Texture Analyzer Manual

For Training and Further Questions Contact

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1 Introduction

The texture analyzer (TA) is a benchtop instrument that combines a linear actuator with a load cell. This setup enables the user to measure/control force and displacement with respect to time as an experiment proceeds. The model that we purchased is the TA.XT Plus Connect. It has a load cell with a maximum force of 50 N and an approximate resolution of ± 1 mN. The actuator can translate a maximum distance of 37 cm and access speeds over a range of 0.01-40 mm/s with a distance resolution of 1 μm. Displacement is quantified via a stepper motor in conjunction with a known gear ratio and is not monitored by an encoder. Threading on the holes in the base and on the load cell are M6 and not $1/4''$-20.

The TA system has several strengths which are listed below.

1. Specialty fixtures that enable a broad library of tests that can be run.
2. Easy to incorporate custom fixtures (no pins like istron fittings or a single source for fixtures like a DMA or rheometer).
3. Straightforward software control of the instrument that exports the raw data gathered during the test.
4. Can be reprogrammed to create custom experimental sequences.
5. Is light enough to be transported to a fume hood when experiments involve solvents with inhalation risks.

The purpose of this manual is to introduce users to the basics of operating the TA as well as some of the theory and analysis needed to process data.

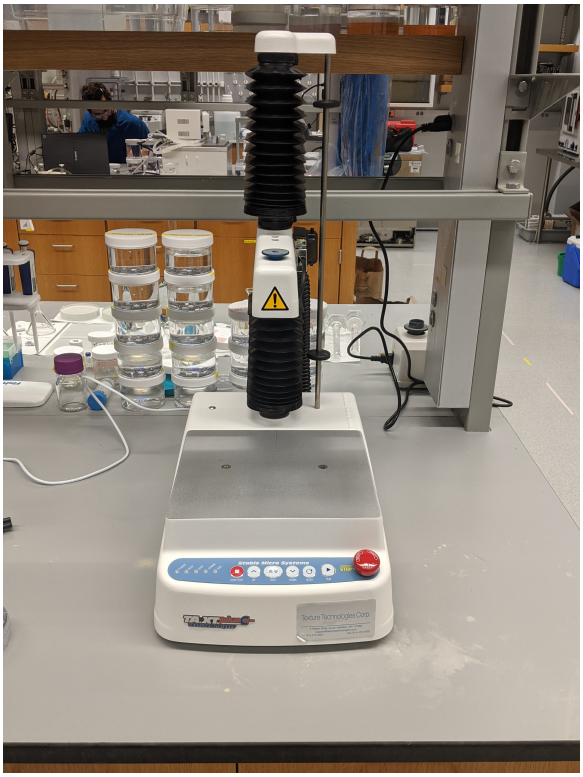
2 Operating the TA

In general there are ten steps for running the TA. These steps are covered in this section.

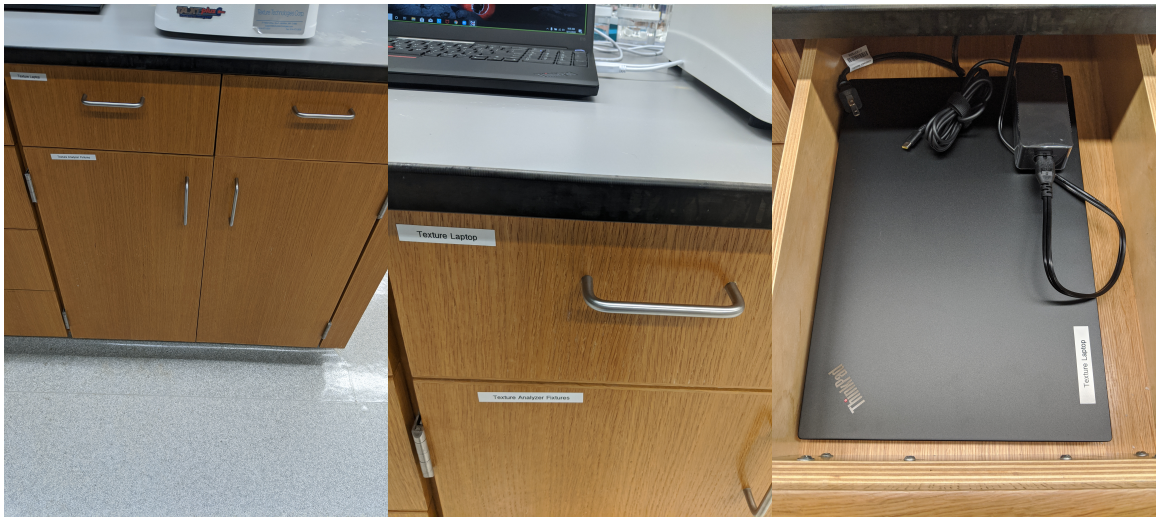
1. Turn the instrument on and start the software
2. Select and attach fixtures
3. Calibrate the instrument
4. Load the sample
5. Tell the TA what to do in the software
6. Tell the TA where to save the data
7. Run the test
8. Export the data
9. Unload the sample
10. Clean the TA and remove the fixtures as you put them on

2.1 Turn the Instrument on and Start the Software

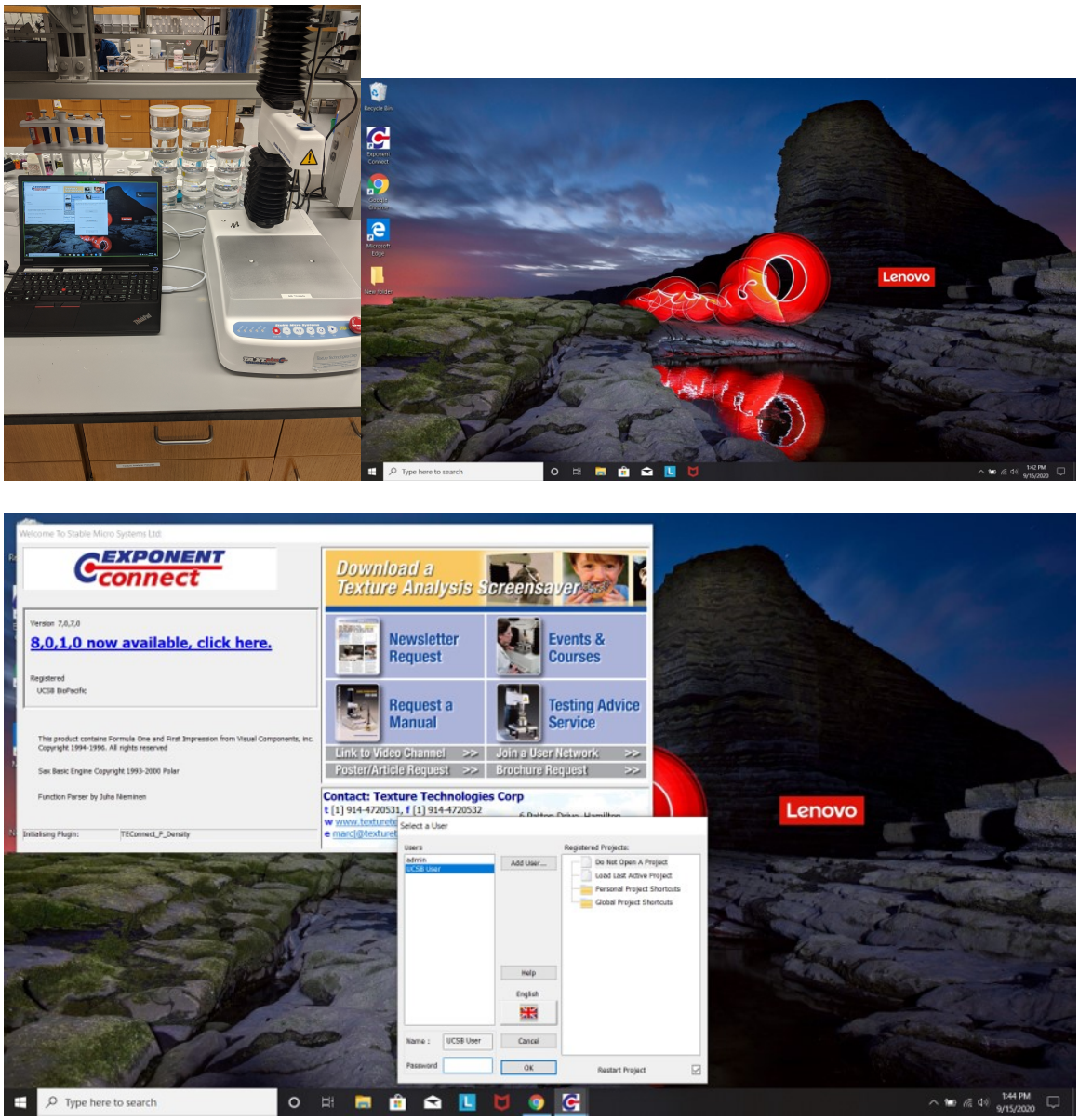
Each user will clean off the instrument and put away any fixtures and laptop after each run. When approaching the TA, it will look as such.



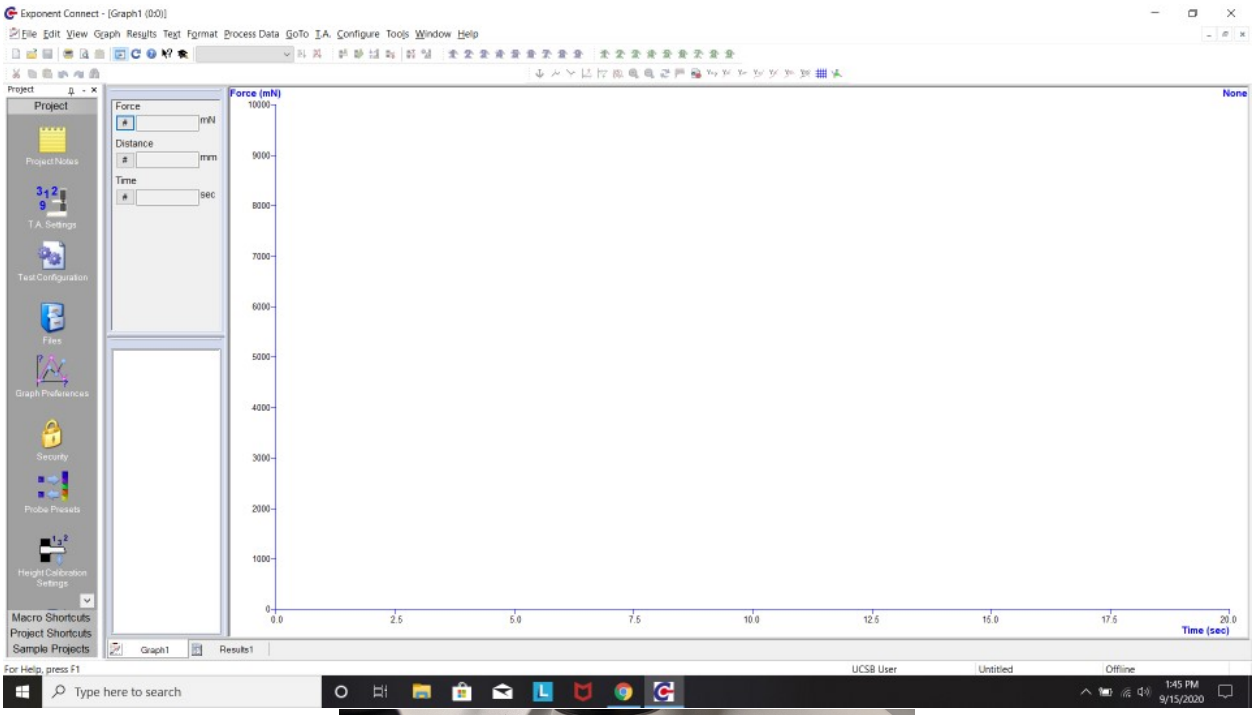
The laptop designated for the TA will be shutdown and in the labeled drawer below the instrument.

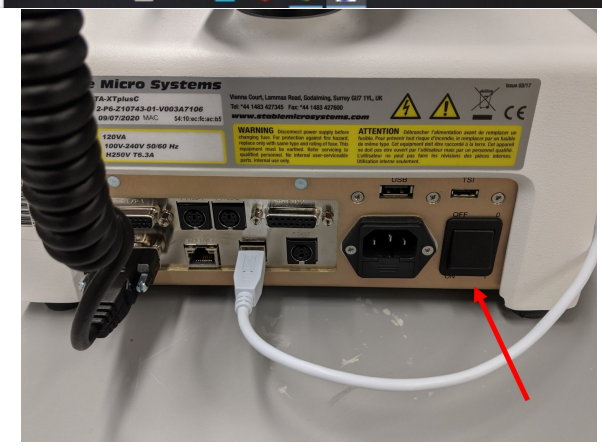


Remove the laptop from the drawer, plug it in, and turn it on. The password for the computer is texture. The software that controls the instrument is called Exponent Connect.

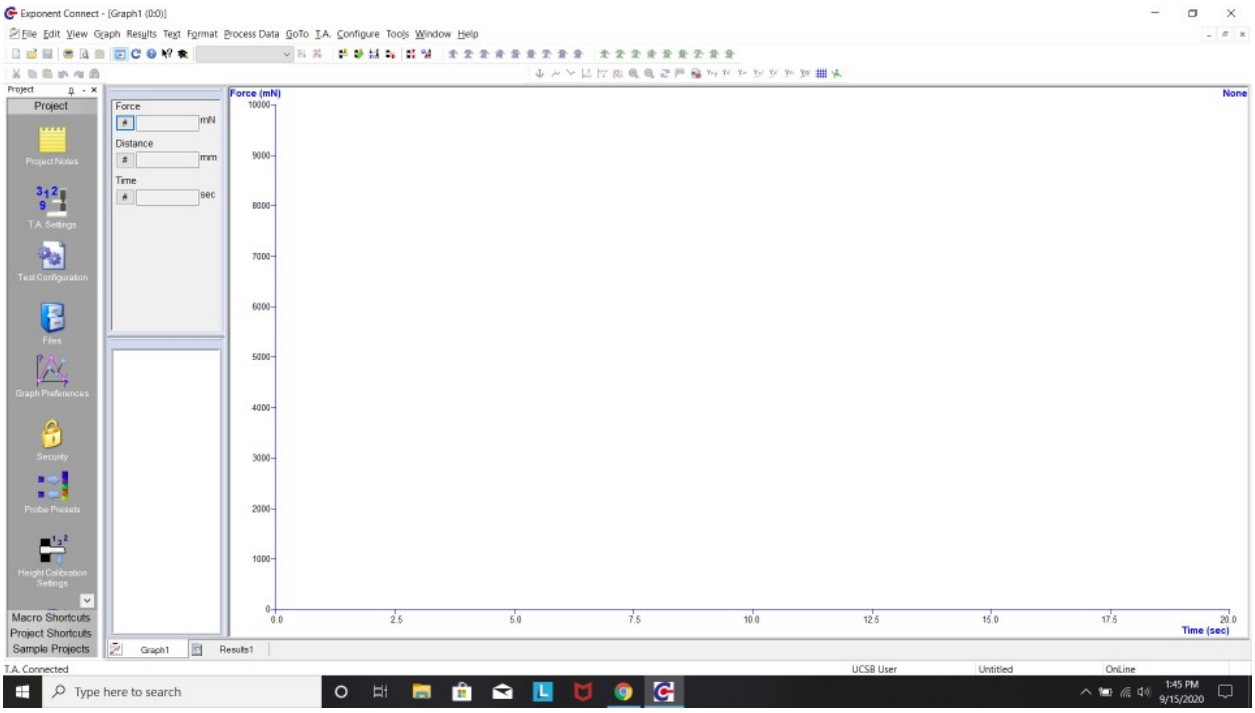


The password for account user UCSB user is texture. This will let you access the software. You will also need to turn the instrument on using the button on the back of the instrument.



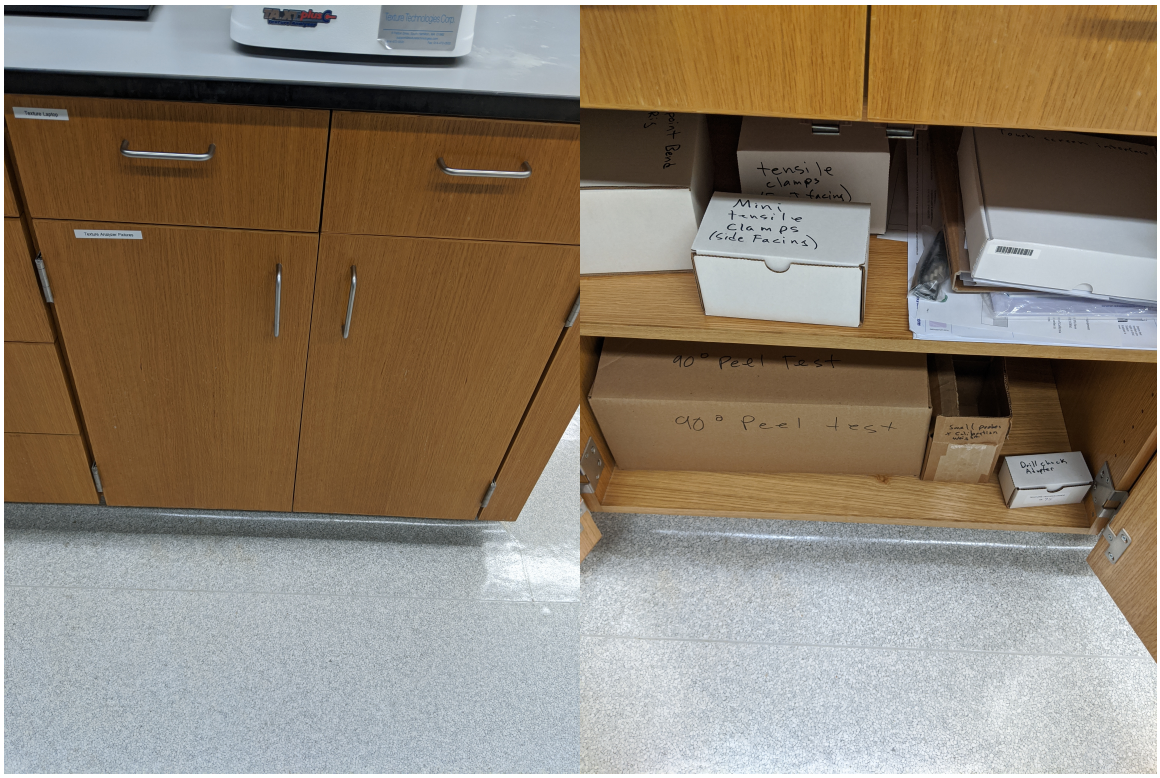


Once the instrument is turned on and connected to the laptop via usb, many instrument control buttons will become available.



2.2 Select and Attach Fixtures

Fixtures for the TA are stored in the cabinet below the instrument. A summary of each figure is contained in the appendix. Select and remove the one that is appropriate for your test.



As an example, this manual will use the 2 mm diameter flat cylinder probe for indentation tests.

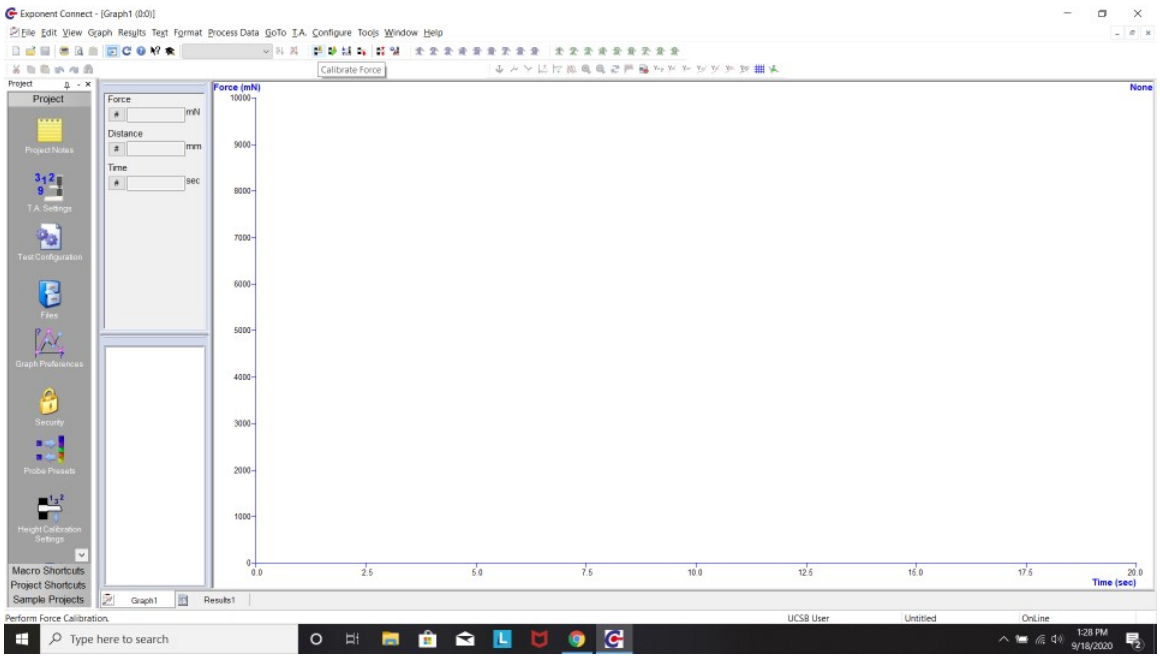
The bottom and top fixtures on the TA screw onto the instrument. The threads on the TA are M6. The indentation fixture screws directly into the hole on the load cell. There is no bottom fixture for this setup so nothing needs to be screwed into the base of the instrument.

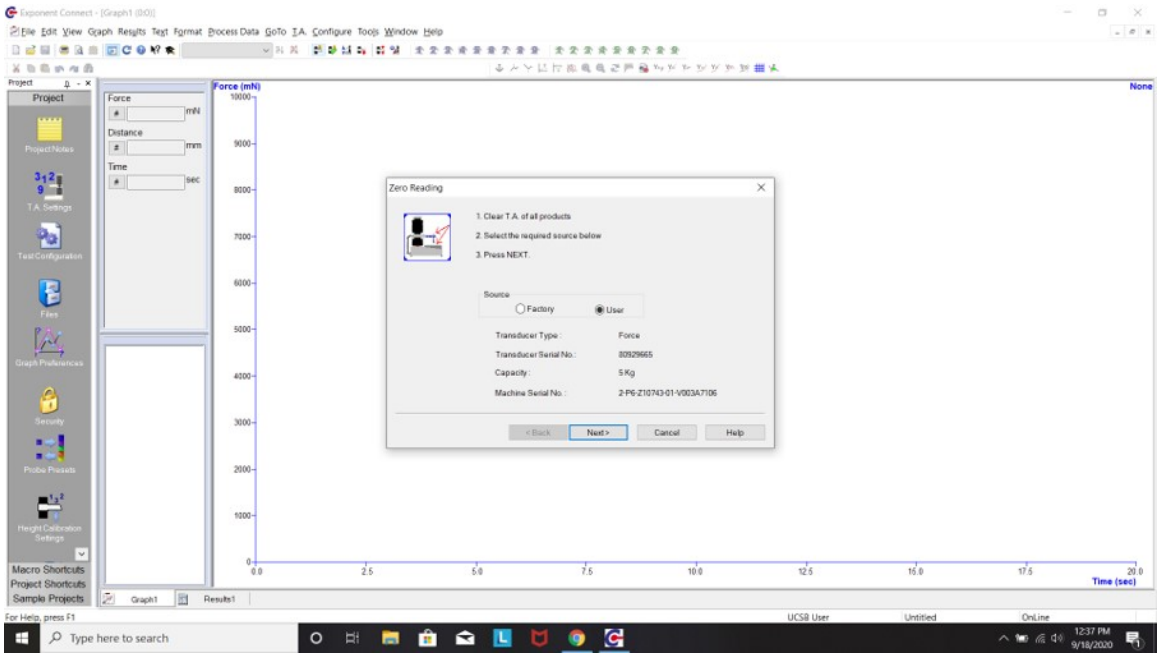
2.3 Calibrate the Instrument

There are three calibrations that need to be performed on the instrument.

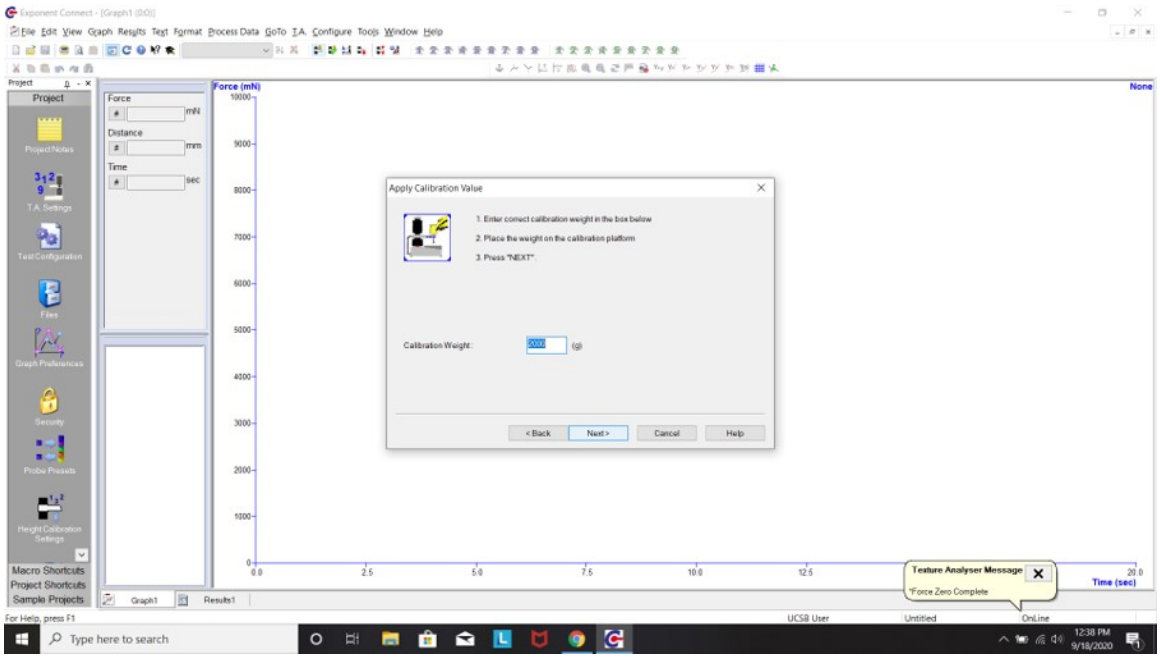
2.3.1 Force Calibration

The first calibration is the force calibration on the load cell. You can have fixtures attached during this calibration, but you must make sure that they are suspended in air and not supported from below.

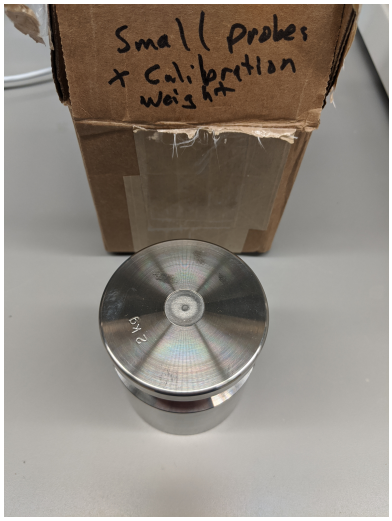


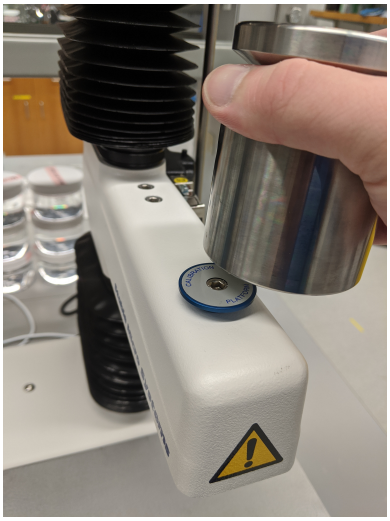


You are the user so you want to select the user option and hit next. The instrument will then zero the force so do not touch the instrument until the next screen shows up.

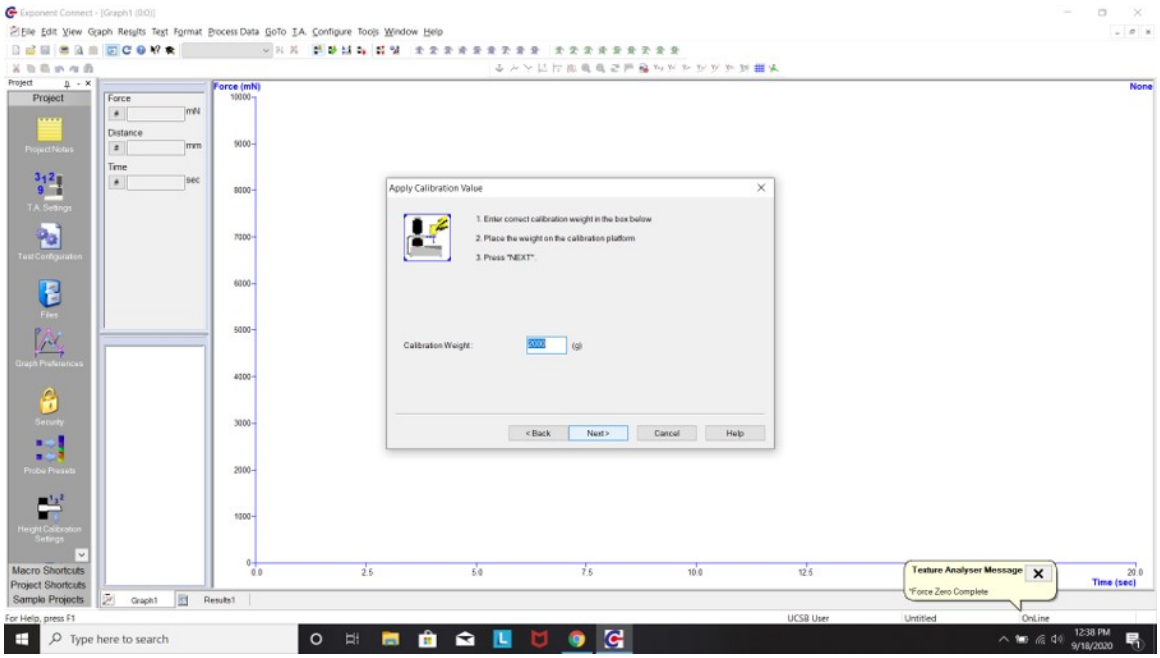


Now you will need to add the calibration weight onto the platform at the top of the load cell.

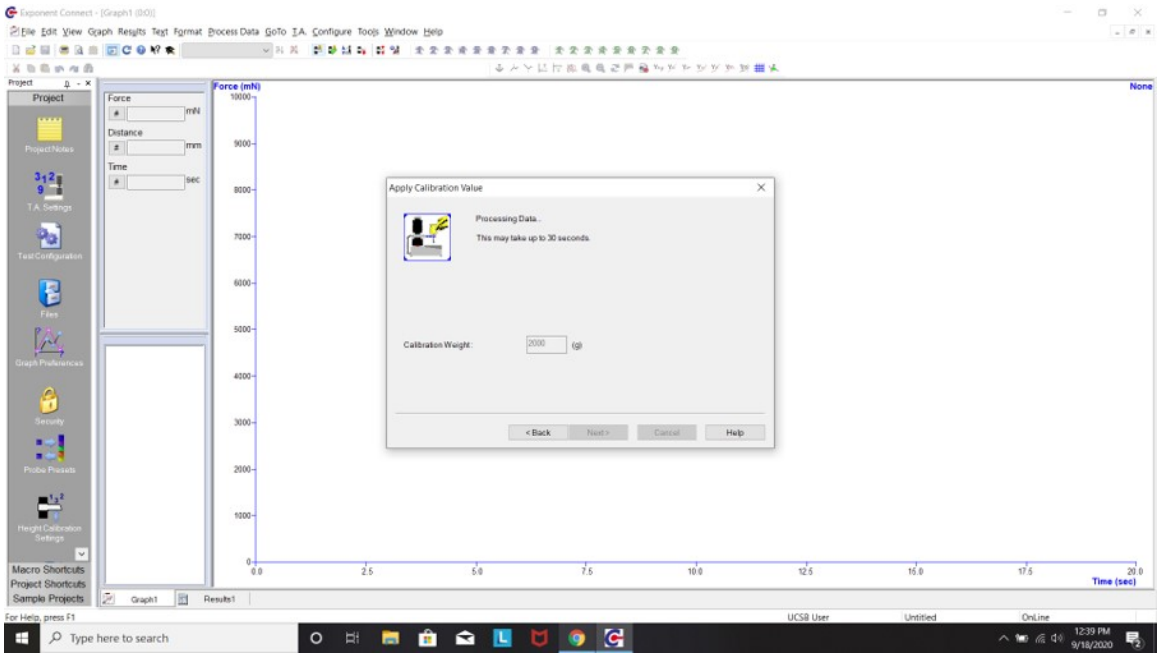


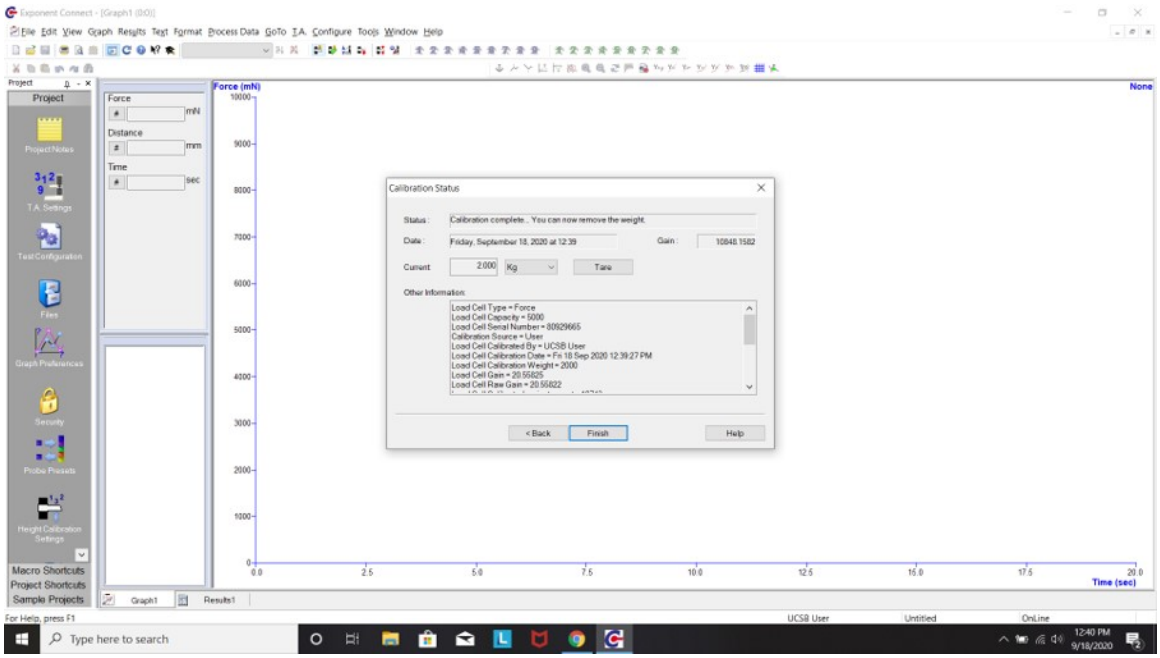


Once this is done hit next in the software.



The software will take about 30 seconds to calibrate the force.



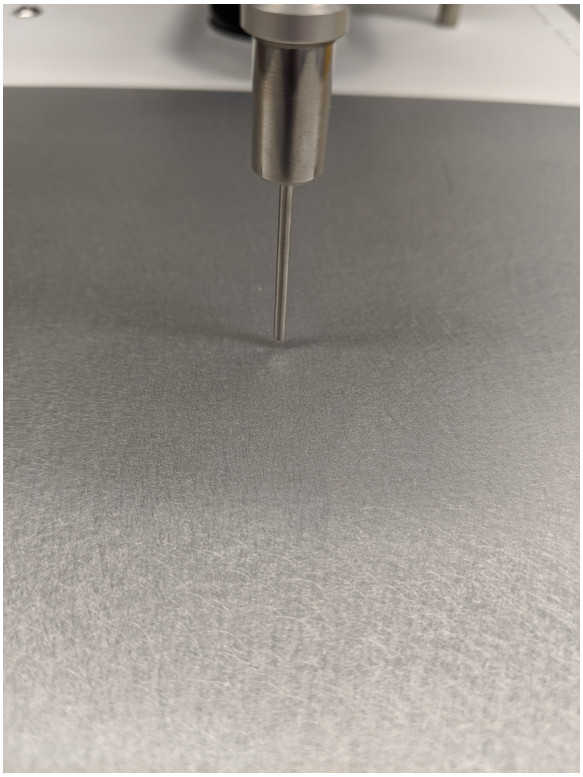


This screen indicates that the force calibration is complete and you can hit finish. Don't forget to remove the calibration weight and put it away.

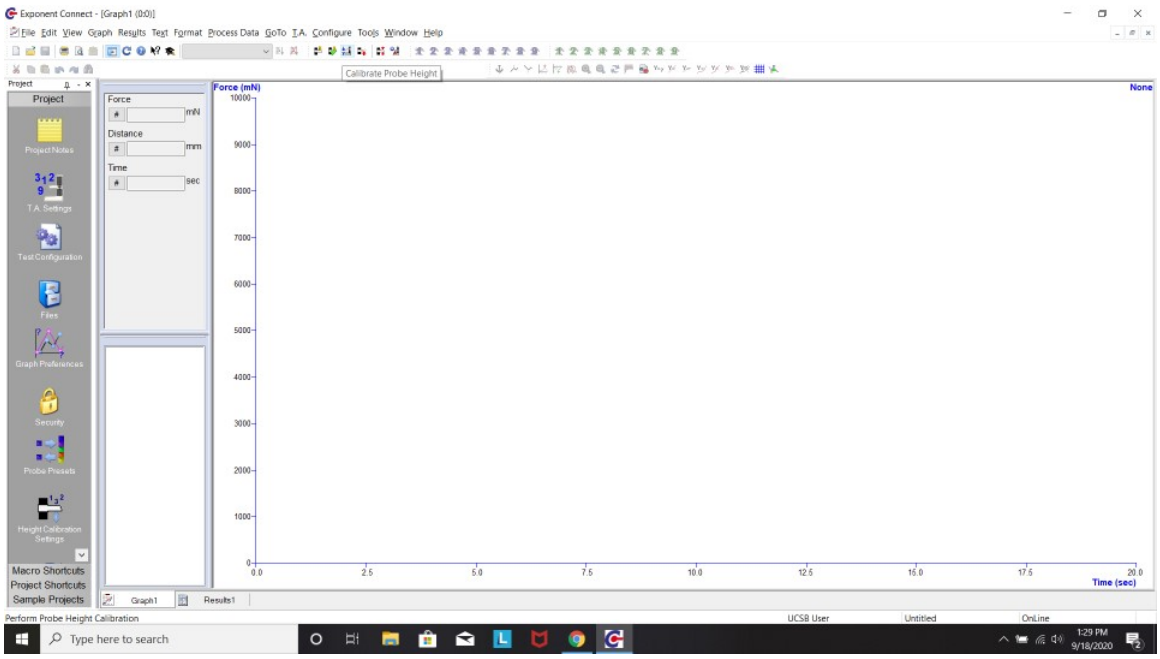
2.3.2 Calibrate Probe Height

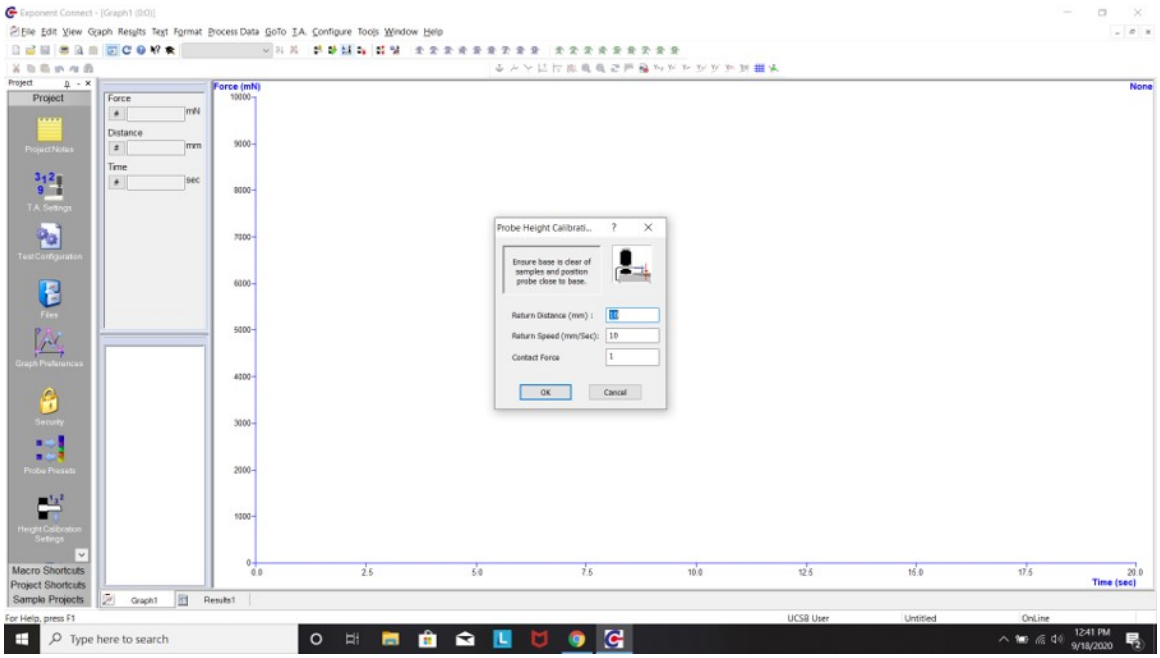
The second calibration is the probe height calibration. **It is important to do this calibration before using the move probe option in the software to make sure that you do not smash the fixtures into one another. This can damage the load cell and is the main way that you could break the instrument.** Before running this calibration, you will need to use the buttons on the front of the instrument to bring the fixtures close (within a centimeter or so).

Note the up and down buttons on the instrument. If you press either of you will get a slow actuation. When you press them in conjunction with the fast button you will get fast translation. Make sure to use the fast translation far away from where the fixtures would contact to get a feel for the speed before making the fixtures/probe approach. The other two important buttons are the red stop test button and the emergency stop button. The red stop test button will stop a test that is currently running and save the data gathered up to that point. The emergency stop button will cut the power to the instrument and any data gathered will be lost. The reset button is used when you run an experimental sequence that requires user input to determine when a certain stage is finished. You will not need to use the run button.

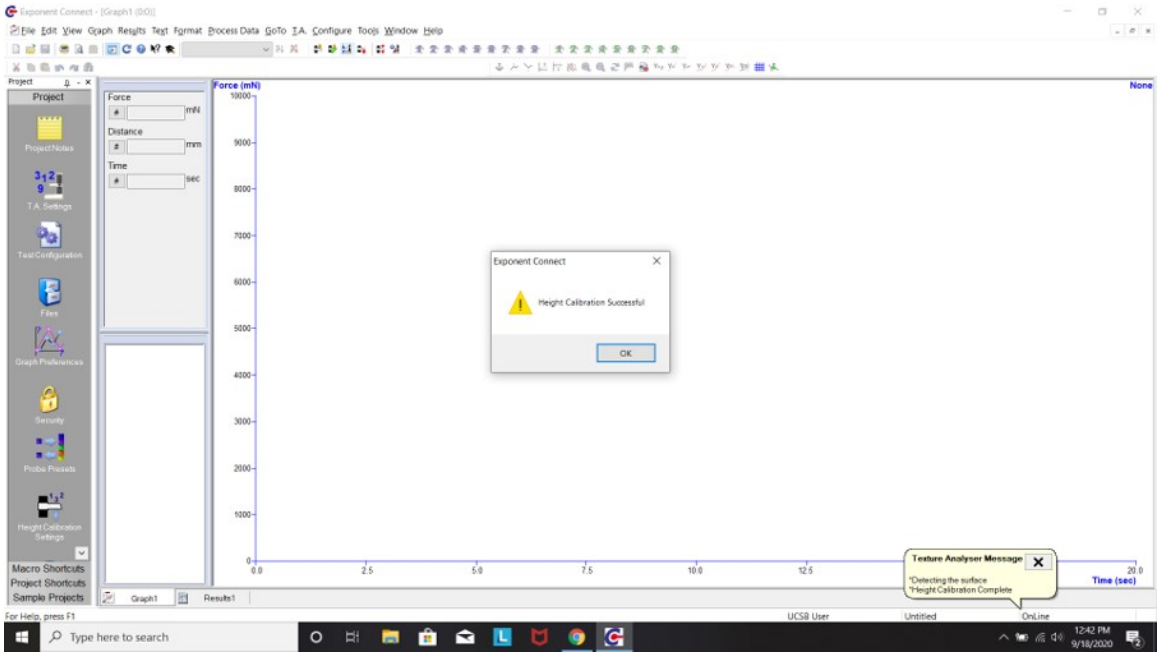


Now that the probe is close to the surface, select the calibrate probe height button.

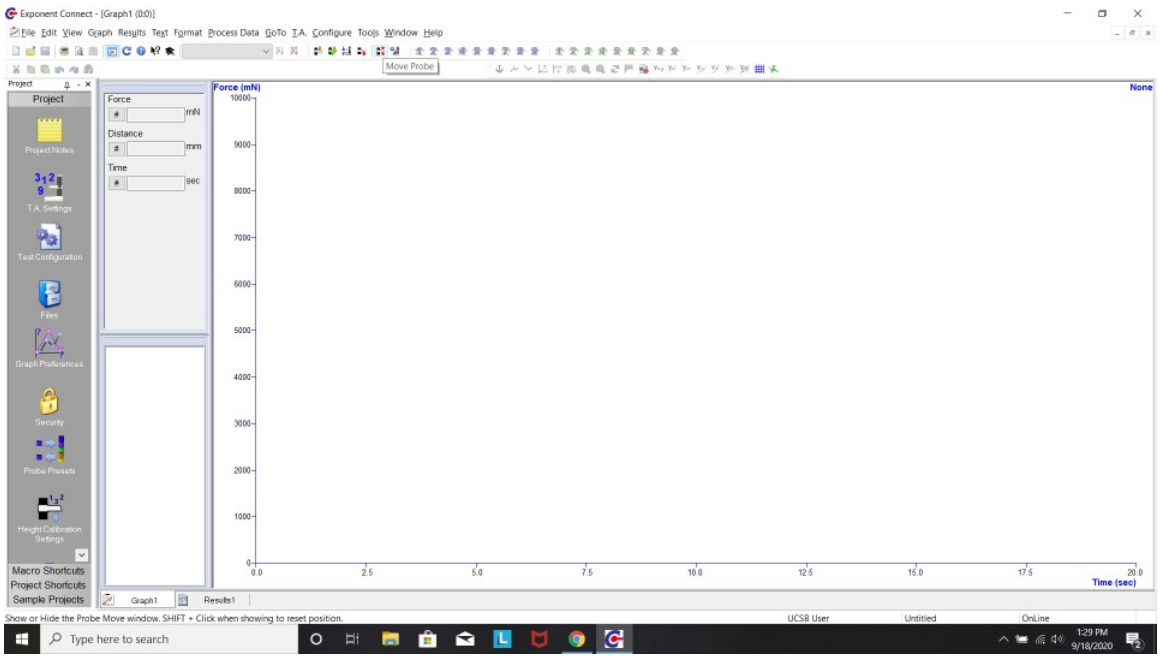


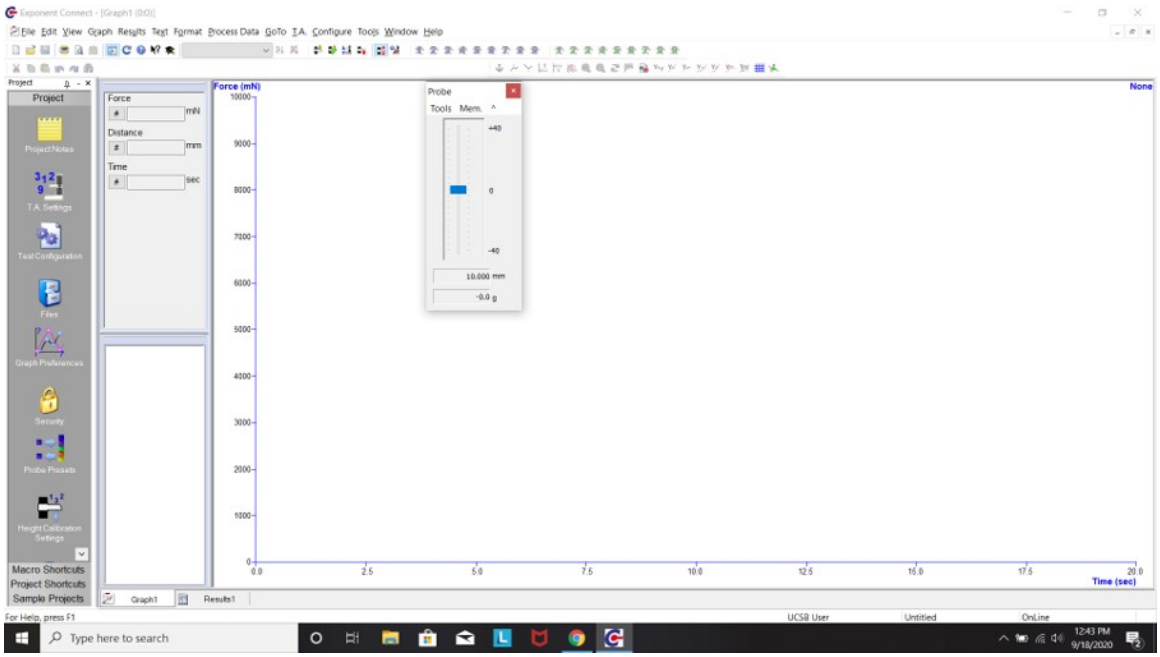


Select ok and the instrument will bring the probe into contact with the substrate until it senses a 10 mN force and mark that as zero displacement. The probe will then retract to 10 mm and the software will display this.

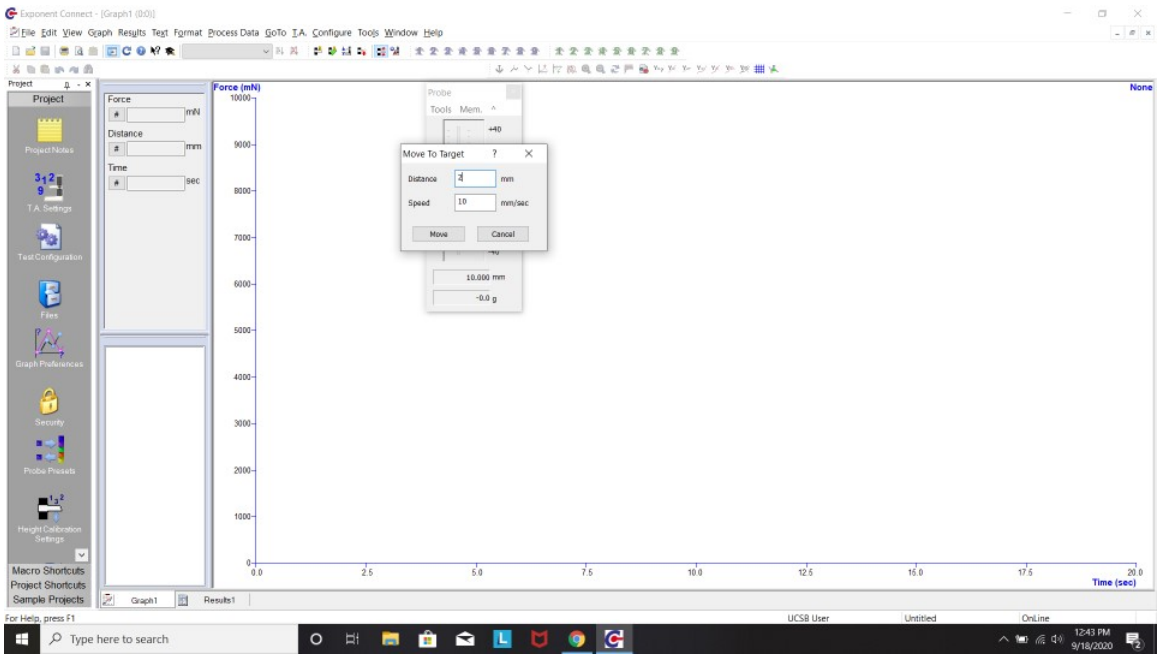


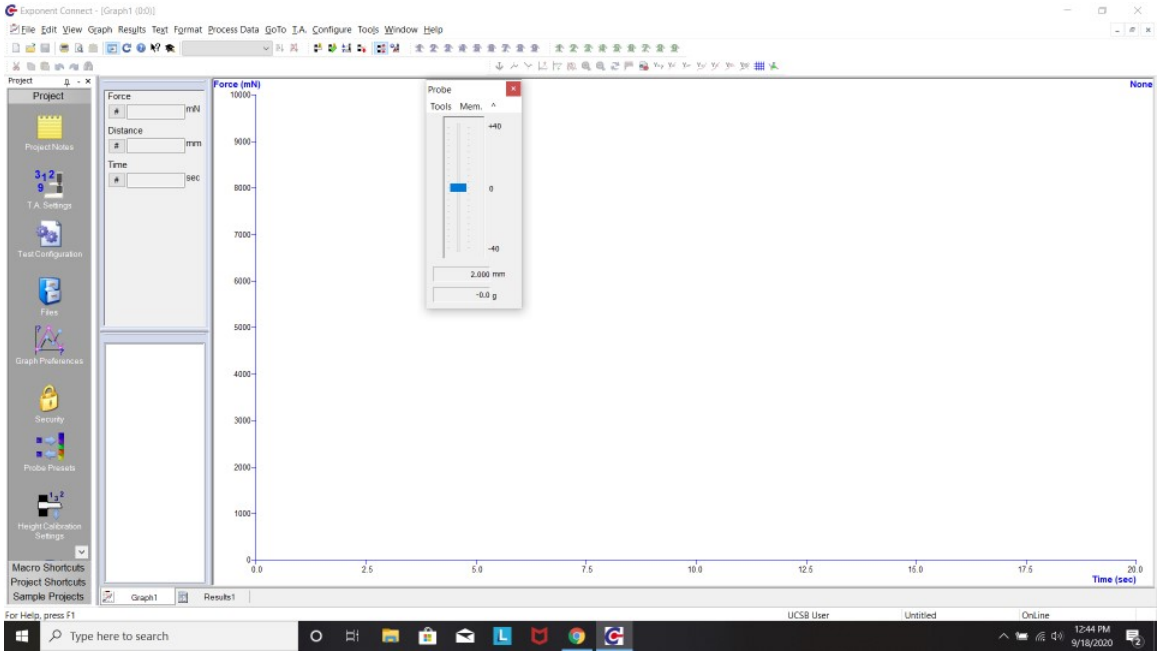
Now that you've calibrated the probe height you can use the move probe button in the software.





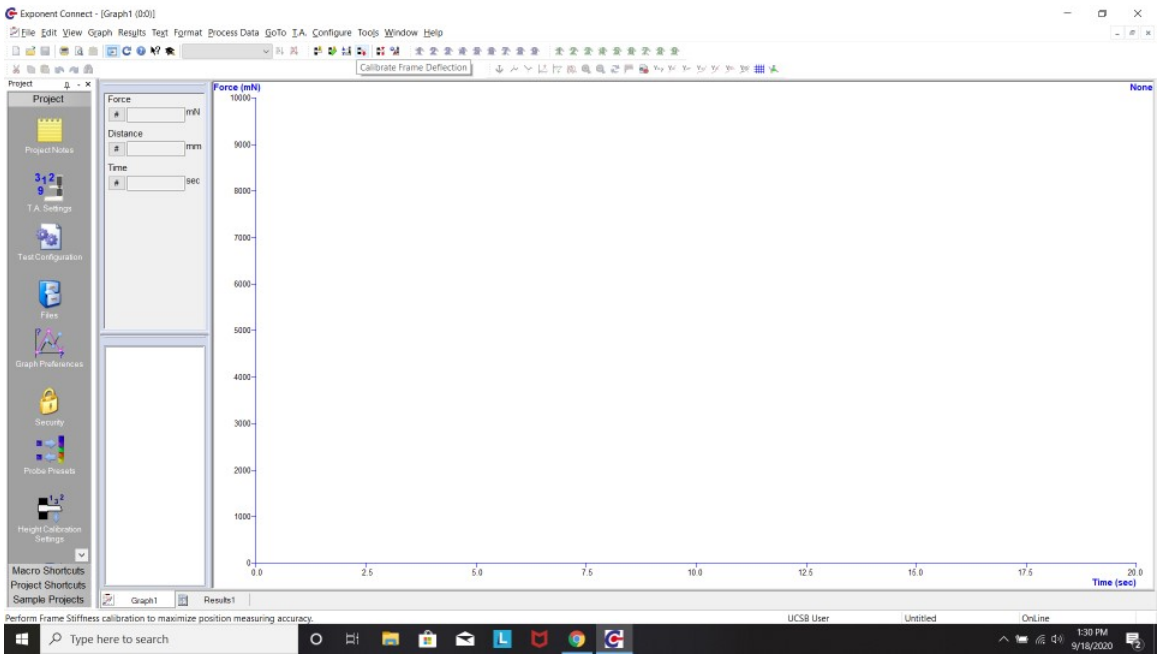
This will tell you the current location of your probe in space and let you set the position of the probe. This can be used to infer the height or thickness of a sample when the position of the probe is recorded before a test.

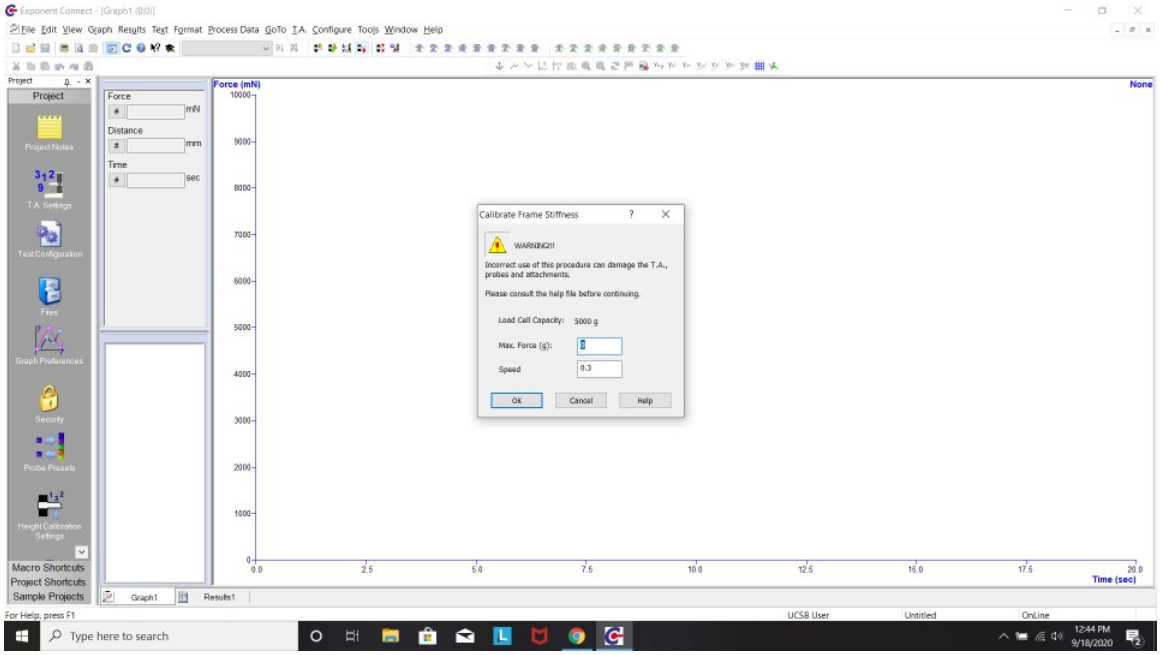




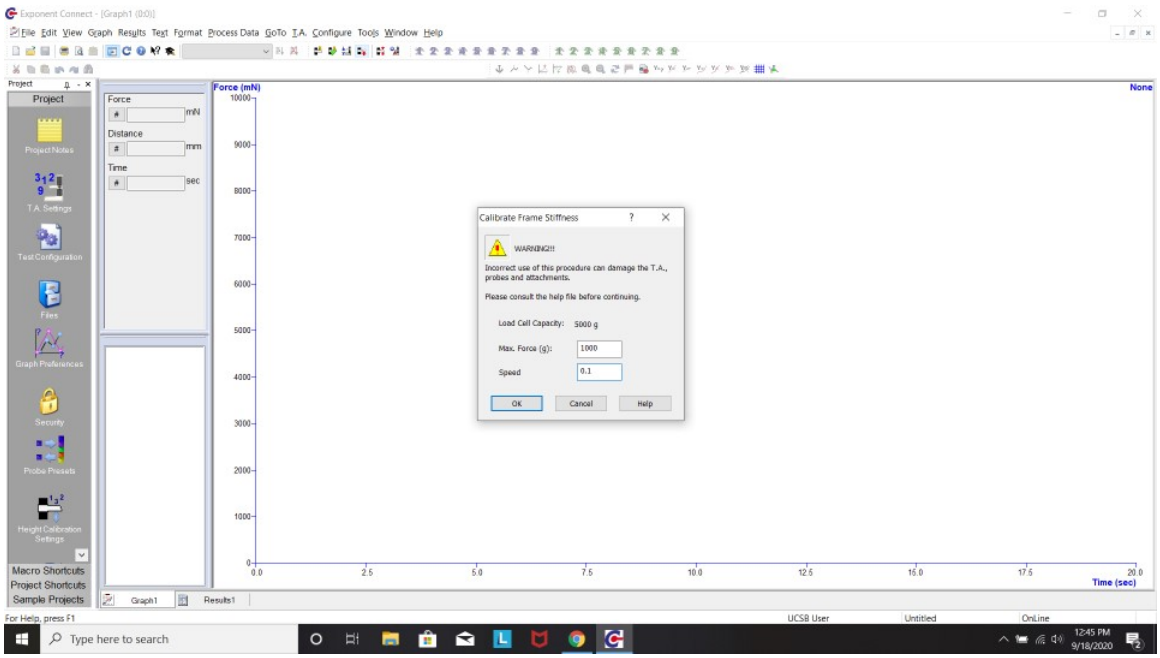
2.3.3 Calibrate the Frame Deflection

This calibration will measure the stiffness of your load cell and fixtures. It will then correct the displacement measured for any deflection that the load cell undergoes during the test. Note that the load cell and fixtures are fairly stiff and so this calibration is only important when using small displacements. Usually, this means that the calibration is important whenever you are testing materials in compression, such as when you are doing small strain contact mechanics or uniaxial compression. During extensional tests, the deformation of the sample is typically so large that this correction is negligible. Bring the probe close to the substrate again and select the calibrate frame deflection button.

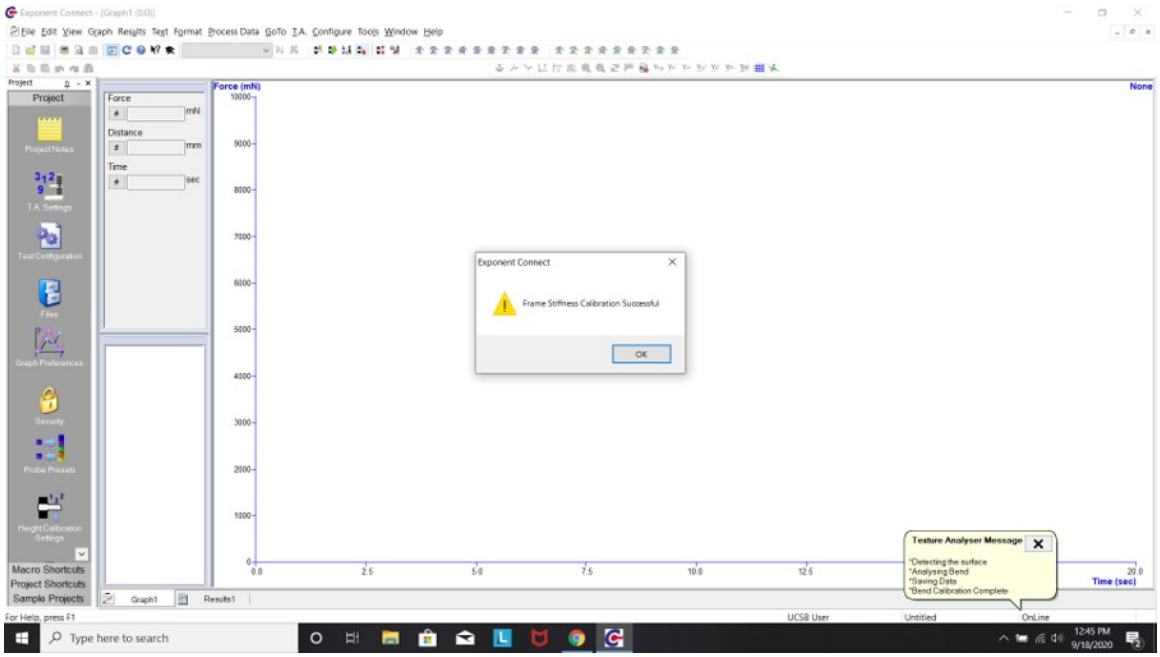




You will then be prompted to enter a translation speed and a force to be used for calibration. For the 2 mm diameter steel cylinder probe I typically use 1000 g with 0.1 mm/s as the speed. **Note that you may need to alter these values when using another probe that is too fragile to handle forces that high.**



Once you hit ok the instrument will bring the fixtures into contact to desired force and record the displacement needed to do so.

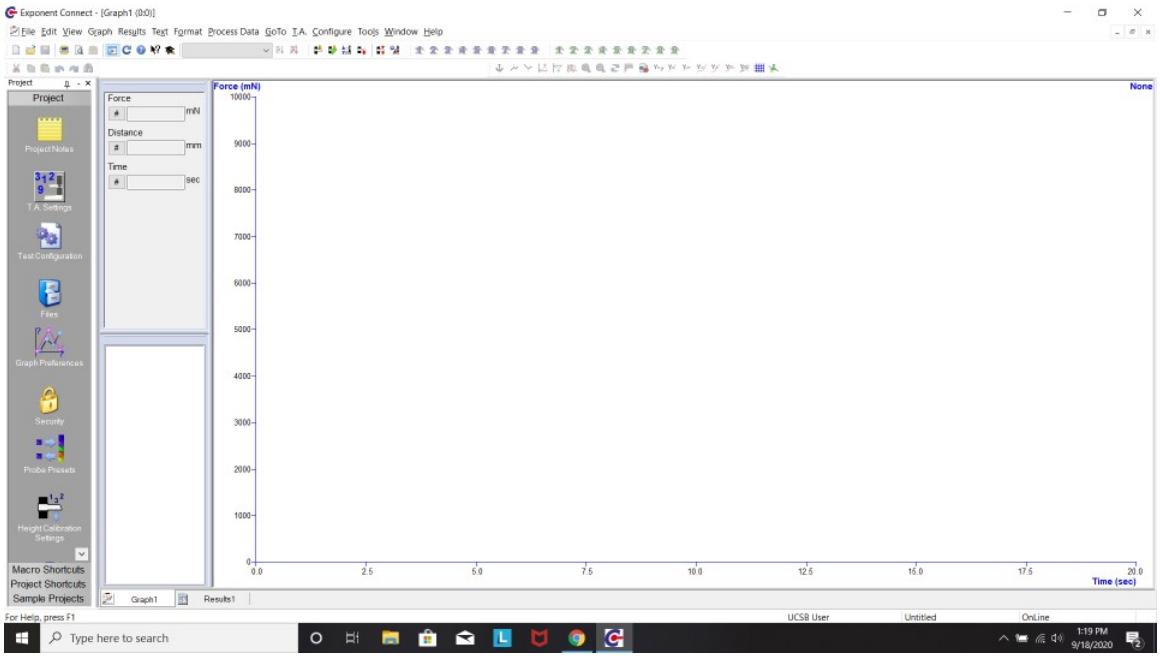


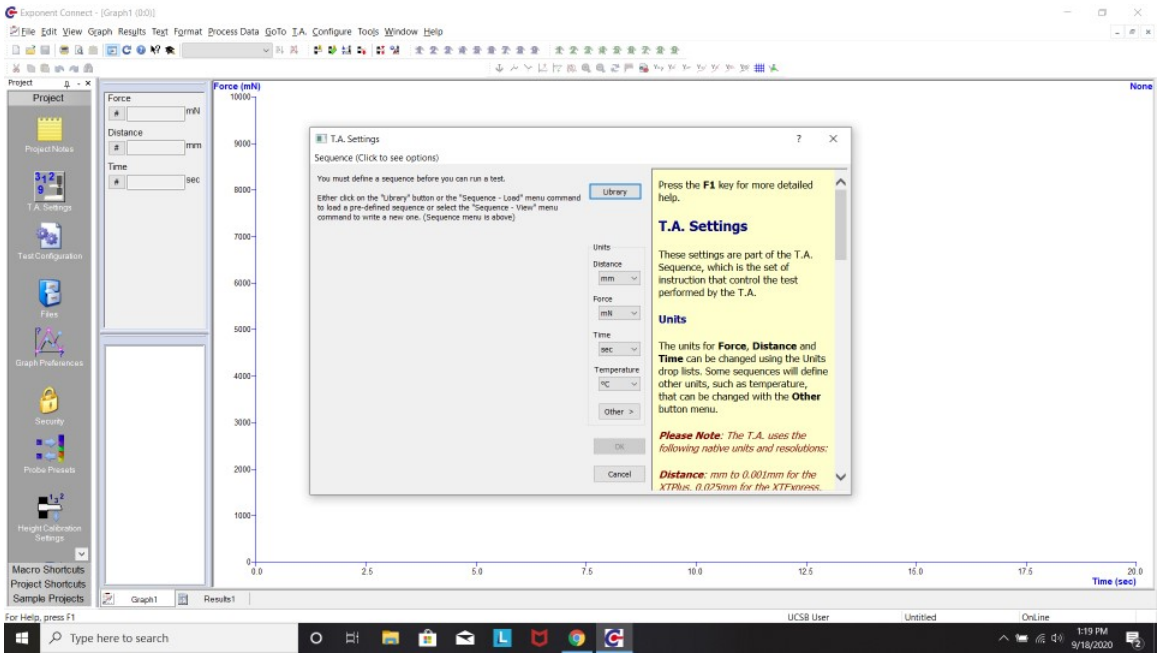
2.4 Load the Sample

Loading the sample is fairly straightforward in the indentation geometry. It consists of sliding the sample underneath the probe and lowering the probe close to but not touching the sample. Depending on the sample, I find it helpful to place the sample on a glass slide and slide it underneath the probe. If you're using a vial to hold your sample make sure that the probe is long enough to touch the sample without hitting the lip of the vial. When using extensional geometries where the sample needs clamped on the top and bottom, it is usually good form to clamp the sample on the top first and then lower the sample into the bottom clamp.

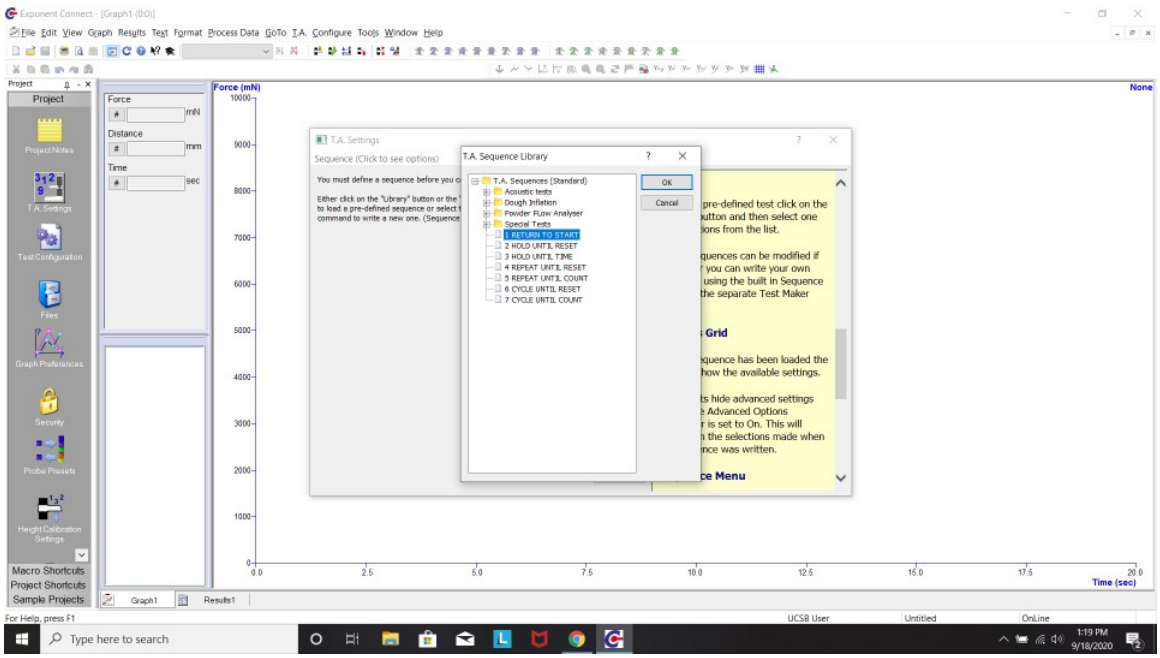
2.5 Tell the TA what to do in the Software

Once the sample is loaded and the calibrations are run, you need to tell the instrument what to do during the test. The goal for this example indentation test will be to translate the probe into the sample at a speed of 0.1 mm/s to a turnaround force of 20 mN and translate back to the initial position at the same speed of 0.1 mm/s without having any dwell time at the turnaround force. We also want the instrument to correct for the frame deflection. This is done by clicking on the TA settings tab on the left hand side of the screen.

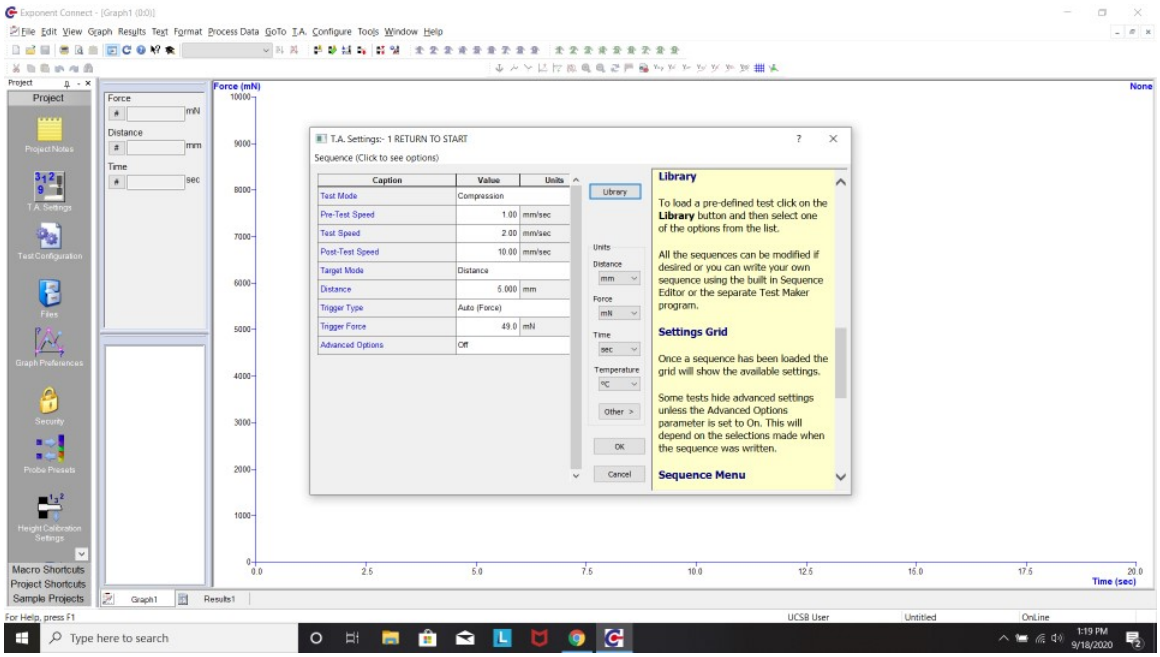




The first thing you will need to do is select an experimental sequence. Many standard experimental sequences come premade and can be found by clicking the library button.

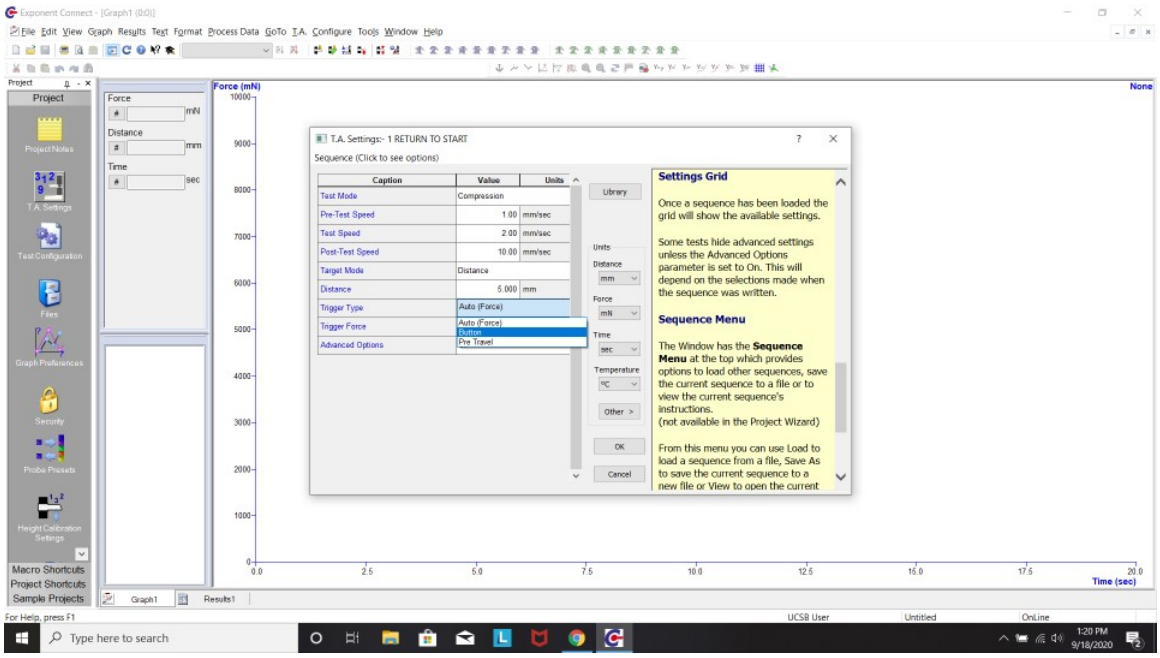


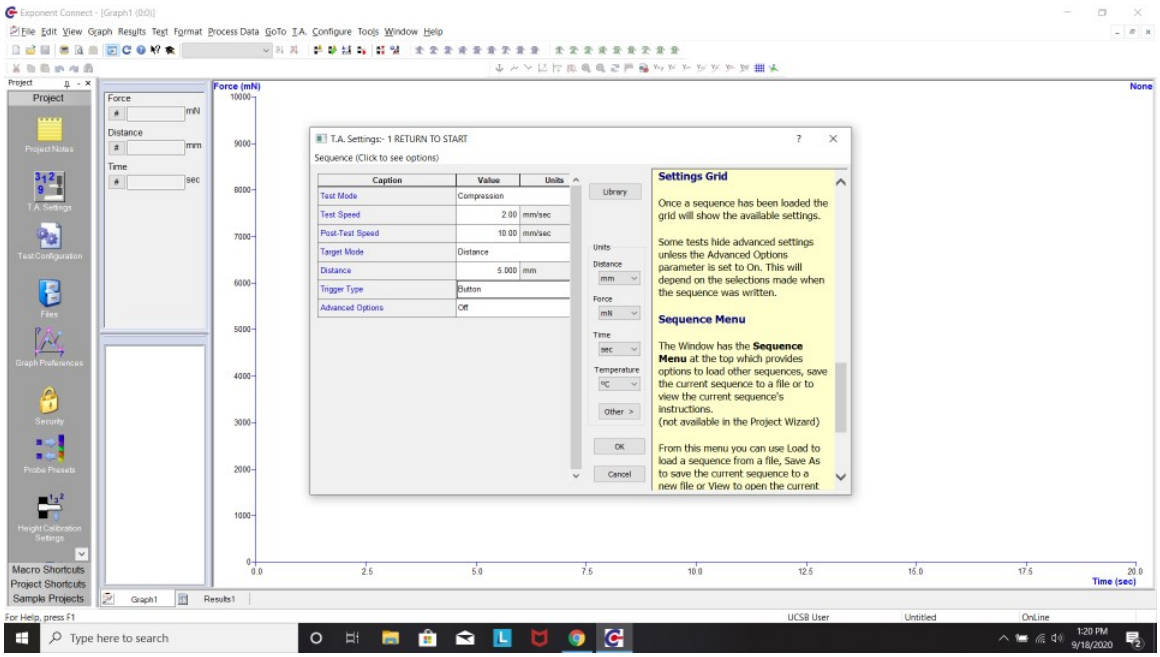
The names of these sequences generally describe what they tell the instrument to do. Note that if you want to do something custom not captured by these sequences you have the ability to reprogram an existing sequence and save it as a custom sequence. For this experiment we will use a return to start test as it will tell the instrument to translate until a turnaround condition is met and then return to the starting position.



Once this is selected a number of inital values will be pop up. First you will chose the test mode as tension or compression. It's initially set to compression for this experimental sequence. That works with this indentation test, but will need to be switched to tension when doing tensile tests. **Note that forgetting to change this during a tensile test can lead to damage if the test is not stopped before the fixtures slam into each other.**

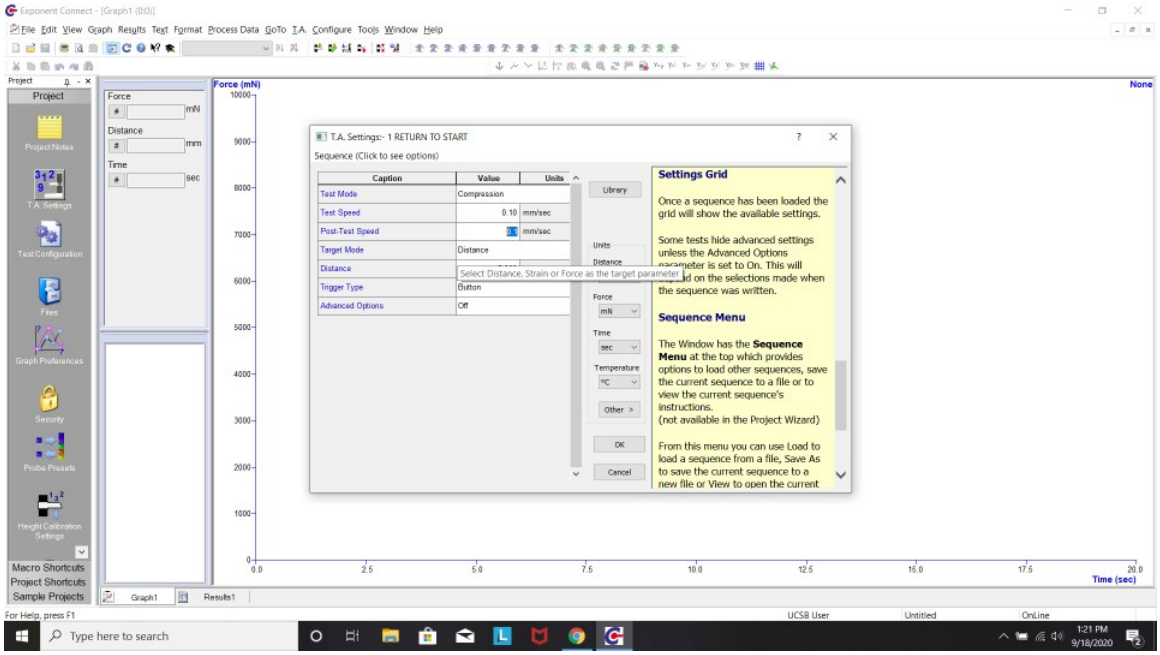
Next you will notice that there are three different speeds. The pre-test speed varies based on the trigger type and controls the translation speed of the instrument before data collection is triggered. Data collection can be triggered in one of three ways. It can be triggered after the load cell reaches a certain trigger force (the inital option). It can also be triggered after traveling a certain distance. Finally, it can be triggered as soon as the probe starts moving (the button option). The force option should never be used. The distance option should only be used in cases where a significant pre-travel is needed. The button option should almost always be used.



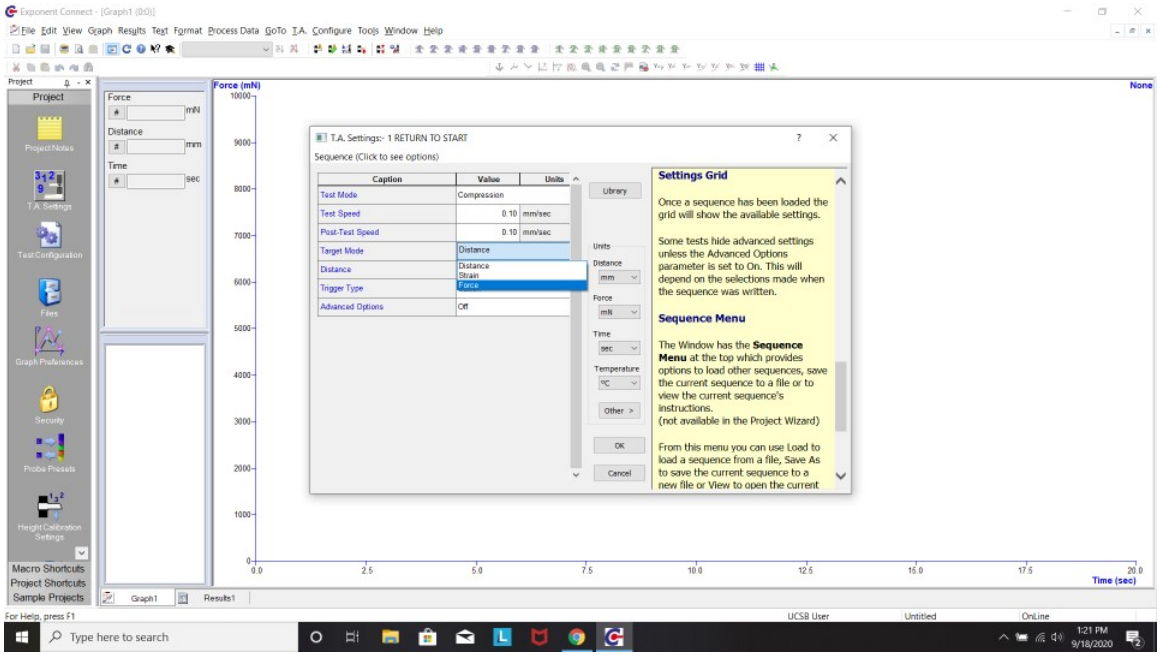


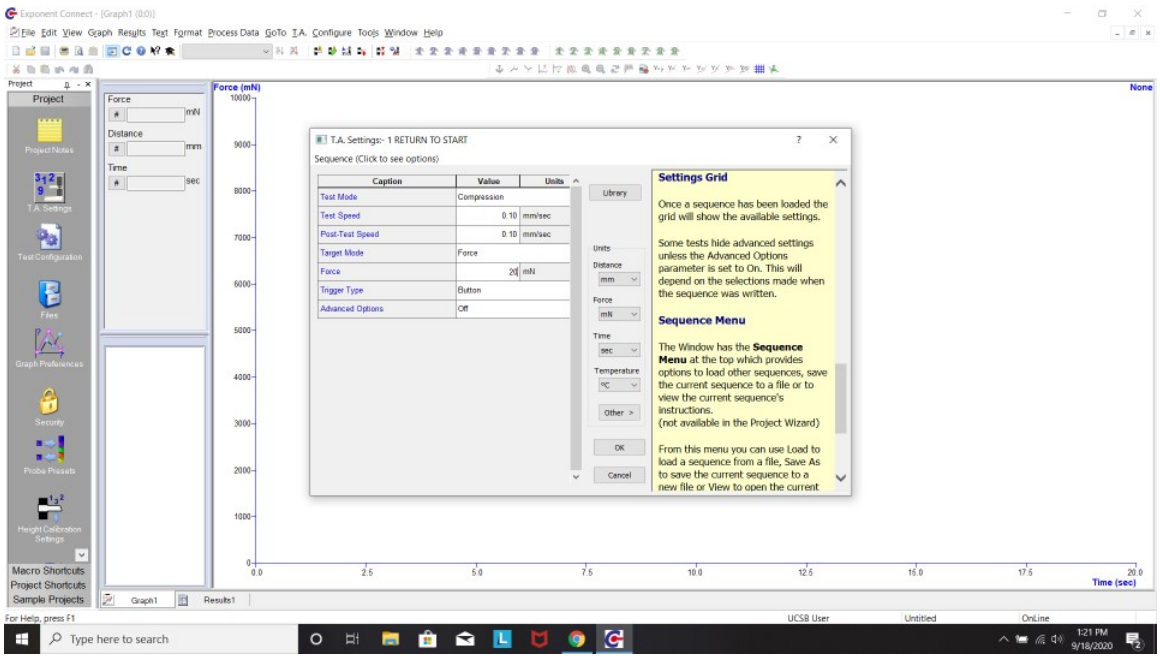
Note that changing the trigger type to button eliminates the option to set a pre-test speed. The test speed describes the translation speed before the turnaround condition is met. The post-

test speed describes the speed of translation after the turnaround condition is met. These are often set to the same value and in this instance will be set to 0.1 mm/s.

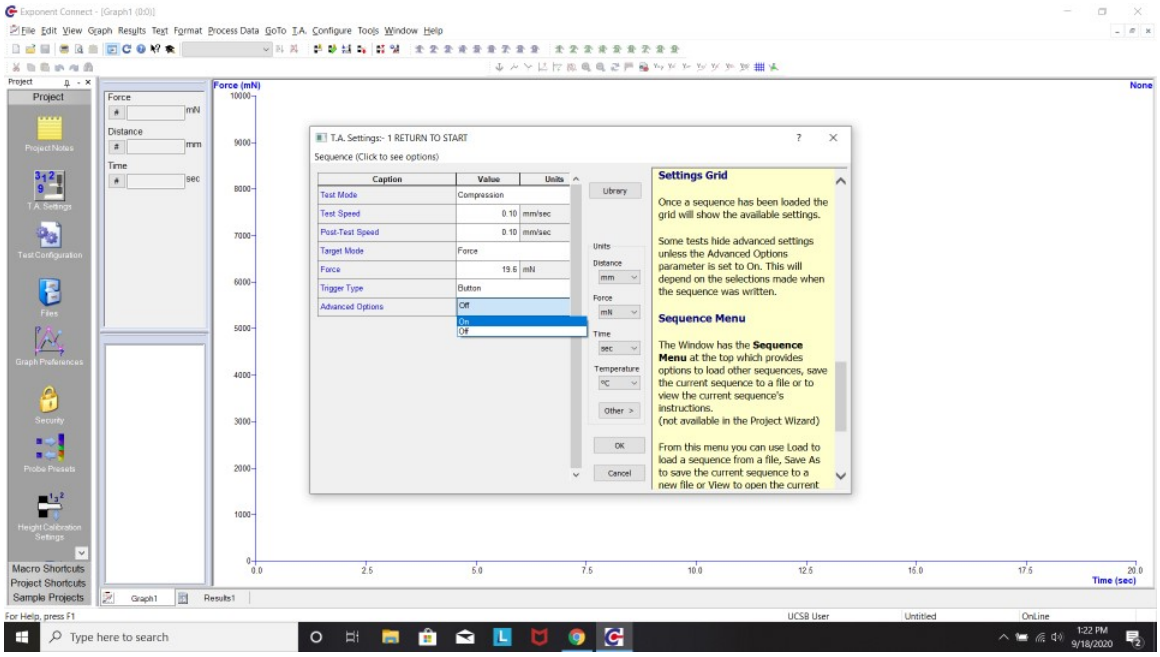


Next you will need to set the target mode. This sets the turnaround condition. It can set to distance, strain, or force. Strain should not be used because you will not be entering the sample dimensions into the software (you can convert a desired strain over to a distance by knowing the sample dimensions). In this case we want a turnaround force of 20 mN so we will choose the force option.

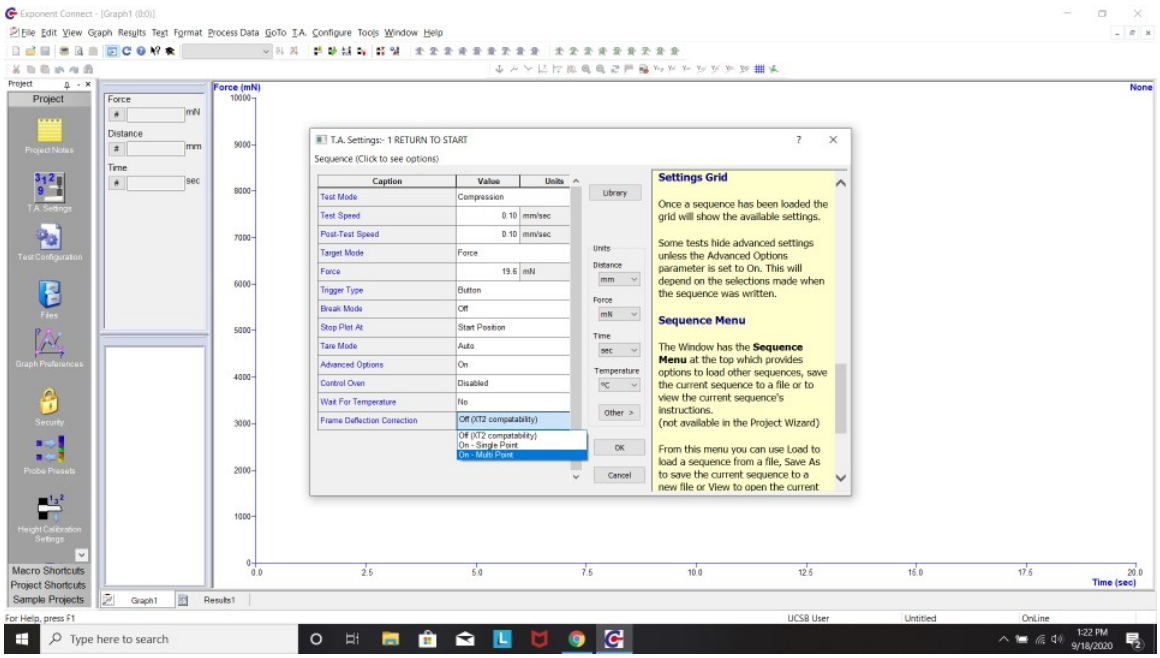




Next you need to turn the frame deflection correction on if you are running a test where you need it. In this case we do so we will turn advanced options on.



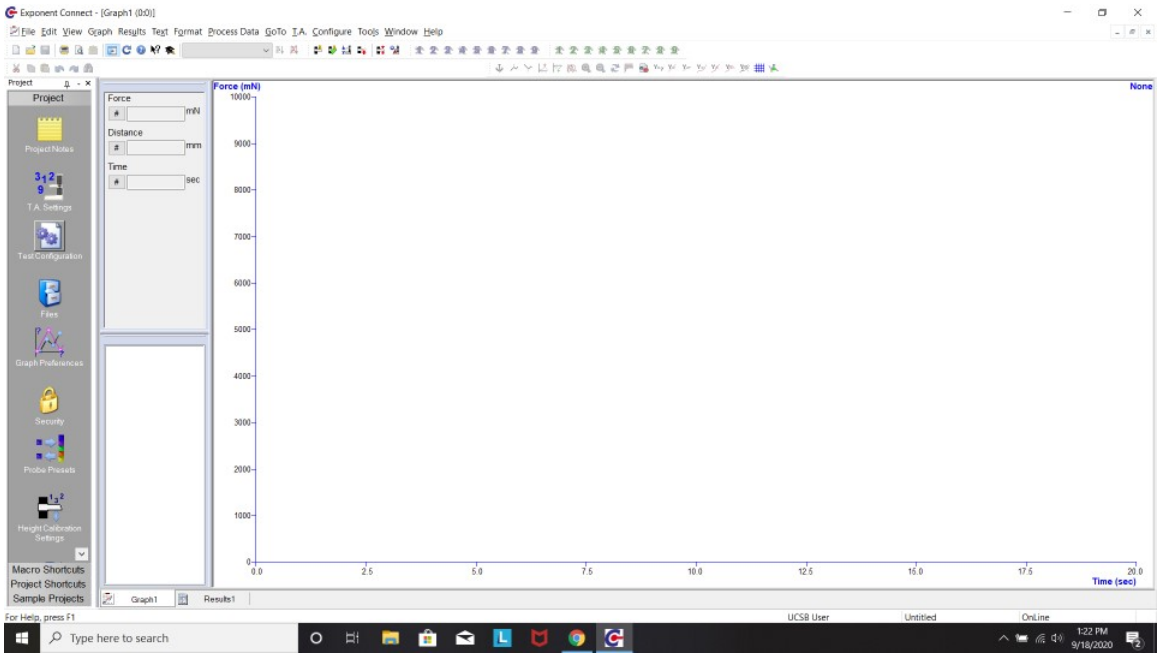
Once you turn advanced options on a number of other choices will appear. You only need the frame deflection correction option.

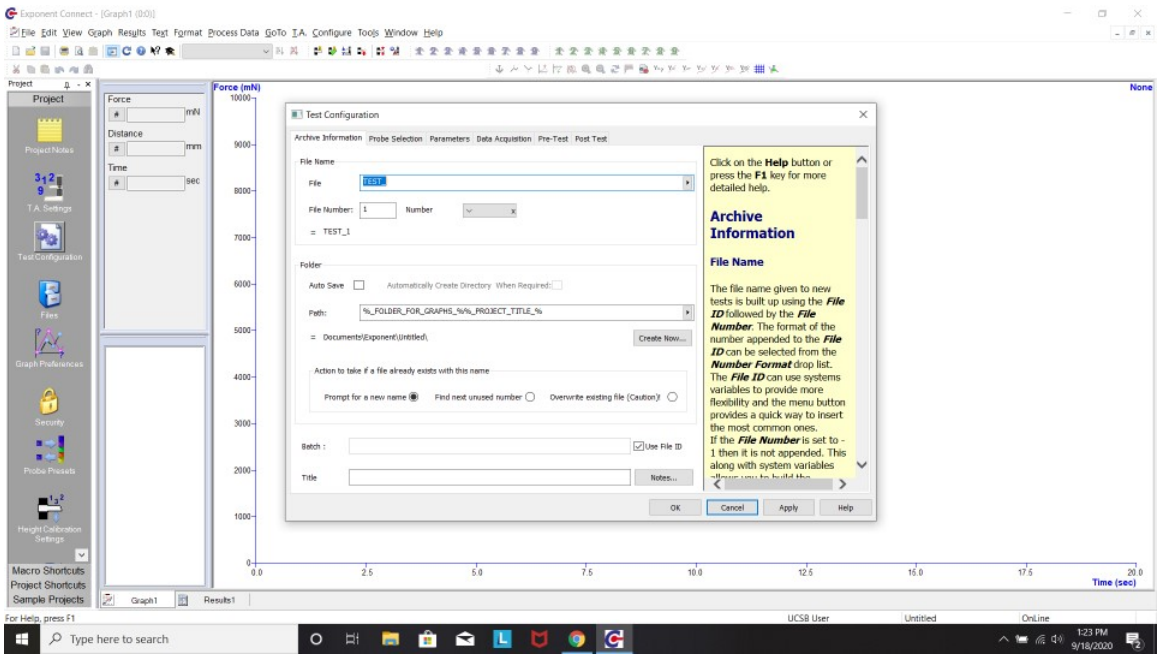


You need to select the On-Multi Point option. This will tell the TA to use the frame deflection calibration and infer the displacement correction. If you select single point, it will substract off one displacement from every data point and effectively just shift the entire displacement profile. This is the last thing you need to set and you can hit ok.

2.6 Tell the TA Where to Save the Data

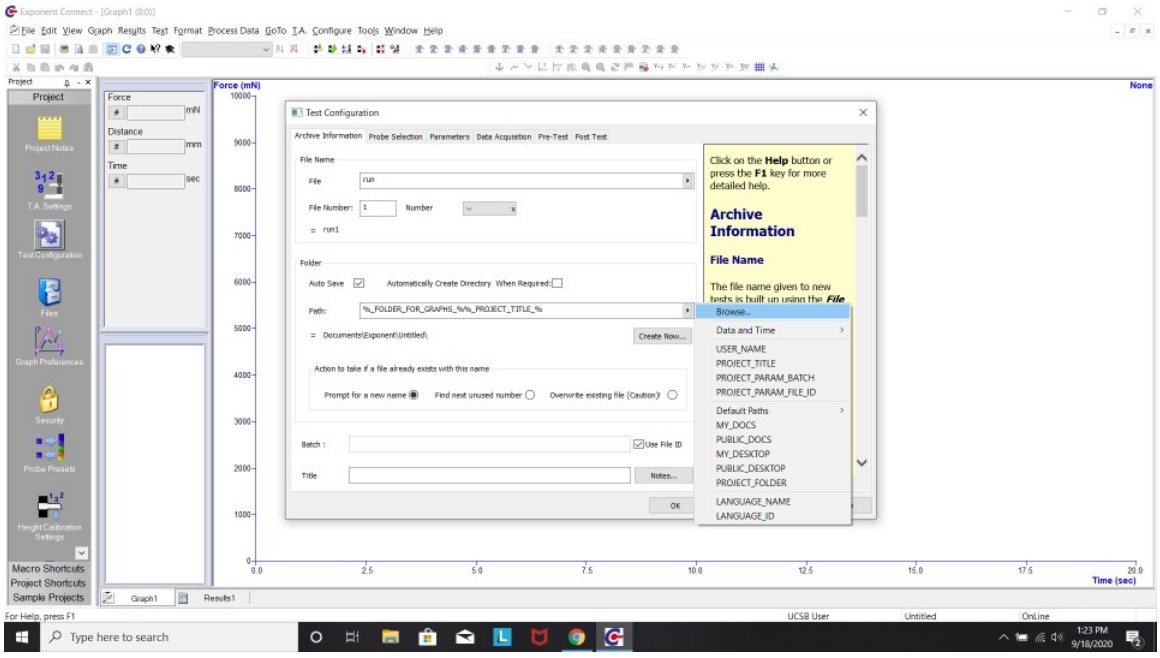
Now that you've told the instrument what to do, you need to tell it where to save the data. This is done with the test configuration button on the left hand side.

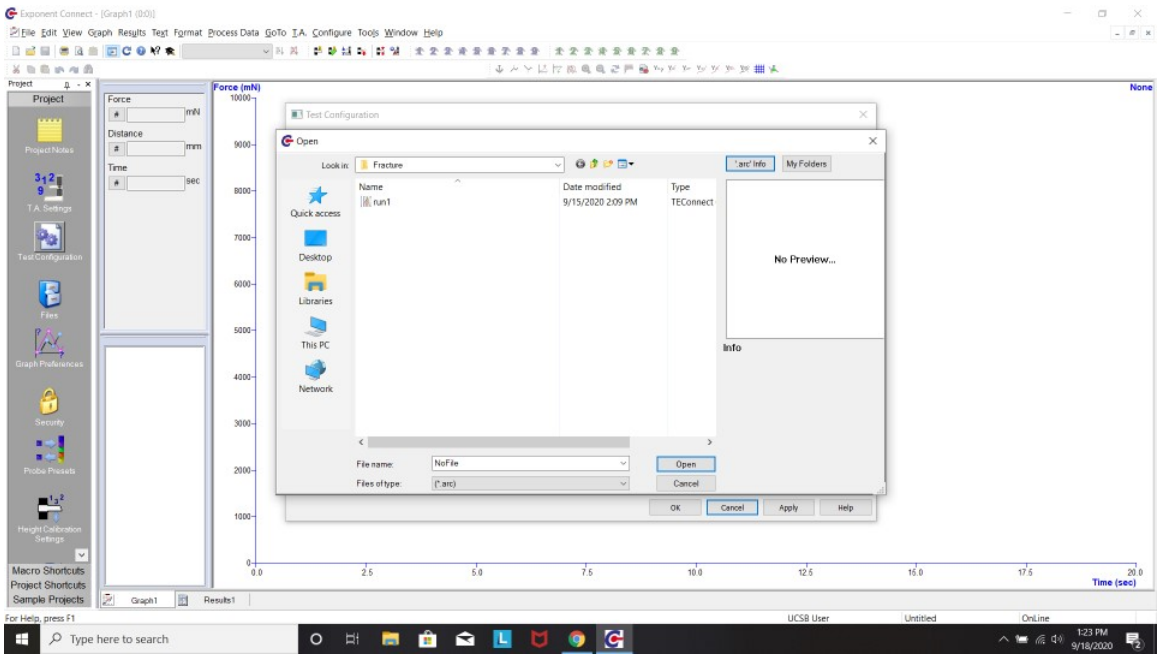




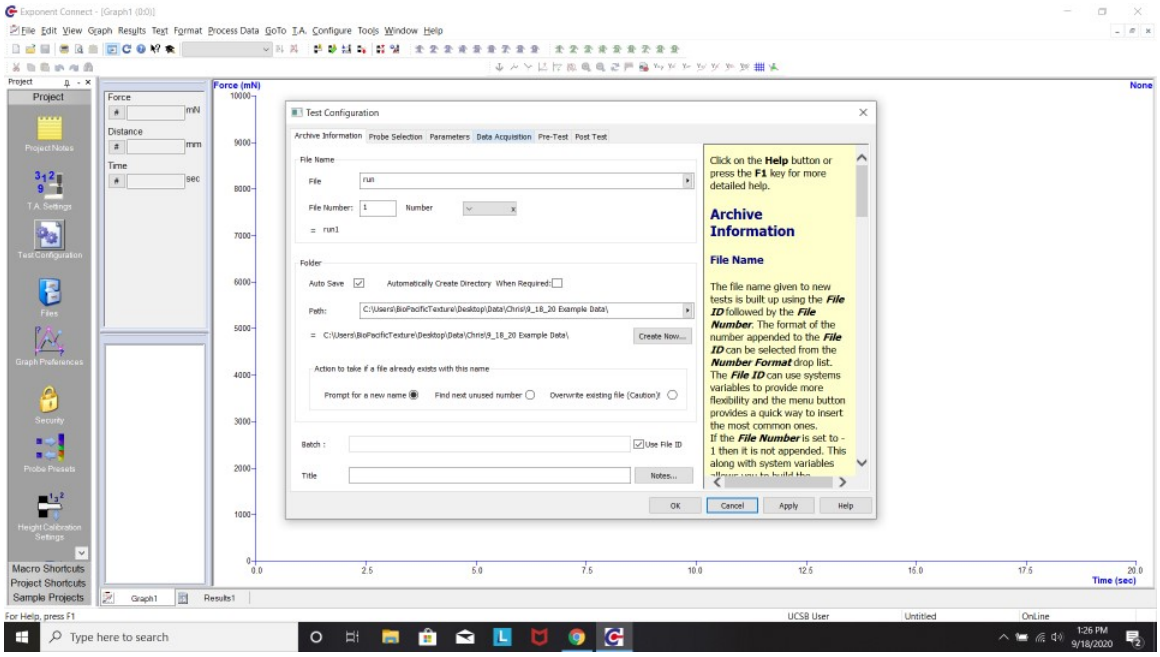
This will bring up the archive information tab where you will set the file name and folder where the data will be saved. For the example, I've set the file name to run. When you run multiple tests under the same conditions, the TA will save multiple tests with the same file name but will iterate a number at the end (run1,run2,run3...). Note that I use a very generic name for the file instead of something long that specifies the test and sample conditions. This is intentional as it makes postprocessing (discussed in Section 3) easier to automate. I typically use the folder hierarchy to store these details.

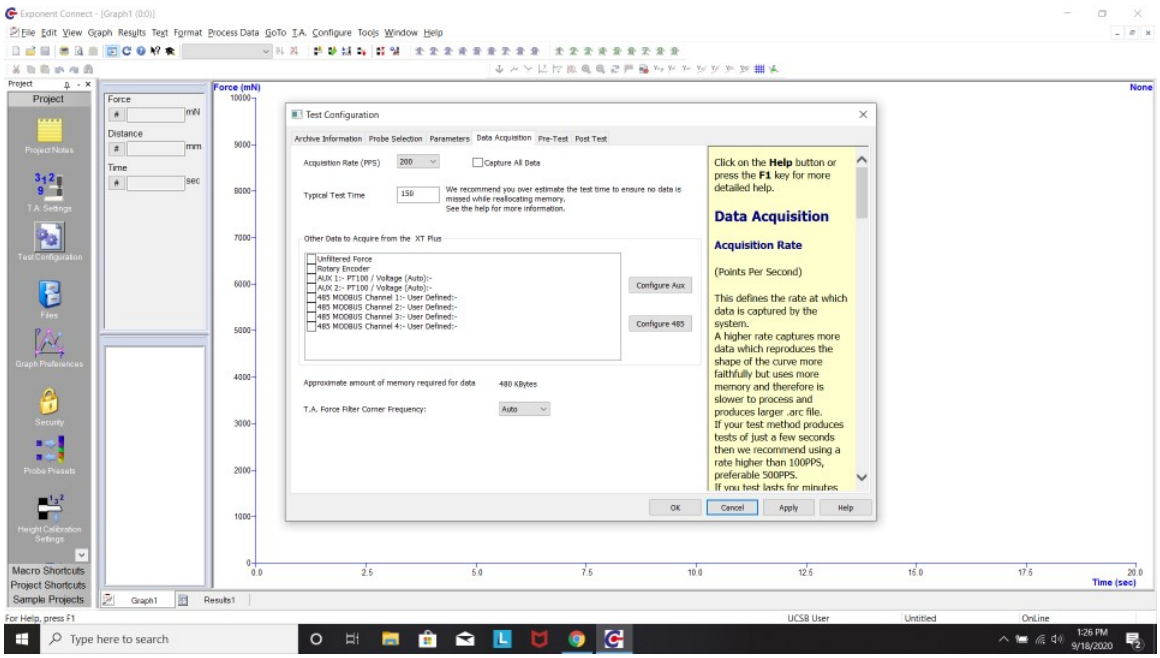
Click the autosave button and choose a folder by selecting the browse option under the arrow to the right of the path. There is a data folder on the desktop where you can create a folder for yourself.



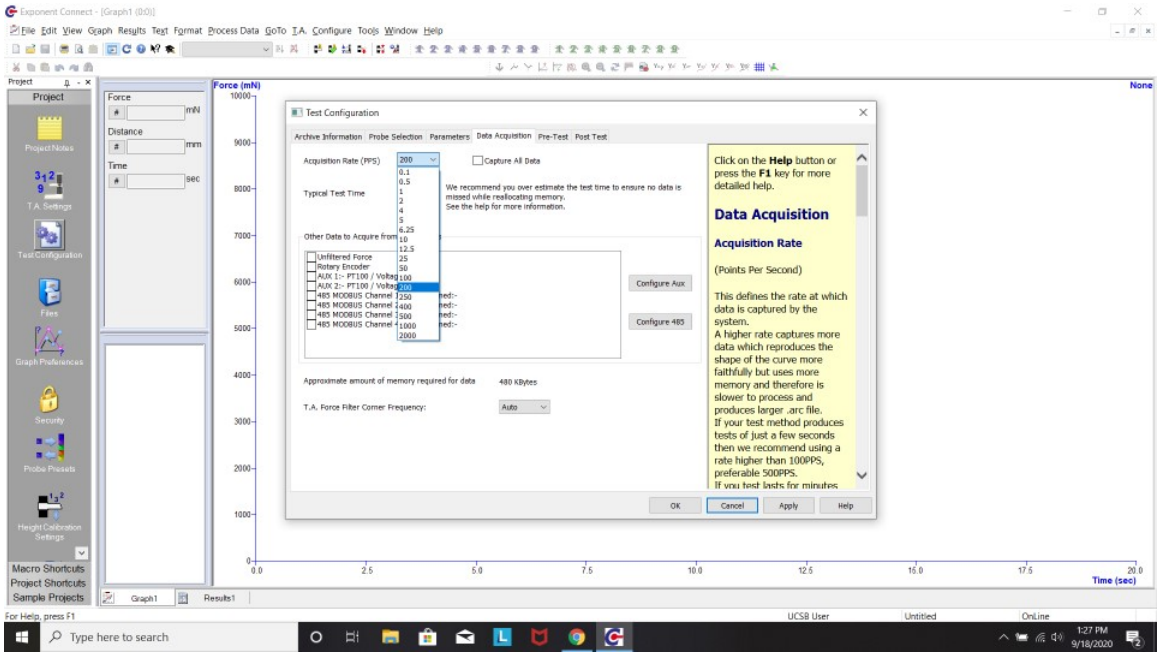


Note that the autosave option will save a file that can only be opened in the exponent software; however, it is still important to setup autosave as it will save you time when exporting data later on for further analysis. Exporting data is discussed later on. Next you will need to access the data acquisition tab.

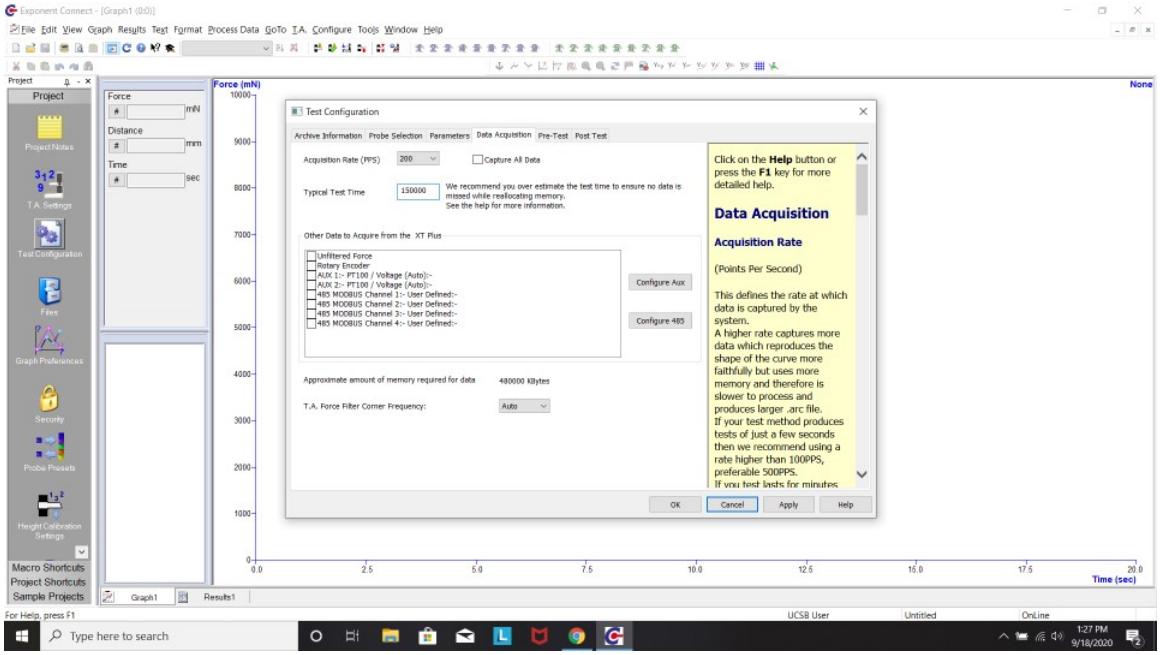




You will see an option to adjust the number of points collected per second. You can turn this up when trying to capture really dynamic events. You can also turn this down if you are running a longer test where time resolution is not as important and you don't want an unwieldy amount of data to analyze.



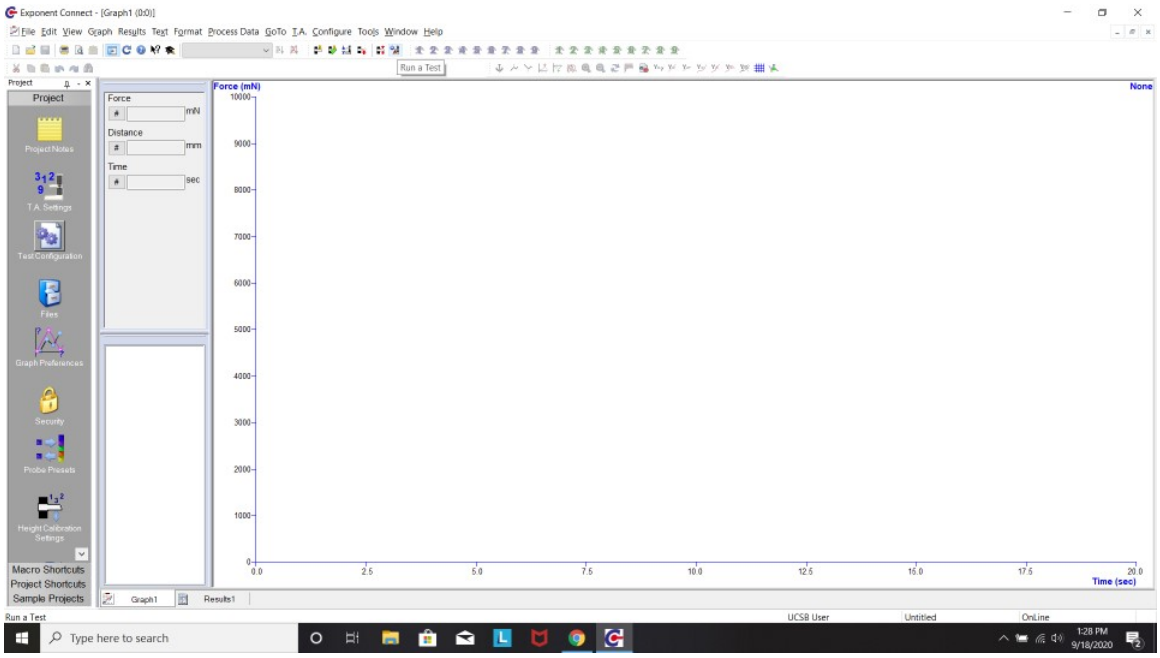
Finally you will need to adjust the typical test time. This tells the computer to set aside enough memory to gather the data during your test. It's typically set to 150 s. I usually set it to 15000-150000 seconds. If you forget to reset this value and gather data for longer than 150 s, the software can crash as it tries to both run the test and reallocate more memory for your data. After a crash you will lose any data during that run and any unsaved data.



Once this is done you are ready and can hit ok to close the window.

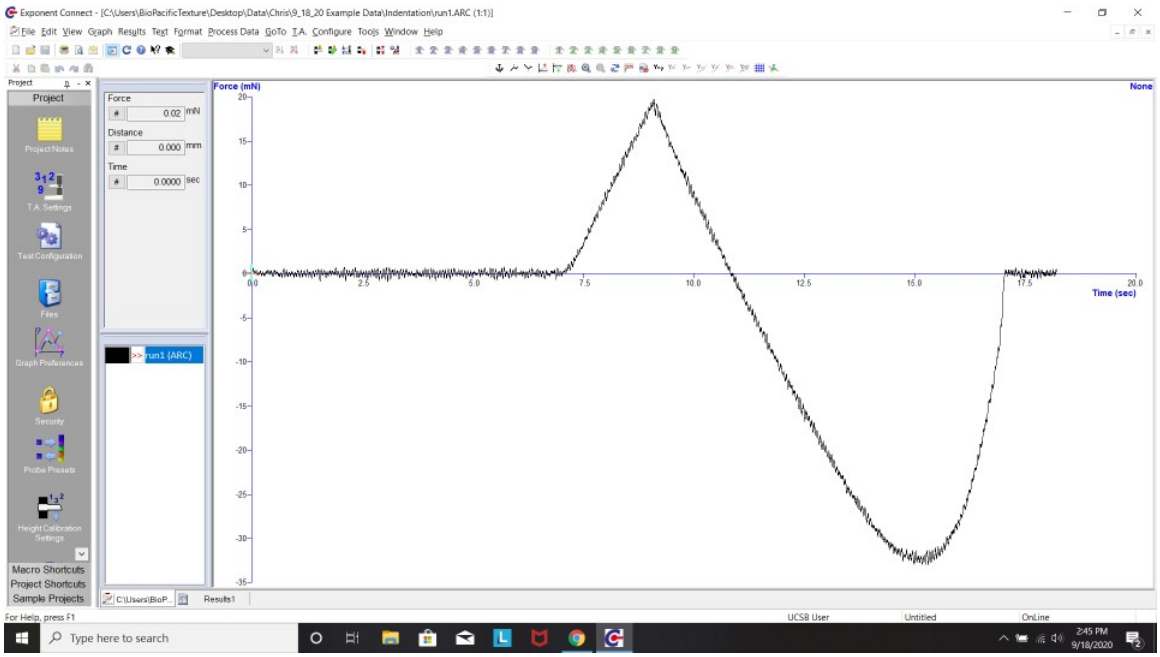
2.7 Run the test

Now that you've calibrated the instrument, loaded the sample, told it what to do during an experiment, and told it where to save the data you are ready to run a test. There are two different ways to run a test. You can either use the run a test button or you use the ctrl q shortcut to do so.



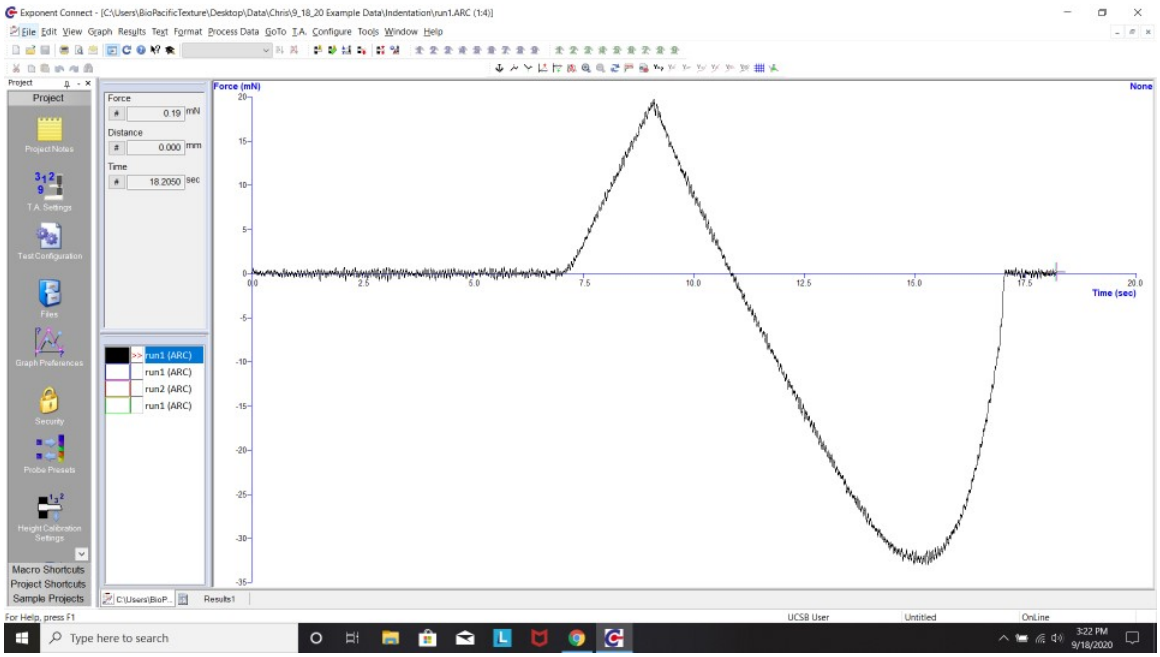
Note that if you've setup an auxiliary camera, it's good form to start capturing video before the test and stop capturing video after the TA is done with test so that you can be sure to capture the entire event on camera. The exception to this would be use of a high speed camera that only captures data during a small window of interest.

After running your test, your data will display on the screen and the graph will automatically rescale. You change the units or axes on this graph by clicking on them.

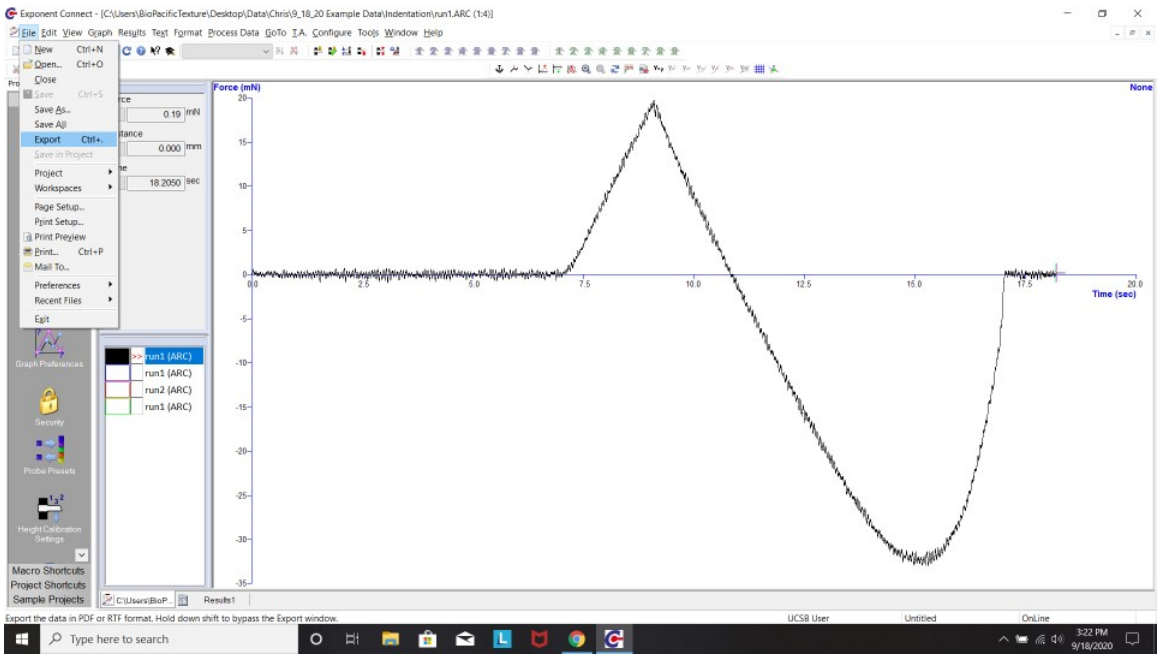


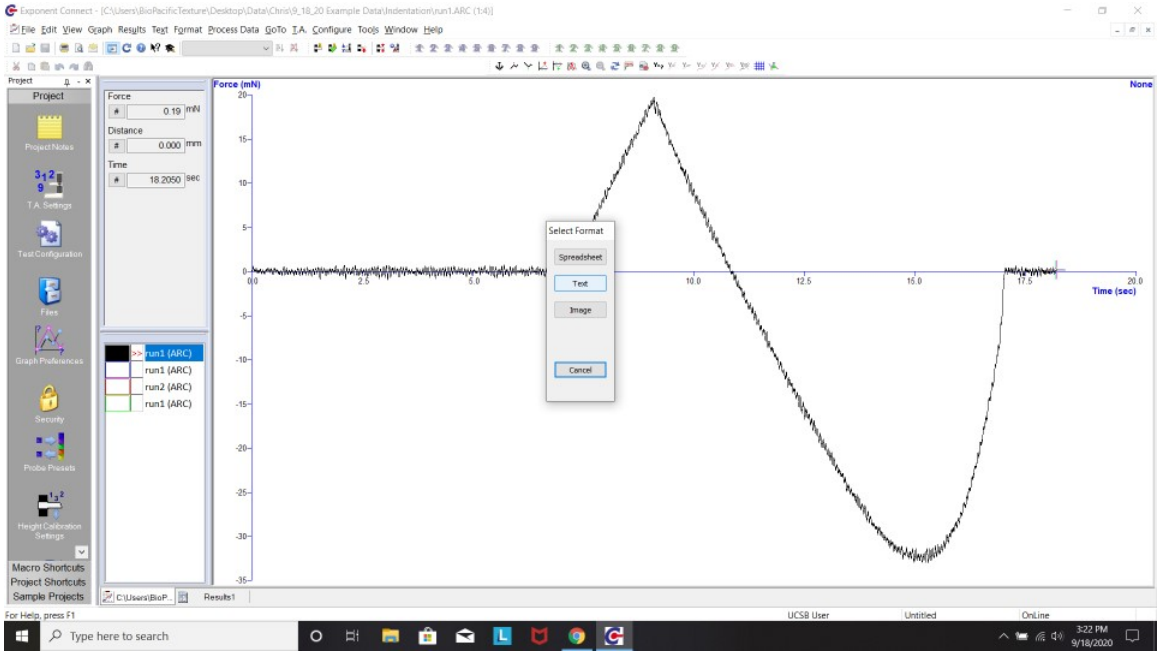
2.8 Export the Data

Now you will want to export the data you gathered in a form that can be processed in another program (i.e. origin or MatLab). First select the file that you want to export.

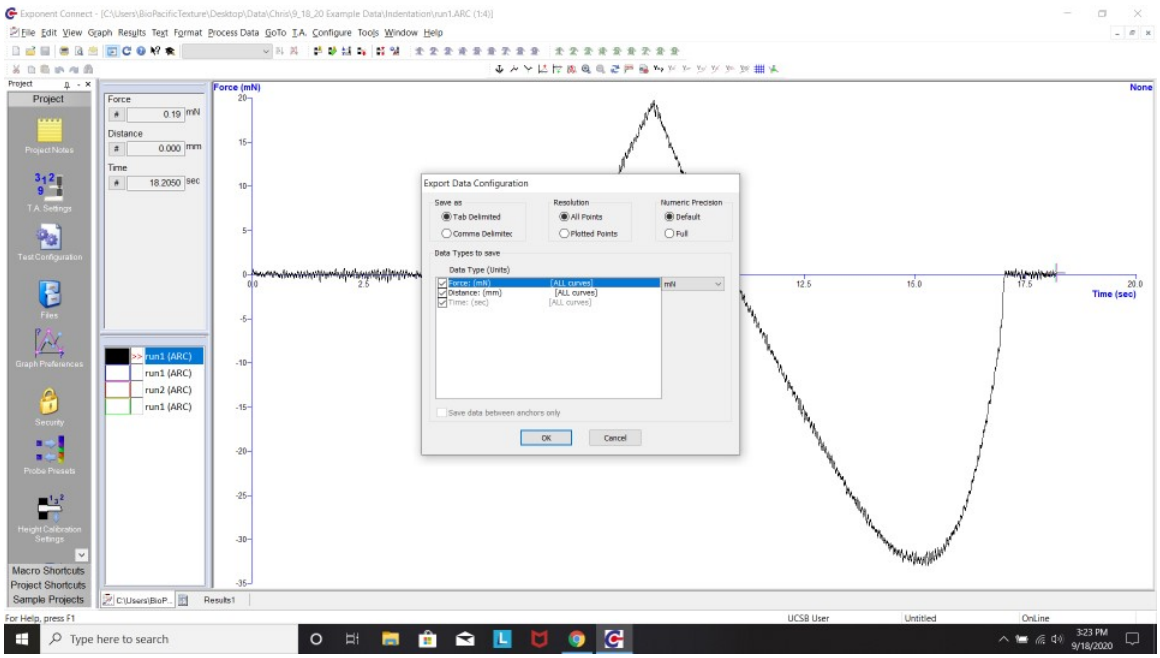


Then go to file and down to export. The shortcut for this is "ctrl ." and will bring up several file options for exporting the data.

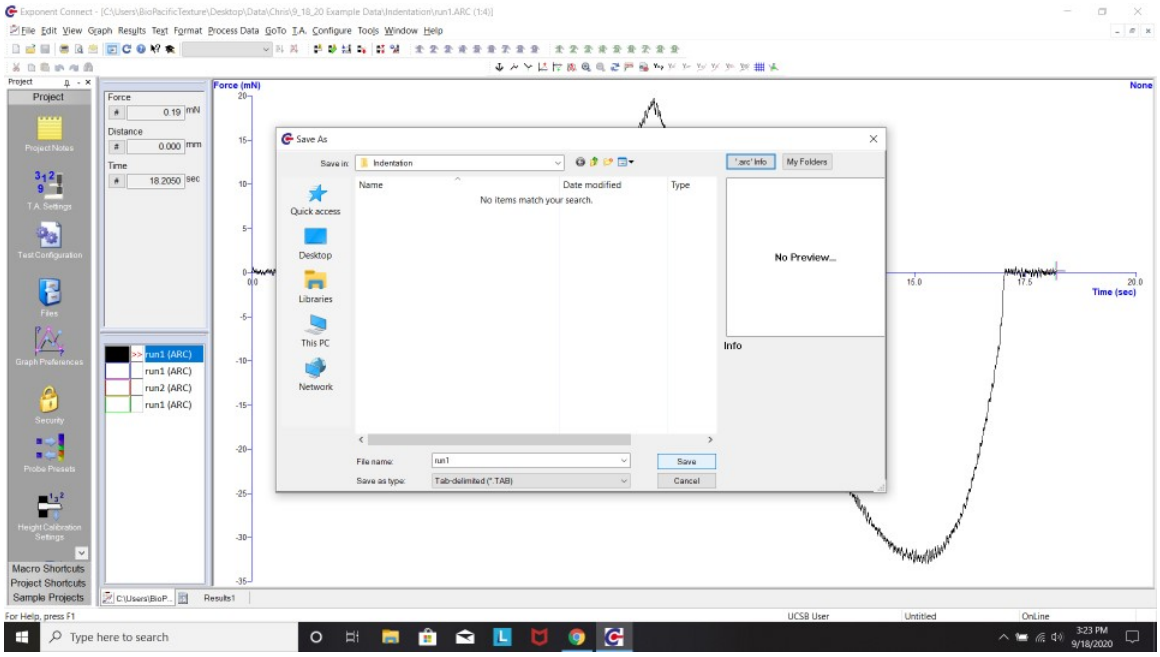




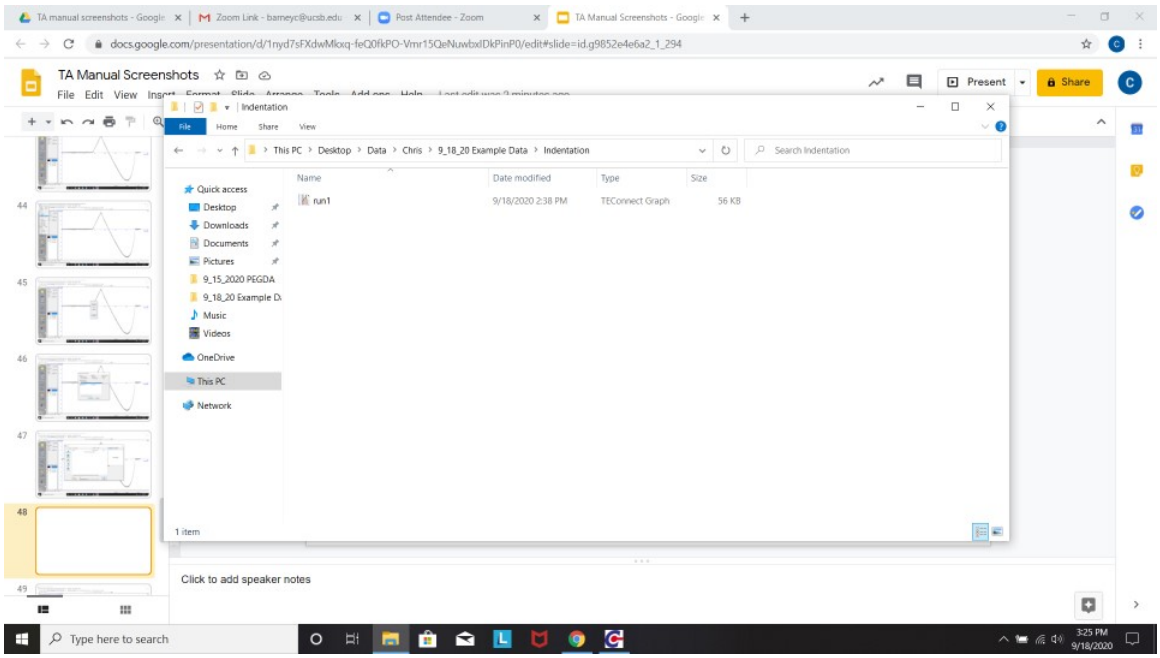
I like to work with raw data so I typically use the text file option.

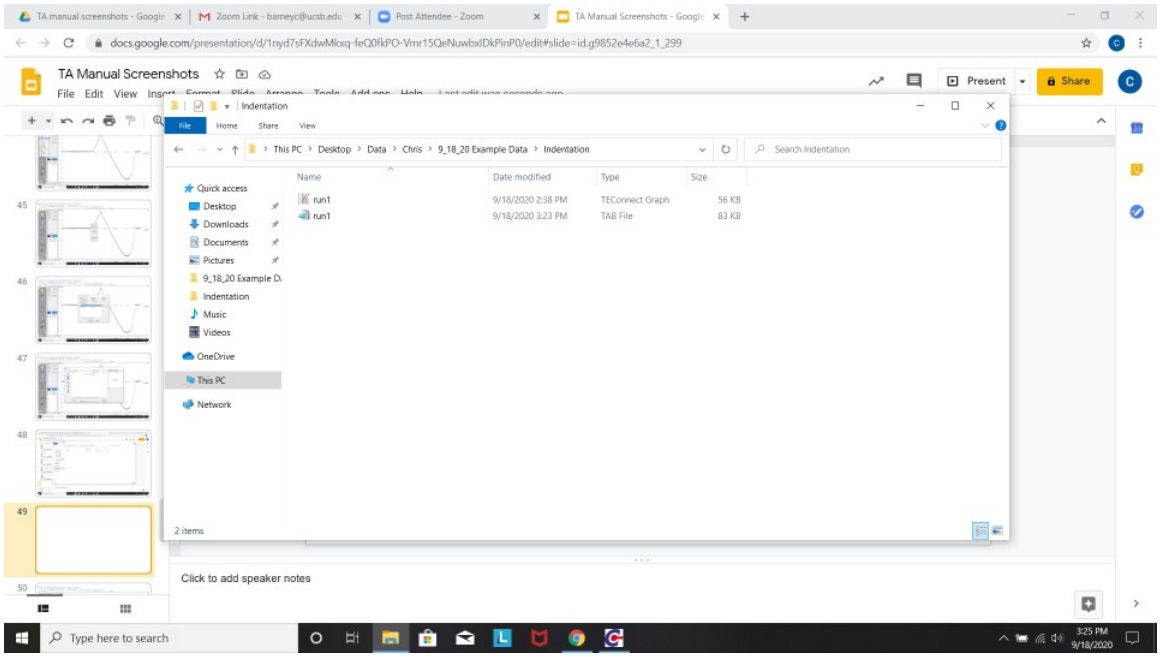


Once you hit ok it will ask where to save the file. If you setup autosave earlier it will automatically use the same directory where you saved the exponent file. If you didn't, you will have to find your folder again.

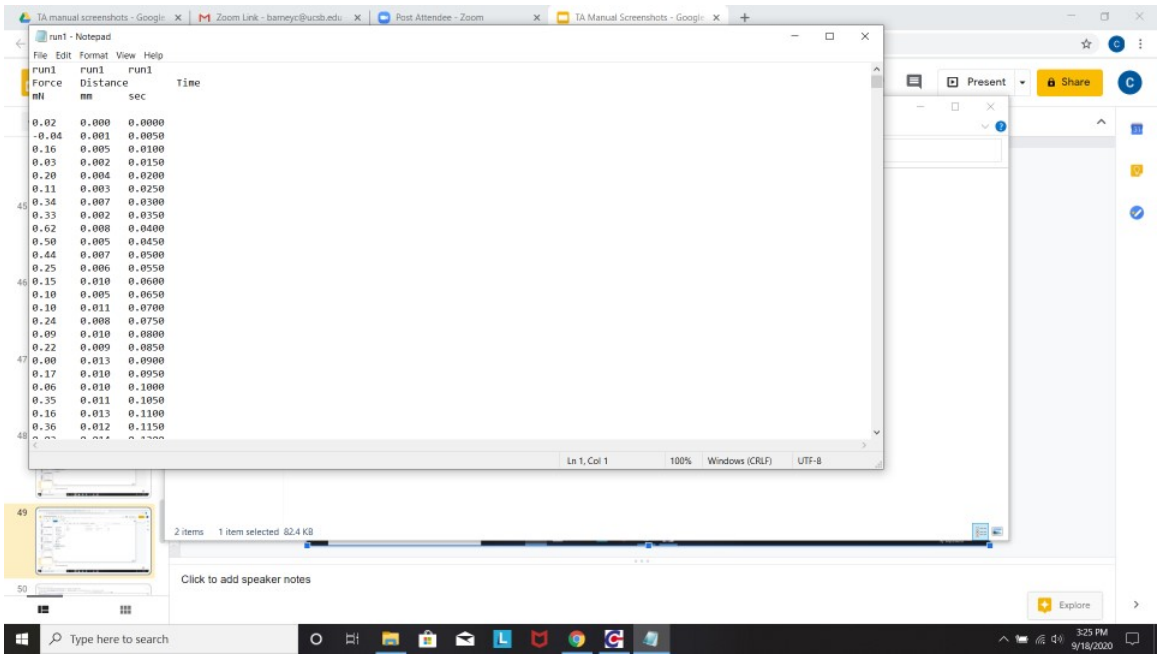


Files are saved as a .tab file.





This is a type of text file and can be opened in notepad. Note that the TA outputs time, distance, and force. Distance is really displacement zeroed to the starting position. The signs on force and displacement vary depending on whether or not the test is run in tension or compression.



2.9 Unload the Sample

Unload the sample making sure to wipe down any oils or solvents that came from the sample. Note that the TA is located in communal lab and is likely not the lab where you made your sample. This lab operates under the idea that you bring everything you need to use (gloves, razor blades, wipes, samples...) and take everything you brought when leaving. **You should not dispose of your samples in this lab.** Instead you should collect them and take the waste back to your lab where you can dispose of them through the proper procedures. 70% IPA and hand sanitizer will be available when cleaning the instrument.

2.10 Clean the TA and Remove Fixtures

Spray the instrument, fixtures, and surrounding area down with 70% IPA solution and wipe everything down regardless of the samples you were testing. This keeps users safe during covid times and makes sure that harmful residue is not left on the instrument.

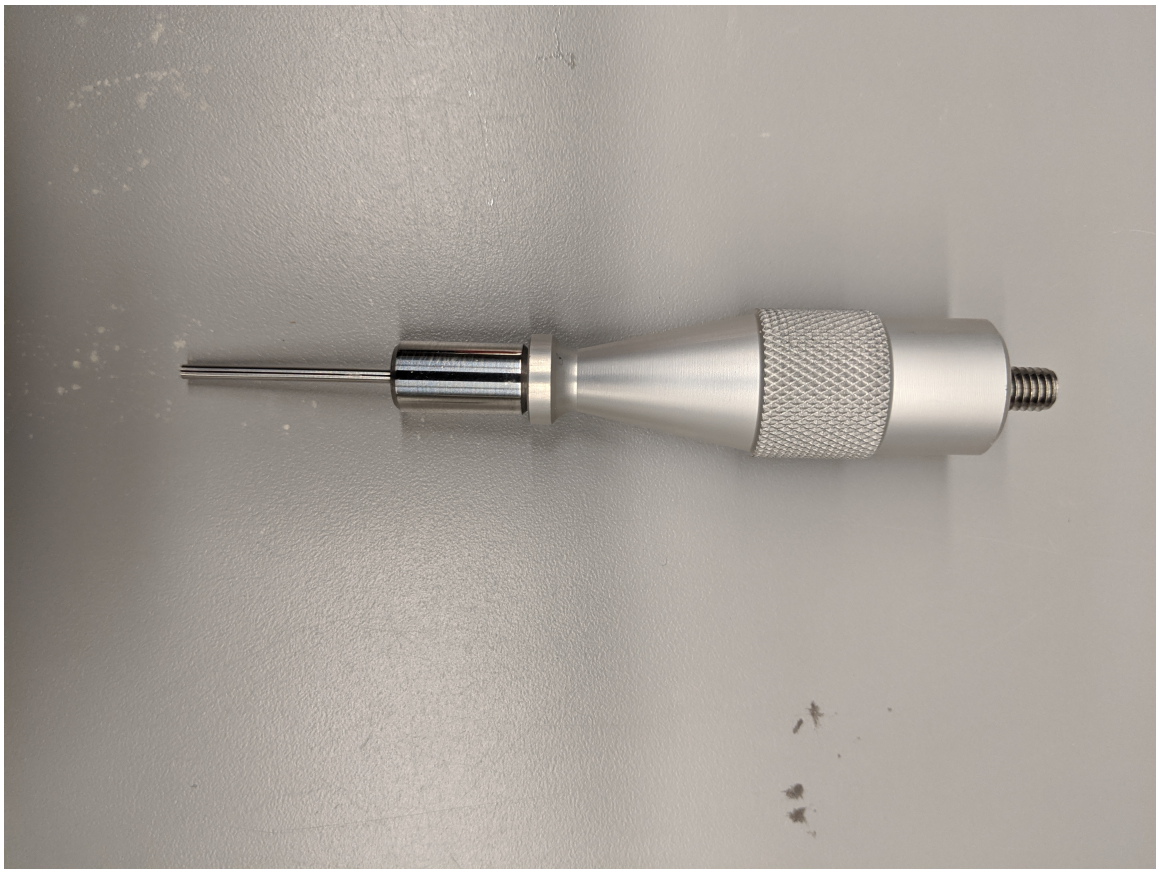
3 Example Tests on the TA

3.1 Indentation

3.1.1 Materials and Methods

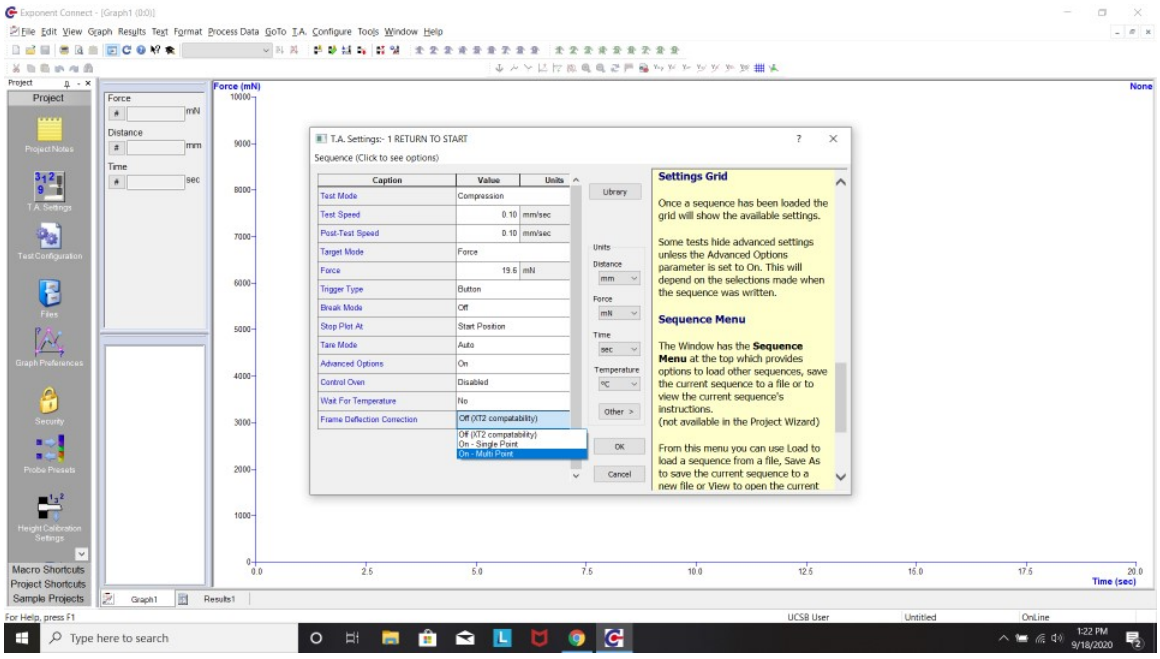
Indentation tests were performed on a poly(dimethylsiloxane) (PDMS) elastomer. The PDMS was prepared by mixing the prepolymer:curing agent from a Sylgard 184 kit together at a ratio of 30:1. The PDMS was then mixed and degassed before curing for 1 hour at 70°C.

A 2 mm diameter flat cylidrical probe (shown below) was indented and retracted from the sample at a rate of 0.1 mm/s. 20 mN was used as a turnaround force and no dwell time was imposed. The starting position of the probe was 5.000 mm above the base of the TA and the sample was placed on a 1 mm thick glass slide resulting in an effective start height of 4.000 mm. First contact with the sample occured at a displacement 0.705 mm giving a sample height of 3.295 mm.



3.1.2 Experimental Sequence

Indentation tests are typically run using a return to start sequence.



The parameters above are fairly typical. Stiffer materials often require a larger turnaround force. Partially due to the steepness of the slope, but also because misalignment between the probe and

the substrate can lead to an initially nonlinear slope as the contact area increases. A common variation of this sequence is the hold until time sequence which works like a return to start test, but builds in a dwell time.

3.1.3 Data and Analysis

The raw data gathered during the experiment is shown in Figure 1 and consists of the imposed displacement and measured force with respect to time. As discussed in Section A.2, we will use the force-displacement profile to measure both E and G_c.

E is measured using the stiffness $1/C$ in the linear loading/unloading regime on the force displacement curve as well as the probe radius $a = R$ and sample height h according to Equation (7). G_c is estimated using the peak peel force P_{peak} and the radius R of the probe according to Equation (8). Both the stiffness in the linear regime and the peak peel force are shown in Figure 2. Together these values give that $E = 27.5$ kPa and $G_c = 1.2$ N/m.

3.1.4 Matlab Code

A big advantage of doing small strain indentation to quantify the modulus is that the test itself only takes about 1 minute to run. This means you can gather data fairly quickly. For example, a person familiar with the TA can set it up, test 10 samples, and break down the instrument in 15-20 minutes. However, the rest of your hour would be spent analyzing the data which limits the ability to make in-lab decisions. For example, you could screen with indentation to determine whether or not your solution gelled homogeneously or if your reactants went bad by comparing your stiffness to a previously measure value. If you have volatile solvents in your system you may have to screen quickly to avoid solvent loss. To do this, I developed some MatLab code. This code takes the analysis presented in the previous section and automates it.

A few preliminaries on this code.

1. To stop a runaway matlab code hit "ctrl c"
2. There are some slight variations between how you can call files in windows vs Mac. Everything I've done is windows compatible, but will need modified if you use a Mac.
3. To use a GUI in matlab you need to take both the .m file and the .fig file and put them into the same folder. The first time that you want to use the gui you need to open the .m file in the editor and hit run from there. For every time after you will be able to just type it into the command window.

   When you open the code it will look like this and need a number of inputs.

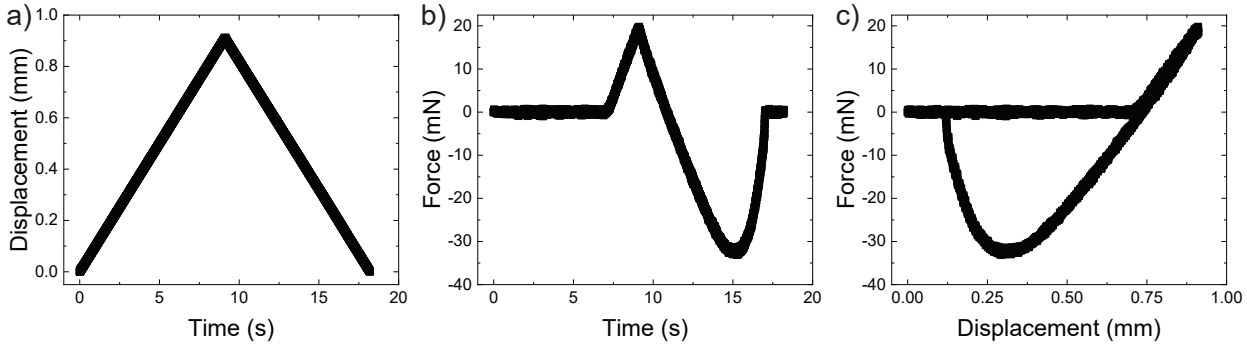


Figure 1: Plots of the raw data showing a) the imposed displacement vs. time profile, b) the measured force vs time profile, and c) the force vs displacement profile. Note that positive force is compressive and negative force is tensile.

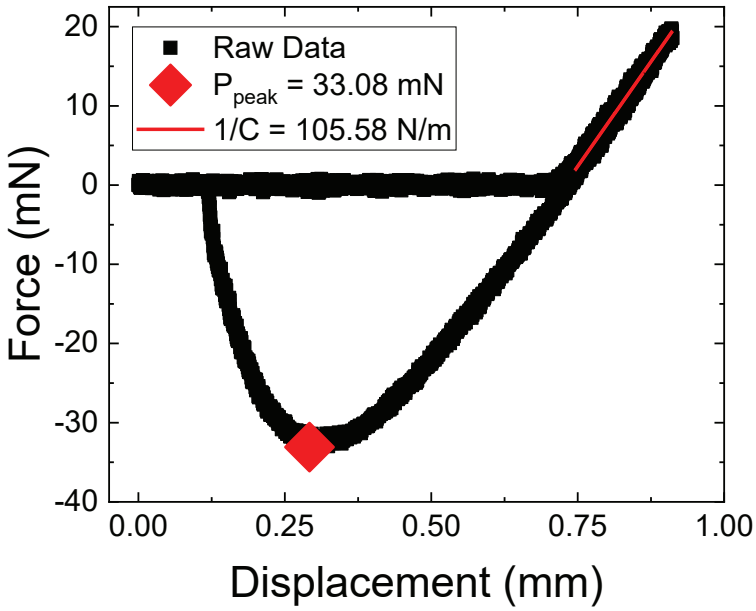
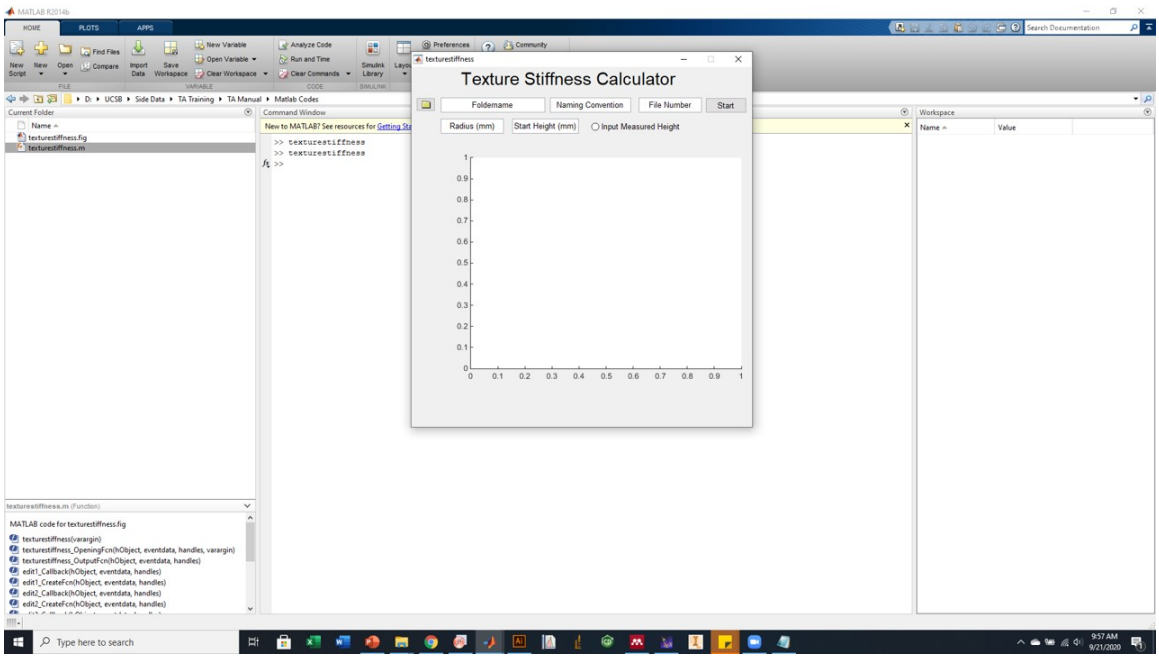
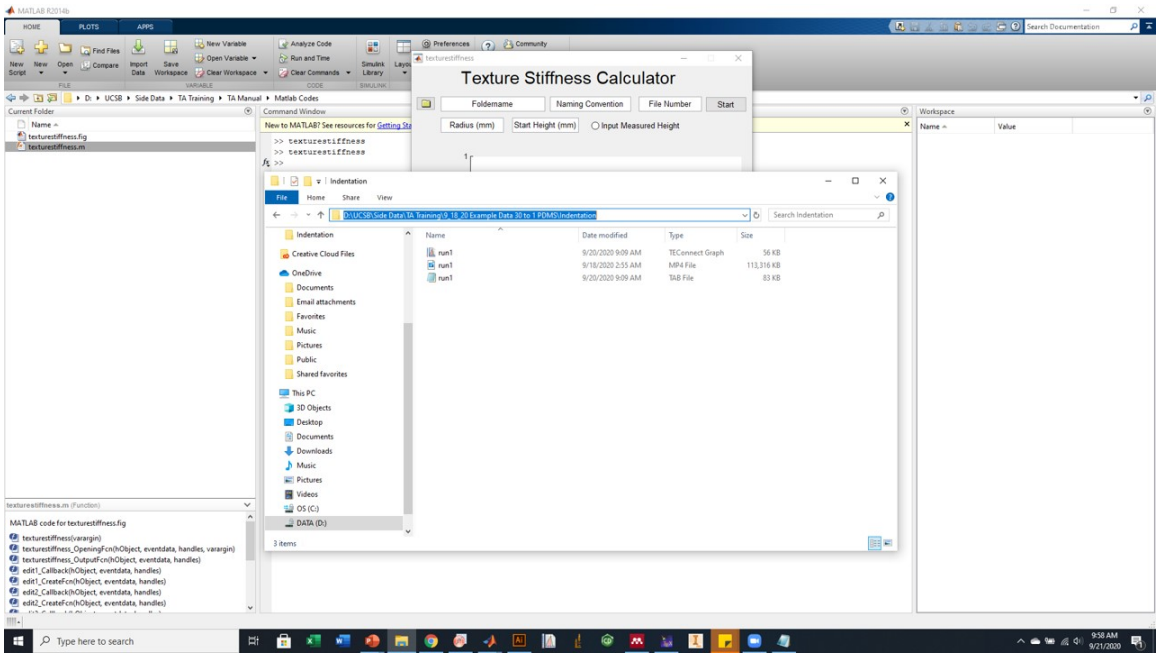


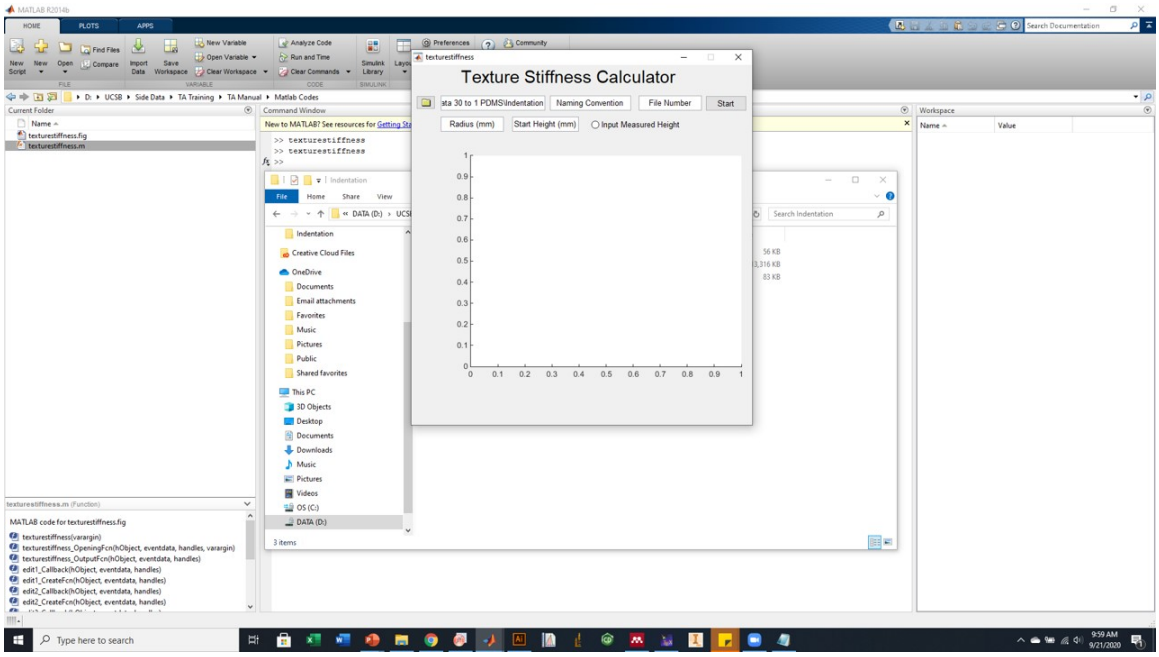
Figure 2: Plots of force vs displacement where both the peak peel force and stiffness have been measured.



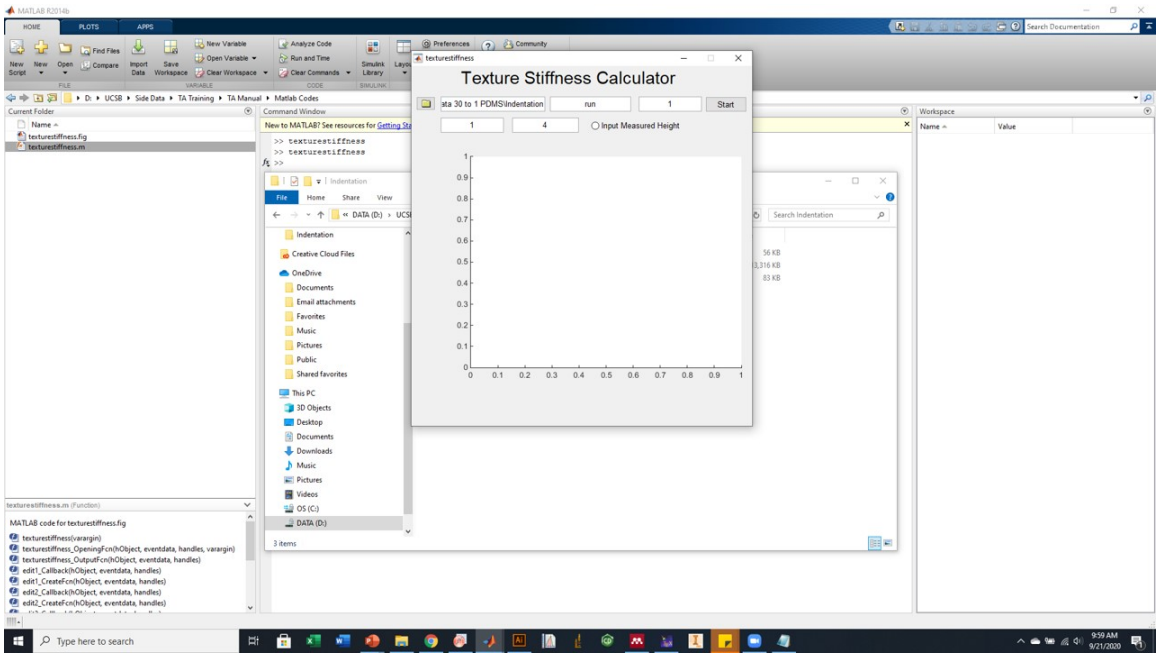
First you will need to enter the foldername where the data is stored.



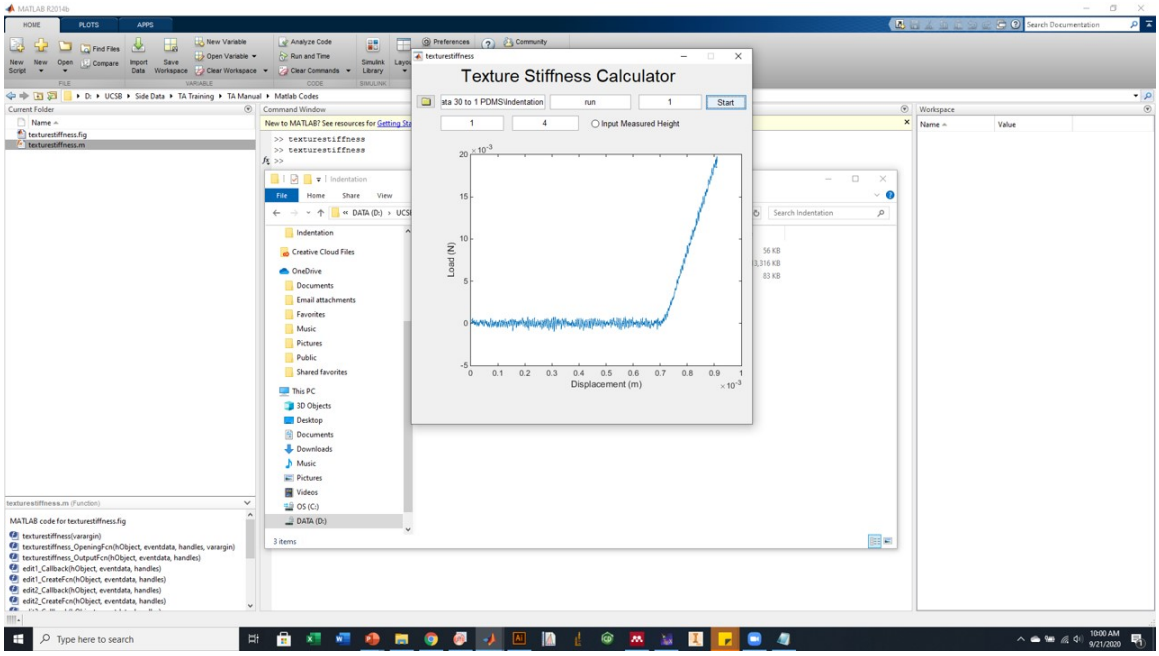
This can be copied and pasted from file explorer or selected using the folder button.



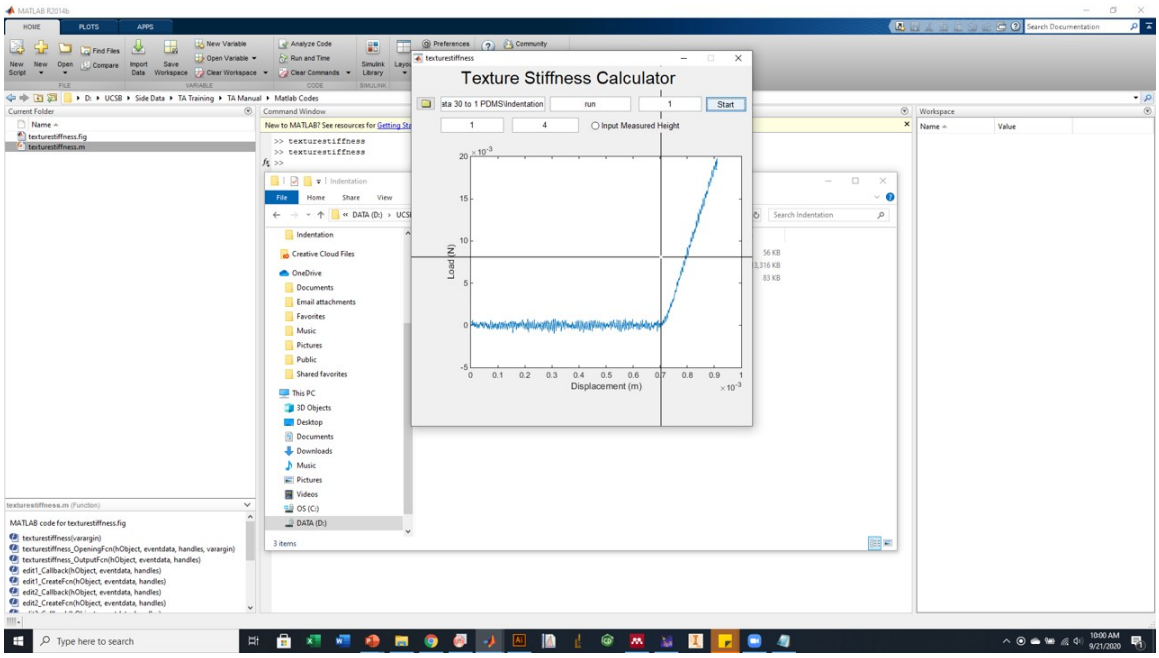
Then you will need to fill in the other inputs. Be careful that your units match those specified within the prompt. Note here that we only have one file so I only entered one file number. If we had multiple files I would enter them into the prompt separate by spaces (i.e. "1 2 5 7 10") and the code would loop and let you analyze multiple files in one go. Also note that we inputted a probe start height instead of a measure sampled height. This means that the sample height will be inferred from the displacement at which contact is made.



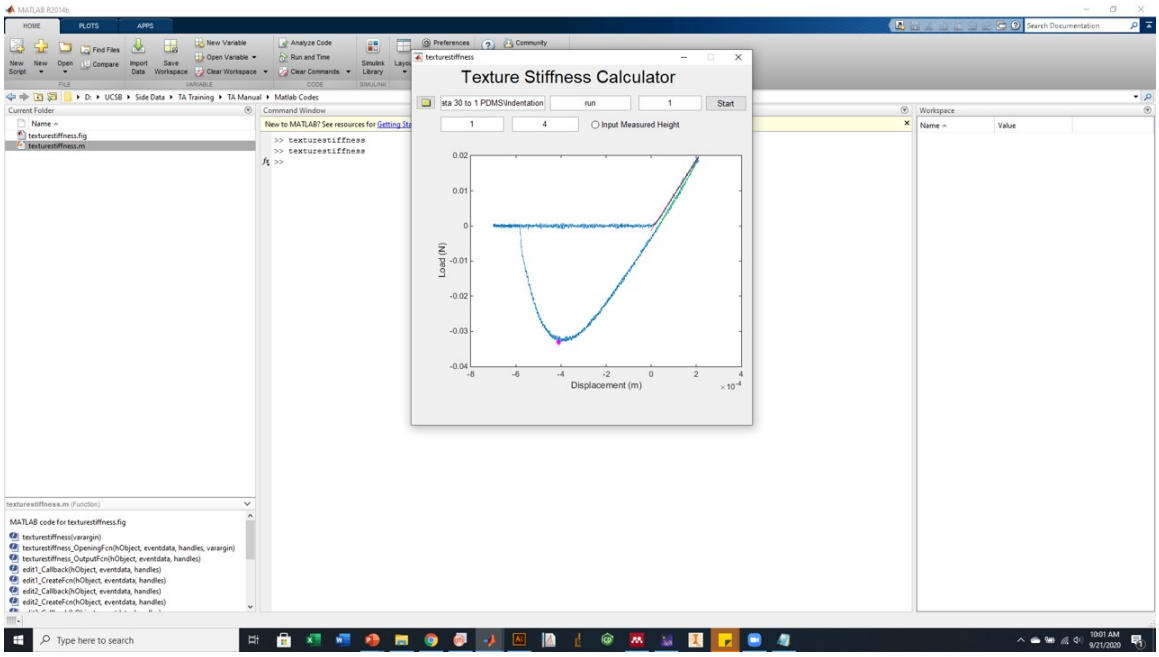
Hit start.



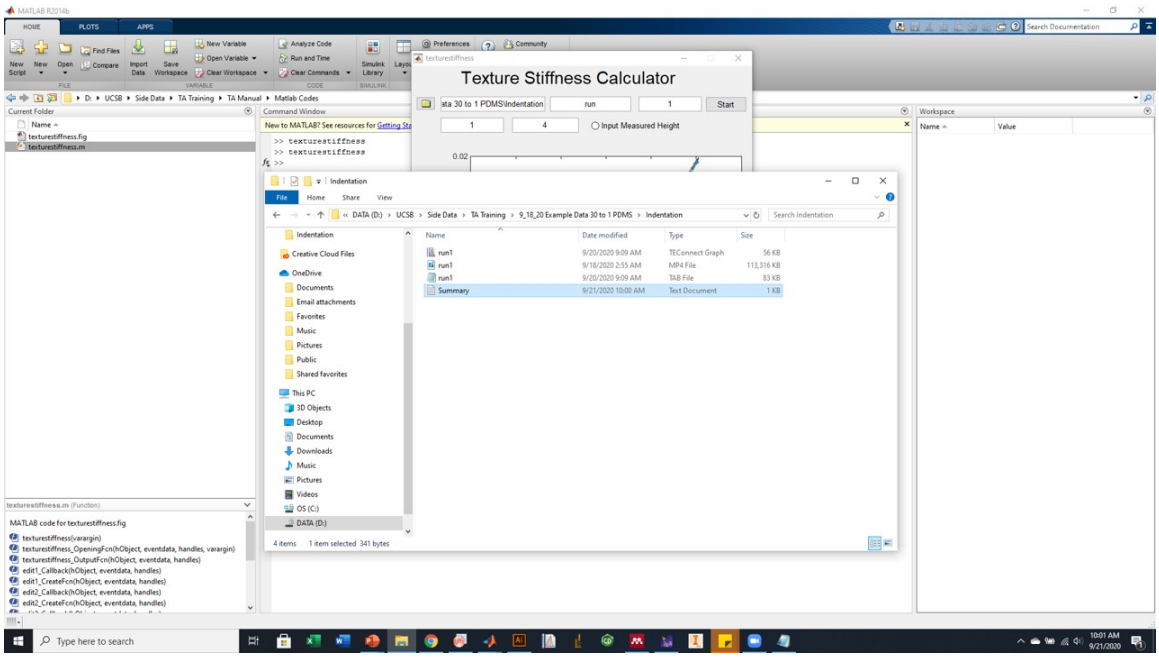
The loading curve will be shown. The code will then wait for you to identify the point at which contact with the sample is made. The x value for your cursor is used, but the y value is not so you can simply move your cursor until the vertical line intersects the point on the curve where contact is made. Note that in the measured starting position mode this value is used to infer the sample height; however, this value is also used to define where to take the slope and so it is important. If you were looking to fully automate the code this is the part that you would want to change.

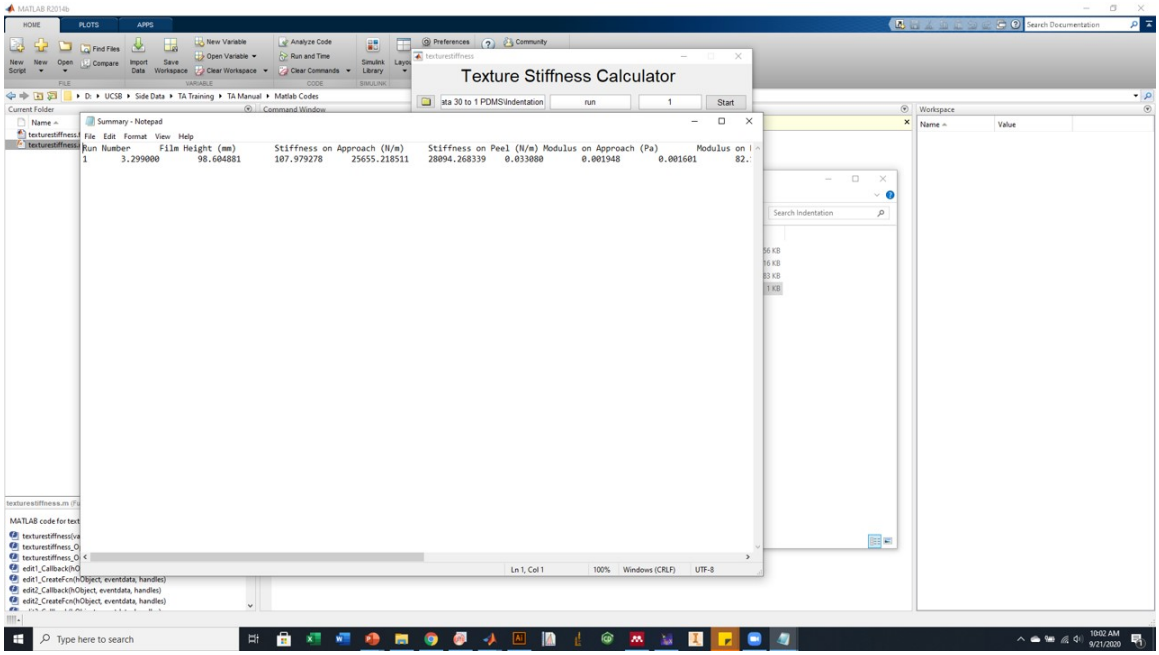


After selecting this value the code will show you a plot with the peak peel force and slope of the loading and unloading curves marked.



There will also be a summary text file created in the folder where your data is stored that contains the summary data values. The code estimated $E = 26.9$ kPa and $G_c = 1.2$ N/m which are very close to the by hand values above. The slight difference is most likely due to a slightly different inferred height of 3.299 mm instead of the 3.295 mm used in the by hand data.



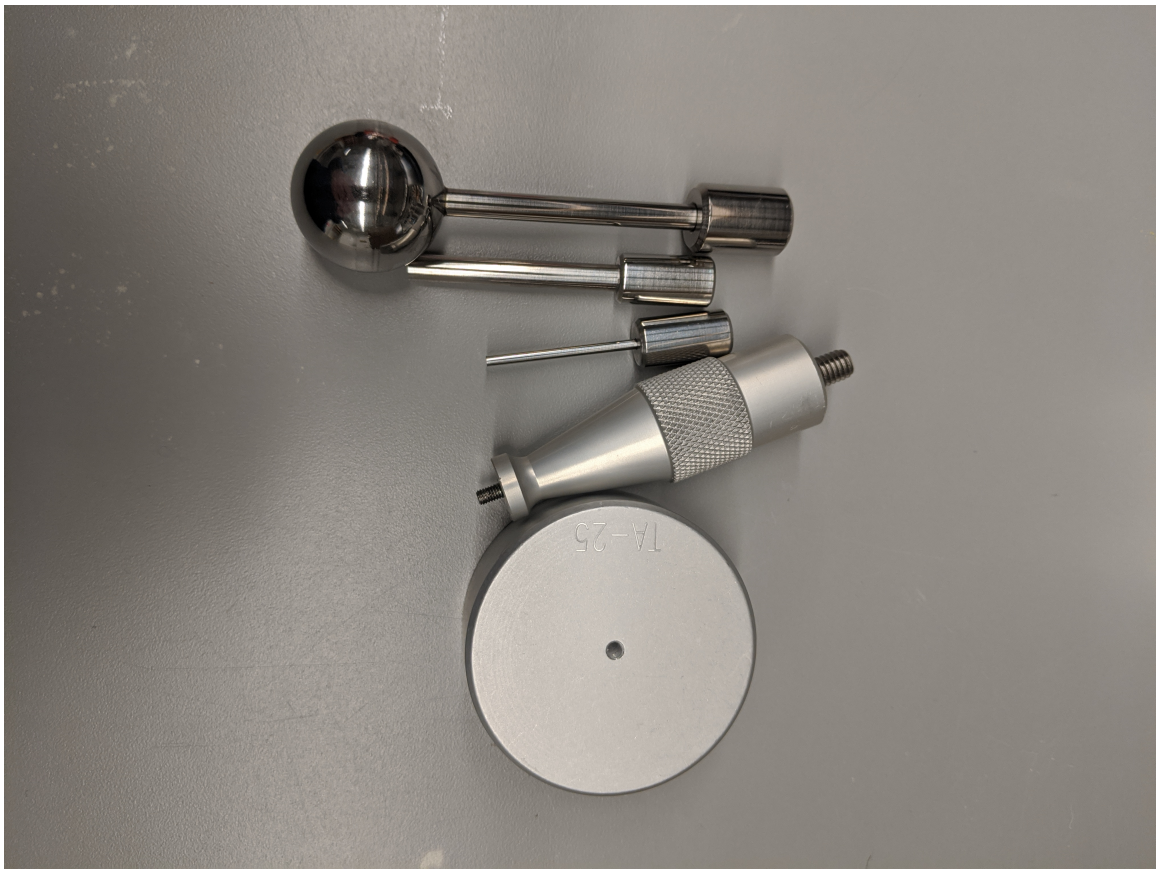


3.2 Uniaxial Compression

3.2.1 Materials and Methods

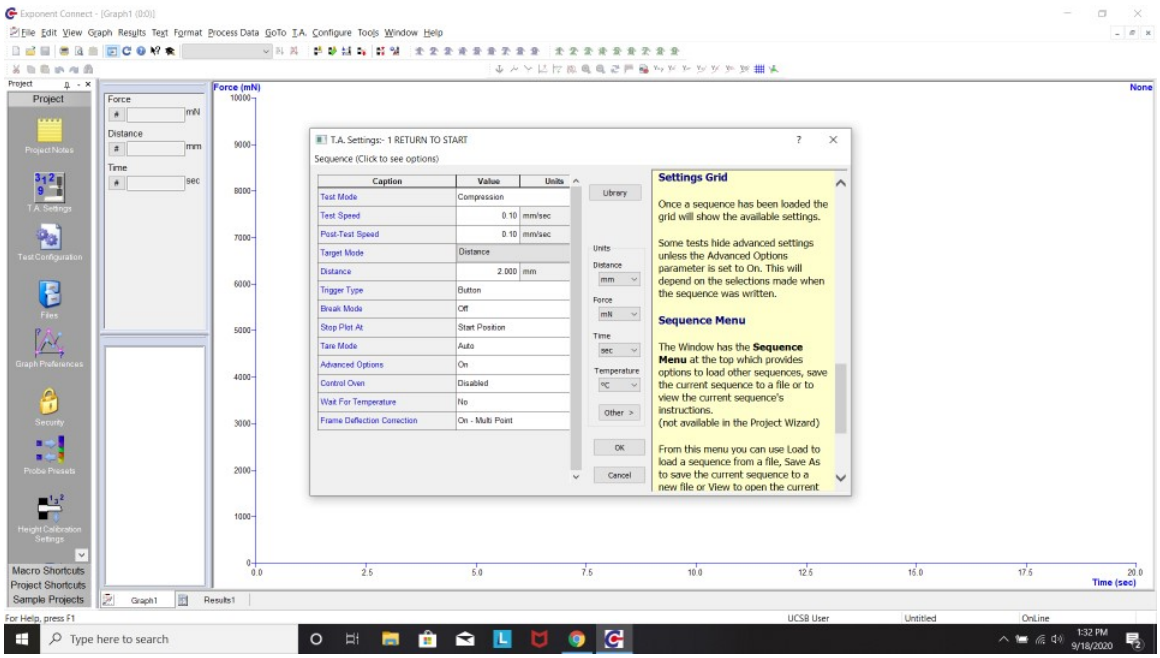
Uniaxial compression was performed on a poly(dimethylsiloxane) (PDMS) elastomer. The PDMS was prepared by mixing the prepolymer:curing agent from a Sylgard 184 kit together at a ratio of 30:1. The PDMS was then mixed and degassed before curing for 1 hour at 70°C.

Compression was applied to a 16.6 mm diameter sample that was 3.295 mm thick. Both plates were lubricated with 100 cSt linear PDMS chains. This test used the 50 mm diameter aluminum cylinder shown below.



3.2.2 Experimental Sequence

An example sequence is shown below. It uses the return to start sequence and incorporates the frame deflection correction. Note that you should be extra careful when doing uniaxial compression both because the sample can generate high forces and because getting the turnaround condition right is crucial. In the example sequence distance is used as the turnaround condition. **You should only do this if you have calibrated the probe height and have used the move probe button to be sure that your initial gap height is greater than your turnaround travel distance.**



3.2.3 Data and Analysis

The raw data from this test is shown in Figure 3. It consists of the imposed displacement and measured force with respect to time. To measure E, the force data is normalized by the initial area to calculate the engineering (nominal) stress and displacement is zeroed at the contact point and normalized to calculate compressive strain ε_c as well as the stretch ratio λ. Note that some of the force picked up near the beginning and end of contact is from the capillary bridge formed by the lubricant.

Plots of the normalized data is shown in Figure 4. Note that positive stress on these graphs is compression and negative stress is tension. This is opposite the sign convention used in Section B.2 so the slopes on plot b) and c) are $-E$ and $-\mu$ respectively. Note also that the axis from c) is that derived in Equation (21).

If we consider the loading curve for each style of normalized plot, we can fit to estimate E where plots a-b) give $E = 26.05$ kPa when fitted between 0-10% strain and plot c) gives $E = 27.15$ kPa. Note that this is in excellent agreement with the estimate of $E = 26.9$ kPa from the indentation measurements in the previous section. Also note that converting to c) enables us to use a larger fraction of the data gathered to fit for modulus. This can be useful because small errors in things

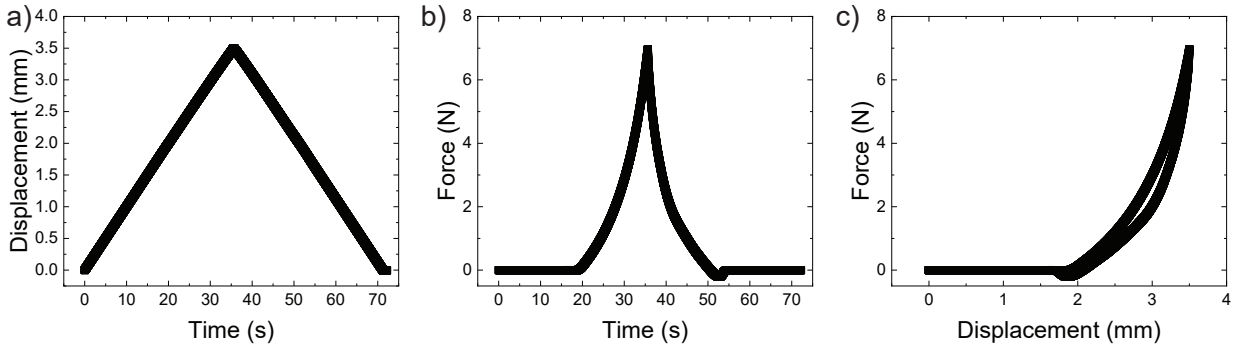


Figure 3: Plots of the raw data showing a) the imposed displacement vs. time profile, b) the measured force vs time profile, and c) the force vs displacement profile. Note that positive force is compressive and negative force is tensile.

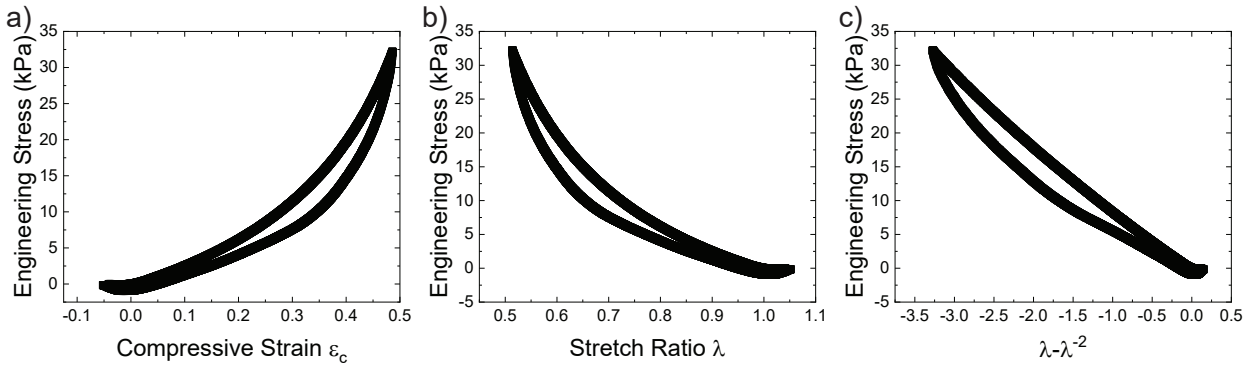


Figure 4: Plots of the engineering stress against a) ε_c, b) λ, and c) $\lambda - \lambda^{-2}$.

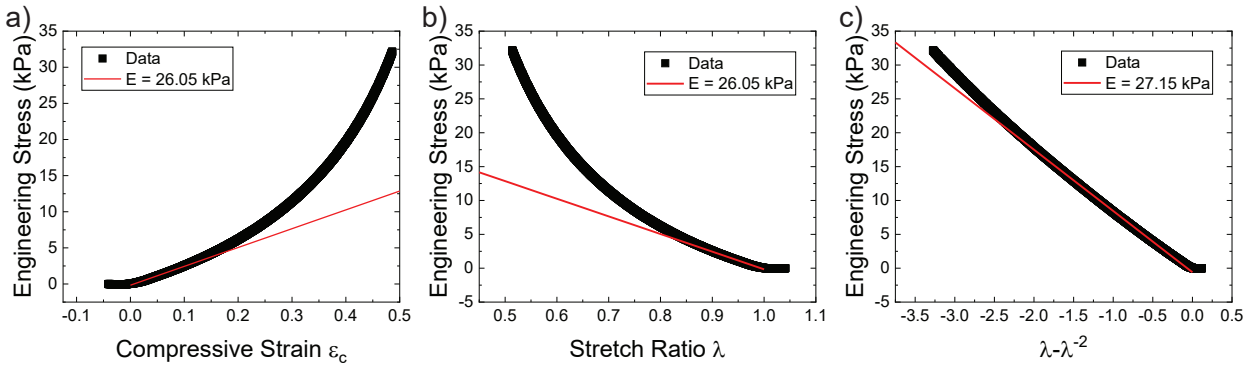


Figure 5: Plots of the loading engineering stress against a) ε_c, b) λ, and c) $\lambda - \lambda^{-2}$ where fits have been performed to measure E.

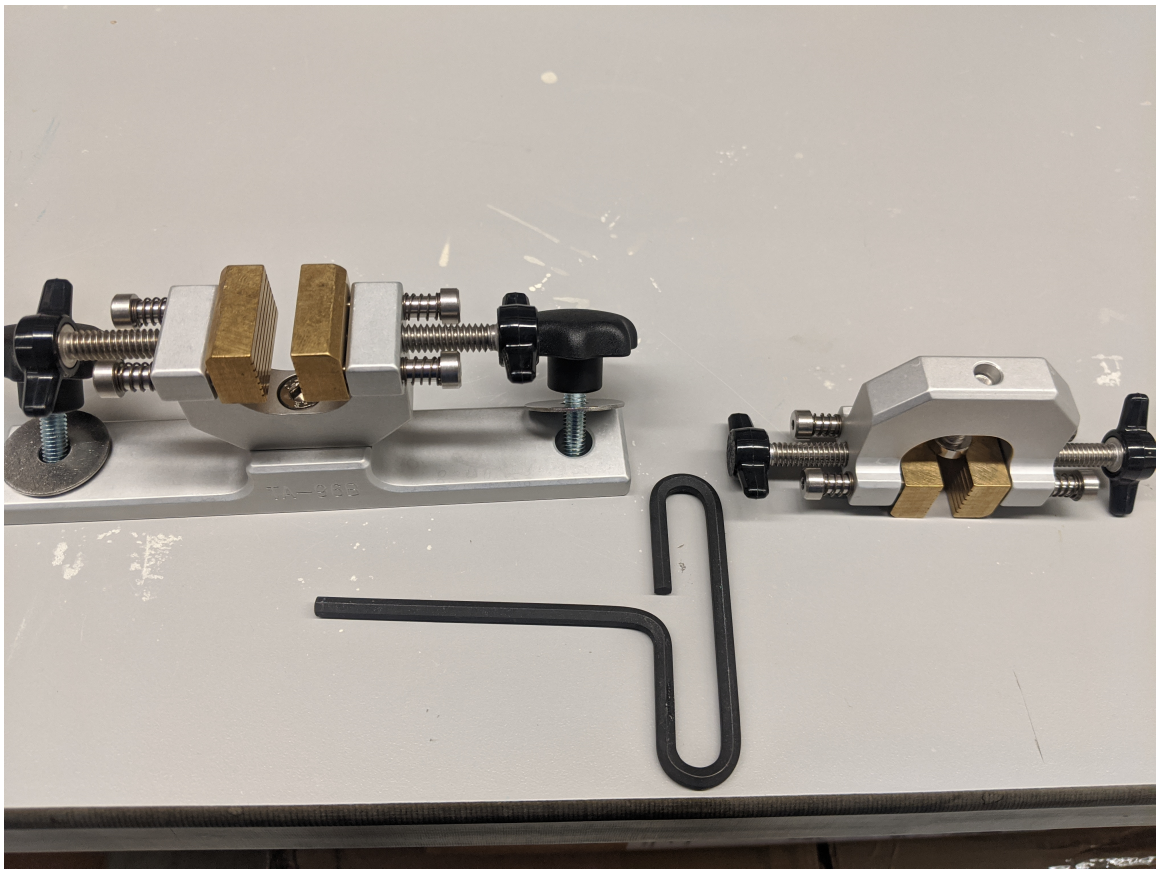
like the contact point can lead to significant error in strains in the small strain regime.

3.3 Uniaxial Extension

3.3.1 Materials and Methods

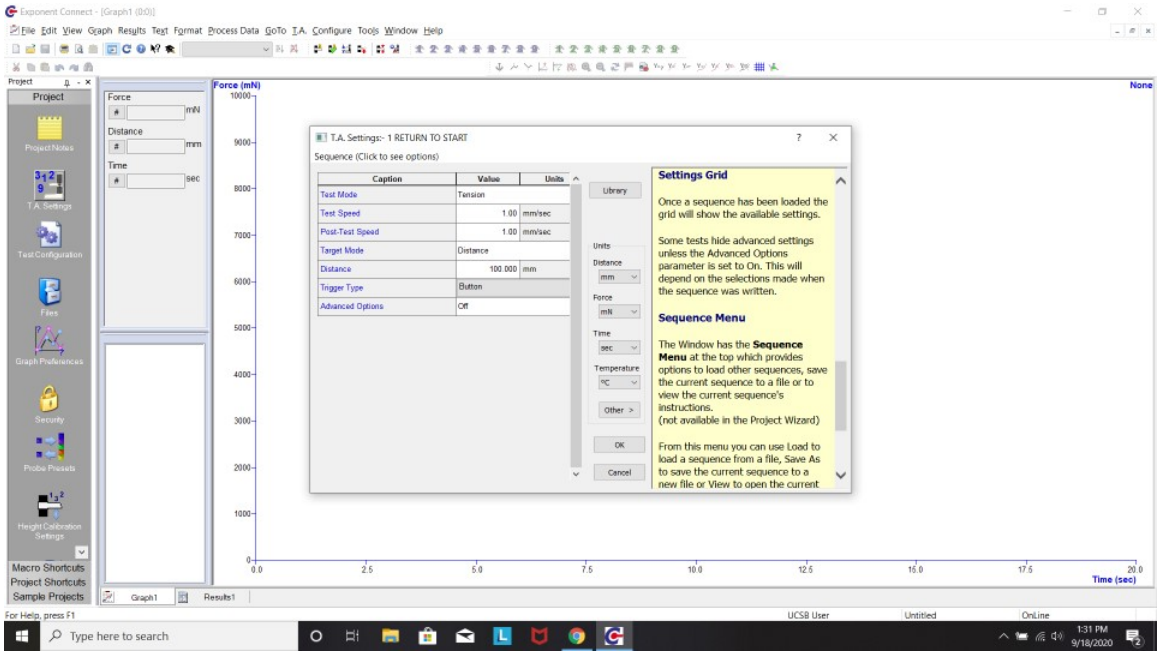
Uniaxial extension was performed on a poly(dimethylsiloxane) (PDMS) elastomer. The PDMS was prepared by mixing the prepolymer:curing agent from a Sylgard 184 kit together at a ratio of 30:1. The PDMS was then mixed and degassed before curing for 1 hour at 70°C.

A rectangular sample with a 3.295 mm thickness, 13.25 mm width, and 45.485 mm height was used for testing. The sample was pulled at a speed of 1 mm/s and the test was stopped by using the stop test button on the TA. The test ended in slip out of the clamp and not rupture of the sample. This test used the mini tensile clamps shown below.



3.3.2 Experimental Sequence

An experimental sequence for uniaxial extension is shown below. It uses the return to start sequence and set the turnaround distance to a large distance of 100 mm. This is intentional as the sample will most likely fail before this distance and so the test can be stopped using the stop test button on the TA.



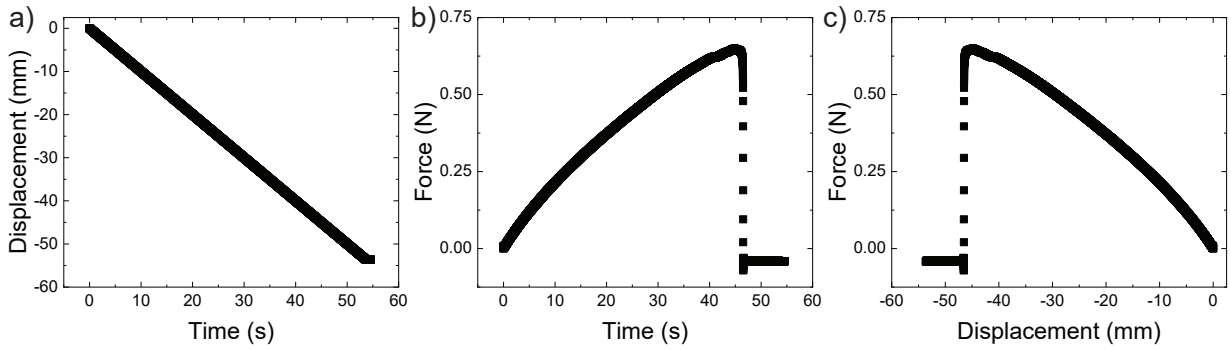


Figure 6: Plots of the raw data showing a) the imposed displacement vs. time profile, b) the measured force vs time profile, and c) the force vs displacement profile. Note that positive force is compressive and negative force is tensile.

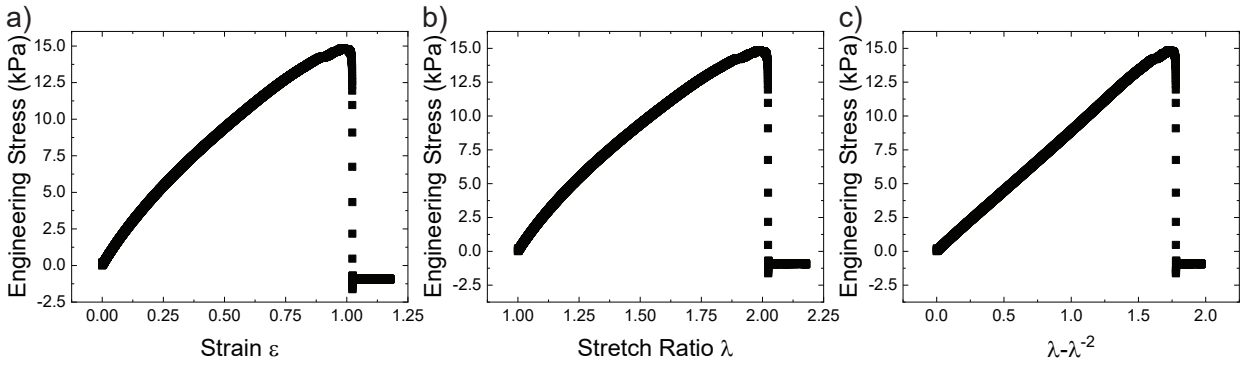


Figure 7: Plots of the engineering stress against a) ε, b) λ, and c) $\lambda - \lambda^{-2}$.

3.3.3 Data and Analysis

The raw data from this test is shown in Figure 6. It consists of the imposed displacement and measured force with respect to time. To measure E, the force data is normalized by the initial area to calculate the engineering (nominal) stress and displacement is normalized to calculate strain ε as well as the stretch ratio λ.

Plots of the normalized data is shown in Figure 4. Note that positive stress on these graphs is tension. Also not that the sign on the displacement from the raw data has been flipped to meet the common convention of tension is positive, compression is negative. Note that the axis from c) is that derived in Equation (21).

If we consider the loading curve for each style of normalized plot, we can fit to estimate E where plots a-b) give $E = 25.70$ kPa when fitted between 0-5% strain and plot c) gives $E = 26.4$ kPa. Note that this is in excellent agreement with the estimate of $E = 27.15$ kPa and $E = 26.9$ kPa from the uniaxial compression and indentation measurements, respectively, in the previous sections. Also note that converting to c) enables us to use a larger fraction of the data gathered to fit for modulus. This can be useful because small errors introduced by slack in the system can lead to significant error in strains in the small strain regime.

When the uniaxial compression data from the previous section is plotted on the same axis as the uniaxial extension data (Figure 9), it becomes apparent the this sylgard sample is largely neo-Hookean over the range tested. It appears that there might be some slight stiffening in the compressive regime, but this could also be due to traction at the interface.

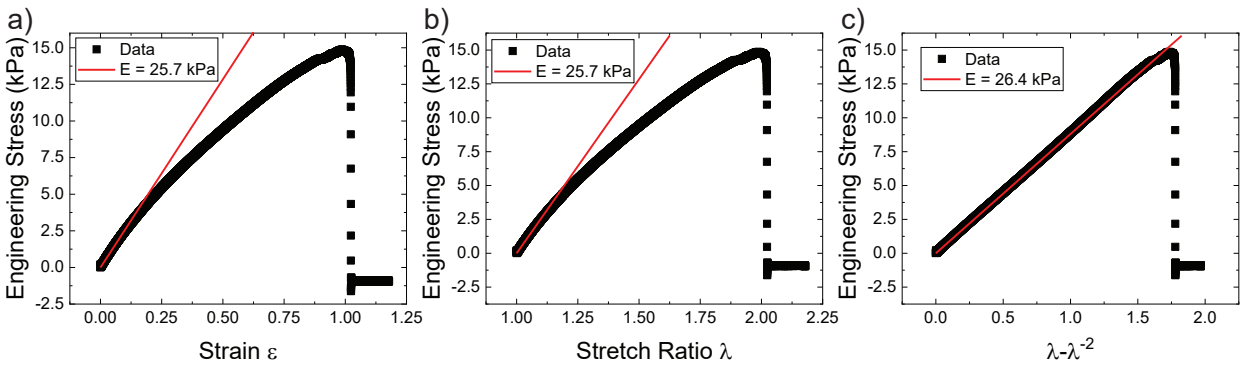


Figure 8: Plots of the loading engineering stress against a) ε_c, b) λ, and c) $\lambda - \lambda^{-2}$ where fits have been performed to measure E.

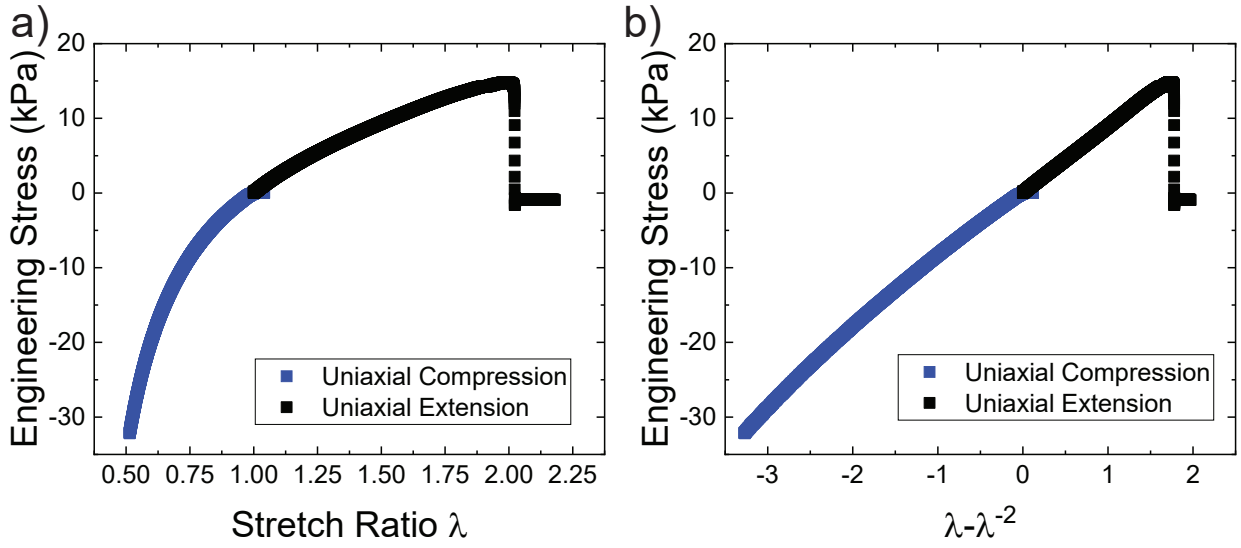


Figure 9: Plots of the loading engineering stress against a) λ and b) $\lambda - \lambda^{-2}$ that largely show a neo-Hookean response.

3.4 Cyclic Extension

3.4.1 Materials and Methods

3.4.2 Experimental Sequence

3.4.3 Data and Analysis

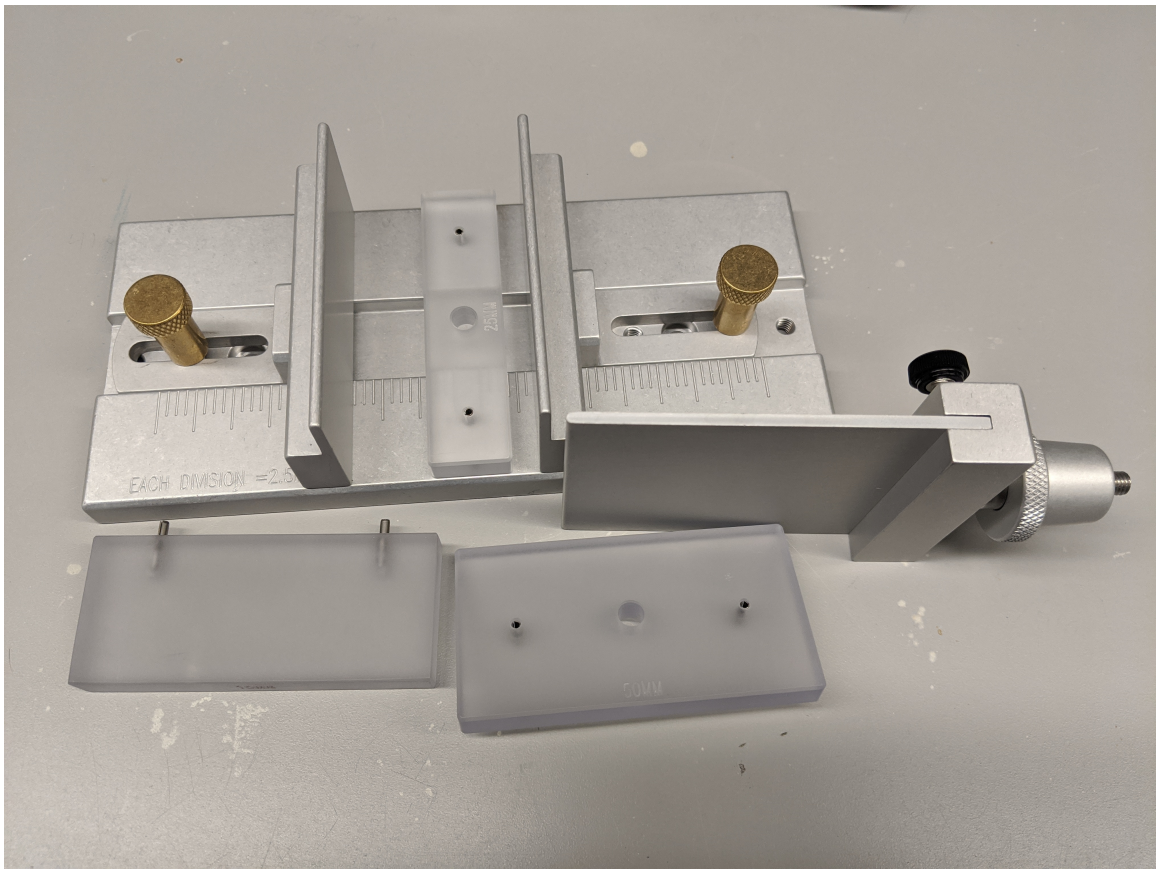
3.5 Pure Shear

3.6 Peel Test

3.7 3 Point Bend Test

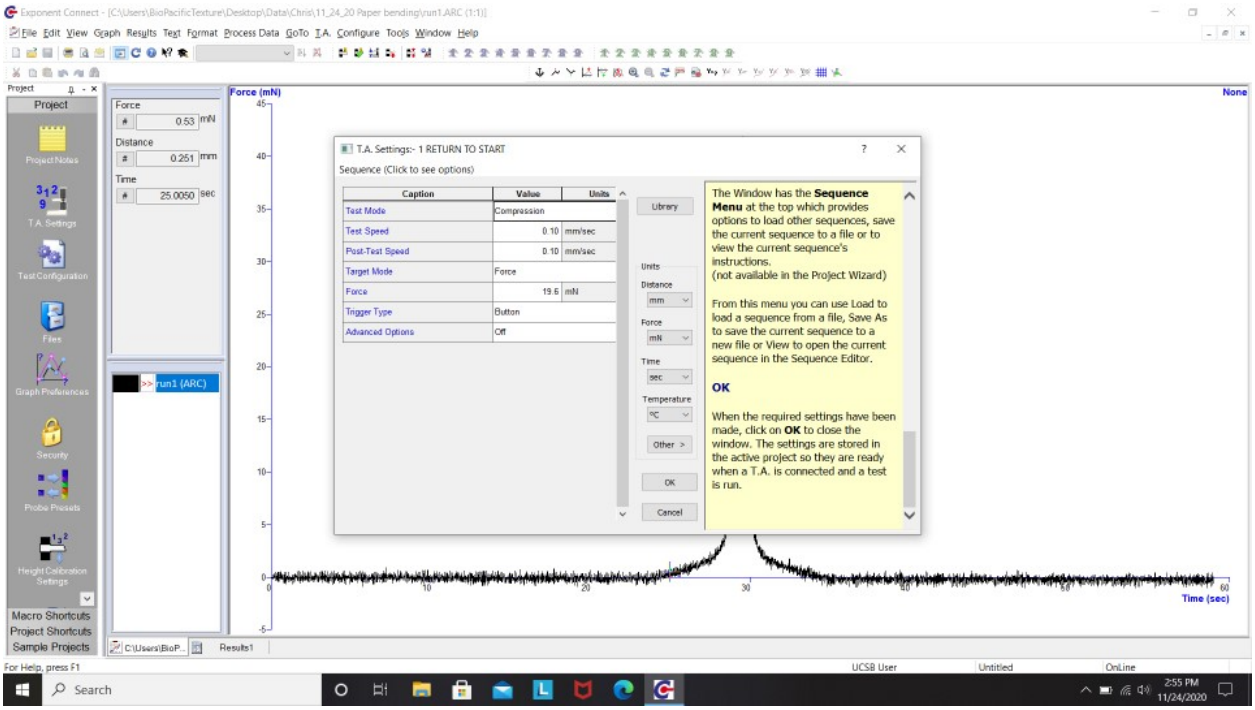
3.7.1 Materials and Methods

3 point bending was performed with on a piece of paper that I found sitting around in lab. It was 15 mm in length, 5 mm in width, and 0.1 mm in thickness. The sample was loaded at a rate of 0.1 mm/s to a turnaround force of 20 mN using the 3 point bend test rig shown below.



3.7.2 Experimental Sequence

An experimental sequence for 3 point bending is shown below. It uses the return to start sequence with a turnaround force of 20 mN at a loading and unloading rate of 0.1 mm/s.



3.7.3 Data and Analysis

The raw data gathered during the experiment is shown in Figure 10 and consists of the imposed displacement and measured force with respect to time. As discussed in Section A.2, we will use the force-displacement profile to measure E.

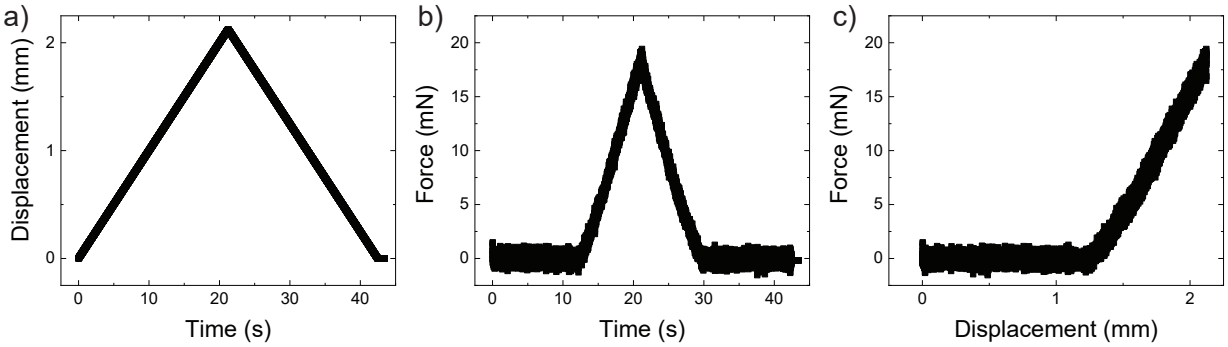


Figure 10: Plots of the raw data showing a) the imposed displacement vs. time profile, b) the measured force vs time profile, and c) the force vs displacement profile.

E is measured using the stiffness K in the linear loading/unloading regime on the force displacement curve as well as the sample dimensions as shown in Equation (1) where L is beam length, I is second moment of inertia, b is beam width, and h is beam thickness.

$$E = \frac{KL^3}{48I} = \frac{KL^3}{4bh^3} \tag{1}$$

The stiffness in the linear regime is shown in Figure 11. This measurement shows that $E = 3.9$ GPa.

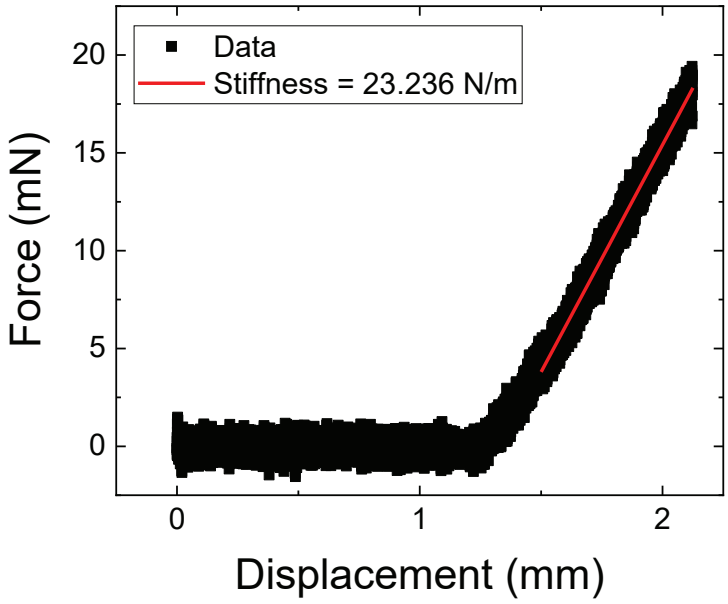
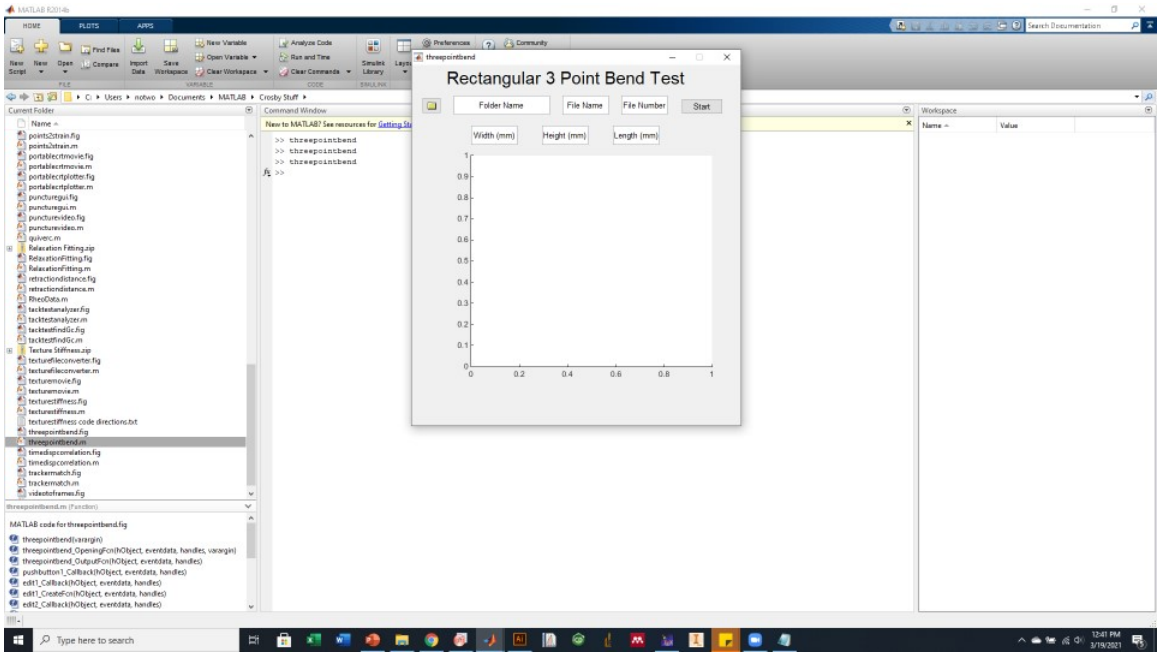


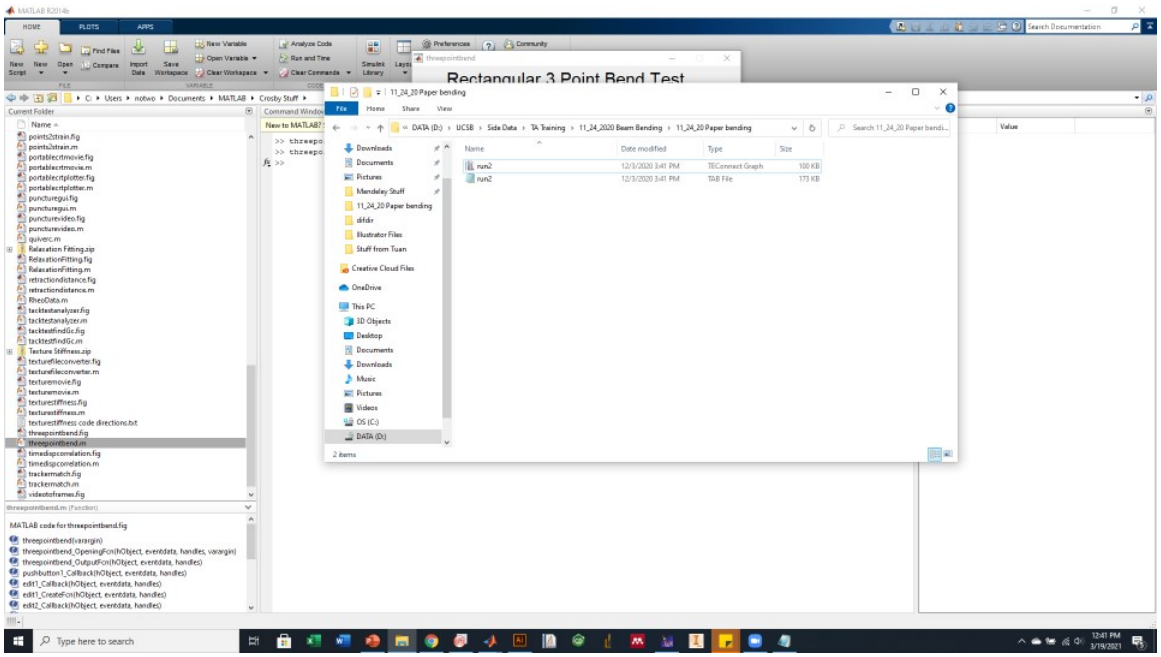
Figure 11: Plots of force vs displacement where the stiffness has been measured.

3.7.4 Matlab Code

There is also some Matlab code designed for beam bending. It assumes that you have a simply supported rectangular beam and are applying force at the center of the beam. Preliminaries on Matlab codes are covered in the indentation section. When you open the code it will look like this and need a number of inputs.

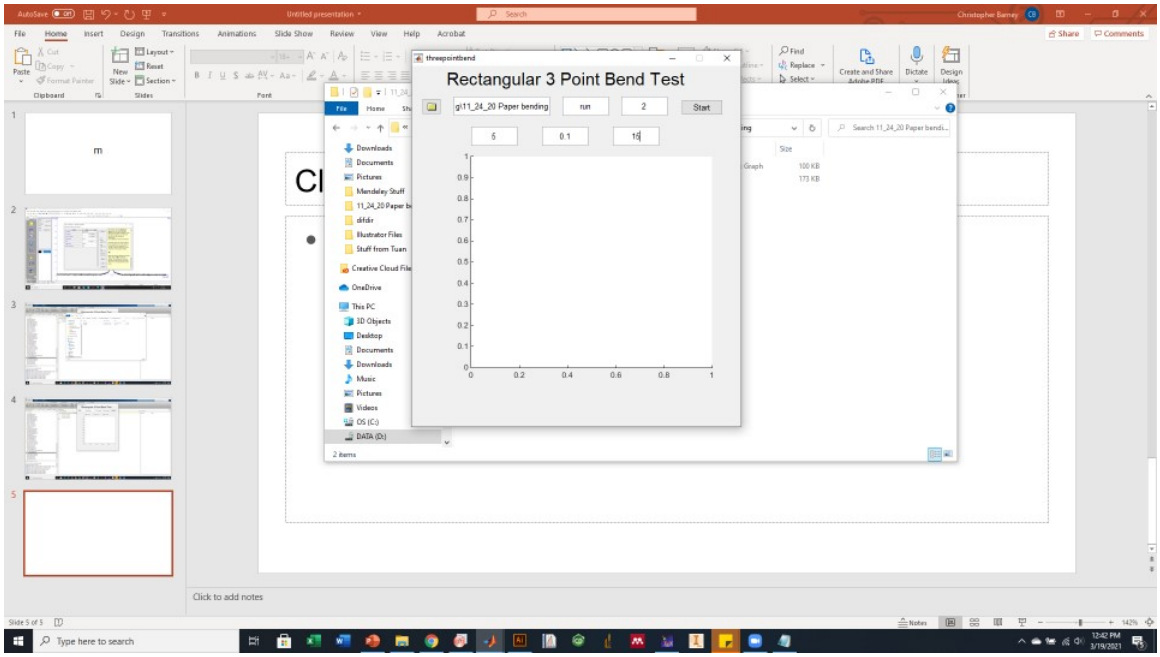


First you will need to enter the foldername where the data is stored.

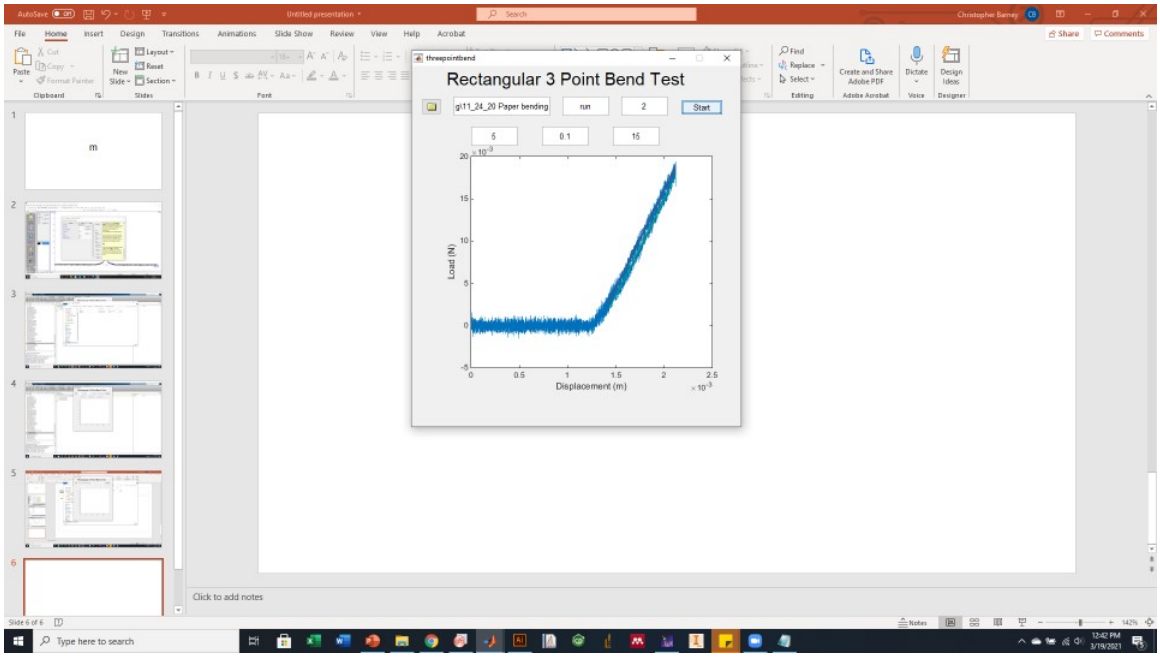


This can be copied and pasted from file explorer or selected using the folder button. Then you will need to fill in the other inputs. Be careful that your units match those specified within the prompt. Note here that we only have one file so I only entered one file number. If we had multiple

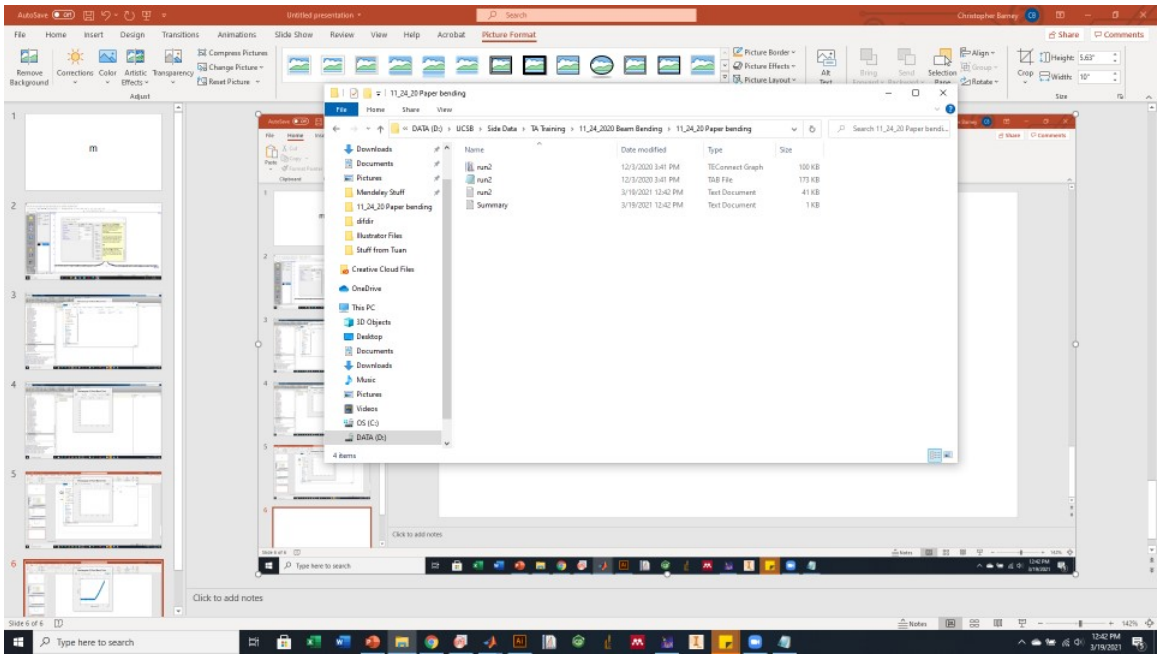
files I would enter them into the prompt separate by spaces (i.e. "1 2 5 7 10") and the code would loop and let you analyze multiple files in one go.

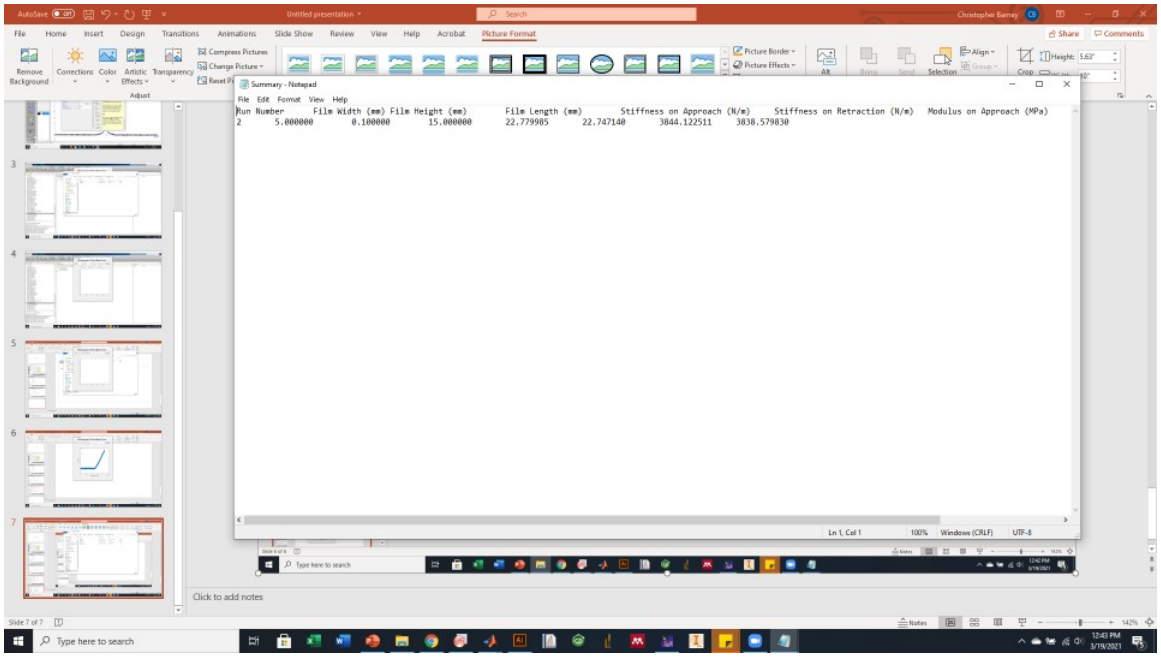


Hit start.



Two files were created in your folder. The summary text file contains the summary of the properties extracted from the data. Note that the value of $E = 3.8$ GPa is in good agreement with the value of $E = 3.9$ GPa when the data was analyzed by hand.





3.8 Sliding Friction

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[1] K. L. Johnson, *Contact Mechanics*. Cambridge University Press, 1985.

[2] K. R. Shull, D. Ahn, W.-L. Chen, C. M. Flanigan, and A. J. Crosby, "Axisymmetric adhesion tests of soft materials," *Macromolecular Chemistry and Physics*, vol. 199, no. 4, pp. 489–511, 1998.

[3] K. R. Shull, *Contact mechanics and the adhesion of soft solids*, vol. 36. 2002.

[4] H. Hertz, *Miscellaneous Papers*. Macmillan and Co., LTD., 1896.

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A Contact Mechanics

This section gives a crude/functional introduction to contact mechanics. For a more complete discussion, I recommend you find a copy of *Contact Mechanics* by K.L. Johnson [1] or one of the reviews from Ken Shull. [2, 3]

A.1 Hertzian Contact

The geometry considered in this section consists of a sphere of radius R indented on a flat substrate of thickness h by an imposed displacement δ. The imposed displacement results in a force P (P, not F, is used to represent force for historical reasons) and contact radius a. Note that this sphere on plane geometry is equivalent to contact between two cylinders of equal radii crossed at an angle of 90^o. [1] Heinrich Hertz first solved the elastic problem of how to relate P, δ, and a through materials properties in the absence of adhesion in the late 1800s. [4]

$$\delta_H = \frac{a^2}{R}, \quad P_H = \frac{4E^* a^3}{3R}, \quad E^* = \frac{E}{1-\nu^2} \tag{2}$$

E^* is the plane strain modulus which is defined in terms of the elastic modulus E and Poisson's ratio ν($\nu = 0.5$ for incompressible materials). Equation (2) can be used to calculate the Hertzian compliance C_H.

$$C_H = \frac{d\delta}{dP} = \frac{d\delta}{da}\frac{da}{dP} = \frac{1}{2E^* a} \tag{3}$$

Note that the relationship between force and displacment for a cylinder indented onto a flat substrate can be calculated from this by assuming a constant contact radius $a = R$ where R is the radius of the cylinder.

$$C_H = \frac{\delta}{P} = \frac{1}{2E^* R} \quad P = 2E^* R\delta \tag{4}$$

While the equations developed by Hertz are powerful, they contain a number of assumptions. Below is a list of the assumptions built into the Hertzian contact equations.

1. Materials are isotropic
2. No adhesion between the two contacting bodies

3. $a << R$, in practice $a < 0.2R$
4. $a << h$
5. No friction at the interface between the contacting bodies
6. Materials are linear elastic

Hertz originally considered contact between glass spheres ($E = 70$ GPa) meaning that the assumption of no adhesion between the bodies was reasonable. [4] However, this assumption is no longer valid when considering softer materials where $E < 10$ MPa. Formulation of the critical strain energy release rate G_c using contact mechanics requires that one separate the energy dissipated through adhesion from that recovered elastically in the system as the contact area changes. Johnson, Kendall, and Roberts (JKR) were the first to solve this problem using an energy balance approach in 1971. [5] Maugis and Barquins later generalized this solution through a fracture mechanics formulation in 1978 by treating the contact line between the probe and substrate as a crack. [6]

$$G = -\frac{(P' - P)^2}{2}\frac{dC}{dA} = -\frac{(P' - P)^2}{4\pi a}\frac{dC}{da} \tag{5}$$

P' is the force needed to establish a given contact area in the absence adhesion (given by Hertzian contact) while P is the observed force. When $P' = 0$, Equation (5) collapses to the classic compliance method formulation. [7] Equation (5) is a general formulation but is still limited by the assumptions of the equations plugged into it(minus the no adhesion assumption).

For clarity, derivations in the following sections will be performed with the classical Hertzian equations presented above. However, in practice modified versions of the classic Hertzian equations are employed. These confinement corrected equations were developed by Shull and coworkers and eliminate the need for Assumptions (4-5) above. [2, 3]

$$\delta' = \delta_H\left(0.4 + 0.6e^{-\left(\frac{1.8a}{h}\right)}\right), \quad P' = P_H\left(1 + \beta\left(\frac{a}{h}\right)^3\right), \quad C' = C_H\left[1 + \frac{4}{3}\left(\frac{a}{h}\right) + \frac{4}{3}\left(\frac{a}{h}\right)^3\right]^{-1} \tag{6}$$

β is a coefficient describing the friction at the interface. When $\beta = 0.15$ there is no friction and when $\beta = 0.33$ there is full friction. For most analyses, it is assumed that $\beta = 0.25$ implying that there is friction at the interface but it is not full friction.

A.2 Contact with a flat cylinder

A.2.1 Determining E

The contact compliance can be measured as the inverse of the slope on a plot of force and displacement in the linear regime. This compliance can be used to calculate E by substituting Equation (4) into Equation (6).

$$E = \frac{3}{8R}\frac{1}{C}\left[1 + \frac{4}{3}\left(\frac{a}{h}\right) + \frac{4}{3}\left(\frac{a}{h}\right)^3\right]^{-1} \tag{7}$$

Here the contact radius a would be equal to the radius of the cylinder R.

A.2.2 Estimating G_c

G_c can be estimated by substituting Equation (3) into Equation (5) and assuming that $P' = 0$ and $a = R$.

$$G_c = \frac{{P_{peak}}^2}{8\pi E^* R^3} = \frac{3{P_{peak}}^2}{32\pi E R^3} \tag{8}$$

P_{peak} is the peak separation force

Note that this formulation treats G_c as a property that is independent of strain rate and temperature. This is not the case and you will need language addressing this in your manuscript. See the experimental section from Barney et al. for an example of such language. [8]

A.3 Contact with spherical probe

A.3.1 Determining E

Assuming you haven't made alterations to the TA system, we are currently unable to monitor the contact area during indentation. E can still be estimated from indenting with a spherical probe, but the assumptions necessary to do so introduce a large amount of error. For this reason, I would suggest that you use a flat probe when you want to estimate E. If you want to use a spherical probe, I suggest you set up visualization of the contact area and measure the modulus as Shull and coworkers do, but it's much simpler to use a flat probe. [2]

A.3.2 Estimating G_c

This walkthrough of estimating G_c assumes that the contact radius is not monitored during indentation. G can be estimated by substituting Equation (2) into Equation (5).

$$G = \frac{\left(\frac{4E^*a^3}{3R} - P\right)^2}{8\pi E^* a^3} \tag{9}$$

Rearranging and solving for a^3 gives

$$a^3 = \frac{3R}{4E^*}\left(P + 3\pi GR + \sqrt{6\pi GRP + (3\pi GR)^2}\right) \tag{10}$$

which is only valid so long as the quantity in the square root is positive. Setting this quantity equal to 0 gives the force at which crack propagation becomes unstable (assumed to be the peak separation force P_{peak}).

$$G_c = -\frac{2P_{peak}}{3\pi R} \tag{11}$$

Note that this formulation does not rely on the modulus of the material.

B Useful Nonlinear Elastic Relationships

This section gives a crude/functional introduction to nonlinear elasticity. For a more complete discussion, I recommend finding a copy of *The Physics of Rubber Elasticity* by L.R.G. Treloar. [9] This explanation defines constitutive relations of incompressible materials as a function of the stretch ratio λ. λ is defined by the current length L and intial length L_o of a material and can be related to the engineering strain ε.

$$\lambda = \frac{L}{L_o} = \frac{L_o + \Delta L}{L_o} = 1 + \varepsilon \tag{12}$$

Strain energy density functions, W, relate material deformation to the stored strain energy per unit volume. They are useful when using the TA as they can be used to calculate the relationship between stress and strain for different geometries. True stress and engineering stress can be related to strain energy functions as shown in Equation (13) where σ is stress and λ is stretch ratio. [10]

$$\sigma_{engineering} = \frac{\sigma_{true}}{\lambda} = \frac{dW}{d\lambda} \tag{13}$$

Engineering stress, also known as the nominal stress, is defined as the force divided by the initial

area of a sample. Most often this is the stress used during uniaxial extension/compression. In other fields, such as biology, it is more common to use the true stress, also known and the Cauchy stress. This means that if a strain energy function is known, one need only specify the deformation to calculate the stress response.

The Neo-Hookean strain energy function shown in Equation (14) is the simplest strain energy function possible for an isotropic, incompressible material where $I_1 = {\lambda_1}^2 + {\lambda_2}^2 + {\lambda_3}^2$ is the first strain invariant and μ is the shear modulus. [11]

$$W = \frac{\mu}{2}(I_1 - 3) \tag{14}$$

Note that μ can be related to the elastic modulus E through Poisson's ratio ν, which is 0.5 for an incompressible material.

$$\mu = \frac{E}{2(1-\nu^2)} \tag{15}$$

While the behavior of gels and elastomers deviates from Neo-Hookean at large strains, most nonlinear strain energy density functions converge to Neo-Hookean behavior at small strains which gives the physical principles predicted by this model general application. [9] For analysis of the experiments in this manual, it is instructive to derive the elastic response of a Neo-Hookean material in common test geometries.

B.1 Pure Shear

An incompressible material subjected to pure shear deforms as described by the stretch ratios

$$\lambda_1 = \lambda, \quad \lambda_2 = \frac{1}{\lambda}, \quad \lambda_3 = 1 \tag{16}$$

where stretching is imposed along the 1 direction. Substituting Equation (16) into Equation (13) gives Equation (17).

$$W = \frac{\mu}{2}(\lambda^2 + \frac{1}{\lambda^2} - 2) \tag{17}$$

Differentiating Equation (17) with respect to stretch gives the engineering stress. [9]

$$\sigma = \frac{dW}{d\lambda} = \mu(\lambda - \frac{1}{\lambda^3}) \tag{18}$$

B.2 Uniaxial Extension/Compression

An incompressible material subjected to uniaxial extension deforms as described by the stretch ratios

$$\lambda_1 = \lambda, \quad \lambda_2 = \lambda_3 = \frac{1}{\sqrt{\lambda}} \tag{19}$$

where stretching is imposed in the 1 direction. By substituting Equation (19) into Equation (13) one gets Equation (20).

$$W = \frac{\mu}{2}(\lambda^2 + \frac{2}{\lambda} - 3) \tag{20}$$

Differentiating Equation (20) with respect to stretch ratio gives the engineering stress in Equation (21) [9].

$$\sigma = \frac{dW}{d\lambda} = \mu(\lambda - \frac{1}{\lambda^2}) \tag{21}$$

The predicted behavior is clearly nonlinear, but at extremely low strains ($\varepsilon << 1$) the model collapses down to the Hookean Equation Equation (22) where ε is engineering strain.

$$\sigma = \mu\Big(\frac{\lambda^3 - 1}{\lambda^2}\Big) = \mu\frac{(\varepsilon+1)^3 - 1}{(\varepsilon+1)^2} = \mu\frac{\varepsilon^3 + 3\varepsilon^2 + 3\varepsilon}{\varepsilon^2 + 2\varepsilon + 1} \approx 3\mu\varepsilon \tag{22}$$

Table 1: Different strain energy density functions with engineering stress-stretch relationships worked out.

Strain Energy Function	$\frac{dW}{dI_1}$	$\frac{dW}{dI_2}$	Shear Modulus μ	Shear $\lambda_1 = \lambda, \lambda_2 = \frac{1}{\lambda}, \lambda_3 = 1$	Uniaxial Extension $\lambda_1 = \lambda,\ \lambda_2 = \lambda_3 = \frac{1}{\sqrt{\lambda}}$	Equibiaxial Extension $\lambda_1 = \lambda_2 = \lambda,\ \lambda_3 = \frac{1}{\lambda}$	Cavitation
Neo-Hookean $W = C_{10}(I_1 - 3)$	Constant	0	$2C_{10}$	$\tau = \mu\gamma$	$\sigma = \mu\left(\lambda - \frac{1}{\lambda^2}\right)$	$\sigma = \mu\left(\lambda - \frac{1}{\lambda^5}\right)$	$P = \frac{5}{2}\mu - \frac{2\mu}{\lambda} - \frac{\mu}{2\lambda^4}$
Mooney-Rivlin $W = \frac{C_1}{2}(I_1 - 3) + \frac{C_2}{2}(I_2 - 3)$	Constant	Constant	$C_1 + C_2$	$\tau = (C_1 + C_2)\gamma$	$\sigma = C_1\left(\lambda - \frac{1}{\lambda^2}\right) + C_2\left(1 - \frac{1}{\lambda^3}\right)$	$\sigma = C_1\left(\lambda - \frac{1}{\lambda^5}\right) + C_2\left(\lambda^3 - \frac{1}{\lambda^3}\right)$	$P = \frac{5}{2}C_1 - \frac{2C_1}{\lambda} - \frac{C_1}{2\lambda^4} - C_2 + 2C_2\lambda - \frac{C_2}{\lambda^2}$
Yeoh $W = C_1(I_1 - 3) + C_2(I_1 - 3)^2 + C_3(I_1 - 3)^3$	Variable	0	$2C_1$	$\tau = 2C_1\gamma + 4C_2\gamma^3 + 6C_3\gamma^5$	$\sigma = 2C_1\left(\lambda - \frac{1}{\lambda^2}\right) + 4C_2(I_1 - 3)\left(\lambda - \frac{1}{\lambda^2}\right) + 6C_3(I_1 - 3)^2\left(\lambda - \frac{1}{\lambda^2}\right)$	$\sigma = 2C_1\left(\lambda - \frac{1}{\lambda^5}\right) + 4C_2(I_1 - 3)\left(\lambda - \frac{1}{\lambda^5}\right) + 6C_3(I_1 - 3)^2\left(\lambda - \frac{1}{\lambda^5}\right)$	$P = 5C_1 - \frac{4C_1}{\lambda} - \frac{C_1}{\lambda^4} - \frac{177}{10}C_2 - \frac{C_2}{2\lambda^8} - \frac{4C_2}{5\lambda^5} + \frac{3C_2}{\lambda^4} - \frac{4C_2}{\lambda^2} + 8C_2\lambda + \frac{12C_2}{\lambda} + \frac{2909}{30}C_3 - \frac{C_3}{2\lambda^{12}} - \frac{2C_3}{3\lambda^9} + \frac{9C_3}{2\lambda^8} - \frac{4C_3}{\lambda^6} + \frac{36C_3}{5\lambda^5} - \frac{27C_3}{2\lambda^4} + 8C_3\lambda^3 - \frac{8C_3}{\lambda^3} + \frac{36C_3}{\lambda^2} - 72C_3\lambda - \frac{54C_3}{\lambda} + 24C_3\ln\lambda$
Gent-Thomas $W = \frac{C_1}{2}(I_1 - 3) + \frac{3C_2}{2}\ln\frac{I_2}{3}$	Constant	Variable	C_1	$\tau = C_1\gamma + C_2\left(\frac{\gamma}{3} + \frac{1}{\gamma}\right)^{-1}$	$\sigma = C_1\left(\lambda - \frac{1}{\lambda^2}\right) + 3C_2\left(\frac{\lambda - \frac{1}{\lambda^2}}{\lambda^2 + \frac{2}{\lambda}}\right)$	$\sigma = C_1(\lambda - \lambda^{-5}) + 3C_2\left(\frac{\lambda^5 - \lambda^{-3}}{\lambda^4 + 2\lambda^{-2}}\right)$	$P = C_1\frac{5}{2} - \frac{2}{\lambda} - \frac{1}{2\lambda^4} + \frac{C_2}{2}\left[\ln\left(\frac{3\lambda^6}{2+\lambda^6}\right) + 2\sqrt{2}\left(\tan^{-1}(\sqrt{2}) - \tan^{-1}\left(\frac{\sqrt{2}}{\lambda^3}\right)\right)\right]$
Gent $W = -\frac{\mu J_m}{2}\ln\left[1 - \frac{J_1}{J_m}\right]$	Variable	0	XX	$\tau = \frac{\mu\gamma}{\left[1 - \frac{J_1}{J_m}\right]}$	$\sigma = \frac{\mu\left(\lambda - \frac{1}{\lambda^2}\right)}{\left[1 - \frac{J_1}{J_m}\right]}$	$\sigma = \frac{\mu\left(\lambda - \frac{1}{\lambda^5}\right)}{\left[1 - \frac{J_1}{J_m}\right]}$	$\int_0^{-P} dP = -2\mu\int_1^{\lambda} \frac{\lambda^{-2} + \lambda^{-5}}{\left[1 - \frac{J_1}{J_m}\right]} d\lambda$