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TriboIndenter Manual

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Summary: The following manual describes how to operate the Hysitron Triboindenter in quasistatic, dynamic, or imaging modes and acquire and analyze data. It also presents common pitfalls, troubleshooting techniques, and describes key differences in the testing of hard versus soft materials and uses of different tips.

I. Introduction

The Triboindenter may be used to perform indents in quasistatic or dynamic modes. It also may be used to image a specimen's surface. Indents should be done on relatively smooth surfaces, preferably having a roughness less than the radius of the indenter tip. *(Nature* **411**, 656 (07 June 2001)). Depending on how deep you indent, surface roughness may affect the accuracy of your results. It is preferable to indent at least 10 times deeper than the roughness of your surface (Tsui & Pharr, Journal of Materials Research, 1999). Sample preparation guidelines of the common materials tested in our lab can be found in other protocols. It is also important to make sure the sample doesn't move during testing. The stage has several magnets within it so that gluing or securing your sample onto a metallic base will be sufficient to ensure that the sample doesn't move. The basics procedures for operating the Triboindenter and logging your experiments are described below. In addition, there are guidelines for data acquisition and explanations of common pitfalls and troubleshooting techniques that we have encountered during the years.

<p style="text-align:center;color:red;">Before you start please remember to fill out the log book!</p>

II. Standard Values and Pitfalls

Calibrations
- Do advanced z-axis calibration every time you turn on the software
  - If the air indent calibration does not appear to be normal, try removing the tip and putting it back on again. The tip may have been place incorrectly or something may have gotten stuck in the transducer.
- Do tip-optics calibration every time you change the tip
  - The aluminum will be in focus around 25.8 mm.
- Do tip area calibration every few months
  - Er for quartz = 69.6 GPa
  - Er for polycarbonate = 2.95 GPa
  - Er for PS-6 Sheet = 0.007 GPa (7 MPa)
- Machine Compliance= 4.0
- Always check that the displacement gain (in transducer constants) is 100
- Check that the microscope feedback gain matches in the software (in piezo controls) and on the TriboScope.

Indenting
- Always make sure that you have the correct *Tip Area Function*. From the *Load Function* window, select **Setup >> Tip Area** or click on the **Tip Area** button. Open the correct **.ara file for your tip.
- Set up samples so that the tallest samples are on the right and are the first to be indented.
- If the samples are not totally flat, you can add a sample height variation to prevent the machine from making unexpected contact. From the *Safety Limits* window, select your sample from the pull-down menu. There is a height variation space where you can enter the value.
- Trapezoidal loading functions are usually used for polymers

- If the tip does contact the surface with excessive force during testing, the program will stop automatically. You will need to manually raise the tip using the yellow knob above the transducer and from the *Positioning* window, select **Enable Motors** and then **Setup >> Home >> Z-axis**. To begin again, remove the completed points from the positions group and change the name of the base file (ie from UHMWPE to UHMWPE_b) and save the workspace. It is best to repeat the *advanced z-axis calibration* and the *tip-optics calibration* again. Then, check the methods and begin the method again.
- To add drift correction for stiff materials, go to the *Load Function* window and select **Setup >> Drift Correction**. This will toggle the drift correction on and off.
- Doing a *Quick Approach* will save you some time. Save the workspace, before you do anything else! From the *Video* window, focus on your indent position and click **Quick Approach**. ** WARNING** : This is especially important to do with the current version of the software (7.0) at each position before starting a method. It will greatly decrease the chance of crashing.
- For soft samples, change Z Piezo Analog Feedback Settle Time to 15 s and Z Piezo Digital Feedback Settle Time to 10s by going to **Setup >> Preferences >> Piezo** tab.
- Standard video settings: White = 30, Black = 10.
- Turn off the stage light when the *Video* window is not open.
- Spare light bulbs for the stage light are in a cardboard box under the desk.

III. Turning on the Instruments
- Turn on the *computer* first to prevent a power surge.
- Turn on the *DSP Lock-in amplifier* if you are going to do dynamic testing.
- Turn on the *motor box*.
- Turn on the *TriboScope, TriboIndenter,* and *Universal Motor Drive* (order doesn't matter).
  - The *TriboScope* should flash some numbers on the LCD and settle on –1.
  - The *Display Gain* should be on 10. Switch the *Display Gain* to 1 and check that the LCD reads about –400. (For each tip, this number will be slightly different since this value is a measure of the tip weight.)
  - Chance the *Display Gain* back to 10 and hit *Auto Zero*. Manually zero the value with the *Manual Zero coarse and/or Fine* knobs. You want the Tare Value (number on the LCD) to be +/- 0.05 before starting the software or you will get an error message. This value may drift a little as you continue setting up, so zero the Tare again before you start indenting. This should equilibrate in about 1 hour.
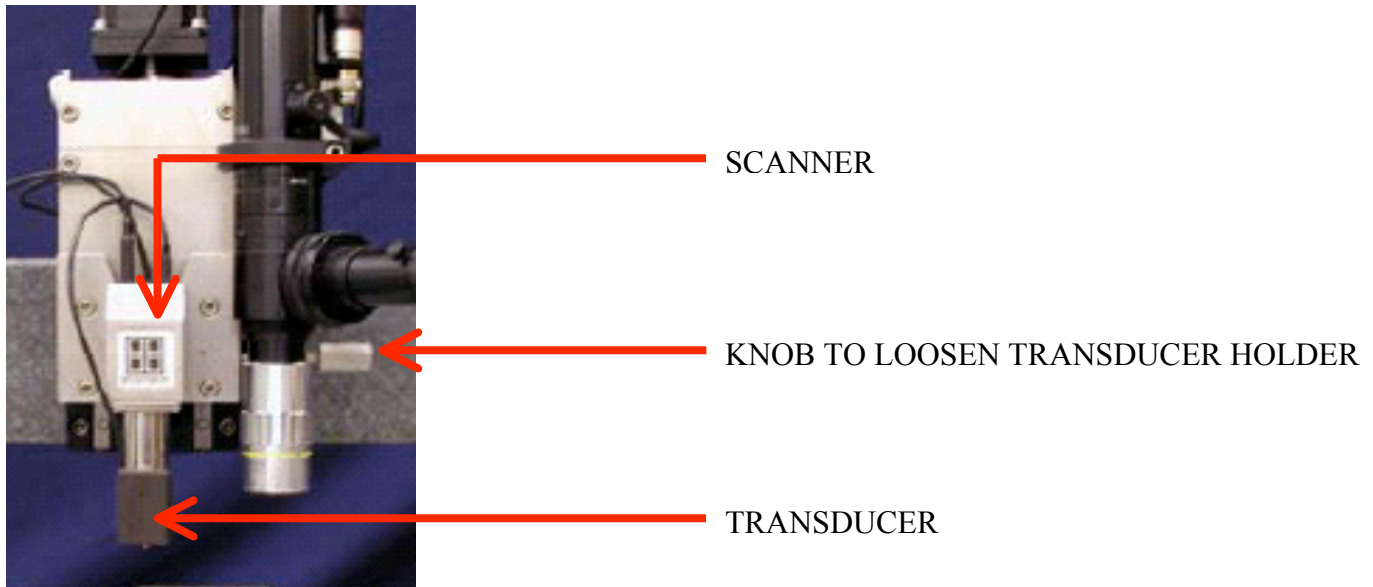
IV. Starting the Software
- Start the software using the shortcut *TriboScan* on the desktop. To avoid software crashes, do not have any other programs running while you run *TriboScan*.
- The first pop-up window asks if you are doing *Analysis Only*. Hit **Cancel** if you are planning to indent, or just wait for the program to load up by itself
- After the axes have been homed, another popup window will ask you to rezero the Tare value. Press **OK** AFTER you have rezeroed the Tare! Values between -.02 and +.02 are considered zero.

- In the *Positioning* window go to **File >> Open Workspace** and choose the most recent test done on the same material. Then save the workspace by selecting **File >> Save Workspace As**. We normally use your initials and the date.

V. Changing the Tip

- With the computer on and *TriboScan* software closed, turn off the piezo and transducer controllers. Carefully remove the tranducer by grasping the scanner (silver part). *Make sure the tip is high enough from any thing else so that you will not hit anything as you remove it.*
- *DO NOT touch the transducers directly!!!!!!!!!! Gently hold scanner.*

SCANNER

KNOB TO LOOSEN TRANSDUCER HOLDER

TRANSDUCER

- Open the *Triboindenter* door and unplug the left cable (from your perspective) from on top of the transducer holder. Then unplug the cable on the top left of the horizontal beam. This plug also connects to the top back of the transducer holder.
- Loosen the transducer holder by unscrewing the horizontal silver knob on the right side of the transducer.
- Once you have done all of this, the transducer holder should be free to move. Slide it up vertically.
- Unscrew the tip using the tip tool and place the tip into the plastic hollow tube; only the tip end will fit. To stabilize you may hold on to scanner, but do not touch the transducer. You can touch the non-tip end with your fingers to remove the new tip from its tube.
- Now screw in the tip you desire. **Stop tightening the tip tool at the first sign of resistance.**
- Slide the transducer holder back into its place. Screw the horizontal knob on the right and plug in both cables that you had originally removed.
- Turn both the piezo and transducer controllers back on.
- **Rezero the tare value.**
- Unpause the TriboScan software.
- Check to see that correct Tip Area Function is selected. In the *Positioning* window, go to **Setup >> Tip Area Function. Select the correct function for your tip.**

VI. Cleaning the Tip

Materials: cue tips, 70% ethanol

- *You should clean the tip every 2 weeks with hard materials.*
- *Clean the tip after every use for tissue or sticky polymer (e.g. PDMS) samples*
- Dip a cotton swab into 70% Ethanol.
- Then lightly brush the tip in strokes that only go from the top to the bottom of the tip.
- ***Do not try to wet much above the tip because you don't want alcohol to seep into the transducer!!***

VII. Advanced Z-axis Calibration

- **Change the microscope feedback gain to 100** on the *TriboScope* panel and in the software. From the *Positioning* window select **Setup >> Piezo Controls** and change the value for **Front-panel Microscope Feedback Gain** to 100. Minimize this window as a reminder to change the value back.
- From the *Positioning* window, go to **Setup >> Indent Open Loop Load Function**
- Set up the Load Function Editor to perform a 600uN 2-segment indent in air, for a total of 20 seconds. For soft materials, this load should be increased so that the air indent goes approximately 5um, but 600uN should be sufficient. Or open the existing load function AIR INDENT.ldf.
- From the *Open Loop Load Function* window, select **Setup >> Advanced z-axis Calibration**.
- Click "Calibrate Transducer". The instructions at this point from the pop-up windows are fairly self-explanatory.
- The calibration window will pop up. Check that the following values are correct.

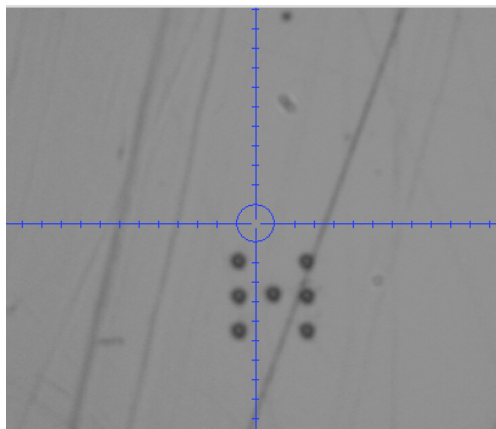| Parameter	Correct Value
Machine Compliance	4.0
Displacement Gain	100
Bias Offset	0.2
Displacement & Microscope Feedback Gain auto detect	Unchecked

- Click **Done** and follow the instructions.
- A graph will appear with a fitted function of the Electrostatic Force Constant as a function of displacement. Verify that the fit is reasonable, and close this graph. (This last step is important – the values will not update in the software until you close the graph and the data analysis window.
- **Return the microscope feedback gain to 1000** on the *TriboScope* and in the software by selecting **Setup >> Piezo Controls** in the Positioning Window or opening minimized window. Change the value for **Front-panel Microscope Feedback Gain** to 1000.
- If you forget to do this before the indent, you can correct your data by opening the *Indent Data Analysis* window and selecting **File >> Update Multiple Files**.
- *Make sure to write the ESF and ZVG in the log book.*

VIII. Tip-Optics Calibration

Aluminum (For Berkovich or 20um Conical Tips)

Materials: aluminum sample used for calibrating which is glued on a metal plate

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- From the *Positioning* window, open the *Video* window by clicking on the Video button.
- Using the arrows in the *Positioning* window, move the optics to the alumninum sample and find a "clean" space to indent.
- You will probably need to disable the **X-Y Safety Height**. From the *Positioning* window or the *TriboScan Toolbar*, select **Safety Limits**. From the *Safety Limits* window, select the **X-Y Safety Disable** button.
- Change Zoom to 5X. Change Video Settings, White =30 and Black =20, so that indents will be easier to see.
- Click on the **Create Boundary** button on the video window to create a sample boundary.
- From the *Positioning* window, select **Setup >> Indent Open Loop Load Function.**
- Open a load function appropriate for your tip (examples: berk_cal.ldf, 20um.ldf)
- Zero the Tare value.
- From the *Positioning* window, select **Setup >> Calibrate Optics >> Stage Offset >> New "H" pattern**
- The software will prompt you to adjust the tip height to about 1 mm from the sample's surface, using the green arrows. Click on **OK** after you have done this. The machine will then perform 7 indents. (You can turn off the stage light at this point.)
- After the H-pattern is completed, the software will prompt you to move the reticule to the center of the H. Then select **OK**.

- To verify your calibration and to prevent anyone else from mistaking this H-pattern for their own, move the reticule to be between the two legs of the H, below the center indent. From the *Positioning* window, select **Setup >> Single Indent Open Loop**.
- Zero the Tare value and hit **OK**.
- If the calibration is complete, the optic should return and focus on that spot once the indent is completed.

IX.   Tip Area Calibration

Quartz (For Berkovich Tips)

- First perform the other two calibrations (See Sections VI and VII)
- Set up a sample boundary on the quartz sample (See Section IX)
- Set up a new group of positions within the boundary (See Section X)
- Set up a new pattern (See Section XI)
- Create a new method to do a large number of indents over a large range of forces. For example, you could do 72 indents from 100 to 10,000 µN.
- Run the method. Usually overnight. It is often a good idea to do at quick approach at each position before running the method to lower the likelihood of crashing. Definitely do this if you will run the method unattended.
- After all indents are complete, perform a multiple curve analysis. In the *Analysis* window, select **Analysis >> Multiple Curve Analysis**. Select all files created from your method. Save the results as a text file.
- In the *Analysis* window, select **Analysis >> Calculate Area Function**. Select the text file you just created. The correct reduced modulus for quartz is 69.6 GPa.(This should be the default in the software.) Enter this value and press OK.
- Enter 24.5 into the C0 dialog box. Make sure that the Vary C0 check box is NOT checked. Press Execute Area Fit.
- Save the area function (Click on the Disk icon) with the name of the tip and date.

Polycarbonate (For Conical Tips)

- First perform the other two calibrations (See Sections VI and VII)
- Set up a sample boundary on the quartz sample (See Section IX)
- Set up a new group of positions within the boundary (See Section X)
- Set up a new pattern (See Section XI)
- Create a new method to do a large number of indents over a large range of forces. For example, you could do 72 indents from 100 to 10,000 µN. If doing displacement control indents, keep displacements lower than 800 nm for most tips.
- Run the method. Usually overnight. It is often a good idea to do at quick approach at each position before running the method to lower the likelihood of crashing. Definitely do this if you will run the method unattended.
- After all indents are complete, perform a multiple curve analysis. In the *Analysis* window, select **Analysis >> Multiple Curve Analysis**. Select all files created from your method. Save the results as a text file.
- In the *Analysis* window, select **Analysis >> Calculate Area Function**. Select the text file you just created. The correct reduced modulus for polycarbonate is 2.95 GPa. Enter this value and press OK.
- Enter 55 into the C0 dialog box. Make sure that the Vary C0 check box IS checked. Press Execute Area Fit.
- Save the area function (Click on the Disk icon) with the name of the tip and date.

X. Defining the Sample Boundary

- Set up the samples so the tallest samples are placed to the right of the shorter samples on the platform. This prevents the tip from bumping into the specimen surfaces as the safety limits for each sample are defined.
- Turn on the stage light.
- From the *Positioning* window or the *TriboScan Toolbar*, select **Safety Limits**. The image of the stage should show a green circle for the tip, a pink circle for the optics, and black dots for the locations of the magnets.
- From the *Safety Limits* window, click on **Edit Samples >> New Sample**. (The annoying thing here is that it automatically numbers your samples and you have to reselect them and click **Rename** in order to give them your own names.) Select and delete any old samples using **Remove**. *Always leave the stage as a sample.* After you have defined all your sample names, select **Close**.
- From the *Safety Limits* window, select the sample for which you want to define a border, using the pull-down menu. Make sure that the *Video* window is also visible.
- If you have not done so already, click on the **X-Y Safety Disabled** button, which will turn red. This will allow you to move horizontally below the Z Fly Height.
- To define the boundary of your sample, move the optic so that it is above your sample, using the +/- X and Y (blue and red) arrows on the *Positioning* or the *Safety Limits* window. Then, use the +/- Z arrows (green) to focus on the sample. Move the optic so that the reticule is on the sample boundary. In the *Safety Limits* window, select **Add**. Select as many points as you wish around the boundary. If you want to remove a point, click on the point with the left mouse button and select **Remove**.
- To manually define the boundary, double left click with the mouse in the *Safety Limits* window on the space where you want to add a point.
- To move the optics to a point, single right click with the mouse in the *Safety Limits* window.
- Go to the next sample by using the pull-down menu and define its boundary. Once all the samples' boundaries are defined, *save the workspace*!!! This will also determine the Z Fly Height, which is automatically adjusted according to the height of the tallest sample. To save time, it's best to define the boundaries from shortest to tallest.

XI. Individual Indents

- All samples require a Quick Approach.
- Using the Video window, optically focus on position where you would like to indent.
- Performing a Quick Approach prior to an indent will lower the chance of crashing and decrease the time during approach.
- Select **Setup >> Open Loop Load Function** from the *Positioning Window*.
- Move the reticule over the space you want to indent and select **Setup >> Single Indent Open Loop** from the *Positioning Window*.
- Single Indents can also be done for Load Control and Displacement Control indents.

XII. Setting up Position Groups

- Remember: You do not want to indent too close to the edge of the sample a) to avoid edge effects and b) your sample may not be sufficiently supported by its substrate at the

edges. Rule of thumb – Don't indent deeper than 0.1 of the substrate thickness or within 2 x tip radius of curvature of the edge.
- From the *Positioning* window, select **File >> Positions**. In the *Positions* window, select **Edit Position Groups**. In the *Edit Position Groups* window, remove any position groups that you don't want by highlighting the group and selecting **Remove.** *Do not remove the Calibration Indent position!* Add as many new groups as you would like, naming them after your specimens.
- In the *Positions* window, use the pull-down menu to select the correct position group for the specimen which is being viewed in the *Video* window. It is helpful to have the Video, Safety Limits and Positions Window all visible so you can see where a position is and whether it is within the boundary as you add positions to the group. Use the **Add** buttons (**above** or **below** changes the order in which the indents are performed) to add positions to your position group. Do the same for each position group.

XIII. Setting up Patterns
- Select **File >> Patterns** from the *Positioning* window.
- Click on the **Grid** or **Circle** button in the *Patterns* window depending on which type of pattern you would like to create.
- To make a **Grid**:
    - You can change the various values to make the grid for your test.
    - Choose an appropriate separation distance – at *least* 2 x tip radius of curvature!
    - Choose how many indents across and high the grid should be.
    - Click on the **Create** button and save the grid under either your test name (ie uhmwpe_1) or grid type (3_by_2) so that you can find it later.
    - Notes on grids: The indents are always done from lower left to upper right. You can set the origin to change the position of the grid with respect to the origin. You can remove selected points from the grid pattern in the *Patterns* window. You can also duplicate or rename the patterns with **Edit Patterns** in the *Patterns* window.
- To make a **Circle**:
    - You can change the various values to make the circle for your test.
    - Choose an appropriate radius – at *least* 2 x tip radius of curvature!
    - Choose how many points will be used to form the circle.
    - Click on the **Create** button and save the circle.
    - Notes on circles: You can set the origin to change the position of the circle with respect to the origin. You can also change the angle of the first position.

XIV. Setting up the Methods
- From the *Positioning* window or the *TriboScan Toolbar*, select **Methods**.
- In the *Method Editor* window, select **New Method** (if you are only doing one method) or **Edit Methods** if you are planning to do several. In the *Edit Methods* window, add new methods using **Add**. Give each method a name, usually relating to the name of the specimen. This will become your base file name.
- From the *Methods* window,
    - *Method Name*: Choose your method from the pull-down menu.
    - *Method Type*: Open Loop Indent, Displacement Control, or Force Control

- o *Base File*: Double-check, but should be your method name, automatically.
- o *Drive and Directory*: Click on **Browse** and select the folder in which you want to save the data. We usually save in the data folder (NOT the workspace folder) under your name and today's date. Once you've selected the folder, click **Done**.'
- o *Pattern*: Select your pattern from the pull-down menu. Option for adjusting?
- o *Positions*: Select your position group from the pull-down menu.
- o *Load Function*:
  - ▪ *OPEN LOOP:* Select your load function from the pull-down menu, or browse in the *LoadFunctions* folder to find the one you want. There are 4 options for combination rates of changing loading rate and peak force. We usually try to keep the loading rate constant for testing polymers. There are 3 parameters (of which you can specify only 2 – the 3rd will change automatically to fit those 2) relating to initial load, final load, and load increment.
  - ▪ *LOAD CONTROL & DISPLACEMENT CONTROL: See Section XV Dynamic Testing*
- ▪ Repeat the previous steps for each method and if you want to link them together, make sure that there are no loops – this can cause the software to crash!
- ▪ *Save the workspace!!!*
- ▪ Go to the first method (if you have linked several together) or each method individually (if you have not) and select **Check Method**. Make sure that the number of indents is what you expect. You can save this as a .txt file if you want to have a record of the method.
- ▪ If you want to duplicate a method (using **Edit Methods >> Duplicate**) make sure that you change the base file name of the new method.
- ▪ When ready, from the *Methods* window, click on the **Start Method** button.

XV. Force / Displacement Control

It is best to refer to Hysitron's Feedback Control Manual that can be found in our log book. Be aware you will need much patience to use these features because you'll need to tweak the load or displacement function a lot before getting them to behave properly.

- ▪ Once you *FINALLY* get the load or displacement function to behave properly, save the function. In both the *Load* and *Displacement Function* windows *Panel>>Save Load Function* and type what you would like to call the function.

XVI. Imaging

The Z-axis measurement needs to be calibrated. Until this occurs do not use imaging for absolute dimensional measurement. Also referred to as AFM mode, this technique is described in Chapter 6 of the Hysitron Manual. Briefly you can get a very high resolution image by moving a sharp probe tip (e.g. Berkovich) in a raster scan pattern over the sample. The image files can then be viewed in TriboView.

XVII. Lateral Force

Samples may be scratched to determine a coefficient of friction. The process for calibration and scratching is described in Chapter 7 of the Hysitron Manual. It is very similar to the steps used for indentation.

XVIII. Data Analysis
- From the *Positioning* window, select **Setup >> Open Loop Indent Data Analysis** (or from the *TriboScan Toolbar*, select **Open Loop Indent Data Analysis**).
- Open and analyze one plot (**File >> Open**) – make sure that you have the correct segment highlighted for analysis. Use **Execute Fit** for the analysis.
- To analyze all your curves together, from the *Indent Analysis* window, select **Analysis >> Multiple Curve Analysis**. In the subsequent window, highlight all the desired files, hit the **Add** button, and then hit the **OK** button. Choose an appropriate filename for your data summary file.
- To export multiple text files of the data, from the *Indent Analysis* window, select **File >> Export Multiple Text Files**. Highlight all desired files, select the **Add** button, and then hit the **OK** button.
- It can also be useful to plot multiple files. From the *Indent Analysis* window, select **Analysis >> Plot Multiple Files**.
- At this point, use zip disk or e-mail to transfer the data off of the computer. If your e-mail doesn't have much room you can always temporarily use the nanoindenter@yahoo.com account.

XIX. Shutting Down
- First turn off the software
- Shut down the rest of the instruments in any order
- Make sure to turn off the stage and optics light!
- Shut down the computer
- *Do not shut down the computer before the Triboindenter system as that may damage the instrument!!!!*

XX. Nanoindenter Calendar
- Go to calendar.yahoo.com
  - Sign in as
    - Username: nanoindenter
    - Password: hysitron
- Mail Tab
  - The latest software and manuals are stored here
- Addresses Tab
  - All users of the nanoindenter are listed with contact info. Contact other users to move appointments.
- Calendar Tab
  - Sign up for appointments here
  - In general, all users should sign up for All Day appointments
  - An appointment lasts from 10 AM the day you sign up until 10 AM the next day to allow for overnight testing