General Instructions, Precautions, and Troubleshooting/FAQ for the MTS Nano Indenter XP at Drexel University

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*All users should read this document before using the Nano Indenter XP.

Beginning Note:

Whenever you are using the Nano Indenter, you must sign out your time on the sheets kept at the Nano Indenter, as well as reserve the time on the Faces online scheduling program. This is linked from the facilities page of the Nano Material's Group website at the URL: http://nano.materials.drexel.edu/ or directly at http://faces.ccrc.uga.edu/. If you do not have a Faces account, please contact Tim Kelly (tkelly@cbis.ece.drexel.edu) concerning this. It is important that both are reserved. Pricing and other general information about the Nano Indenter can be found at: http://nano.materials.drexel.edu/Facilities/facilities.html

An introduction to indentation can be found here:
http://nano.materials.drexel.edu/Facilities/TomNanoindentationBasics.pdf or here:
http://www.atos-online.de/atosneu/UMISTheory.pdf

Precautions

Before anyone uses this machine, they should be fully aware of certain things that need to be followed.

- Never eat or drink around the machine. This is obviously a general rule for any testing equipment. Also, open-toes shoes (i.e. sandals) are not allowed in the Nano Indenter lab.
- Know the name/composition of the material that you are testing, along with consideration for whether it will react with or cause damage to the diamond indenter. **Any material that possibly contains diamond particles should never be tested on this machine! If there are any questionable materials, please consult with Professor Gogotsi.**
- Research the typical and expected properties or range of properties of your sample before you run any of the tests. This includes the hardness, modulus, and Poisson's ratio. This is necessary to get reliable data, and will help you determine

if there is something wrong with your sample treatment. It is also necessary because very hard materials (i.e. carbides, nitrides) may damage the indenter tip at higher loads (>40-50 mN).

- Make sure that your sample is prepared to be as flat as possible. If your material is very hard, it is possible that the tool tip and machine hardware may be damaged when contacting the surface at an angle other than normal, in addition to giving useless results. Know the surface finish of your sample. Keep in mind that a rougher surface will not yield homogenous modulus and hardness values because of indenting at angles. An acceptable surface finish will be 1 μm or below. The optimum surface finish should be a lesser value than the penetration depth, in the nanometer range. The lower, the better for testing. You can use a profiling method to check your surface roughness.
- Never turn the power off from any system components (excluding Imagelite and monitor) while any test is running, or while Testworks is running.
- While a test is running, do not open the hinged door to the Nano Indenter. This will cause fluctuations in the gathered data. Also, while any test is running, try to keep noise in the room down to a minimum. If necessary, run your test overnight. Keep all doors to the room closed.
- Never touch the components inside of the Nano Indenter box except for the loading tray components. If you would like to change the objective (currently, 40X and 10X are available), please ask for Tim Kelly or the *super user*'s help. Do not try to do this yourself. If you would like to check or change the indenter tip (a Berkovich [normally used], a 1 and 13.5 μm radius conical tip, and cube corner tip are currently available), again, ask for Tim's help if you have no experience doing this. There are sensitive spring mechanisms that can get damaged without taking the proper precautions.
- Do not indent or scratch very hard materials with the cube corner indenter. Consult Tim, Professor Gogotsi, or the *super user* if you are not sure. This expensive indenter has the smallest radius (~40 nm) and builds the highest stresses at the point.
- If you are encountering a problem not addressed below under "Troubleshooting", please contact Tim or the *super user* and do not try to fix it yourself.

General Instructions

Starting the Program/Machine

- Assuming power is off when you reach the machine, make sure that the Dell computer power is turned on, the monitor power is on (power is far right button), and that the Imagelite power is on (front knob). If the machine doesn't appear to be energized, check that everything is plugged in, and that the red switch that turns on the power strip is to the on position. The control switch will most likely be found next to the Data Acquisition/Control Unit. When machine is energized, wait for Windows to boot up. You currently must use a login and password that is in the 'MATERIALS' domain. Then, start Testworks 4 by clicking the icon on the desktop.

- Assuming power is on and machine is running when you reach it, the first thing you want to do is log in. This is done by selecting the "User" menu, and then "Login". If your name is not on the pull-down list, please let the *super user* know. Although it is not critically important, please log out when you are done using the machine. The next thing you will do is choose your method. If you are doing indentation tests, you will usually choose "XP/XP Basic Hardness, Modulus, Tip Cal, Load Control". If you are doing scratches, you will choose "Scratch/Standard Scratch With Cross Profile". If you want to do only profiling, choose "Scratch/Single Profile". For the interested user, other available methods are described later in this document.

Loading Your Sample

- The next step is to mount your sample into the loading tray. Right click the screen with the schematic of the sample tray and choose "Load Sample Tray". This will move the sample tray away from the vicinity of the indenter head. To remove the loading tray from the machine, open the front of the machine, locate the black knob, and twist it counterclockwise *as much as you can* to loosen the tray. Be aware that some people have gotten confused when the piece with the black knob is loose, but the tray is not. Make sure to twist it all the way out until the tray is loosened. After the tray is removed, pick an empty spot to put your sample. For analysis, the sample should have a flat surface and be mounted tightly on one of the aluminum cylinders kept near the machine. To mount a sample, the hot plate near the sink in the room may be used in conjunction with the hot melt glue kept in the top drawer of the cabinet. If no empty cylinders can be found, do not remove anyone else's sample from one. Ask, and let Tim know about the shortage. If it is more convenient, the samples may also be mounted in a similar shape (cylinder of 3 cm diameter) in Bakelite, epoxy, or other suitable material. Please note that using epoxy as a mount introduces a high amount of compliance (which is bad and can alter modulus/hardness results). It is advantageous to mount in Bakelite if possible. To load your sample into the tray, use the hex key kept in the top drawer of the nearby cabinet to loosen the setscrew found in the holes of the loading tray. Place the tray on a flat and hard surface upside down, and make sure the tray is resting on something clean and smooth, like a piece of paper for instance. Put your sample through the back of the bored hole and let it gently fall until it is flush with the flat surface. Now use the hex key to tighten the setscrew back up. Your sample should be level now with the protruding flanges on the loading tray. If it is not, try again. It is extremely important that the sample is level with this flange! If it is not, there is a good chance that the indenter tip will crash into the sample if the sample is mounted just slightly higher. This will render the machine useless for quite some time. At this point, you can load the sample tray back into the place where you took it out, tightening the black knob again.

Important Note: It has been noticed that the flange on the tray might sometimes be directly below the indenter head after the tray has returned after indentation. It is possible for the flange in this position to come in contact with the indenter head if the tray is tilted down when it is almost removed. For this reason, make sure you always get into the practice of pulling the tray straight out (level) when pulling it out or replacing it.

Changing The Tip

If a tip change is required, you may do it yourself granted that either Tim or the *super user* has seen you change it at least once before and has given you permission to do it. Otherwise, ask an experienced user. Available are five different tips: Berkovich, #B1576, #B17--, a spherical indenter (radius ~13.5 um), a spherical indenter of radius 1 um (purchased and kept by Prof. Barsoum's group), and a cube corner indenter. To change to one of these tips, first move the stage all the way over to the right. Insert the two blue-flagged pins found on the Nano Indenter vibration table into the holes found in the indenter head. This locks the sensitive spring mechanisms in the indenter shaft so they cannot be damaged. Next, get the black tool from the drawer next to the Nano Indenter. It has a head on it fit for loosening a square nut. Gently place this into the indenter shaft and turn it loosely until you feel it grab the square nut. Turn it until the nut is fully loosened. Take care not drop the tip after it's been loosened (it's happened more than once before, even by yours truly), and set it in a safe place. Using a tweezers, remove the tip from the nut and put it in its respective container, and replace it with the new tip. Use the tool to replace it firmly into the indenter shaft, but do not over tighten it. Make sure to remove the two pins.

At this point, you need to do a microscope to indenter calibration test on the fused silica (Tools/Microscope to Indenter Calibration). The position of the current and previous tips will be different. The calibration will make five indents, and you will be asked to place the cursor in the middle indent. After this is done, make sure to go to "Tip/Select Tip" and choose the tip you just changed to. Results will be grossly wrong if you do not have the correct tip selected, as the area coefficients to describe contact area for each are very different.

Finding Your Location of Indentation

- You are now ready to find the location to make your indentations, profiles or scratches. First, you will want to make sure that you are in "Batch Mode". To do this, you can either click the icon near the top of the screen that shows a folder with 3 "III"'s coming out of it, or select "Mode/Batch Mode". Next, to find your location to indent, go to the inset on the screen on the left of the screen (video monitor). If you don't see an inset, make sure you are on the "Test" tab. You should see a grid and a shape like a five on a dice. If you don't, right click on the inset area, and choose "Nano Handset". This is the coarse positioning. It corresponds to the loading tray. Use this first to find the general location to indent. Simply click with your mouse to move the location of the cone, and then right click and select "Move To Target" to have the machine move there. The other icon on this inset corresponds to the position of the indenter head, and will usually be red.

- Once you have the coarse positioning, right click in the area again and choose "Nano Video Handset". Now, you should see a black and white screen corresponding to what the video monitor is seeing. Therefore, if it is black, the level of light on the Imagelite needs to be increased. If it is white, likewise try turning the light down. To focus, use the icons found directly below the video inset to bring focus up or down, fast or slow, until your sample surface is in view. Now, you can click your mouse to move in any direction. Simply click the mouse on the screen location that you want to move to. By holding the mouse button down, the stage will continuously move in that direction. Find the starting location for your indentations, scratches, or profiles.

Running The Test For Indentation

- Making sure you're in batch mode, click on the "Define" tab. Throughout the setup, you will be clicking the "Next Step" icon to proceed. Select Batch - choose "Start A New Batch". Select Options For This Batch - select any of that that you wish. The option "Delay Before Running" is especially useful when doing indentation at small depths, since there is generally less noise in the building at late hours. If you anticipate running shallow indentations, try to schedule during the late evening hours. Create Sample - type in any sample name that will allow you to later identify what you indented. Surface Find Parameters – you can usually leave all of these to default. The "Delta X" and "Delta Y" are the locations relative to your defined indentation where the machine will crudely determine the surface height by quickly approaching the sample. "Allowable Drift Rate" should not be set any higher than .5 nm/s for any reason, and .1 or .05 nm/s is considered optimum. This is the allowed velocity of the indenter head (usually due to vibration and noise) that the tool is allowed to electronically drift in a closed loop before the machine proceeds to begin the test. Surface Approach Parameters – you can usually leave them at the default values. "Surface Approach Distance" should be increased to 2000 or 3000 nm if you have run tests before on your sample and frequently receive the error similar to "Test did not run because indenter was already on the surface". After the crude surface height determination, this value is how high above the perceived surface the indenter starts its more sensitive approach.

- Required Inputs – "Percent to Unload" is usually set to 90%. This is the unloading value that a hold for thermal drift occurs. If you do not wish to consider thermal drift, set this value to "99.9". Entering "100" is known to cause a zero error in the machine. "Maximum Load" is the peak load on your sample. If you are not sure what to enter here, enter loads in the range of 15-35 mN. Usually, if your material is sufficiently hard (>20 GPa), you should stick to smaller loads as to not damage the indenter head. If the material is very soft, such as single-crystal aluminum, 15-35 mN will create large indents, with effective diameters of about 30 um across. "Load Rate Multiple For Unloading" corresponds to time for the sample to unload in relation to the loading time. In most cases, this should be set to "1". A number less than one increases unloading time; greater than one decreases unloading time with respect to loading time by that factor. For example, if loading time is 30 seconds and you wanted an unloading time of 3 minutes, this number should be set to .5 min/3 min = .167.

"Number Of Times To Load" will calculate material properties at different loads in the sample indentation space. The different loads are set by a decrement factor of .5. So, if your maximum load is 100 mN and it is loaded 5 times, it will first take properties by loading to 6.25 mN, then 12.5, 25, 50, and finally 100 mN. "Peak Hold Time" is the amount of time that the machine will hold at the maximum load. This should be set usually to 15 or 30 seconds, but if your material creeps, set this higher. "Time to Load" should usually be set from anywhere from 30 to 60 seconds. Again, the more the material creeps, the higher this number should be. The collection rate of the machine is 5 Hz, so too short of a loading time results in not enough data points collected for analysis. "Poisson's Ratio" is the value of the indented material. For most metals, it is about .33. For other stiffer, harder materials, you can use a value between .17 and .25, but look this up if possible.

- <u>Define/Review Test Locations</u> – Here you tell the machine where to place the indents or scratches. You will see a number of icons to choose, and most are self-explanatory. If you have individual locations to test, choose the place within the crosshairs and click "Add Test At This Location" until you have enough to make a good data analysis (usually 30-40 indentations are enough for statistics). The other option is to create an array of tests. Select "Define Array of Tests…" and enter the size of the array as well as the spacing. You may also save this array. <u>*One important thing to note: whoever made the direction sense in this program used the tray movement sense and not the screen movement sense. On the screen, consider what you "normally" think of as positive x and y directions. This is actually the sense that the stage moves. On the screen, in their coordinate system displayed in relation to normal positive senses, negative x actually points in the positive direction, and negative y points in the positive direction. When you define spacing for the array, a "positive" value (i.e. 20 um) defined for x and y will yield spacing in the traditional positive sense, making your starting point the lower left-hand corner of your array.*</u> If you wish to move the stage to specific coordinates during this step, you can right click in the optical inset area and select "Move Abs/Rel", keeping in mind the direction sense mentioned above. After clicking "Next Step", you will be asked whether you want to add another sample. If you wish to do indentations with different conditions, i.e. maximum load, choose "yes". Everything will be run successively. If you now go to the "Test" tab, you should see parameters for all of the indentations you have just defined, including coordinates. The last step is to hit the green "play" button. This starts the test. However, it is not usually wise to walk away at this point. Watch the machine gather information for at least two indentations to make sure everything is agreeable. Most commonly, the machine may lock up at the point where the drift correction is just beginning. You should see in real time a load/displacement curve being generated on the screen after about 5-10 minutes. Unless really pressed for time, it is usually a bad idea to cancel the drift correction – this correction has been known to take upwards of 45 minutes to 1 hour (but this normally isn't the case). Never cancel the drift correction before a measured number is displayed as it will cause the machine to crash.

After your tests are running, turn off the Imagelight.

Running The Test For Scratches

- Follow the same directions for running the test for indentations until "Required Inputs"…

  If scratching very hard materials, consult Tim or the *super user* first concerning inputs. In general, one should not be making scratches with the cube corner tip, unless it is on something very soft, like a polymer.

  Required Inputs – "Scratch Length" is typically 100 or 200 um, but any value is accepted. "Scratch Velocity" is usually between 2 and 10 um/s. Harder materials will need slower speeds. "Scratch Orientation" is the direction of the scratch to go from your starting point. 0 is down, 90 to the right, 180 up, 270 to the left. If using the Berkovich tool to scratch, you should always scratch in the direction of a point for the best results. The tool is only allowed to be fixed in two orientations, so you will always get a point by entering 0 or 180. It is best to check the orientation by an indentation first though. ! Note !: Avoid using negative degree values here, because experience has shown that it is not as it would seem, i.e. –45 doesn't yield the same as 315. "Maximum Scratch Load" should be set at around 15-20 mN if you are not sure about how your material reacts to scratching. The upper limit should normally be about 80-100 mN for this parameter. "Starting Scratch Load" is 0 if you wish to have a scratch of linearly-varying depth, i.e. for 100 mN scratch of length 100 um; 0 mN at the beginning, 5 mN at 5 um length, 60 mN at 60 um, etc. "Cross Profile Length" is the distance to profile your scratch after it has been performed. "Perform Cross Profile" is set to 0 for no post profiling, 1 for profiling. "Load Applied During Profiling" should be set to 50 uN, but if the profile yields poor/unstable results,

try increasing this amount of force applied. “Cross Profile Location” is the location on your scratch that is to be profiling in terms of load.
*Follow same directions as for indentations for the “Define/Review Test Locations”.

Note: It has been observed that the defined starting position of your scratch isn’t actually where the machine begins the scratch. According to MTS, there is a profile offset of 20% of total scratch length, which apparently cannot be altered. So, for a 500 um scratch, it will begin 100 um away from your defined position.
The method first profiles the original surface (your scratch length plus 20% on each end), makes the scratch, and profiles the scratch length again. If you select post profiling, it will do a cross-profile afterward, across the scratch length.

<u>Running The Test For Profiling</u>

- All aforementioned directions are the same as for indentations except for “Required Inputs”…
  <u>Required Inputs</u> – “Profile Load” should be set to 50 uN (or lower), but if the profile yields poor/unstable results, try increasing this amount of force applied. “Number Of Points Per Profile” is the data points collected. Higher resolution is usually better, but per 100 um, 300 is usually good. “Profile Length” is just that. “Profile Orientation” is direction of profiling. 0 is to the left, 90 is down, 180 is to the right, and 270 is up. <u>! Note !: Avoid using negative degree values here, because experience has shown that it is not as it would seem, i.e. –45 doesn’t yield the same as 315.</u>

<u>Post-Test Analysis</u>

- After your test has fully completed, you will notice that the “play” button has restored. This is when the test is considered done. The test will mainly have saved data relating to modulus, hardness, time, loading, and stiffness, along with all of your defined parameters. Note that it will also have saved a “batch file” that includes the coordinates of your indentations.
- You may also monitor your data gathered as it is being collected. For example, you may click on the “Review” tab at any time, without interrupting the test, to see the results obtained for indents already made.
- After the test has completed and you are viewing the results, it is good to choose the tab “Excel” and “Output Sample To Excel”. This outputs your data in Microsoft Excel format so that you can analyze it with your own software

programs (i.e. SPSS, Origin, or Excel). The output includes all gathered raw data and results, along with defined experiment parameters. This file will not include the coordinates of each indentation.

- You can view different collected information for any indentation test under the "Review" screen. By default, it will show you modulus vs. depth. However, you can right-click in this plotted area and will see a list of options pop up. You may choose "Y-Axis Channel" or "X-Axis Channel" and change the display to whatever you wish (time, hardness, load on sample, etc.). Note that the channel units may also be changed under this pop-up menu.
- More than one indentation test result may be displayed on this screen at a time. Simply check the boxes next to the indentation number on the left-hand side of the screen.
- The currently displayed graph can be captured as a picture in Microsoft Word by using the old "Control+C" to copy and "Control+V" to paste it in Word.
- You can access your results via the generated Excel or .mss file through the Drexel network. The group will be "MATERIALS" and the computer is named "Nanoindent". You should choose the directory "Samples", then the folder with your name, then the folder of the day the experiment ran. If you don't see it, chances are you were logged in under someone else's name. In this case, you will need to move the dated folder into your own. Also, please note that once every few months the "Samples" directory will be moved to another folder for older samples. If you need to know the user name and password for access, please contact the *super user*. To access your results directly from the Nano Indenter's computer, use the path C:/Program Files/MTS Systems/TestWorks/Samples. Older samples will be kept under D:/.
- You may save your files to CD or floppy disk. It is recommended to use Roxio CD creator (installed on the computer) to copy your files to CD.

Currently Available Testing Methods

Scratching Methods:

There are three methods available. One is just a simple profiling, "Single Profile", of a surface where the distance, profiling speed and applied force are asked for. The second is "Standard Scratch with Profile" which is discussed earlier in this document. The third is "Standard Scratch with No Profile" which does the same thing as the second, without profiling in the beginning or the cross-profile. Doing just a profile on your sample surface may be very useful to gauge what your surface roughness is.

Indentation Methods:

All of the useful indentation methods should be able to be found in the "XP" method folder. Below is the current order that they are listed.

Quick XP Basic Hardness, Modulus, Tip Cal, Load Control - no thermal drift.msm

This method is the same as "XP Basic Hardness, Modulus…" except that the thermal drift hold and collection times have been set to 1 second. If thermal drift correction is not necessary for your experiment, it is recommended to use this method so that each test is about 1 minute shorter. Inputs for this method have been discussed on pages 6 and 7.

Quick XP Prescribed Partial Unload Hold (for Si).msm

This method was originally made for silicon, but may be useful for other materials. It does not allow for a thermal drift correction to be made – rather this test segment is replaced by a hold at a prescribed unload value while data continues to be collected. Besides the usual test inputs, the user can specify "Partial Unload Hold Time" and "Partial Unload Value".

XP Basic H and E at a Series of Displacements (no thermal drift).msm

If you would like to go to a specific depth instead of a load, you may use this method. Required inputs are a bit different than the standard method. You will choose a "Depth Limit First Cycle" and "Loading Rate First Cycle" in mN/s. The other input different than the standard method is "Depth Limit Increment Factor", which if you are doing multiple loadings will increase the "Depth Limit First Cycle" by a certain percent. For example, if you set "Number of Times to Load" as 3, "Depth Limit First Cycle" to 500 nm and "Depth Limit Increment Factor" as 50%, the first cycle would go to 500 nm, the second to 750 nm and the third to 1000 nm. As the name suggests, no thermal drift is considered in this method.

XP Basic H and E at a Series of Displacements (thermal drift).msm

This is the same as the above method except that thermal drift is collected.

XP Basic Hardness, Modulus, Tip Cal, Load Control Const Cycle - no thermal drift.msm

This method has the same set of inputs as the usual method, except that the cycles are repeated at the same maximum load – not at 50% decrement factors. No thermal drift is considered in this method.

XP Basic Hardness, Modulus, Tip Cal, Load Control Const Cycle, Strain Rate.msm

This method is essentially the same as the previous one, except that the loading rate is defined by an inputted "Strain Rate", or a constant value for the loading rate divided by load – useful for generating stress-strain curves with spherical indentation. Required inputs include an "Unload Rate" in mN/s and an "Unload Value" in uN (as opposed to a %). Thermal drift is not considered in this method.

XP Basic Hardness, Modulus, Tip Cal, Load Control Const Cycle.msm

Identical to “XP Basic Hardness, Modulus, Tip Cal, Load Control Const Cycle - no thermal drift.msm”, except that thermal drift is considered.

XP Basic Hardness, Modulus, Tip Cal, Load Control Varying Cycle.msm

This method is to do incremental loading steps for maximum loads in loading cycles. One required input is “Load Decrease Factor”, which is a % of your maximum load. The best way to illustrate the type of thing you can do with this method is to give an example… If your “Maximum Load” is 100 mN, “Times to Load” is 8 and “Load Decrease Factor” is .1, then the cycle load limits will be 100, 90, 80, 70, 60, 50, 40, 30 mN on the same indent. There is no safeguard in place that makes sure the next load is a positive value, so take care to make sure that each load will be positive (in other words, that “Load Decrease Factor” times “Time to Load” is not greater than 1). Otherwise, the machine will hang forever waiting for the negative load. Thermal drift is currently considered in this method.

XP Basic Hardness, Modulus, Tip Cal, Load Control, Strain Rate.msm

This is the same as the standard method, except that “Strain Rate” is asked as a required input and unloading rate conditions are not asked for. Rather, the unloading rate will be the same as the loading rate value at the end of the loading segment (usually produces quick unloading times).

XP Basic Hardness, Modulus, Tip Cal, Load Control.msm

This is the standard method whose inputs are described on pages 6 and 7.

XP Basic Tip Cal.msm

As the name suggests, this method is mostly used for running tip calibration indents. This method will produce an array of indents, each which has a different load than the others. The parameters for input include “Load Limit of First Test”, which will be the maximum load considered (if you are calibrating a tip, set this to 700 mN), “Do Drift Hold When Load Below”, which is the critical load below which thermal drift collection is taken, and “Load Decrement Factor” (for tip calibration, set this to about .8 or .85). The “Load Decrement Factor” is the multiple of the previous indent value. For instance, if a 7x7 indent array was defined with max load at 700 mN and decrement factor of .8, the first indent would be at 700 mN, the second at 700*.8 mN, the third at $700*.8^2$ mN, the fourth at $700*.8^3$ mN, etc. until the last indent would be at $700*.8^{48}$ or .0156 mN.

*Other methods, namely those used for the continuous stiffness measurement (CSM) option, are also listed but not currently of use for us. Some of the above methods have been created by the *super user* because they were required for a specific application. If you are interested in creating a method with a specific purpose in mind, please let the *super user* know – it may be possible to create.

Troubleshooting/FAQ

Below are known questions to come up and things that have happened with the machine. If you have any additional problems or questions, please contact the *super user*.

Q: I need to get trained. How do I do that?
A: Currently, Tim Kelly is in charge of training people on the Nano Indenter. He's a friendly guy and will be able to help you with your needs. He's usually here from about 8-4 if you need assistance or training. Training must be scheduled with Tim.

Q: The sample tray seems like it's stuck. I'm frustrated and want to punch the machine.
A: First, try to calm down. The piece with the turning knob is probably removed, so place it back in. Then, turn it counterclockwise all the way to loosen the tray out.

Q: In the middle of an experiment, the program just suddenly quit on me. What happened?
A: Unfortunately, the current Test Works software is still a little buggy. The best remedy is to restart Test Works. Remember that your defined batch should have been saved in the dated directory, so you may not have to redefine everything.

Q: I command the stage to move, but it looks like molasses and takes forever to go anywhere. I also look at the stage and it's not moving. Why?
A: You probably didn't do anything wrong, but again it is a bug in the program that comes up once in awhile. If you notice this happening, press "Cntl+Alt+Delete", go to the "Task Manager" and close any applications of "Testwrks.exe" running. There will probably be two running if you have this problem. Restart the program.

Q: When I try to type in a name for my sample during the experiment setup part, the computer simply doesn't respond to the keyboard.
A: Restart the program.

Q: Right before my test was going to start, the machine locked up. Is everything lost?
A: No! You can quit the program by pressing "Cntl+Alt+Delete", go to the "Task Manager" and close any applications of "Testwrks.exe" running. When you restart the program, chose "Batch Mode" and right click in the area where your programmed tests were displayed. You will see an option "Recall Last Batch". Choose this. Hit the "play" button again and hope for the best.

Q: I notice that the positioning on the schematic of the loading tray on the screen and the actual positioning are not the same in one or both axes. It seems like something got offset.
A: You need to go to “Configure”, “Device” and select first the “X-Position” channel. Click on calibrate and wait for the machine to do its duty. Repeat this for “Y-Position” channel. Now, everything should be ok. Alternatively, you can also reboot the machine and it should be correct.

Q: When I look at my results, I notice that the curves look very strange – maybe not even physically possible. What’s going on?
A: Chances are, one of two things occurred. If the curve is REALLY messed up (i.e. max load is 0.01 mN, displacement is only negative), the indent likely was not made. If it is shifted unrealistically, perhaps the thermal drift was accidentally on and taken at 99.9% unload. To turn it off, in the results screen, look for “Thermal Drift”

Q: I can’t seem to see my sample under the objective. The display screen is black/white.
A: Chances are, either the Imagelite is off, set too high or too low, or the objective has run into the sample. Check both of these things. Try also to focus on the partition space between your sample and mount. Usually, they will be completely different colored and even with little focus you can see the partition enough to clearly focus on it.

Q: I walked in to use the machine and the power is off. Nothing turns on.
A: If the rest of the room has power, chances are that the red switch for the power strip is turned off. This red switch can usually be found in the space between the Data Acquisition/Control Unit and the Nano Indenter box.

Q: Why am I getting completely scattered results on my indentations? Is the machine broken?
A: It’s possible that the machine is not working properly 100% of the time, but more likely is that your sample is not flat enough, or you are indenting on a highly inhomogeneous sample.

Q: In my results, I see some things that have only stars for the indentation results. What is this?

A: This means that the machine wasn't able to locate your surface. This is semi-typical to happen once or twice over 50 indents, but if it is happening more than that, again check the quality of your surface.

Q: Somebody else's sample is currently running, but I'd like to see some results that I got on the Nano Indenter computer from before. Do I need to wait until it's done?
A: No, you don't. You can minimize the application of Test Works that is running the current experiment. Then, start a new application and login. Choose "File", "Open Sample", and go about your business. The only thing that is not a good idea is to export large sample indent arrays (>50) to Excel while another experiment is running. It may cause a program error or instability.

Q: I need to get my sample out of the Nano Indenter, but someone else's sample is running. What do I do?
A: The best approach is to try to foresee if you will need this sample right after indentation. Then, leave a note for the next user to take it out before they start their test. Otherwise, you will need to wait. It is of utmost importance to have respect not to disrupt any testing that is going on.

Q: I can't see the indentations on the screen after the test completed. Why?
A: The reason is most likely that they are too small to be seen. Also, keep in mind that the objective will probably be at 40X, so some smaller indents will need to be viewed under higher optical objectives or SEM.

Q: My indentations didn't go where I wanted them to go. They were "offset". Why did this happen?
A: This is because of the microscope to indenter distance calibration. From time to time, this distance needs to be calibrated. Pick an area that is not important for indentation under the current view, and go under "Tools" and "Microscope To Indenter Calibration". The machine will create five deep indents (make sure not to do this on a very hard material) and will tell you to position the crosshairs in the central one.

Q: I would also like to have this Testworks software in my computer because it does some super neat things easier than other analyzing programs. Can I install it on my computer?
A: Yes, you can. Contact Tim if you are interested in doing this.

Q: Why does it take so long sometimes at the beginning of an experiment for the drift to reach the acceptable value?
A: When the machine is trying to reach this drift value, it is applying a voltage on the indenter head in a closed-loop manner. It continually tries to get the tool to really be still before it starts. However, some things disrupt this process so that it takes very long, including air currents, noise in the room, vibration, and a “cold” machine. Sometimes, it is known to take up to 1 hour for this process to happen. Typically, if you cancel it prematurely, your results won’t come out as good.

Q: I zoom in on my data near the origin and find that there are actually *negative* displacements in my data at the beginning of loading. What’s going on?
A: I’m so happy you asked! The way that the Nano Indenter finds the surface of the material is that it oscillates the indenter tool with a certain frequency and is lowered. When that frequency is disrupted, the machine defines this point as the touching point. Consequently, this can produce an artifact in the curve where you will find one or two data points corresponding to a “negative penetration depth”. This is not real, but an effect of the way the surface is found.

Q: I keep seeing and hearing about this “drift correction” thing. What is that?
A: The drift correction is used to try to get rid of any mechanical, electrical, or thermal effects that are skewing the collected data one way or the other. To attempt to get rid of this, the machine first unloads from the maximum load to the defined “percent to unload”. Then, it waits for 60 seconds at this percent, and measures the displacement of the tool over that time. It uses that number as the drift velocity, and will add this as a function of time to all of your data locations (resulting in a shift). This has been known to cause a problem when there is a sufficiently long holding time (5 minutes or more especially) or “Percent to Unload” is very close to 100%. In the reported data, you will see a large plateau at the maximum load because of this applied shift. This is not real. The best way to correct this for large loading times is to turn the drift correction analysis off. You can do this by selecting the tests in the “results” section, and in the upper right-hand area you will see a value for performing a drift correction. Set this value to “0”. It will previously have probably been a “1”.

Q: Positioning of the stage is WAY off from even the coarse positioning. What should I do?
A: The best thing is to go to Configure/Device… and select first the channel “X Position”, and then “Calibrate”, then “Y Position” and “Calibrate”. Then, it should

be corrected. Under no circumstances should you need to select “Edit”, “New”, “Delete”, or “Reset”. Messing with these options will only do bad things.

Q: When I look at my results, I notice that the indenter did not find the surface depth correctly. How can I make it more reasonable?
A: In the Testworks results, on the load-displacement curve display, zoom in to the location where you believe the actual surface is. Position the crosshairs on the point and hit “s” on the keyboard.

Q: Are there any plans for future upgrades of the Nano Indenter?
A: There sure are. Eventually, we plan to add certain options to the Nano Indenter such as high load capacity (up to 1 kg), lateral force measurement, and continuous stiffness measurement. More details, if you are interested, are at the MTS website. Let us know if you see options that may be of interest to your work.

Theory

How the Machine Works

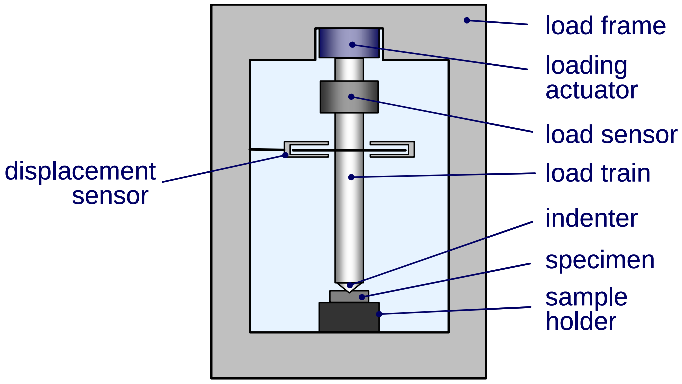
Major components inside of the MTS Nano Indenter XP are shown below. This indenter sits on a mechanical vibration isolation table and its container has sound-dampening materials inside. The load frame is essentially the gantry of the indenter and should be as stiff as possible, so not to contribute significantly to the displacement of the load train. The load frame stiffness can be calibrated by an iterative procedure in which a number of deep indents are made in a standard material (i.e. fused silica).[1] First, it is recognized that the total compliance is:

$$C_t = C_s + C_m\,,$$

where C_s is the indenter/specimen contact compliance and C_m is the machine compliance. Because compliance values are the inverse of stiffness, the relation:

$$C_t = \frac{\sqrt{\pi}}{2\beta E_r \sqrt{A}} + C_m$$

can be used to determine machine stiffness. The intercept of the plot of C_t versus $A^{-1/2}$ will give the machine stiffness directly, provided that E_r and A are well known, which is why a reference material and deep indents are used for this procedure. Scanning electron microscopy (SEM) or atomic force microscopy (AFM) can also be used to experimentally find A by imaging residual impressions in highly plastic materials, such as aluminum. This type of calibration was not performed in this study and the load frame stiffness was taken as 8.1 MN/m, as calibrated by the manufacturer.



The force on the indenter is generated using a coil in a permanent magnet assembly (loading actuator). The generated force is simply the vector product of the current through the coil and the magnetic field strength of the permanent magnet (**F** = **B** x **I**). This type of force application is very simple as well as very linear in its calibration. It allows for very quick closed-loop feedback control over the displacement as it completely separates the force application system and the displacement measuring system. The current through the coil is generated using a 24-bit digital to analog (DA) converter which drives a constant current source. The resolution of the loading system is based on the load calibration (typically 30 mN/V), load voltage range of the DA converter (~4V) and the number of bits of resolution of the DA converter (2^{24}). To sense the load, directly beneath the loading actuator is the loading sensor. The sensor will be either a load cell or a strain gauge. A load cell works by sensing the voltage change across a piezoelectric material (i.e. quartz or barium titanate) when load is applied. A strain gauge directly measures strain, but knowing the stiffness of the gauge this strain can be correlated to a force measurement. For a strain gauge, the change in resistance across a series of resistors in a Wheatstone bridge configuration results when very small forces are applied. Theoretically, this actuator/sensor setup will result in a typical resolution of about 5 nN, but in practice a resolution in the μN range is realistic. For the Nano Indenter XP, the load head can apply forces up to about 733 mN.

The displacement of the indenter is measured using a three-parallel-plate capacitive position sensor. This consists of two outer plates maintained at equal and opposite drive voltages (2V at 12.5 kHz) and a center pick-up plate. The output voltage of the center-pickup plate is uniquely related to the position of that plate in the capacitive gap through the relation:

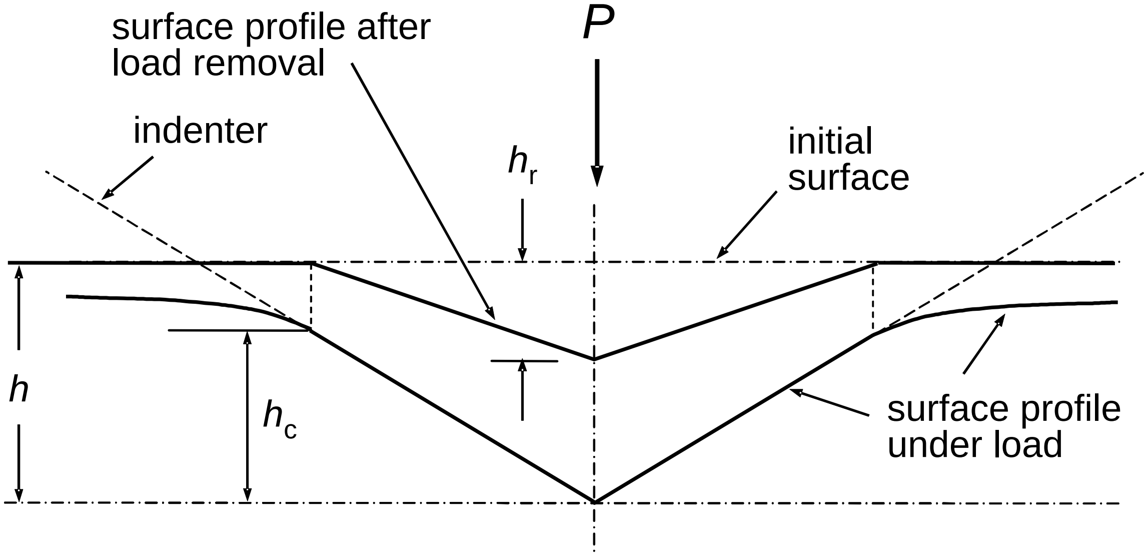
$$V = \frac{d_p Q}{\varepsilon_o A_p},$$

where d_p is the distance between the center plate surface and one of the outer plate surfaces, Q is the charge maintained on each plate, ε_o is the permittivity of free space

(8.854 x 10^{-12} F/m), and A_p is face area of the plates. This plate position is taken as the raw displacement measurement. The pick-up voltage is measured using a 16-bit analog to digital converter (ADC) running at a typical rate of 4 kHz. The displacement system is calibrated using laser-interferometry. For the Nano Indenter XP, it has the ability to operate the head over a range of displacements – 15 mm of allowed travel total. This is achieved by using a high-resolution gain circuit that is applied to the center-plate pick-up voltage. This circuit has gains ranging from 1 (2^0) to 128 (2^7). Such a range allows sufficiently sensitive measurements to be made even at relatively far plate separation distances. The theoretical displacement resolution (~5 pm) can be calculated based on the displacement calibration (~0.2 mm/V), displacement voltage range (10 V), displacement range gain (1-128) and resolution of the displacement ADC (2^{16} bits). However, the noise floor of the machine, around 0.1 nm, limits this resolution and this is the approximate resolution seen in practice.

Oliver and Pharr Method

Currently, the most common method for extracting the hardness and modulus values from an indentation curve is known as the "Oliver and Pharr" method.[1,2] This is the method Testworks software uses to determine hardness and modulus. Their method is based on contact mechanics solutions developed for a solid of revolution indenting a half-space by Sneddon.[3] This method applies to both sharp and spherical indentation. The Oliver and Pharr method begins by assuming a flat, smooth, isotropic and orthogonal contact between the tool and the material. Elastic deflections of the material are taken into account, such as that shown below. This consideration allows for more precise measurement of hardness and modulus properties, as well as more accurate determination of phase transformation pressures when combined with other contact mechanics relations.



From the generated curve, the three most important pieces of information that are used to calculate hardness and modulus are the maximum applied load, P_{max}, the displacement into the surface at maximum load, h_{max}, and the slope at the beginning of the unloading curve which is unloading contact stiffness, *S*. As described by Oliver and Pharr, this unloading curve is fit to the function:

$$P = C(h - h_r)^m ,$$

where *P* is the applied load at a point in the unloading curve, *C, m,* and residual depth h_r are empirically fit constants, and *h* is depth into the surface corresponding to a load *P*. The value of *m* will be between about 1 and 2. This fit equation is then differentiated, and the stiffness is found by evaluating:

$$S = \left(\frac{dP}{dh}\right)_{h=h_{\max}} .$$

While h_r can be found experimentally for complete unloading, for this analysis it remains a fit constant. To find material hardness, the relation:

$$H = \frac{P}{A}$$

is used, where *A* is the area of contact between the indenter and material at load *P*. Different hardness definitions exist,[4] but this is the definition used in depth-sensing indentation. This is similar to the way hardness is found for Brinell, Vickers, and Knoop methods, except that in this case the elastic deflection of the surrounding material is taken into consideration. Hardness is typically evaluated at P_{max}, but this relation can be used to describe contact pressure at any point in the loading curve as well, provided the tip geometry is known.

To calculate modulus, one first finds the reduced modulus derived as:

$$E_r = \frac{S\sqrt{\pi}}{2\beta\sqrt{A}} ,$$

where β is a constant close to 1 that varies depending on indenter geometry.[5] This relation was originally derived from purely elastic theory for unloading between a conical indenter and surface, but also holds true for Berkovich and spherical indenters. The reduced modulus is related to material modulus *E* by the relation:

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^{\,2})}{E_i} ,$$

where ν is Poisson's ratio and the subscript *i* refers to those properties of the indenter. An inherent weakness of this method is that ν of the tested material must be known or closely estimated beforehand.

Tip Calibration

Until now, little attention has been paid to the determination of the contact area between the indenter and sample, *A*. For any indenter shape, *A* is a function of contact depth h_c, where:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S},$$

and ε is a constant (usually 0.75) that depends on the indenter's geometry. To find the relation between *A* and h_c, one should run a series of indentations of various depths into fused silica or other well-characterized material. *A* is found at various values of h_c using the "Basic Tip Cal" method, knowing all other variables in the above Oliver-Pharr equations. The function $A(h_c)$ is found by describing the indenter geometry as:

$$A(h_c) = Bh_c^{\,2} + \sum_{n=0}^{\infty} C_n h_c^{\frac{1}{2^n}},$$

where *B* is a constant which is a strong function of regular indenter geometry for sharp tips, and C_n are constants that describe the imperfections of the tip from ideal geometry. For the area function which describes a spherical tip, *B* will be equal to -π and C_0 equal to $2\pi r$, where *r* is the effective radius of the indenter tip. For the Berkovich indenter, the ideal geometry is with $B = 24.56$ and other coefficients are 0. For the ideal cube corner indenter, $B = 2.5981$. It has been found that Microcal Origin is useful for performing good curve fits for indenter calibrations.

Field and Swain Method

A method popularly used for determination of modulus and hardness by spherical indentation is the "Field and Swain" method.[6] This method is not used in the Testworks software. Although different parameters are considered for determination of modulus by both the Oliver and Pharr and Field and Swain methods, both have been shown to be equivalent contact mechanics analyses.[7] Field and Swain's method is ultimately based on considering the plastic and elastic regimes during a spherical indent, and assumes a flat, smooth, isotropic material and orthogonal tool and material contact. Unlike sharp indentation, the spherical geometry will induce first elastic behavior, and then with load, increasingly plastic behavior. Eventually, fracture will take place around the indent for brittle materials. Equations for purely elastic contact between two spherical bodies (in this case, the substrate has an infinite radius) have been developed first by Hertz[8,9] and further by many others including Johnson.[10]

For a completely elastic response, the reduced modulus of elasticity is found by the expression:

$$E_r = \frac{3P}{4ah_e},$$

where h_e is elastic penetration depth and a is the radius of contact between the tool and material.[10] The relation of E_r to E was given earlier. Since load can be measured directly, one needs to determine only h_e and a. The key assumption made in this method as well as the Oliver and Pharr method is that the initial part of the unloading curve is a completely elastic response (and this has been shown experimentally for some materials). Through the entire loading and unloading, the relation for total penetration depth is:

$$h = h_r + h_e.$$

This is a simple statement that the total depth is comprised of elastically recoverable depth and residual depth. Also, h can be described by the relation:

$$h = h_c + \frac{h_e}{2}.$$

This is based on the assertion that total depth is the contact depth plus elastic surface deflection. It has been shown and observed experimentally that elastic deflection is evenly divided above and below the circle of contact for elastic loading portions.[3] From contact mechanics of Johnson,[10] the load-displacement relation for elastic contact of a sphere on a flat surface is:

$$P = Ch_e^{3/2},$$

where C is a constant dependent on material properties. Therefore, upon unloading, if the residual component h_r of the total depth is subtracted out, then it holds that the change in h with respect to P will be due to only elastic unloading, and can be described by the above equation. Considering two points, (1) at maximum applied load and maximum displacement, and (2) at partial unloading of the material:

$$\left(\frac{P_1}{P_2}\right) = \left(\frac{h_1 - h_r}{h_2 - h_r}\right)^{3/2},$$

since C is constant for the material. Because everything is found experimentally except for h_r, this can be solved for. Now, h_e and h_c can be solved for. From geometry, the radius of the circle of contact can be calculated as:

$$a = \sqrt{2Rh_c - h_c^{\,2}}\,,$$

where R is the indenter radius. Finally, all variables are known to find the modulus of elasticity of the material.

Finding the value of hardness with a spherical indenter is a bit less straightforward than for a Berkovich or other sharp indenter. The determination of hardness is the same, where A is equal to πa^2. However, because a spherical indenter does not induce plasticity on most materials right away, an indentation must be at a depth that is constitutive of full plastic flow, which for metals is when mean pressure of contact divided by the yield stress of the material is equal to about three.[11] This can be determined from finding this quotient at various maximum loads. Otherwise, experimentally one will find that the hardness value measured by indentation will increase with maximum applied load until it remains constant. The load at which this occurs is a strong function of indenter radius.

How to Generate Stress-Strain Curves

For spherical indentation, one may wish to generate an indentation stress-strain curve from the load-displacement data. The steps below are illustrated by Iwashita et al.[12], but in my opinion this is more straightforward. ☺ To do this, first consider the definitions of indentation stress and strain:

$$\sigma = \frac{P}{\pi a^2}, \varepsilon = \frac{a}{R},$$

where R is the effective radius of the indenter, found by calibration of the tip (explained later). All of these variables are known throughout the loading curve, except for a. To find this contact radius, we first attempt to separate the elastic portion of depth of penetration and the plastic part. The elastic depth is computed from the following Hertz equation:

$$h_e = \left(\frac{3P}{4E_r}\right)^{2/3}\left(\frac{1}{R}\right)^{1/3}.$$

Notice that the reduced modulus must either first be assumed or known. When compared to the loading curve in the beginning, this theoretical curve should lie on top of your experimental curve since purely elastic contact usually exists during the onset of loading.

The plastic penetration depth component is then just the difference between the elastic and total penetration depth, or

$$h = h_e + h_p \ .$$

The contact depth needed to find a is dependent on both the plastic and elastic components. From elastic contact theory, contact depth is equal to half the total elastically deformed depth. The entire plastic portion will contribute to contact depth in that amount, so that

$$h_c = \frac{h_e}{2} + h_p \ .$$

Now, a can be solved for from the following based on spherical geometry:

$$a = \sqrt{2Rh_c - h_c^{\ 2}} \ .$$

Your stress-strain curve can now be plotted throughout the duration of the loading and unloading curve. Note that this method assumes a smooth, flat, isotropic material and a perfect spherical geometry. It is recommended to use a “strain-rate” (or loading rate over load is constant) loading method, so that many more data points are collected at the beginning of loading. Because the indentation strain jumps a large amount for small initial displacements, simple “loading rate” methods will show large jumps in the beginning for stress-strain plots. Also, it may be necessary to “shift” your surface find point a few nanometers to insure that your stress-strain curve intersects the origin.

Average Contact Pressure

For spherical indentation, the average contact pressure (ACP) is the same as the indentation stress described above. For sharp indenters, it is a bit different. From contact mechanics, it is known that materials indented with Berkovich and other sharp indenters undergo a purely elastic response during the beginning of the unloading cycle.[10] Using this fact and considering the geometry of the indenter, it can be shown after some mathematical rigor[3,13] that the elastic penetration depth at a point i during unloading is related to applied load by:

$$(h_e)_i = (h_e)_{\max} \sqrt{\frac{P_i}{P_{\max}}} \ .$$

Values for P_i and P_{max} may be taken directly from indentation data points. The elastic deflection at maximum load may be calculated using the equation[2]:

$$(h_e)_{\max} = \varepsilon \frac{P_{\max}}{S} \ .$$

Next, the depth of the material that is in direct contact with the tool at a point i is:

$$(h_c)_i = h_i - (h_e)_i \ ,$$

or the total measured displacement into surface minus the elastic displacement depth. At this depth, the contact area may be determined by using the appropriate calibrated area function. To find the ACP at an unloading point, it is then just:

$$p_i = \frac{P_i}{A_i}.$$

<u>Reference:</u>

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If you feel that additional information should be included in this document, please contact Tom Juliano (tfj22@drexel.edu). If you have any other general questions concerning the Nano Indenter, please first look on the MTS website http://www.mts.com/nano/nano_indenter_xp.htm or refer to the user's manual for the Nano Indenter that is kept near the machine. If you cannot find satisfactory answers, please contact Tom.