

Nanoindentation Hints

Version 1.0

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Nanoindentation hints

1. First things to do prior to experiments.

- a. Turn on the black Hysitron box (need to warm-up, 2 hours recommended)



Switch is here!

Usual setting for 4 dials are:

- a. Low Pass filter: 300
- b. Output Gain 1 : 1000
- c. Output Gain 2 : 1000
- d. Display : 100

- b. Final polish the specimen (0.25 to 0.01 μm) keep it dry or moist

- c. Turn on AFM

1. Turn on the box under the desk. Switch is at the right back side

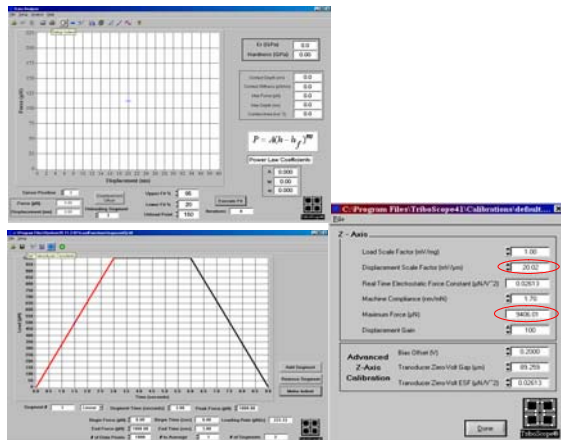


2. Turn on the AFM PC, if it is not on. Open NanoScope version 5.12r3

2. Prepare Hysitron computer

- You may want to reboot the PC, while doing this, turn off the Hysitron box.
- Open “Tribo scope” software
- Find a transducer in a black plastic box. Turn the power off while connecting it to the Hysitron box (backside), turn the power back on.
- Check if the initial setting values match to those on a info sheet in the black transducer box

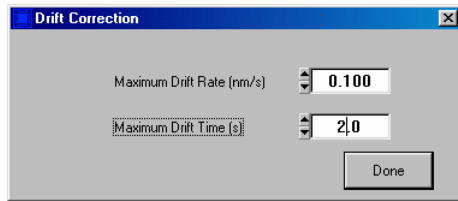
by Clicking “setup indent” → “Set Transducer Constants” icon



*Check Displacement Scale Factor matches to the value on the sheet

*Max Force is smaller than the value on the sheet

*Machine compliance is 1.7 for black shaft tip, lower for white ceramic base(0.8) Please refer to the note on the wall for right machine compliance value for individual tip.



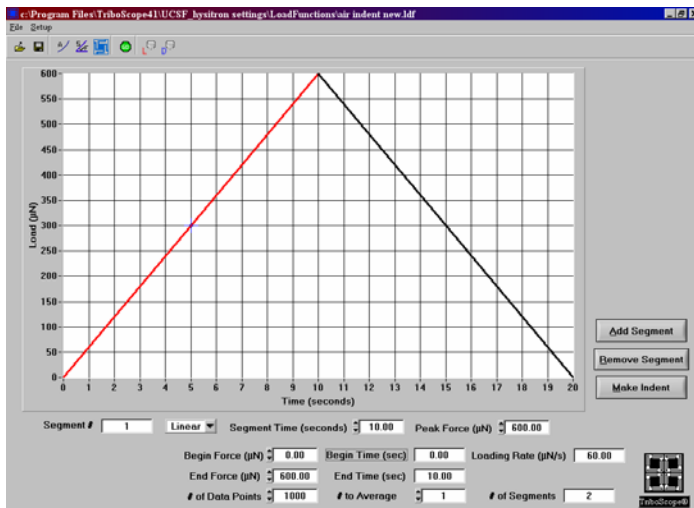
- Drift correction Click “setup” → drift correction. Drift correction: change to 2 sec.
- Spring force compensation should be checked “V” too.

3. Prepare AFM

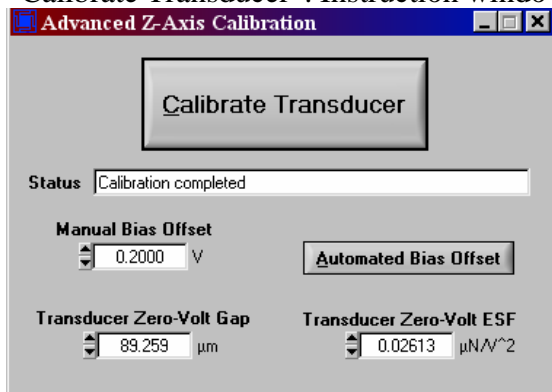
- Remove the optical lens.
- Flip the switch to select STM
- Click “microscope icon”, select “STM” mode.
- Type in initial values for engaging (do not engage, yet) → refer Appendix D
- Set up fused silica quartz reference on the stage.
- Lower the stage to have enough clearance before placing the indenter by using lever (to UP)
- Place the transducer+indenter over the silica reference.

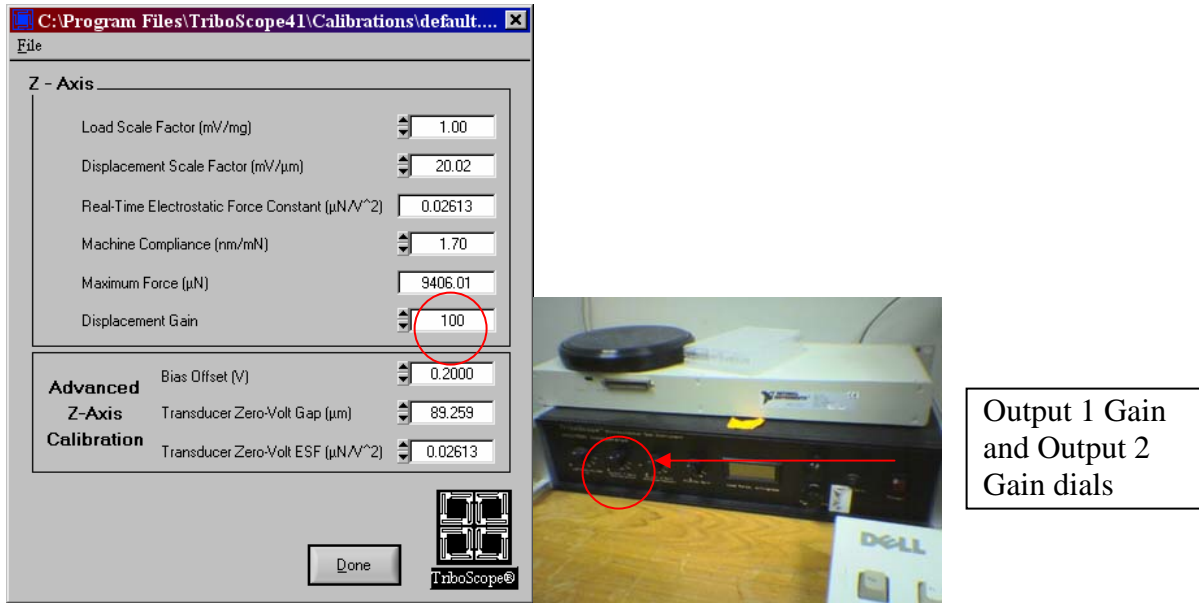
4. Air indent to adjust Electro static force constant.

Open load function “air indent new” (Loading 10sec, Unloading 10sec, peak force 600uN)

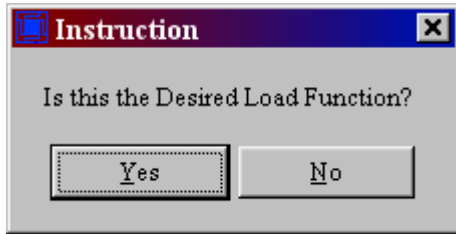


From indent screen, click setup → Advanced Z-Axis Calibration, following window will pop-up. Click “Calibrate Transducer”. Instruction window will appear. Click Yes if you are ready.

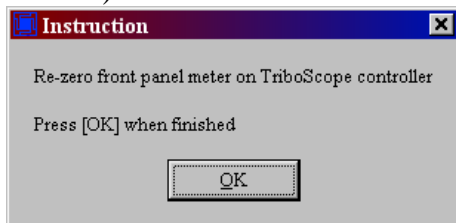




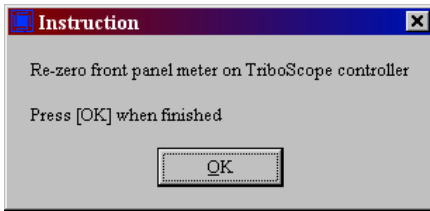
- 1) Change Displacement Gain to 100 (usually it is 1000 for the real measurement) and change Output 1 Gain and Output 2 Gain dials on the front panel of the hysitron box to 100 (1000 for measurement also).
- 2) Make sure “Displacement Scale Factor matches the value on the sheet in the transducer box.
- 3) Click Done



Click Yes if you already chose “air indent new.ldf”. If you click “No”, the program will stop at this point. You have to close all the open windows manually, then select right load function and start z-axis calibration again. (I think this is one of the bugs in the newer version software they’ve installed after upgrading PC. I inquired about possible bug fix, but haven’t heard from hysitron, yet. I can remove this sentence since right load function should be already selected if the user followed this instruction. --Kuniko)



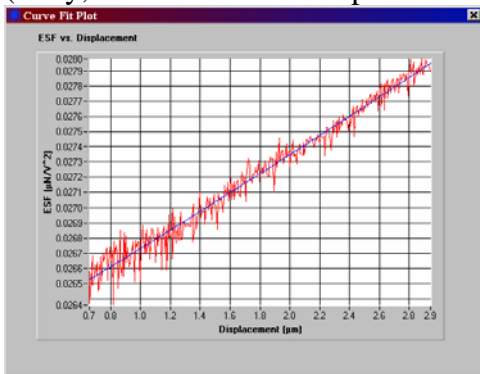
Use two dials in front of the hysitron box and adjust display value to 0.00., click OK. You will see real time plot window display force displacement curve going downward on the screen.



Readjust the dials to 0.00. and click OK.

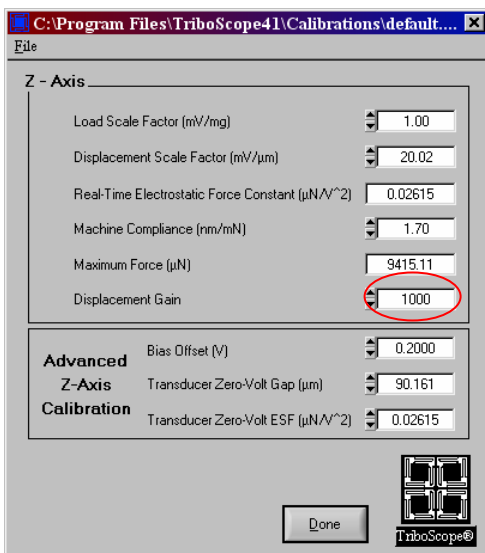
You will see Curve Fit Plot ESF vs. Displacement screen. If the blue line is fitted straight up. You have successfully finished calibration.

(Sally, I will add bad example if I luckily get one in next revision)



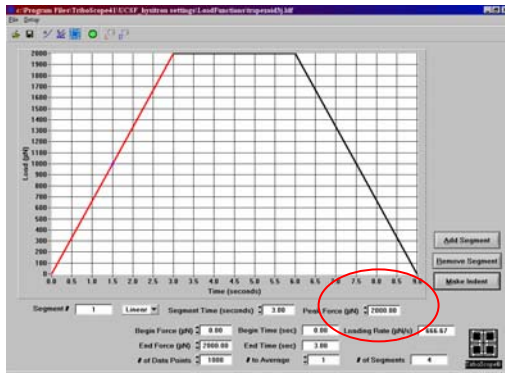
New values were automatically imported to the setting. Close “Advanced Z-Axis calibration” window.

Before starting experiment go back to setup → Calibration. Change Displacement Gain back to 1000. Change Output 1 Gain and Output 2 Gain to 1000 on the Hysitron Box.




5. Indentation to fused silica standard

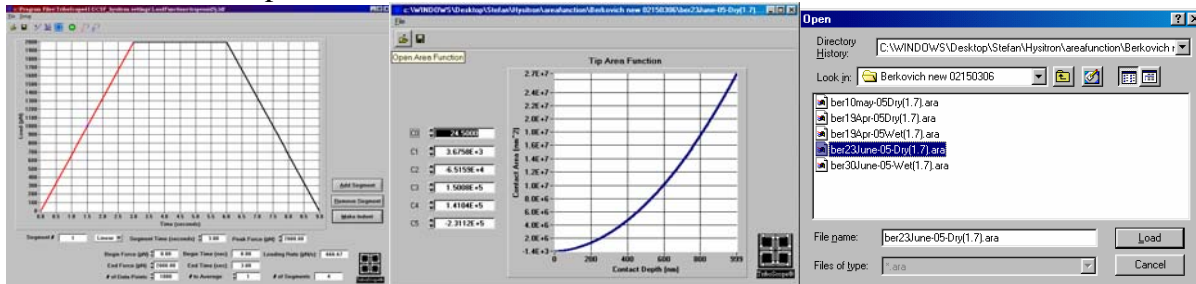
- Click “open load function” icon from “set up indent” screen, choose “trapozoid3j.ldr” to be ready to make real indents.
- Type in the value for the “Peak Force” (usually we start with 8000 μN).



Change Peak Force to 8000

6. Select an Area Function File (calibration file)

- From Set up indent screen, click “Define Area Function” icon 
- Click “Open Area function” icon



- Select an area function file (Check tip number and wet/dry condition, right for your application) and load it.

7. Indentation to Reference

- Lower the indenter very very close to the surface of the fused silica by using lever on AFM, use binoculars for magnified view.
- Adjust Hysitron box to approximately “00.00”
- Make sure Scan Size is “0” at AFM computer
- Make sure gains are connected, i.e. different from 0
- Click Green Arrow icon to engage

If you can not engage (false engagement, z position bar drops to limit), you have 2 options to try.

Option 1: Disengage. Then increase value of setpoint, integral gain and proportional gain. (8, 8 and 8, 10, 10 and 10, instead of 5, 5 and 5). If you still can not engage with higher values, you should consider the contamination of the tip or specimen surface or the tip is broken.

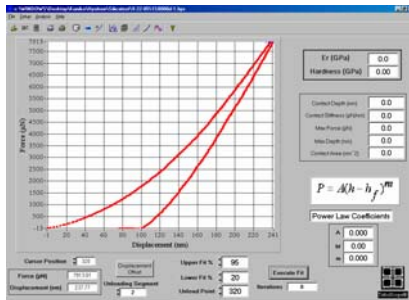
Option 2: Click “Motor” and “Step motor” and manually lower the tip until it engages correctly. (z : -20 to 20 V). To safely do this, please do following. Type “0” in setpoint box, then adjust display of hysitron box to “00.00” manually with “zero” dial. Then type the value you used to engage (such as 5) back into the setpoint box to control the force of contact, Use stepmotor function to move the tip up and down. After the z-range bar is in good position, repeat typing in “0” in setpoint box and confirm the hysitron box displays “0”, before you move on.

- Increase scan size (10 to 30 μm)
- Look for a flat clean area to make indentations.
- Offset to the area (Click offset \rightarrow execute), then change the scan size to “0”.
- Is the Hysitron computer side ready to indent? (setup indent screen is on with correct value typed in, no large drift observed in hysitron box display)
- Change integral gain and proportional gain to “0”.
- Immediately click “Make Indent” in the Hysitron side.

8. After the indentation is completed, save file screen will pop-up. Then you must immediately change the integral and proportional gain back to 5.0 (or something you are using) with the AFM computer.

9. Save the indentation file.

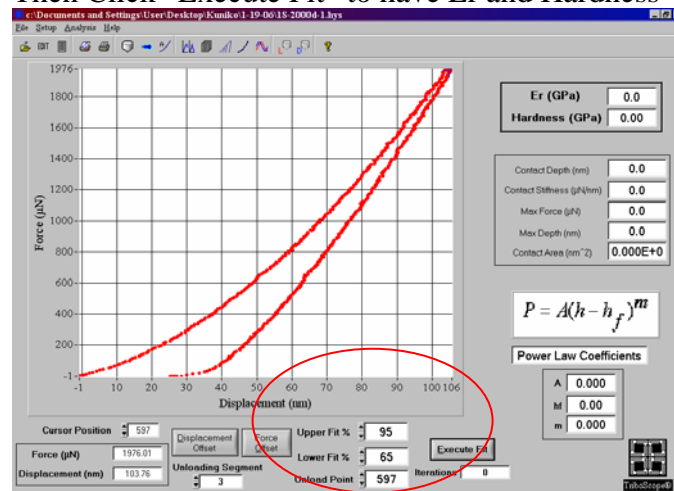
Use filename easy to identify it. Factors suggested to be included in the filename; (load, wet/dry, material), For example, if you nanoindented a dentin sample 1, with 400uN load in wet cell, you can name the file as “den1-400w-1”.



This kind of screen will appear after you save the file.

Change “Unloading segment” to “3” and “Lower Fit %” to “65”.

Then Click “Execute Fit” to have Er and Hardness value calculated.



You have successfully completed one indentation now!

Accepted Er and Hardness values on standard (Silica glass) are

Er: 69.6 (65-75) GPa,
H: 9.5 (8.5-10) GPa

Important!! Please record the values in Log Book.

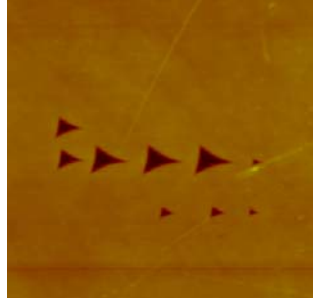
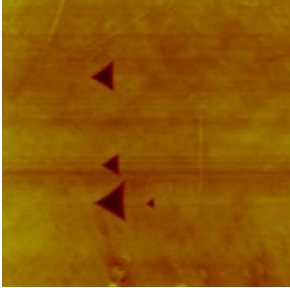
10. Repeat indentations on standard with smaller peak forces such as 4000, 3000, 2000 to confirm the system is calibrated and working properly.

- You do not have to disengage the tip if it is working happily at the right z-limit (-20 to 20)
- Use X or Y offset window in AFM PC to manually move the tip to a new position.

Berkovich tip has a triangular pyramid shape, if you indent with a large load or have a soft sample, you will have a large indented and deformed area. In these cases, make sure to move the tip far enough from the adjacent indents. On silica standard, separate indents at least $5\mu\text{m}$ with more than $5000\ \mu\text{N}$ load.

11. Scan the area you've indented to evaluate the shape of indents after finishing the series.
 - Increase Scan Size in AFM PC by 10 to $20\ \mu\text{m}$.
 - Indents should be equilateral triangle shape for Berkovich indenters

Example of good indents



Example of elongated indents

12. Disengage the tip by clicking red arrow in AFM PC
13. At the end of the experiment please do following procedure to make sure everything is working in good condition for your colleagues.
 - Clean the tip (Follow the instruction)
 - Make an indent with $8000\ \mu\text{N}$ peak force to the fused silica
 - Save the file on "Daily Log" folder on desktop
 - Record the values in blue log book.
14. Indentation of your real sample.
 - Follow the same protocol.
 - Record "Set Point" value, "Area Function" you used in your lab notebook.
 - Indent depth around 150nm is ideal.

Sample preparation hints

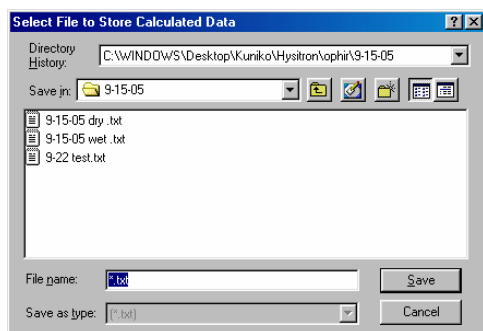
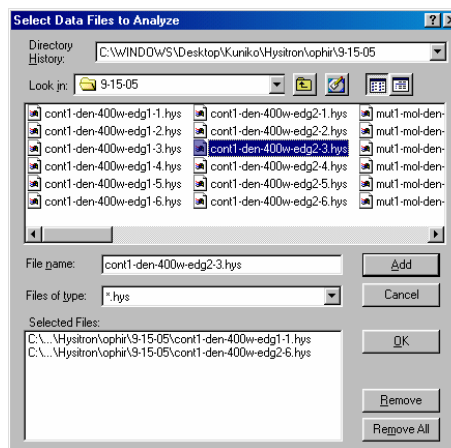
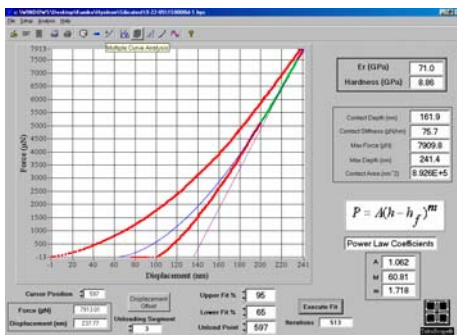
- Height should be less than 2.5 mm, more than 0.5 mm
- Surfaces are polished parallel.
- Sample has to be glued to the metal stub using Cyanoacrylate adhesive (RDS Inc). You cannot use double stick tape. (You end up with measuring modulus of the tape)
- Be aware if the glue infiltrates or crawls up the sides of your sample and alters mechanical properties. (use minimum amount of glue. Sample has to have enough thickness after final polish after gluing)

Indentation using wet-cell

- Prepare a plastic ring from a clear test tube
- Make a thin rope of blue periphery wax
- Assemble sample and ring with periphery wax, so the wax seals the entire ring.
- Add liquid in the wet cell using dropper or syringe. Depth should be half of the length of indenter but should not touch the transducer.
- **Transducer and Piezo scanner are not waterproof. They can die if you wet them accidentally. This experience was costly.**
- Perform indentation on fused silica reference in wet cell.
- Use Area function for wet measurement.

Transferring the data to text or excel format

1. Select right “Area Function file” for the data executed and exported.
2. Click “Multiple curve analysis” icon. Add files to be exported, click OK.



3. Then, name the file and save. Hysitron software will automatically execute fit the area function and save it in text format, which you can open from Microsoft Excel.

Cleaning the tip.

Please get training if you are not familiar with indenter cleaning.

--- How to clean indenter ---

1. Turn off hysitron box
2. Dip Kim-Wipe strip or Q-tip in acetone or ethanol, gently wipe the tip of the indenter. Acetone can be found in yellow flammable cabinet in big lab(Rm.2201)
3. Rinse with DI water, gently dry with canned air
4. Screw back to the transducer and turn on the hysitron box

Calibrating a tip (Making new area function file)

See Appendix C

Trouble shooting

1. Indent shape is not equilateral triangle.
 - a. Indenter was bent!!
 - b. AFM scan rate and angle is making false elongation of image. → change scan angle and rate
 - c. Indent was not done correctly e.g. false engagement, set point etc.
 - d. Sample surface is not perpendicular to the tip
 - e. Tip is contaminated.
2. Value of Hysitron box keeps drifting in wet-cell measurement.
 - a. Possible leakage of liquid!!
 - b. Air bubbles are trapped on the indenter or sample surface
 - c. System has drifted, test for drift at scan size = 0, put current set point = 0, Hysitron display should show 0.0; if different, zero the system and put current set point back to its initial value (probably 3 and higher)
3. Cannot get good image
 - a. Tip may be contaminated. → Disengage and clean the tip
 - b. Integral gain and Proportional gain values are incorrect.
 - c. Scan rate is not correct
4. Transducer making funny buzz noise.
 - a. Board and transducer tend to oscillate if you have been working for long period. → Disengage the tip. Turn off the Hysitron box then shutdown the computer. Let the AFM have a nice break.

Appendix A. Materials currently used

- Cyanoacrylate adhesive (RDS Inc)
- Periphery Wax (Surgident, Heraeus Kulzer Inc.)

Appendix B. Currently used system

- Multimode AFM Nano Scope III (DI instrument)
 - Nanoscope software 5.12r3
 - Piezo Scanner : 4875jv
- Tribo Scope (Hysitron)
 - Tribo scope software v4.0.0.0
 - Sensor bias setting : -0.0320(V)
 - Machine compliance: Unique for each tip.
1.7 for black shaft Berkovich tip, 0.8 for white Berkovich ceramic shaft tip.
 - Berkovich indenter, 142.3°, 100nm tip radius
- On the transducer information sheet
 - Transducer ID: S/N5-060-68
 - Z axis
 - Tare: -348mg
 - Load Scale Factor (Force): 1mV/mg
 - Displacement Scale Factor (Deflection): 20.015mV/ μm
 - Electrostatic Force Constant: 0.0278 $\mu\text{N}/\text{V}^2$
 - Self Calibration Check: 476 mg
 - Maximum Force: 10.008mN