Bruker Multi-Mode 8 AFM

Rochester Institute of Technology

Department of Chemistry and Material Science

SOP prepared by Hayley Richardson on July 23, 2019

I. Purpose
To promote the effective use of the Bruker Multi-Mode 8 AFM to establish an instrumental method.

II. Scope
This SOP is intended for in-group use by trained and certified personnel in the Chemistry Department

III. Prerequisites
The experimenter must be trained in proper instrument techniques before using this SOP.

IV. Responsibilities
The responsibility for this instrument lies with Tom Allston
Room: 08-A161
Voice: 585.47.6034
E-mail: tdasch@rit.edu
School of Chemistry and Materials Science
85 Lomb Memorial Drive
Rochester Institute of Technology, Rochester, NY 14623-5603

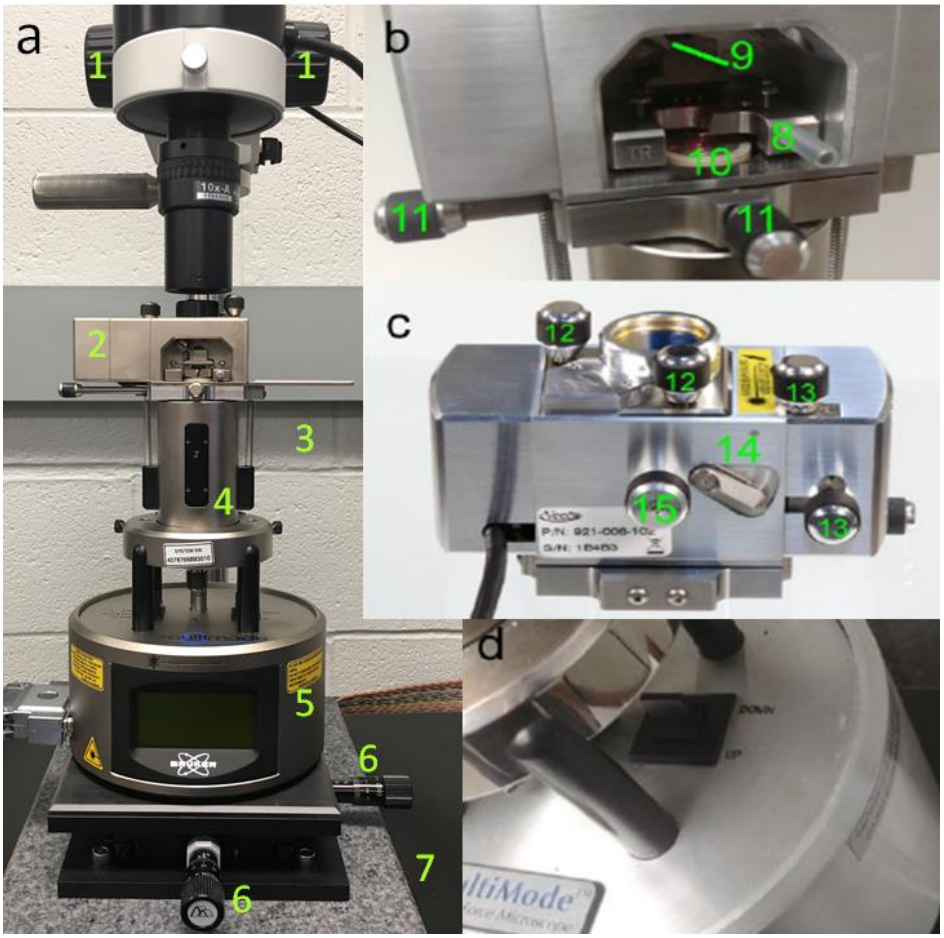


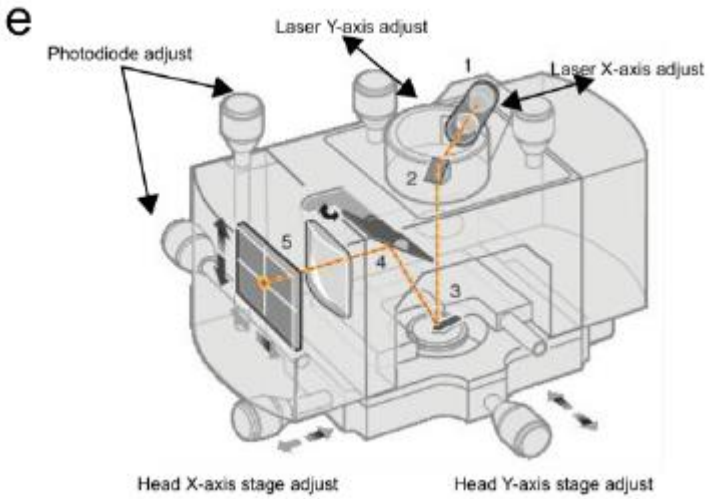
Figure 1)

(a) Bruker Multimode 8 AFM.

(b) front and (c) back of the AFM head

(d) z-scanner motor switch (up and down directions indicate tip movements relative to the sample)

(e) labeled transparent image of AFM head components.



Sample Preparation

Sample preparation will vary based on sample and substrate composition, and many other factors. General solid-state guidelines are provided below.

- Prepare a sample approximately 1cm x 1cm in size
- Samples must be adhered to magnetic pucks, using sticky tabs (similar to double-sided tape)

KEY
1. Laser
2. Mirror
3. Cantilever
4. Tilt Mirror
5. Photodetector

1 – focus knobs (for camera)
2 – AFM head (holds tip assembly and detector)
3 - Springs (secure the head on the scanner)
4- Scanner
5 – Display showing displacement of laser from detector center and laser signal
6 – Knobs to translate the entire AFM in x-y relative to the camera
7 – vibration-isolating optical table
8 – tip holder assembly
9 – mirror (to direct laser to detector)
10 – magnetic sample holder
11 – Knobs to translate AFM head (and tip) relative to the sample
12- Knobs to move laser in x-y
13 – Knobs to move the detector in x-y
14 – Lever to move mirror (9, to direct laser to the center of the detector)
15 – Knob to loosen and secure tip holder assembly (8)

Load Sample and Probe

The tip holder should always be secured in the AFM head when not in use. Please do not store on the counter.

- Remove tip/probe holder assembly **(part 8)** from instrument by unscrewing knob **(part 15)** on back-center of AFM head, and place on a table with the tip facing up
- Press down gently on the probe holder **(part 8)** to lift the clip up
- Using tweezers, insert the probe under the clip
- Make sure upper and left edge are touching the edges of the holder

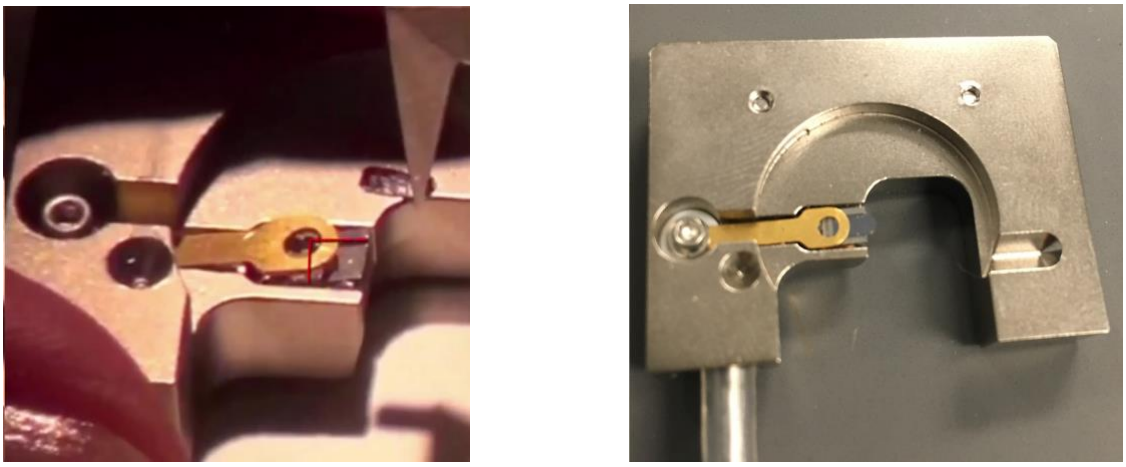


Figure 2) Tip holder assemblies for a solid-state sample.

- Push the lever at the base of the instrument (Figure 1d) in the up direction to move the sample sufficiently far down (tip up) so that the probe will not crash into the sample when the probe holder is placed in the AFM head
- Holding the AFM head **(part 2)** steady with one hand, unhook the securing springs **(part 3)**
- Place sample (adhered to magnetic puck) in the center the magnetic platform **(part 10)**

- Insert probe holder with tip facing down into scope such that pins fit on the protruding pins of the AFM head **(part 2)**, as shown in Figure 1b. Secure probe holder by tightening knob in the back-center of the AFM head **(part 15)** until the clip contacts the tip holder assembly
    - *Do not overtighten, as long as the pins touch the probe holder, it will be secure*
- Replace the AFM head in the same manner it was removed

Set Up Microscope

- On Bruker box, make sure illumination is on and the intensity is half way up
- Power on the microscope controller, which is left of the computer (switch is at the top right)
- Launch Nanoscope 8.15
- Select experiment category, e.g. 'ScanAsyst' (or Tapping Mode, Contact Mode, Quantitative Analysis, etc.), as shown in Figure 3
    - Select experiment group, e.g., Scanasyst in air (or in fluid)
    - Select experiment, e.g., Scanasyst in air (or in fluid)
- Load experiment. The optical microscope video should load immediately

Figure 3) Experiment selection: Screenshots of experiment selection for Scanasyst. *Tip,* use USER experiment if unsure what to use, or contact POC.

Align the laser

- Focus the camera on the tip by using the large black knobs **(part 1)**
- If the cantilever is not visible, move the AFM relative to the camera into the illuminated light circle using the micrometer knobs at the bottom of the instrument **(part 6)** or move the AFM head assembly (and tip) relative to the sample and camera using the knobs on the AFM head **(part 11)**
- Look for the red laser spot, and if needed, move the laser close to the tip in the x and y directions using the knobs on the top of the AFM head **(part 12)**
- **Alignment method 1**:
    - Raise camera using the focus knobs **(part 1)**, and holding the AFM head carefully, unhook the springs **(part 3)**, and hold head such that the laser spot is aimed on a sheet of paper (or your hand)
    - As shown in Figure 5, align laser such that the laser is on the tip using the two laser dials (one on right of head to move laser in x direction and one on the back to move in y direction)
    - Replace head in microscope. Look at the sum reading (want this to be greater than **6.0**)

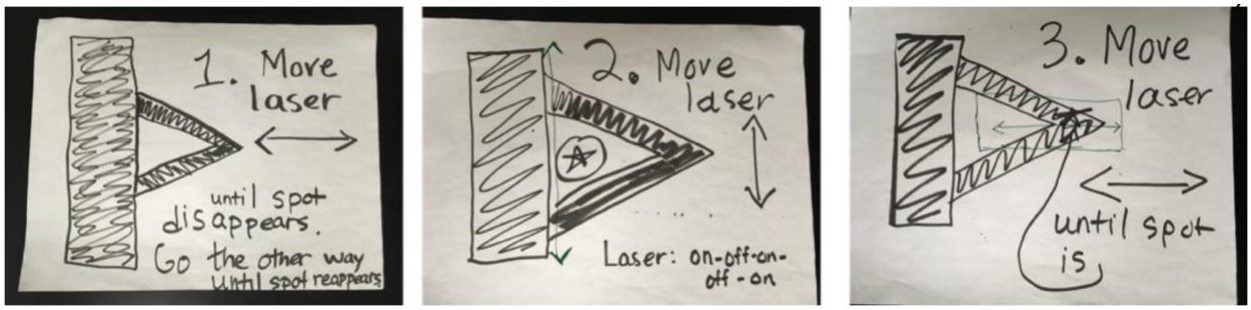


Figure 4) Tip alignment procedure, using method 1. In method 2, the same procedure can be used, except that the laser spot will be visible on the paper covering the detector when it's hitting the cantilever (whereas in method 1, the laser is not visible when it's hitting the cantilever).

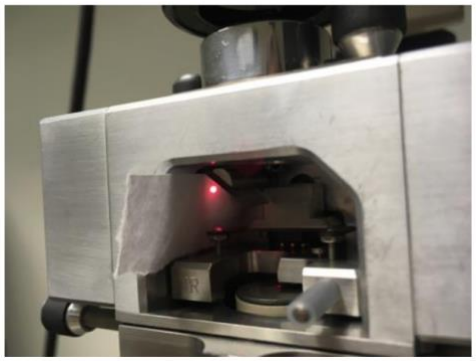


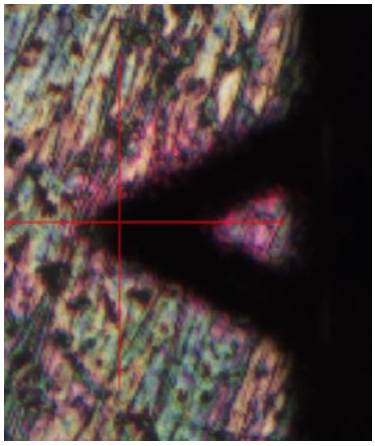
Figure 5) Photo of laser alignment using method 2

- **Alignment method 2**:
    - Slide a piece of paper in front of the detector such that when the laser bounces off the cantilever, it will be visible on the sheet of paper – as shown in Figure 5.
    - Move the laser in x and y to center it on the back of the cantilever closest to the tip following a similar procedure as shown in Figure 4, however in this method, you will see a laser spot when it's hitting the cantilever, unlike in method 1 when the laser is hidden from the paper directly below it when it's reflected off the cantilever
    - Ensure that once the spot is centered and hitting the piece of paper, that it is a round/Gaussian spot rather than elliptical or distorted (otherwise make small x-y adjustments using the knobs, **part 12**)

- This last step allowing us to ensure good laser spot shape represents an advantage over method 1 (in addition to not having to remove the AFM head).
- **Note:** if the laser is aligned and the sum (looks like a speedometer on the digital display, Figure 7) is still below 4-5, try adjusting the lever on back of the AFM head **(part 14)** to tilt the mirror, and thereby redirect laser toward the detector, to maximize the sum

Align the horizontal and vertical detectors

- Move the detector such that the laser spot hits the center of the detector by adjusting the knobs on the left/top of the AFM head **(part 13)** one at a time. This will change the numbers in the displays
- Adjust the position of the detector until one of the display values approaches 0, then adjust the other knob so that both numbers go to 0 (+/-0.1-0.3 is preferable since these numbers will change during imaging)
- If the numbers don't change immediately, it's fine – it just means the detector center is still far away from the laser spot. Turn one of the knobs all the way in one direction, then all the way in the other. At some point, a change should occur. If the detector is at the edge of its movement range and still needs to move more to bring the display numbers (indicative of the displacement of the laser spot from the center of the detector) to 0, adjust the mirror using the lever on the back of the AFM head **(part 14)** to direct the laser to a different spot.



Focus the tip

- Using the optical video on the computer screen, focus on the sample surface using the turning knobs **(part 1)**
- Next, find the tip and focus on it. Just to gauge distance. Then, focus back to surface
- Use the 'tip down' switch to move the tip slowly towards the surface until it's almost in focus. **Do not crash into the sample Figure 6)**

Imaging in PeakForce Tapping/ScanAsyst Mode (most common)

PeakForce Tapping mode combines the benefits of contact and tapping mode, and adds tremendously beneficial automation, allowing users to obtain nice images irrespective of

experience level. At each point along a line, the cantilever is moved relative to the sample until the 'peak force' is reached, and then withdrawn. As such, force-deflection curves are obtained at each point, enabling mechanical mapping in a less destructive way than contact mode.

- **Specify imaging parameters in the software.** Good initial parameters are:
    - Scan size = 1-5 µm is
    - Scan speed = 1 Hz
    - Aspect ratio = 1
    - Samples/per line = 512
        - While 512 points/line is best for publication, 256 is a great way to survey a larger area in half the time
    - Peak force Freq = **2 kHz** ***anything more will cause charging**

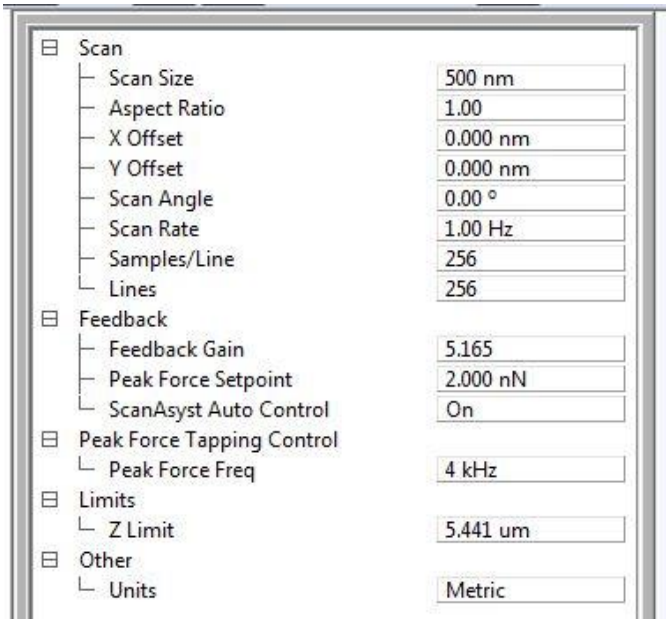


Figure 7) ScanAsyst parameters. Adjust those in the green box if desired and make sure ScanAsyst Auto Control is on; the others are adjusted automatically.

- **Engage** (this moves the tip into contact with the surface)
- The height and peak force error data are collected automatically
    - If you'd like both the trace and retrace data from each line, you'll have to collect this in separate channels (just open and select new channels)

- **Withdraw** the tip when imaging is complete, click several times

Image Capture

- Select capture directory by clicking the file button next to the text box for file name entry (top right of software window) and selecting the folder in which you'd like your data to be saved. Automatically goes to "Capture"
- Name file, and press enter
- Click capture (or continuous capture, which will keep capturing images as the sample is scanned)
- The image will be captured once the scan (from top to bottom, or bottom to top) is complete – though you can always force capture by clicking capture again
- The zoom button at the bottom of the height channel image allows you to draw a box, and then upon clicking execute, an image will be acquired in the place specified by the rectangle (or close to it – the zooming is not always exact)
- Click offset, then place the cursor at a point on the image. Upon clicking execute, a new image with the same dimensions will be acquired, this time centered on the point indicated.

Image Analysis

- Open Nanoscope Analysis, and then open the image file you wish to analyze
- **Flatten** to correct systematic errors/features, such as substrate tilt, which can often be parabolic. Click the flatten icon. 2nd order flattening is a good starting point. Select no thresholding, the whole image. Click execute
- **Set data scale** by right clicking on the image color bar, and click color scale in the window that appears, click the modify data scale tab and set the max and min of each image
- **Save image** as a jpg, by right clicking on the image, select export, then screen display
- **Section analysis**: Use this feature to obtain line profiles (e.g., height vs. x-y dimension)
    - Click the section analysis icon and draw a line to generate a profile, which can be exported as txt files and plotted in origin.
    - It's a good idea to also save an image with the section lines drawn, so you know at which point on the image you took the line profile.