Nanoindentation

Available in both air and liquid. The overall procedure is similar to regular measurements with some dedicate calibration on the system and probe's properties

The Z sensor is assumed to be accurate in this section (whose calibration is introduced in the next section). The deflection sensitivity is calibrated based on Z sensor and a very hard surface (Young's modulus 100 folds of the probe). During deformation, almost all the movement in Z direction is going to be the deflection. This sensitivity is a property of the system, meaning that if the laser is adjusted or the media (air, water) is changed, the value will be different. Based on the experience, if just adjust the laser a little bit, the value is not going to change more than 5 %, which can be assumed identical. With deflection sensitivity, the spring constant is calibrated by thermal tune. This is a property of the probe only. Changing the media and other setting may affect it and causing more than 10 % of changes, but it is fine as long as the method is kept consistent. There is a third parameter, tip radius. This is calibrated separately. It's a property of the probe only. If calibrated in liquid, the tip is abnormally sharp. We better calibrate that in air regardless of air or liquid probe. It's executed by scanning slowly on a rough, sharp sample and using the software to calculate based on the indentation depth. It's more of a contact radius rather than a tip radius. There are other calibrations which we normally don't perform

1. In air
    a. Similar to 'Regular Dry AFM', align the laser and find the sample surface. The sample should be the sapphire sample in the standard sample box. Keep those sample as clean as possible. Sapphire surface might be hard to recognise, just like glass surface. Look for scratches to locate the surface. If the reflection is clear in tip reflection view, it means the head is too low. Rise it up a bit for a clear view in sample
    b. Engage. No need for larger scan size. Once some scans area is available, use offset to find a place without contaminations and go to ramp. Set the trigger threshold to 0.5 V and perform 2 – 3 ramps. Open the files. Correct the baseline and select the indentation region. Find 'Commands > Update sensitivity' to acquire the values and average by calculator. The value should be 60 – 70 nm/V, but currently we have been getting 100 nm/V. The Bruker technician can't find the reason
    c. Open thermal tune window. Enter the sensitivity value and hit 'acquire data' (in air). Then select the region with the peak and fit data. Later, calculate k. It should be 0.35 – 0.4 N/m (marked as 0.4).
    d. Place the sapphire sample back and take the rough, sharp sample out. It has a really good surface so finding the surface is very easy. Make sure adjust the values based on Bruker's document (page 17/35). Need to use extensive mode for more adjustable parameters
    e. Save the image and analysis in the software. For spherical model, tip radius is required. The maximum indentation depth should be the tip radius. Tip radius is a function of indentation depth. Thus, try a few values until they are very similar. Use that value as the calibrated result (new probe < 10 nm)
    f. During measuring, make sure the modulus from spherical and conical models are both turned on. Sample per line is 256 or the software can't calculate so fast.

Set the scan rate to 0.2 – 0.4 Hz. Set Peak force frequency to 1 kHz for better result.

g. Capture a line and find the indentation curves. Export them and make sure the indentation depth is around the tip radius. If not, a just the max force. Few nN is reasonable

2. In liquid
    a. Calibrate the tip radius in air first as it's inaccurate in liquid. Wet and dry is not ideal although it's better to calibrate the tip radius after indentation on a hard surface. It's always a trade off
    b. Similar to in air but in liquid. The sensitivity is ~ 25 nm/V. Fit a good flat line in finding spring constant in liquid (double check the mode). It should be 0.6 – 0.7 N/m (marked as 0.7).
    c. Sometimes in liquid requires larger peak force amplitude. It can go as large as 300 nm. Always auto configure for better force curve. 2 × 2 μm is a good scan against drift in liquid
    d. Sometimes drift can be really serious. We haven't figure out a good way to supress it yet

Calibration in general

Every 6 month, the Z sensor needs to be calibrated. The process uses contact mode and a standard sample with pits marked with known depth and spacing

Basically, it requires aligning the sample to the horizontal direction and scan a certain pit. Measure the depth and check for difference. If within 2 %, then the senser is fine. Otherwise, the sensitivity needs to be updated. The height and height senser need to be calibrated together. Generally, height senser is thought to be more accurate

We use open loop calibration in our system. It's a simplified method from the help document. There is a piece of piezoelectric ceramics (short as piezo) that controls the height of the probe holder. Perform the calibration at -50 V, 0 V and 50 V of piezo voltage. By default, it is around -50 V. This means the head is a little low. The piezo retract a little bit so that the probe is not pushing too hard on the surface. This is preferred. By move the set motor, different value can be acquired