

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

MTS Nanoindenter

Manager: Dr. Dmitri Barbash
Senior Research Scientist

Contact: Drexel University
Bossone 112
3141 Chestnut Street
Philadelphia, PA 19104
Office: 215-571-4611
Mobile: 267-918-3188
dvb36@drexel.edu

Latest Update: 2020-06-22

These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

1. Once you arrive at the nanoindentation room, sign the log book and fill in all of the pertinent information in regards to the tests that you will be running (tip type, size, advisor, # of indents, etc).
2. Start your reservation using iLab Kiosk to activate the Nanoindenter computer.
 - a. Click on the Nanosuite icon. No password is needed on the window that pops up after clicking the icon and so you can just click OK.
 - b. Choose method - XP and select the appropriate testing method for the tests you will be performing. If unsure of which method to use, select CSM, standard, hardness, modulus, and tip cal.
3. Inserting samples (there are two screen modes: schematic of sample holder and microscope. Right click to switch. Schematic = nano handset; microscope = nano video handset):
 - a. On schematic mode, right click and select initialize, then click OK.
 - b. On the machine, unscrew (counterclockwise) until it gets tight, then pass that and it will loosen again. At that point, the screw can be removed and the stage can then also be removed.
 - c. Position the samples on the block, take the block back to the stage, and tighten the screw (clockwise).
4. Changing the indenter tip:

For more MCC SOPs please visit our Training Library at <https://www.drexel.edu/core-facilities/facilities/material-characterization/resources-for-users/training-library/>

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

- a. Click on the 'Tip' tab and select 'Change Indenter Tip'.
 - b. Follow the instructions on screen.
 - c. Remove tip from indenter (clockwise).
 - d. **Pins in - change indenter - pins out.**
 - e. Go to 'Tip' tab and select the tip type that you just inserted.
5. Tip and microscope calibration (for Berk, 1 μm , or 5 μm use SiO_2 calibration sample; for anything larger than 10 μm use Al calibration sample)
 - a. Left click on calibration sample.
 - b. Right click on screen and select move to target.
 - c. Right click on screen and select video handset. (If screen is too dark, increase voltage. It should be around 5.0V)
 - d. Use up and down tabs to focus.
 - e. Find a clean spot to put the calibration index on. Move around on the sample by clicking on the screen.
 - f. Right click and select microscope to indenter calibration.
 - g. Click on advanced settings and set # of indents to 5, distance from center: 50, depth: 2,000, then click next twice and wait for calibration to complete.
 - h. Click on central indentation.
 - i. **Never move out of screen to find indentations. If they can't be found on screen, cancel calibration and call/text/e-mail Instrument Manager. Remember to check and make sure that the pins are not in the indenter tip.**
 - j. Click finish.
6. Moving to samples:
 - a. Right click and select nano handset.
 - b. Left click on sample.
 - c. Right click where sample is and select move to target.
 - d. Right click and select nano video handset.
 - e. Focus the microscope.
7. Program:
 - a. Click on batch mode tab (6th tab) and select define tab.
 - b. Go through all steps.
 - i. Select batch to run:
 1. Select batch - nothing to be done here.
 2. Select option for this batch:
 - a. Uncheck first three

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

- b. In the 4th one (delay before running), select use default start time and enter the desired time.
- ii. Create/edit a sample:
 - 1. Create sample - enter name.
 - 2. Surface find parameters - Allowable drift rate (nm/s): 0.05 (if there is less concern for drift rate then put it at 0.1 or 0.2 nm/s)
 - 3. Surface approach parameters - nothing to be done here.
 - 4. Required input (depth depends on your sample and tip size/strain rate: usually 0.1/2.0/45.0/Poisson's ratio of your material).
 - a. Depth: at least 10 times less than the thickness of your film
 - b. Depth: less than half of the tip radius:
 - i. $1\mu\text{m} \sim 400\text{nm}$
 - ii. $5\mu\text{m} \sim \text{less than } 2\mu\text{m}$
 - iii. ...
 - 5. Define/review test locations: either do it manually and define a few locations or you can define an array of tests by selecting the first tab:
 - a. For example:
 - i. $x = 5$ (number)
 - ii. $y = 5$ (number)
 - iii. $x = 15$ (distance)
 - iv. $y = 10$ (distance)
 - v. angle: 10, click OK.
 - iii. Select next, add another sample (click 'Next Step') and then repeat procedure for additional testing of samples.
- 8. After test:
 - a. Go to review tab.
 - b. Go to the 'Tip' tab and click on select tip, and select Berk.
 - c. Right click on the graph, select y-axis channel, and then click modulus.
 - d. Adjust the depth in each sample (flat line).
 - e. To go to the next sample click on the second tab, select advanced, samples folder, select sample.
 - f. Click on the 9th tab to convert the data collected to an exportable Excel file.