Tetra High-Temperature Bulge Tester SOP

Rev. 0

Dec. 7, 2011

Jeremy Thurn

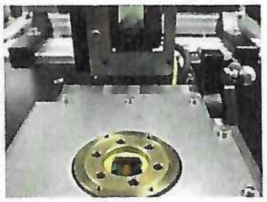
STEP I: UNLOAD THE SAMPLE

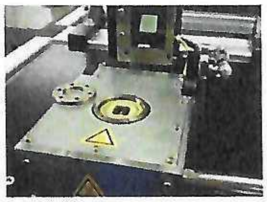
Unload any sample that may be in the tester, if necessary. Otherwise skip to STEP II.

1. Open the cover of the bulge tester.
The laser will shut off automatically.

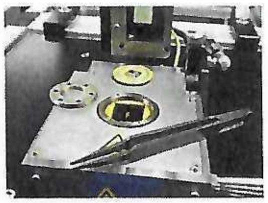
2. Lower the sample fixture.

3. Unscrew the nut. Use the torque wrench tool to get it started, then unscrew by hand.

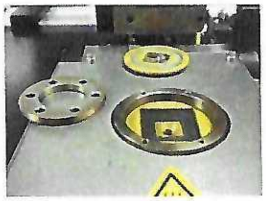




4. Remove the insert using a plastic tweezer.

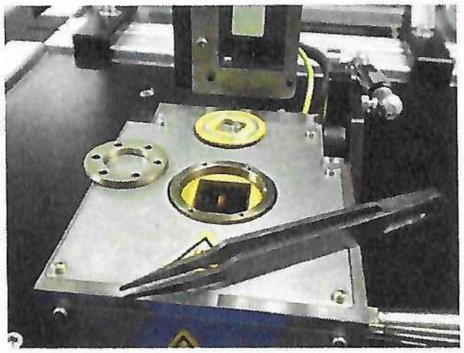


5. Remove the sample using a plastic tweezer.
Leave the graphite seal in place.

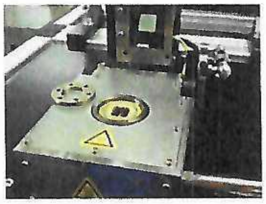


STEP II: LOAD THE SAMPLE

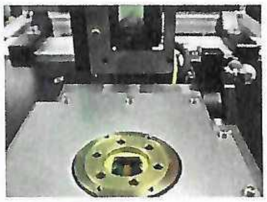
1. Carefully place the sample on the graphite seal using a plastic tweezer.
Align the rectangular membrane as shown in the picture.
The membrane may be very fragile!



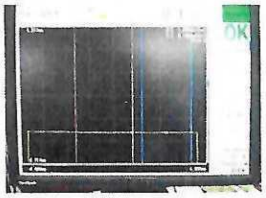
2. Carefully place the insert atop the sample using plastic tweezers



3. Screw on the nut and tighten to 15 inch-lbs using the torque wrench.



4. Raise the sample fixture and lower the cover. Be sure the cover is seated properly such that the laser turns on and displays a (more or less) flat line on the monitor.

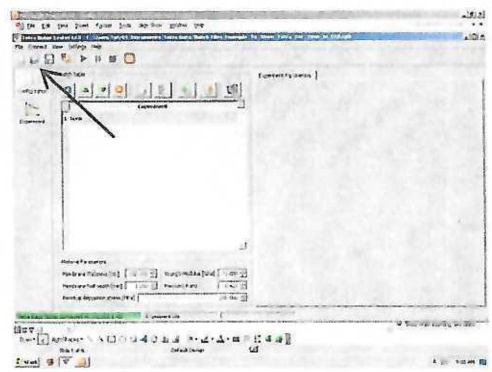


STEP III: SET UP THE MEASUREMENT LOCATIONS

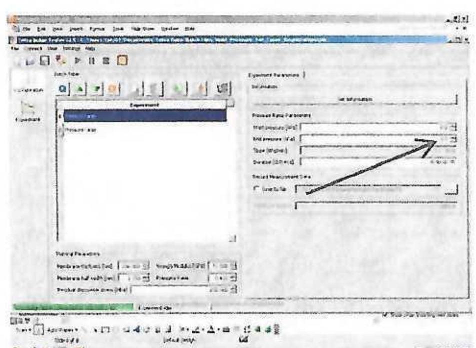
1. Start the GUI ("BulgeTesterGUI.exe")

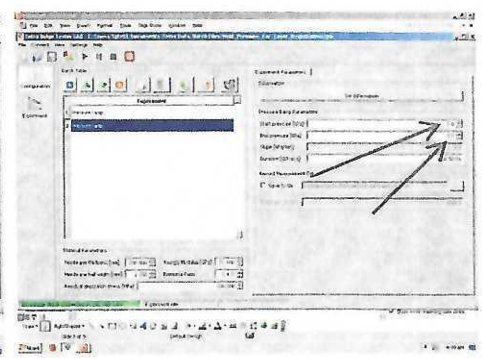
2. Connect to the bulge tester by clicking on the icon that looks like a plug-in.

3. Open the batch file "Hold_Pressure_For_Laser_Registration" in the folder My Documents: Tetra Data: Batch Files



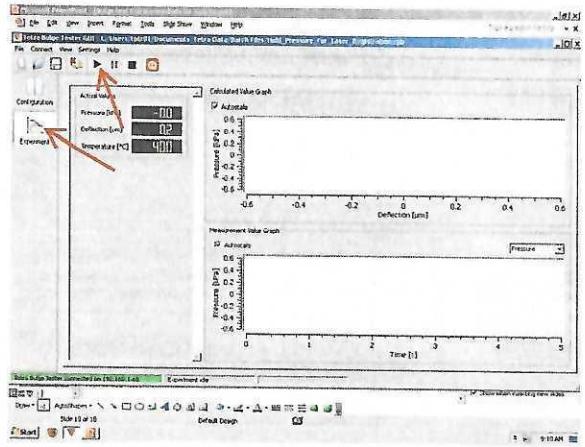
4. Step 1 ramps up to the target pressure, and Step 2 holds at that pressure until the user stops the test. The goal is to make the membrane bulge just enough to be obvious to the naked eye so you can set up the laser measurement & reference regions. You will need to adjust the End Pressure in Step 1, and both the Start Pressure and End Pressure in Step 2.





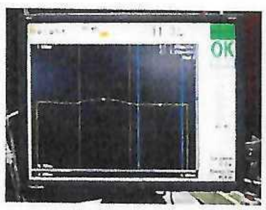
CAUTION: The target pressure should be as small as possible to avoid breaking the membrane. For a 1.5um silicon nitride membrane I use 3kPa and for a 200nm gold membrane I used 0.5kPa.

5. Click on the "Experiment" button at the left side of the GUI, then press the play icon at the top of the GUI.



6. Press the green "Start" button on the front panel of the bulge tester (it will be blinking).

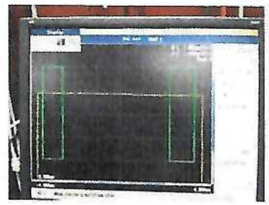
7. Watch the laser profile monitor and when the bulge becomes apparent, on the Keyence remote control pull the "Prog/Run" slide, located on the right side of the remote.

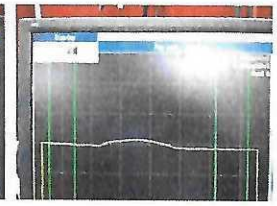


8. Use the small joystick (labeled "Enter") to move the cursor down to "Master Reg" and press down on the joystick (i.e. press "Enter"). Press the "Trig/Hold" button several times. This records the current laser reading of the bulge shape in the laser controller's memory. Press "Enter" when prompted. Press the "Escape" button to return to the menu.

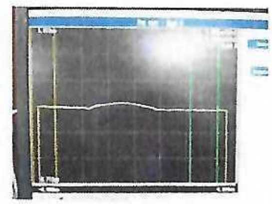
9. Move the cursor to "Poss Corr" and press Enter. Select Head A and press Enter.

10. Zoom in on the laser profile by pressing the "Menu" button, which causes a small menu to appear in the top-left corner of the screen. Then use the joystick to move to the 3rd icon from the left labeled "Auto" and press Enter. The bulge will now be clearly visible. Press Escape.

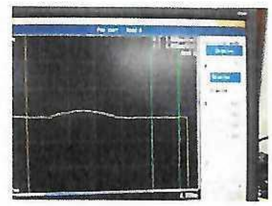




11. If necessary, adjust the location of the Tilt Correction boxes. They should be at the far left and right extremes of the laser image. The picture shows an example where the left-hand box is improperly located; it needs to be moved to the right. Follow the steps below to move one or both boxes, if necessary.



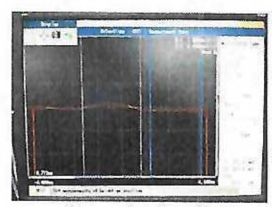
a. Move the cursor to ANG area1 or ANG area2 (ANG area1 is the left-hand box, and ANG area2 is the right-hand box) and press Enter



b. With the cursor on "Drawing" press Enter. Press the "~~Select~~ screen" button twice. Use the joystick to move the box to the left or to the right. When it is properly positioned, press Enter, then press Escape twice.

12. Move the cursor to "OUTsetting" and press Enter. Select "OUT1: Height" and press Enter. Move the cursor to "Measurement Type: Step" and press Enter.

13. Press "Menu" and select "Auto" to zoom in on the laser profile, so you can see the bulge clearly. Press Escape.
There should be two boxes, one near the center enclosing the bulge, from which the maximum bulge deflection will be measured, and one to the right of the bulge, from which an average reference position will be measured.
The average reference position box should be relatively wide, encompassing a large part of the laser profile between the edge of the bulge and the edge of the sample.

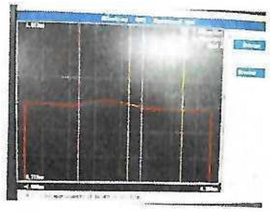


Example of well-positioned boxes

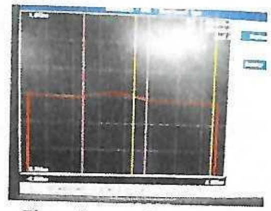
If the box locations are acceptable, pull the Prog/Run slide on the remote ✓ and proceed to STEP IV.
you may see a "No go signal" Error, It's ok, just click "OK"

These pictures show examples of poorly located boxes that must be adjusted.

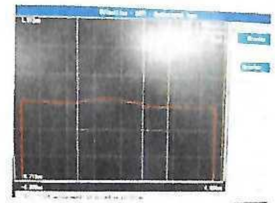
If the box locations are acceptable, pull the Prog/Run slide on the remote and proceed to STEP IV.



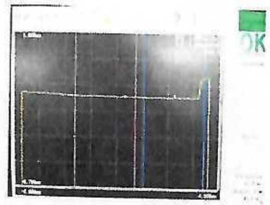
The reference box is too far to the left



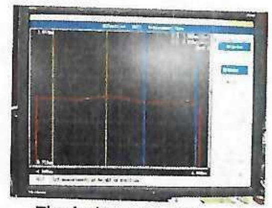
The reference box overlaps part of the bulge and needs to be reduced in width



The reference box is too far to the right



The reference box includes some defect at the right side, and should be reduced in width



The bulge measurement box is too far to the left

14. To adjust the location of the average reference position measurement box, move the cursor to “Area 1” and press Enter. Leave the cursor on “Drawing” and press Enter. Use the joystick to move the left edge of the box, press ~~Select~~ Screen then use the joystick to to move the right edge of the box, press ~~Select~~ Screen again to move the entire box. After positioning is complete, press Enter, then Escape.

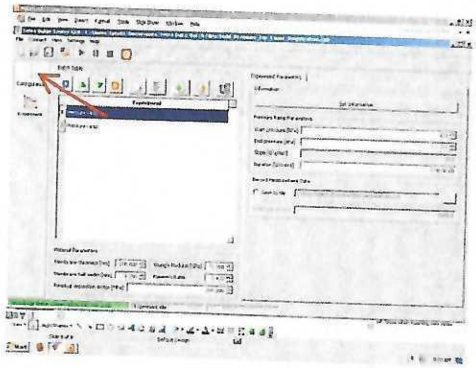
15. To adjust the location of the bulge measurement box, move the cursor to “Area 2” and press Enter. Leave the cursor on “Drawing” and press Enter. Use the joystick to move the left edge of the box, press ~~Select~~ Screen then use the joystick to to move the right edge of the box, press ~~Select~~ Screen again to move the entire box. After positioning is complete, press Enter, then Escape.

16. Pull the Prog/Run slide on the remote.

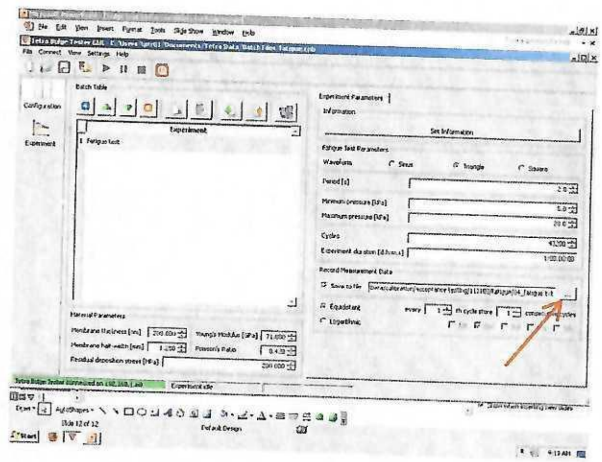
STEP IV: RUN YOUR EXPERIMENT

1. After you pull the "Prog/Run slide" on the Keyence remote in STEP III, the pressure will drop to zero and the bulge will disappear. Press the "Zero" button on the remote to zero out the laser reading.

2. In the GUI, click "Configuration" and open the Batch File you wish to run.



3. Press the icon "..." in the "Record Measurement Data" area to Browse for the file location. Write in an appropriate name for the data file from each step (e.g. "01_load_30kPa.txt" or "01_hold_30kPa.txt" or "01_unload_30kPa.txt" for experiment 1, "02_load_30kPa.txt" etc. for experiment 2).



4. Fill in the appropriate values for Membrane thickness [nm], Poisson's Ratio, and Residual deposition stress [MPa]. The Young's Modulus [GPa] value does not matter. The Membrane half-width [mm] should always be set to 1.25.
If you so desire, click "Set Information" and add a User Name and Material and Description. The text entered in these fields will be included in the header of the TXT files generated during the experiment. (Do not use any Carriage Returns in the Description field, as that will cause an error in the Excel Macro.)

5. Click "Experiment" and then the "Play" icon. Press the green "Start" button on the front of the bulge tester to run your experiment. Press the Stop button at any time to stop the experiment.

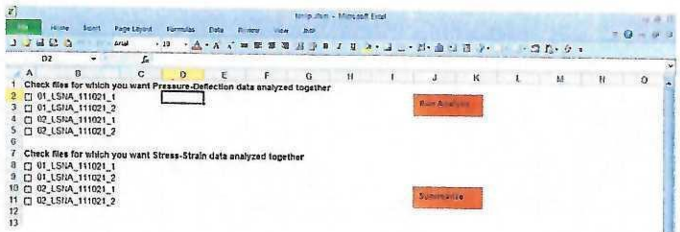
STEP V: ANALYZE YOUR DATA

1. Open the file "Tetra Bulge Test TXT Analyzer Rev 1.07.xlsm" using Excel 2010. Close any other open Excel files.

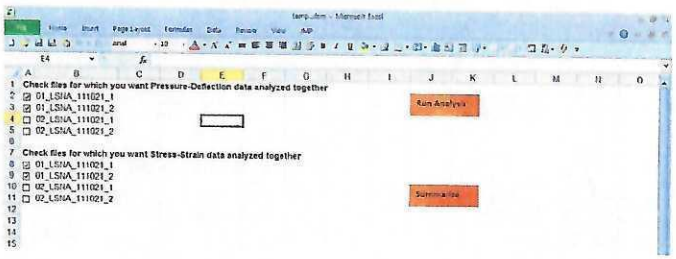
2. Click the "Run Macro" button and Browse to the folder containing your data files. While holding down the Shift or Ctrl keys, select all the Pressure Ramp data files you want to analyze. Do not include any Pressure Hold data files. Click "Open."

3. Type in the name of the Excel file to generate. It will be saved as an XLSM file so that the macros will be enabled for future data manipulation.

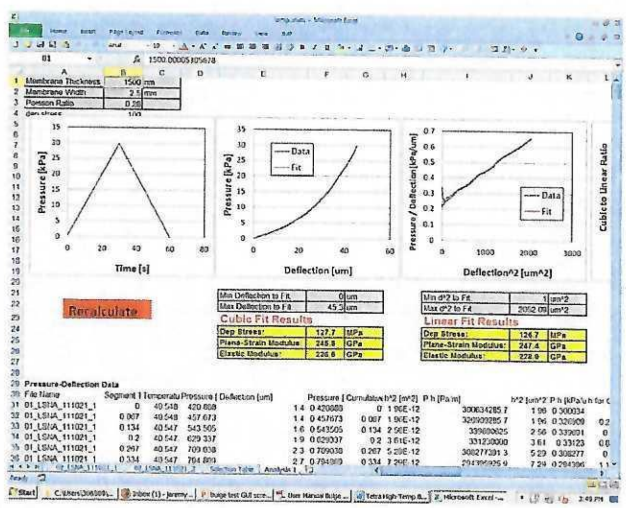
4. Each TXT file you selected will be listed with a checkbox next to it. The top list is used to select the files for Pressure-Deflection data analysis. The bottom list is used to select the files for Stress-Strain data analysis



In the example shown here, file 01_LSNA_111021_1 is the loading part of Experiment 1 and 01_LSNA_111021_2 is the unloading part of Experiment 1



To analyze the loading and unloading data from Experiment 1 together, check both boxes and click the "Run Analysis" button.

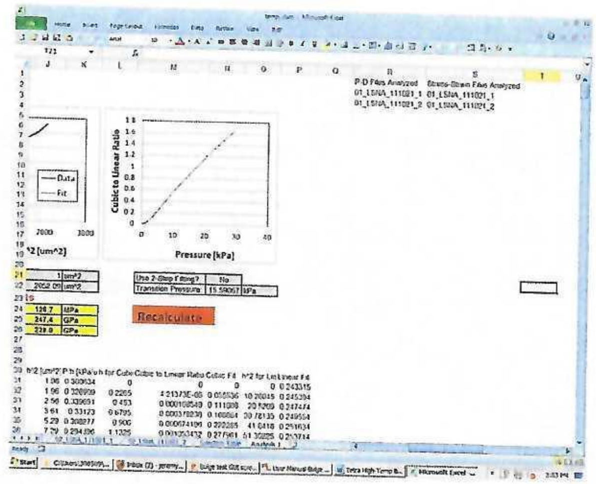


5. The macro generates a new Sheet labeled "Analysis #" where # is the number of times you've pressed the "Run Analysis" button.

User Inputs: You can adjust the Membrane Thickness, Membrane Width, and Poisson Ratio in cells B1-B3. They default to the values from the TXT file header. Press the Reclaculate button after making changes.

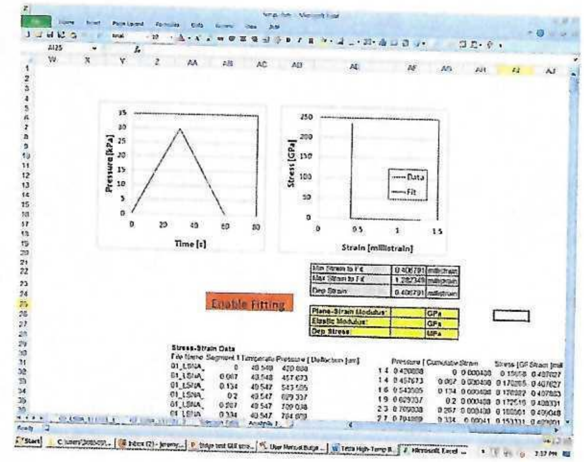
Pressure Profile: The Pressure-Time graph shows both a loading and unloading part because both loading and unloading files were selected in Step 4, above.

Pressure-Deflection Data: The Pressure and Deflection are plotted as raw data in the middle graph, and linearized in the right graph. A nonlinear curve fit is performed on the raw data, and a linear curve fit on the linearized data. You can specify the minimum and maximum values of the abscissa (Deflection or Deflection Squared) and re-do the curve fit. The Young's Modulus and Membrane Stress (or deposition, or "Dep" stress) extracted from the curve fits are shown in the yellow boxes.



Two-Step Analysis: If you scroll to the right, there is a graph of the ratio of the Cubic Term to the Linear Term from the nonlinear curve fit. Some manuscripts recommend extracting the Membrane Stress from that portion of the data where the ratio of the Cubic to Linear is less than one, and the Young's Modulus from that portion where the ratio is greater than one. To perform this "two-step" analysis, type "Yes" or "Y" in cell N21. If there is a number in cell N22, it is the calculated value of the pressure precisely at a Cubic to Linear ratio of 1.0. You can adjust this transition pressure if you so desire. Then click the Recalculate button.

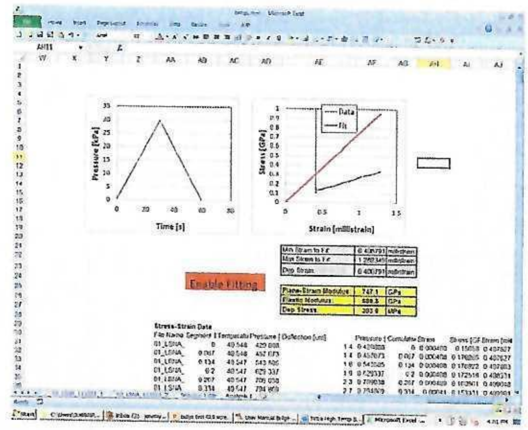
Filenames: Near the top of the page in columns R and S the TXT filenames used in this analysis are listed.



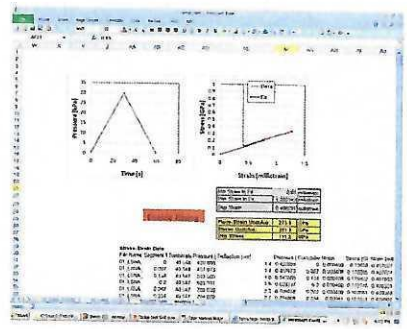
Stress-Strain Analysis: If you scroll further to the right, you'll see another Pressure-Time graph and a Stress-Strain graph. Here the Pressure-Deflection raw data has been converted to Stress-Strain data. You must iterate on the Deposition Strain (or "Dep Strain") to get a good fit between the bold red line and the data shown in black. Start by clicking the Enable Fitting button

CAUTION: Clicking the Enable Fitting button activates a Worksheet Macro. This may make it impossible to effectively use the ReCalculate button over on the left side of the sheet. So either re-save the Workbook under a new name, or make sure you're done with the Pressure-Deflection data.

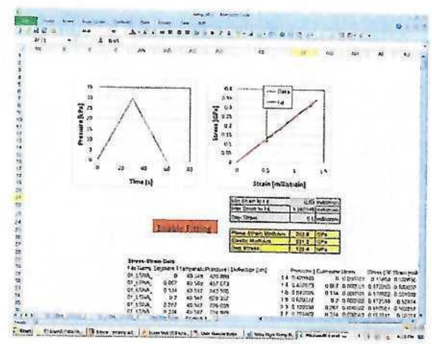
The Stress calculated from small pressure data is usually bad data, so you'll probably have to adjust the Y-Axis scale to see the data. The picture shows what happened after I clicked "Enable Fitting" and changed the Y-Axis maximum to be 1.0.



Next I changed the Min Strain to Fit to be 0.65 to avoid the bad data at low pressures. The fit will be adjusted as soon as you hit "Enter." Now the fit is much better.



I again adjusted the Y-Axis and increased the Dep Strain to 0.5. Now the fit is nearly perfect. The slope of the red line is the Young's Modulus and the value you entered for Dep Strain is used to calculate the Deposition Stress (or Membrane Stress).



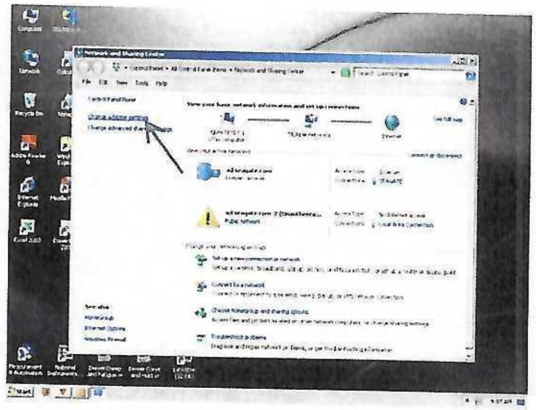
If the stress-strain data is linear only for some portion of the curve, and above some stress it rolls off, then you have exceeded the yield stress of your material. Be sure to set the Max Strain to Fit to be a value in the linear region of the data.

Creating a Summary Table: Return to the "Selection Table" tab. If I un-check the first two filenames, and check the bottom two filenames, then a new tab "Analysis 2" will be created with data from the two checked TXT files. After I'm done analyzing this data, I can return to the Selection Table tab and click the "Summarize" button to create a new tab called "Summary" that lists all the calculated parameters from all the Analysis tabs.

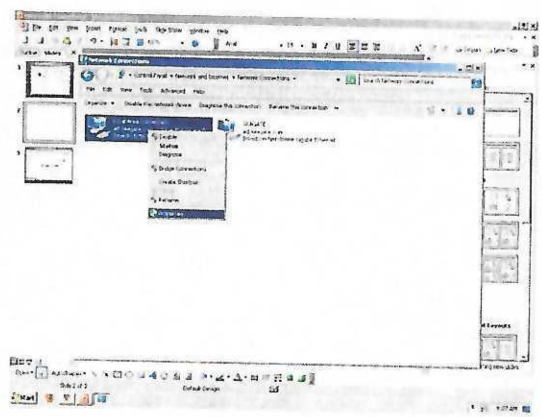
APPENDIX A: HOW TO CONNECT THE BULGE TESTER TO THE COMPUTER

The computer interfaces with the bulge tester via ethernet cable. This document assumes the cable is connected.

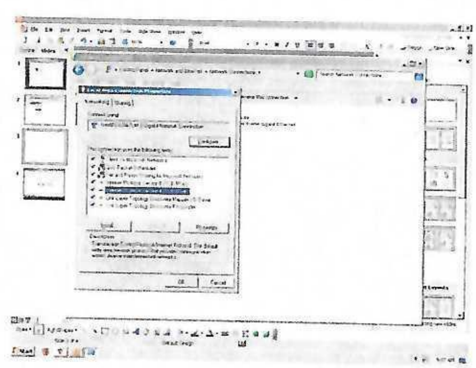
1. In the PC control panel, select Network and Sharing Center, then Change Adapter Settings.



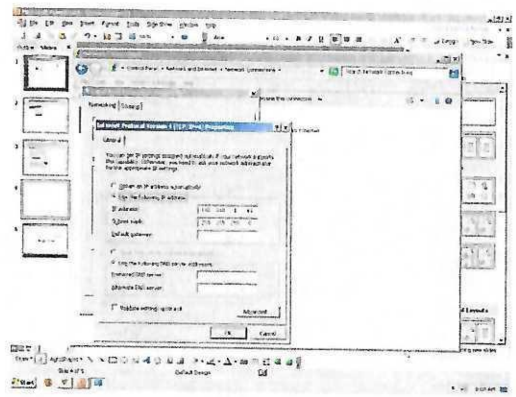
2. Right-click on Local Area Connection and select Properties



3. Select Internet Protocol Version 4 (TCP/IPv4) and click Properties.

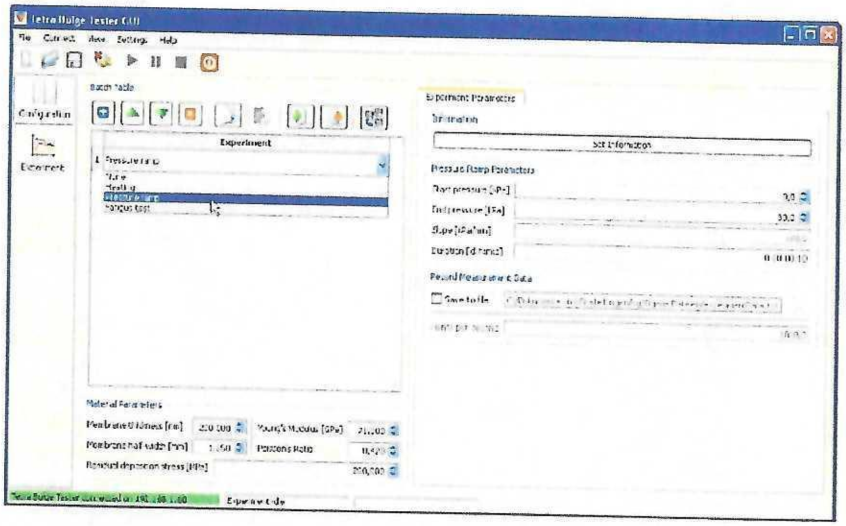


4. Select “Use the Following IP Address” and type in 192.168.1.62. Click OK.



APPENDIX B: HOW TO WRITE YOUR OWN SCRIPT IN THE TETRA GUI

1. Double-click on the region under "Experiment," then click again to show a drop-down list. Select from "Heating," "Pressure Ramp," or "Fatigue."



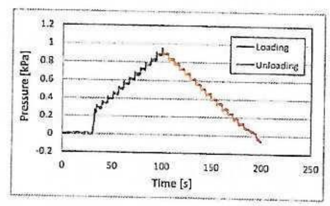
Heating: Change the temperature of the sample. Input the target temperature, and specify whether to proceed immediately to the next step or wait until the target temperature is reached. Temperature range is 40C to 400C.

Note: no data is recorded during a Heating step.

Pressure Ramp: Specify the Start Pressure, End Pressure, Duration, Filename, and data collection rate ("Points per Second"). The Points per second should be set to 15.

The maximum pressure ramp rate is unknown, the fastest I've used is 30kPa/s and it worked OK.

If the pressure ramp rate is very low, then the pressure becomes discretized in steps of 0.04kPa, as shown in the graph.



CAUTION: The first time the pressure is ramped up from zero, there will invariably be a jump in pressure, as shown in the graph. The magnitude of the jump depends on the ramp rate; the higher the rate, the bigger the jump. Thus, it may be wise to split your loading into two steps, one with a low End Pressure and very slow ramp rate, and a second with your desired End Pressure and your desired ramp rate.

Alternatively, you can include a loading step to some low pressure followed by a load of at least 20s, followed by an unloading step to 0.2kPa or so. This "pre-load" will eliminate the jump and subsequent steps are jump-free.

If you set the Start Pressure and End Pressure to be the same value, then the Pressure Ramp step is actually a Pressure Hold step. For exceptionally long Holds, it may be wise to reduce the points per second from 15 to make the file size managable.

You can chain as many Pressure Ramp and Pressure Hold steps together as you want.

Fatigue: The Fatigue test is used to cycle the pressure between a Minimum and Maximum pressure for a specified number of Cycles using a specific Waveform (Sinus, or sinusoidal, Triangle, or Square) and Period. The data file will record the Maximum deflection during the cycle and the pressure associated with that maximum deflection. I am awaiting an upgrade to enable the software to also record the minimum deflection and pressure for a cycle.

The GUI displays the pressure-time profile for each cycle, but only the data point at the peak deflection is saved in the data file.

You can save the data from each cycle, or every n^{th} cycle, or at a logarithmic spacing.

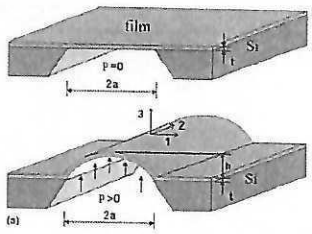
CAUTION: The GUI has failed thus far to run a complete 24-hour fatigue test. Once it locked up after 40 minutes, and once after almost 24 hours. I am running tests to see if the Anti-Virus Software is the culprit.

APPENDIX C: THEORETICAL BACKGROUND

See Xiang *et al.*, J. Mater. Res., Vol. 20, No. 9, Sep 2005, p. 2360 and
Vlassak & Nix, J. Mater. Res., Vol. 7, No. 12, Dec 1992, p. 3242 for a thorough background on the test and the basic analysis techniques.

Pressure-Deflection Data: A theoretical relationship between the applied pressure and measured maximum bulge deflection is obtained from the above references:

$$p = 2\frac{\sigma_0 t}{a^2}h + \frac{4}{3}\frac{Et}{(1-\nu^2)a^4}h^3$$



β

$\sigma = \sigma_0 + d\sigma$
$= \sigma_0 + E' \cdot d\epsilon$

where p is pressure, t is the membrane thickness, a is the membrane half-width, h is the maximum bulge deflection, E is Young's Modulus, ν is Poisson's Ratio, and σ_0 is the membrane stress, which is assumed to be uniform through its thickness. If the sample clamp applies no forces to the membrane, then σ_0 is the film deposition stress (if measured at room temperature). Since the sample clamp does, however, impart forces to the membrane, σ_0 is actually the "membrane stress" in the clamped position.

Linearizing the Equation: The above equation is readily linearized by dividing through by h and plotting p/h as the ordinate (Y-Axis) and h^2 as the abscissa (X-Axis).

Two-Step Procedure: The Young's Modulus deduced from a curve fit is dependent only on the cubic term of the equation. It has been noted in the literature that unless the pressure-deflection data shows considerable curvature, the Young's Modulus from the curve fit is highly variable and unreliable. I similarly found that silicon nitride membranes showed an apparent strange dependence of Young's Modulus vs temperature if I calculated Young's Modulus from the entire pressure-deflection data (linearized or not), but if I instead used a two-step procedure and calculated Young's Modulus only from that data where $p > 20$kPa, then the Young's Modulus was invariant with temperature, as expected. This pressure is approximately the pressure where the ratio of the cubic term to the linear term is one.

This means, unfortunately, that some materials will yield before reaching a cubic-to-linear ratio of one, so this technique cannot be used to measure Young's Modulus in every situation.

Pertinent references include Huston *et al.*, SPIE Proc Vol. 4344, 2001, p. 673 (they provide an inequality to determine if the cubic term is large enough to extract a modulus); Gally *et al.*, MRS Symp Proc. Vol.

594, 2000, p. 195; Ortlner *et al.*, Rev. Sci. Instrum. Vol. 81, 2010, p. 055111; Edwards *et al.*, Exp. Mech. Vol. 44, 2004, p. 49; and Mitchell *et al.*, J. Aero. Eng. Vol. 16, 2003, p. 46.

Anisotropy: Equations have been derived for membrane materials with strong crystalline anisotropy (see Tsakalakos *Thin Solid Films*, Vol. 75, 1981, p. 293, although his derivations are for a circular membrane), or Bonnotte *et al.*, J. Mater. Res. Vol. 12, 1997, p. 2234, where equations are derived for single-crystal membranes, or Mitchell *et al.*, J. Aero. Eng. Vol. 16, 2003, p. 46.).

Bending Stiffness: Bending is normally not important for bulge tests unless the dimensions are changed considerably. For instance, Li & Cima (MRS Symp Proc, Vol. 795, 2004, p. U10.3.1) incorporate bending stiffness in their analysis because they use square membranes with edge lengths between 20um and 200um.

Large Deflections: The equations used here are appropriate for small deflections. Large-deflection elasticity is studied in Bonnotte *et al.*, J. Mater. Res. Vol. 12, 1997, p. 2234, but should not be needed unless the maximum bulge deflection is orders of magnitude greater than the film thickness. (That being said, I've never gone through the large deflection analysis to confirm that its unnecessary. One could imagine that a 150nm Au film with 50um of deflection might be violating the small deflection assumption!)

Wrinkling: If the membranes are wrinkled, even a little tiny bit at the edges (I recommend inspecting the membranes under a microscope prior to testing to ensure no wrinkles), then the data analysis is bad and no data can be extracted. There is one literature article that claims to overcome this, but I could not understand their technique (Kalkman *et al.,* Rev. Sci. Instrum. Vol. 70, 1999, p. 4026). The articles stressing the importance of not having wrinkles are numerous but I only cite two: Vinci & Vlassak, Annu. Rev. Mater. Sci. 1996, vol. 26, p. 431 and Small & Nix, J. Mater. Res. Vol. 7, 1992, p. 1553. The pictures show examples of wrinkled membranes.

wrinkles in a PVD Au membrane

wrinkles in a Comptech alumina membrane

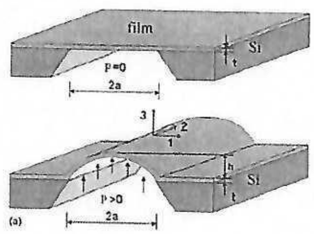
wrinkles in an LPCVD silicon nitride membrane after several minutes in ion mill

Fracture: If the applied pressure causes the stress in the film to exceed the fracture stress, then the membrane will break. Thus, the bulge test can serve as a measurement of fracture strength of thin film materials. See Yang & Paul, Sensors and Actuators A, Vol. 97-98, 2002, p. 520. Also, for bilayer membrane fracture, see Yan *et al.*, J. MEMS, Vol. 17, No. 5, Oct. 2008, p. 1120.

Fracture Toughness: If a pre-crack is generated in the membrane, then the bulge test can conceivably be used to cause controlled crack extension and extract a measurement of fracture toughness. See Xiang *et al.*, In. J. Fract., Vol. 144, 2007, p. 173 and Merle & Goken, Acta Mater., Vol. 59, 2011, p. 1772.

Stress-Strain Data: A theoretical relationship between the applied pressure and measured maximum bulge deflection and the stress and strain in the membrane is obtained from Xiang *et al.*, J. Mater. Res., Vol. 20, No. 9, Sep 2005, p. 2360:

$$\sigma = \frac{pa^2}{2ht} \text{ and } \epsilon = \epsilon_0 + \frac{2h^2}{3a^2}$$



where p is pressure, t is the membrane thickness, a is the membrane half-width, h is the maximum bulge deflection, σ is Stress, ε is Strain, and ε_0 is the membrane initial strain. If the sample clamp applies no forces to the membrane, then ε_0 is the film deposition strain (if measured at room temperature). Since the sample clamp does, however, impart forces to the membrane, ε_0 is actually the "membrane strain" in the clamped position. If the membrane is at least four times longer than its width (as with our membranes), then the stress and strain are in a state of "plane strain," and the slope of the stress-strain curve from the above equations is not the Young's Modulus, but instead the "plane-strain modulus," which is $E/(1-\nu^2)$, where ν is Poisson's Ratio.

Creep Tests: You'll note in the equation for stress that it depends on both p and h. Thus, during a test with constant p, or a "creep" test, the stress is actually decreasing due to any creep in h. So a true creep test cannot be performed with the current equipment. The software would have to be modified to include feedback control of the pressure to maintain a constant stress before a true creep test could be run.

Strain Rate Control: The system operates under pressure control. Some materials may have strain-rate dependent behavior. This can only indirectly be assessed by varying the pressure ramp rate. To control the strain rate would, again, require a software upgrade to include feedback control.