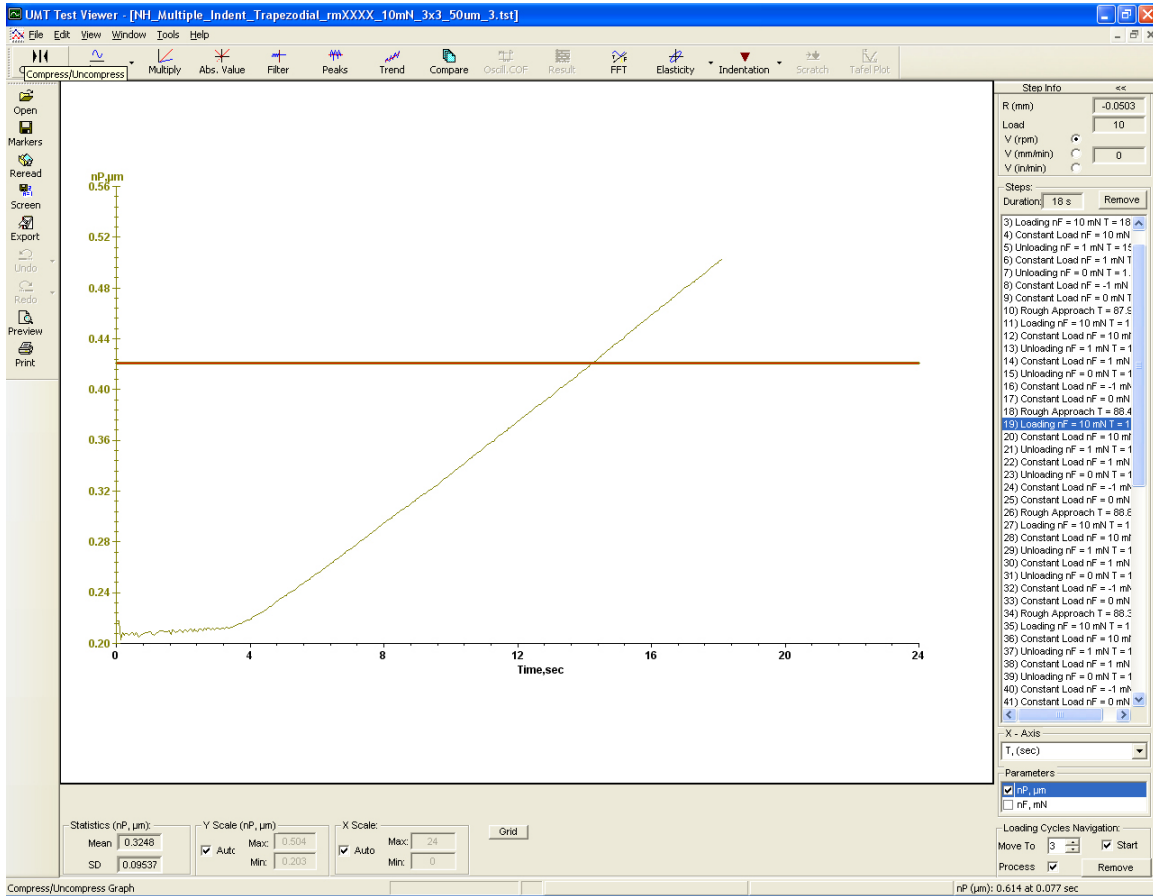


Figure 30: Improper touch point.

- 9) To correct the improper touch point you can right click outside the nP axis to place the touch point manually, or use the “correct by” method described in 4 above.
- 10) To cycle through the indents on the nP vs time curves you can use the “Loading cycles navigation” window highlighted in Figure 31.

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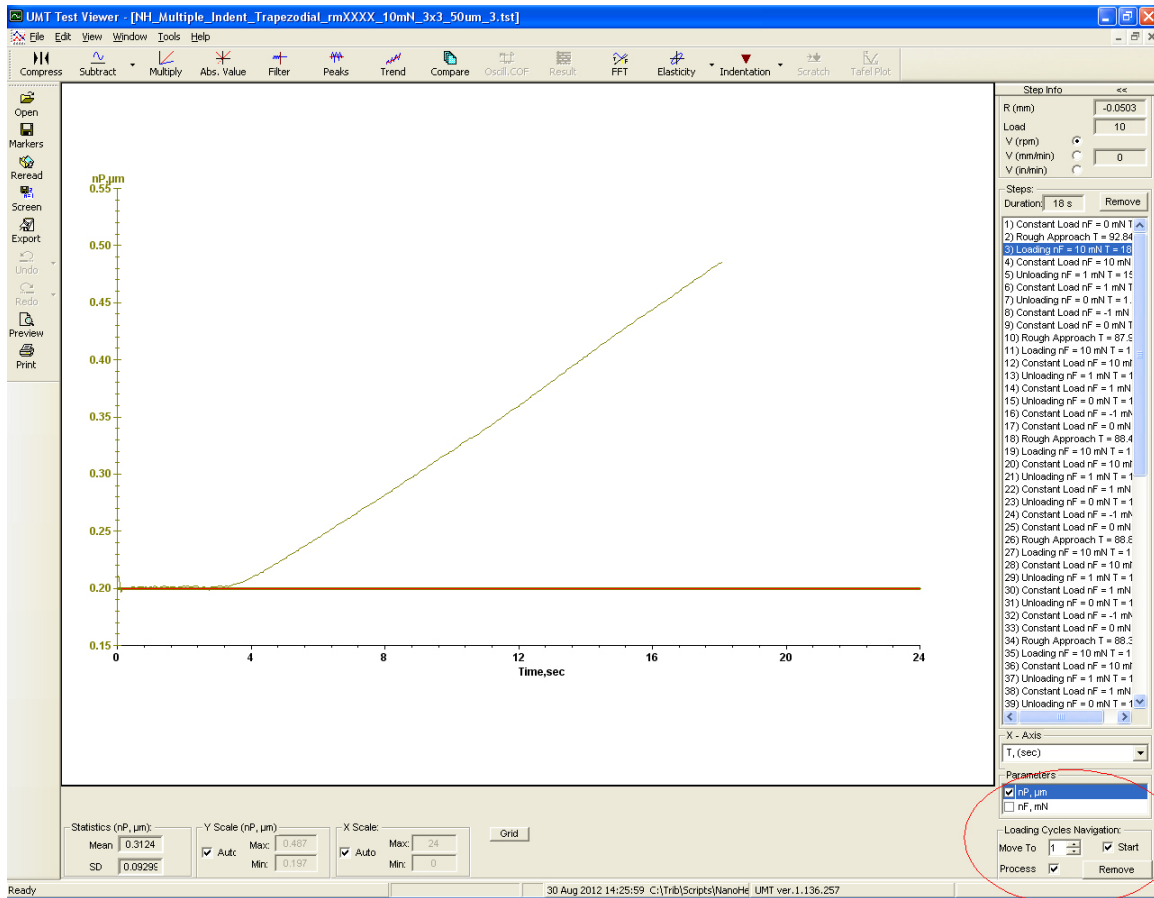


Figure 31: Load Cycle Navigation.

11) After the touch points have been corrected by either method the markers must be saved or the process will have to be repeated when the file is re-opened. To do this push the Indentation Button (data process button) and the Load vs. Depth and Hardness/Modulus vs. contact depth graphs will appear, then push the Marker button on the left as indicated in Figure 32.

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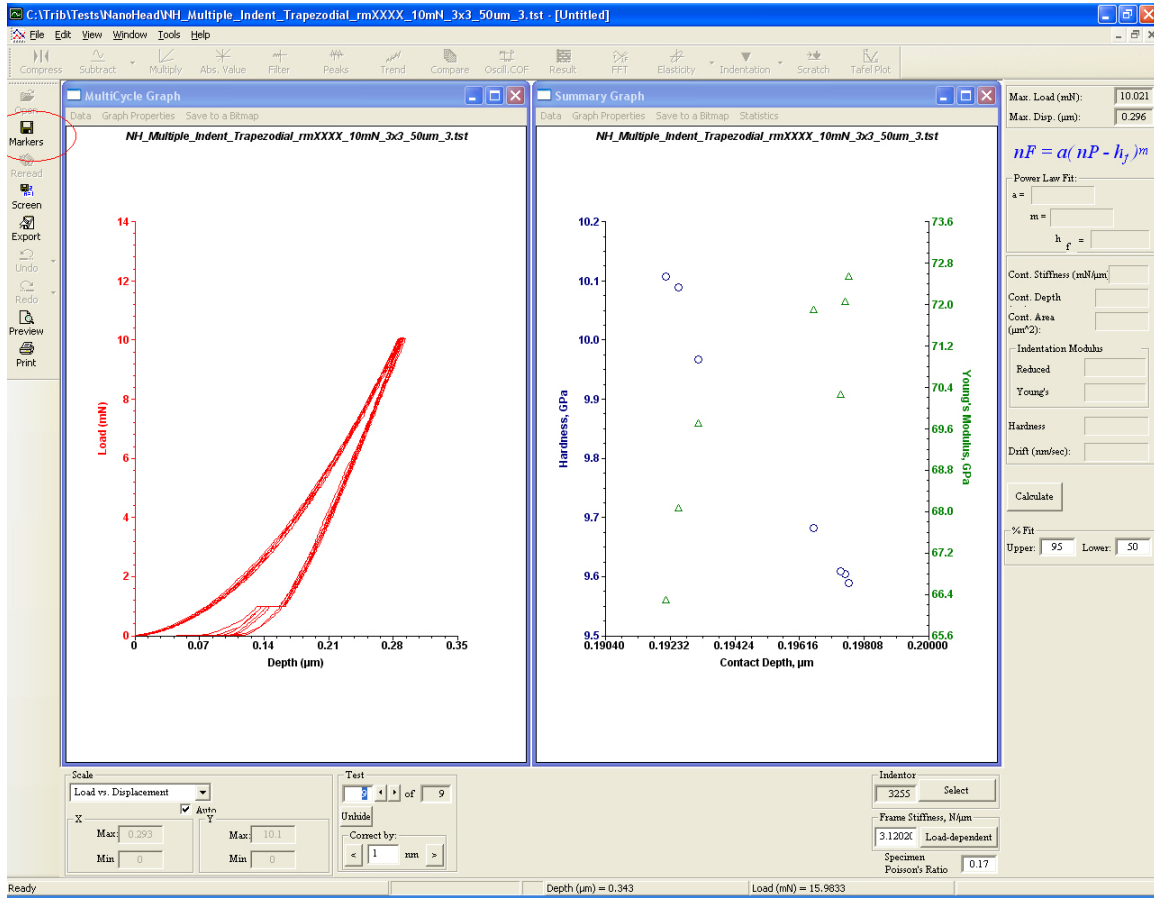


Figure 32: Saving Markers.

- 12) If one indent cycle seems to have a problem it can be hidden from view and data processing by pushing the “Hide” button as shown in Figure 33. Figure 33 has also had the y axes re-scaled for hardness and Young’s Modulus for a different graph appearance.

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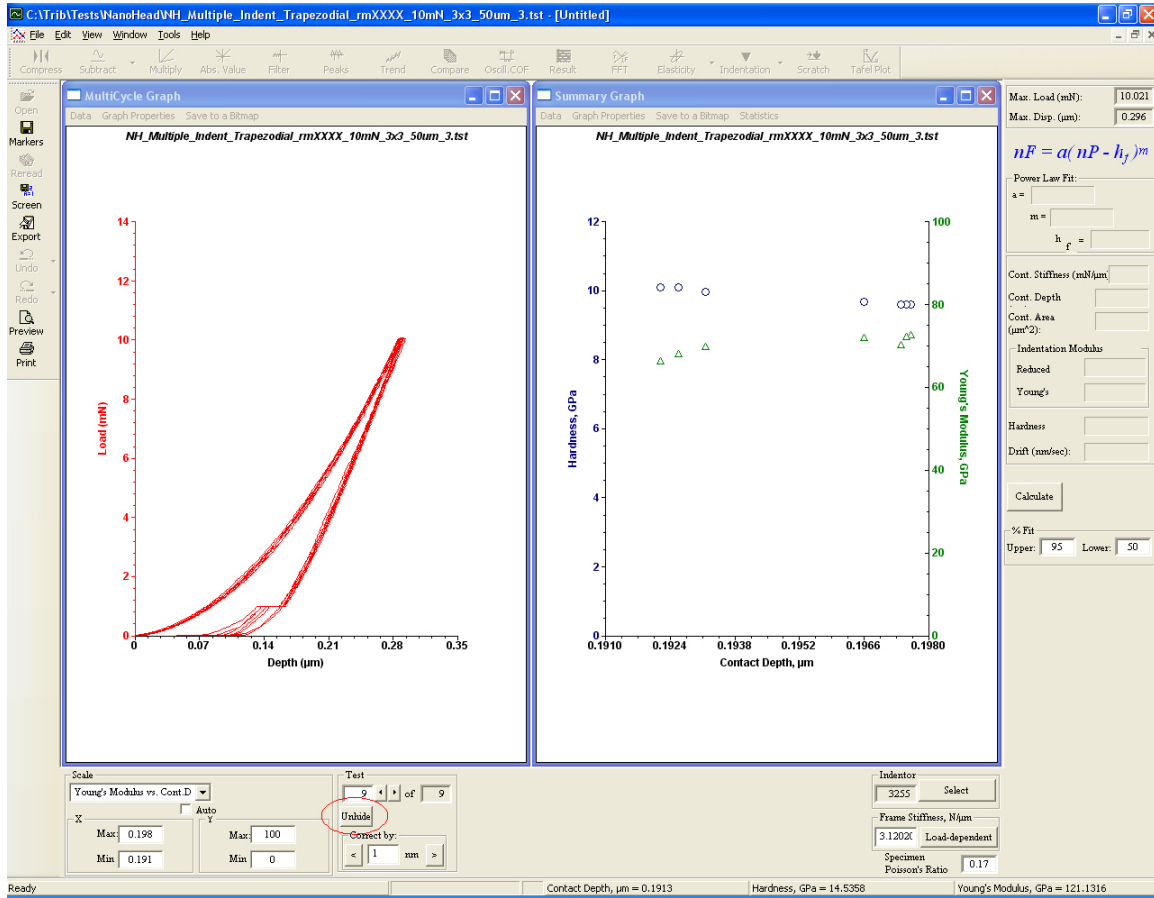


Figure 33: Hiding an indent cycle and re-scaled Hardness and Young's Modulus axes.

- 13) Figure 34 shows the same data as Figure 33, only with the indent #2 not hidden, you can see that its contact depth was significantly greater than the other indents, which is the reason for it being hidden from data processing:

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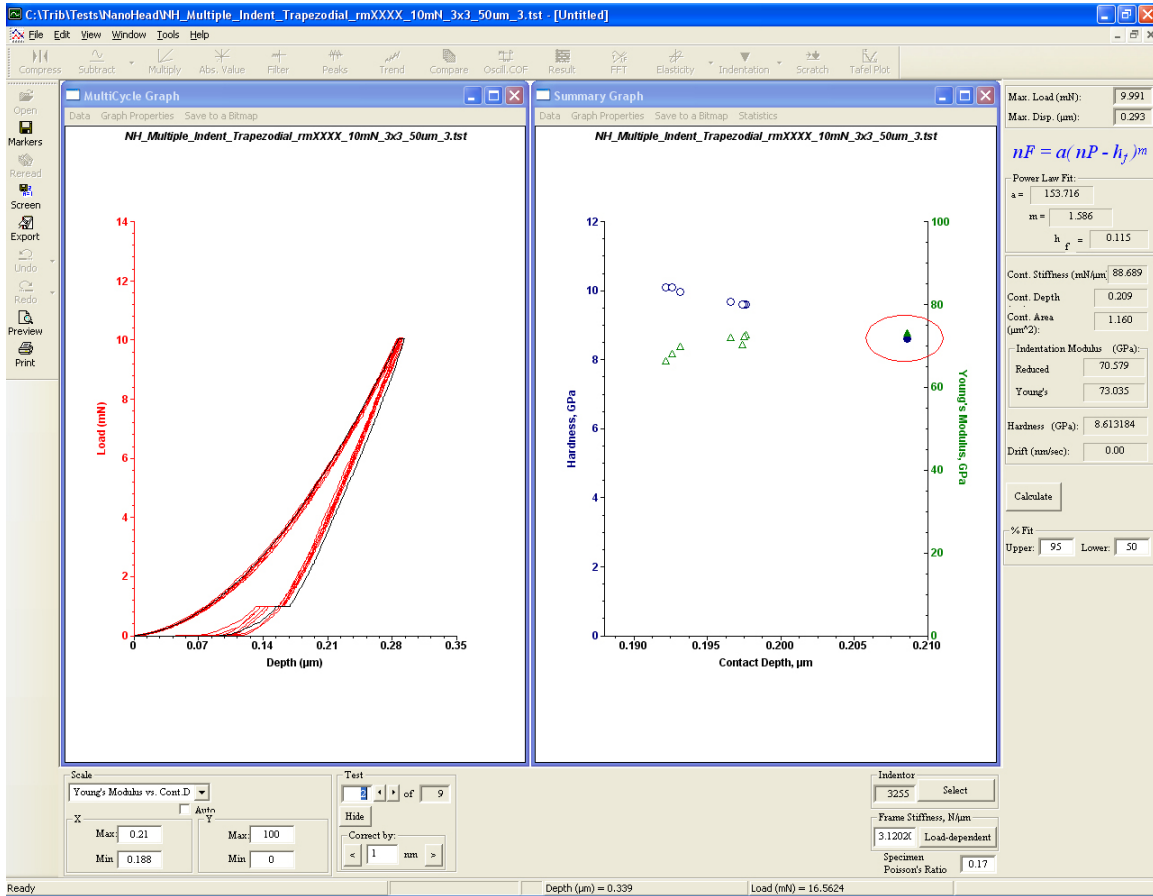


Figure 34: Indentation Data showing one indent with a significantly different contact depth, same data as Figure 33, only with an indent cycle unhidden.

14) For more detailed analysis see the Viewer Manual Provided.

Additional Notes on setting Contact Points:

- Typical Adjustment is 1-5nm
- Adjust until the beginning of the curve starts to appear noisy (this indicates that the “plateau” region of the soft touch).
- Zoom in to do the correction so that only around the first 10% of the indent is visible.
- If curves on different locations on the same surface go over each other in this range it is a good indication the touch point has been adjusted properly.
- For single indent test the touch point correction has to be completed for each test.

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- For multi-cycle tests the touch point is adjusted for the first contact only, with the others automatically being completed – this is for multiple indents in the same location (multi-partial-unloading)

7. Highlights / Critical Control Points

1. Do not open doors during testing.
2. Do not talk during testing.
3. Do not move during testing.
4. **For Optimal Results set a delay timer and test around 10:00 PM.**

8. Reporting

Report any problems to the Departmental Assistant and your Supervisor.

9. Regulatory / Standards

The following SOPs must be followed while using the Mechanical Engineering Labs:

ME0001
Mat0001
Mat0002

10. References

Bruker, 2012. *Frequently Used Test Procedures*, email correspondence.
Center For Tribology (CETR), 2009. *NH type Nanohead Manual*
Center For Tribology (CETR), 2007, *UMT Multi-Specimen Test System Viewer Manual Version 2.15 Build 90.*