

# Step-by-step instruction for using the Digital Instruments Multimode AFM

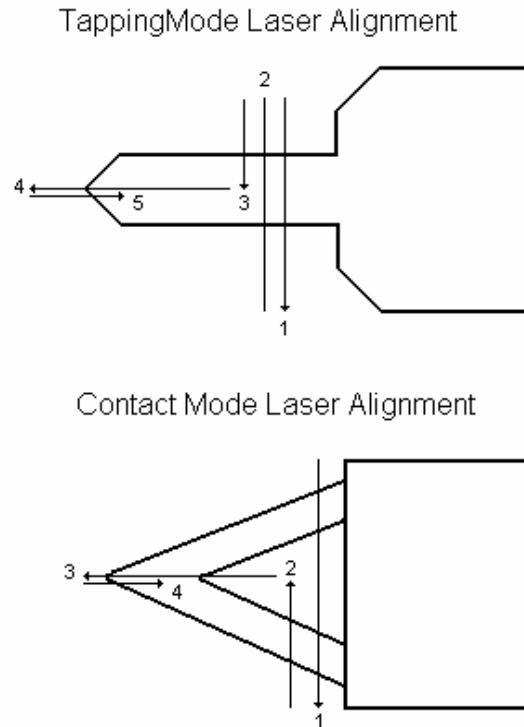
## I. Sample Preparation and related issues

- (a) sample size: sample diameter is approximately 1 cm in diameter and a thickness of not more than 2 mm
- (b) mount your sample on the magnetic metallic disks with double-sticky tape, glue or epoxy
- (c) if you need to clean surface, use isopropanol and dry it with the air gun gently
- (d) Consider the size of the features you are interested to see versus the probe tip (this will affect your resolution of what you can see. Always make sure the probe is smaller than the feature of interest. If the surface has smaller features than the tip, the surface may scan the tip instead!)

## II. Setting up the Microscope, Sample and Tip

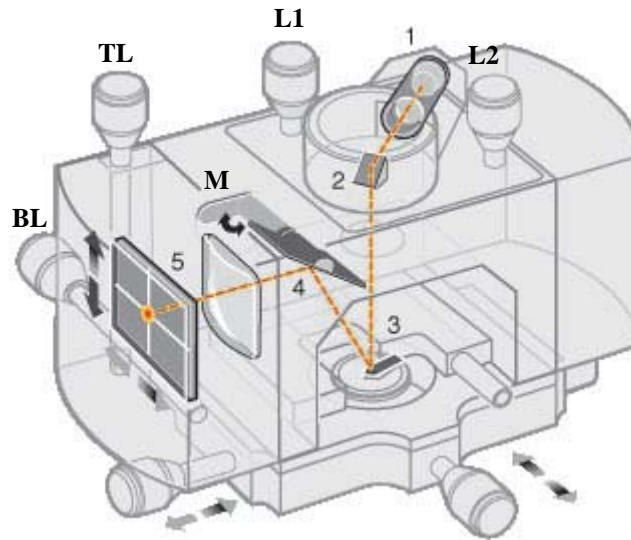
1. Switch power on for the laser and scanner (located at the back of the controller), turn on optical light source and monitor.
2. Align the optical light circle in the middle of the stage and the red laser light spot in the middle of the optical light circle, using micro movement stage controller at the bottom of AFM base.
3. *LOADING SAMPLE ON THE SCANNER.* Put sample/holder onto scanner. The strong magnetic force will hold it tightly. Focus the microscope on the sample surface. Using tweezers to move your sample until you find an area that you want to scan. (**Note:** Keep an eye on the objective lens as you focus on your sample – you may crash on one of the Laser alignment knobs, L1 & L2 on Figure 2 below.)
4. Adjust sample surface position to level with the bottom line of the optical head window using “**UP**” toggle switch that is located below the tube scanner on the right side. This ensures a safety gap of about 2 mm between tip and sample surfaces.
5. *MOUNTING PROBE INTO THE CANTILEVER HOLDER.* Put the tip holder upside-down and then press it down onto the table so that the yellow spring pops up. Insert the cantilever/tip assembly into the tip holder with the tip face up. Make sure that the chip aligns correctly under yellow spring and fits into the end of the groove.
6. *LOADING THE CANTILEVER HOLDER.* Put the cantilever holder back into the optical head. Make sure it's lying correctly and secure the holder by tightening the middle screw at the back of the optical head (do not over tighten).
7. *GETTING THE TIP CLOSE TO THE SURFACE OF THE SAMPLE.* Bring the sample up using “**DOWN**” toggle switch that is located below the tube scanner on the right side. (**Tip 1:** focus on the sample then move it up until you see the probe slightly in focus.) Stop when you can see the tip nearly in focus. It is helpful to go up

and check the focus of the cantilever from time to time to see how close it is to the sample surface. (**Tip 2:** If you are looking at a shiny or clear surface make sure you are not looking at the reflection or shadow of the tip, it is best to use the edge for focusing.)



**Figure 1.** Laser Alignment diagram. (Ref. <http://www.mse.mtu.edu/~jwdrelic/AFMGuide.pdf>)


8. **LASER AND TIP-SAMPLE ALIGNMENT.** Make sure that the mode switch, located below the tube scanner on the left side, is on the AFM&LFM or TM AFM. Align the laser beam at the end of the cantilever (use Figure 1 as a guide), using the two positioning knobs on the **top right side (L2 & L1)** of Figure 2) of the optical head. Check the Sum signal on the elliptical meter on the bottom LCD is at its maximum signal (4.8 – 7.2) - this value is dependent on the reflectivity (i.e.; the coating at the back of the cantilever) of the back of the tip. If the signal is low adjust the mirror (lever on back of optical head, **M** of Figure 1) until you achieve the maximum signal to center the laser reflection into the photodiode cavity. **Tip:** If you are unable to reach the maximum intensity of the laser on the elliptical meter, check if you are focused on the reflection of the cantilever instead of the cantilever itself.



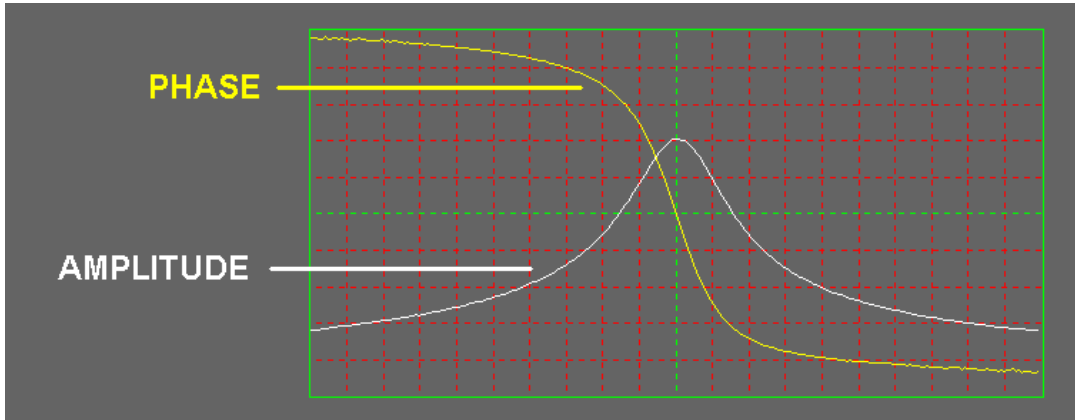
**Figure 2.** Optical head.

9. **ADJUST PHTODETECTOR SIGNAL.** Switch to TM AFM mode. Rotate **Top Left** screw (**TL** of Figure 1) on the optical head until the Vertical Difference is close to 0 (any thing between -0.5 to 0.5 is acceptable; this is the digital number reading on the SUM window. As you do this make sure the signal of the laser i.e. the SUM signal on the elliptical meter remains at its maximum. Switch to AFM&LFM mode and then rotate **Back Left** screw (**BL** of Figure 1) on the optical head until the Horizontal Difference is close to 0 (between -0.5 -0.5 is good). Again make sure the laser signal remains at its maximum during this procedure.
  
10. Switch back to TM AFM mode and double check if the vertical difference is still close to 0. If not repeat step 9 until there is no change in the readings of the Horizontal and Vertical Differences.

### III. Procedure for Scanning in Tapping Mode

1. Check if the SPM is on TM AFM mode.
2. Start NanoScope SPM program. Wait for 20-30 sec. Nanoscope control and NanoScope image windows will show up on the left and right monitors.
3. Click real time icon (Scope icon). And then click Microscope ⇒ Profile, select Multimode Tapping AFM.
4. Click Microscope ⇒ Scanner, select the exact scanner you are using (3417jV).
5. Open Auto Tune icon  on the top bar. Click **Auto Tune** bar in Auto Tune Control window. Execute the auto tuning and wait until Auto Tune finishes. When the Auto Tune is finished; you should see a clear resonance peak on the amplitude graph. The Phase

(bottom) graph should take an “S” shape (as shown below). If you get error message then check the laser alignment. Make sure the frequency range covers the frequency of the three tips on the cantilever/tip assembly; if you have changed the drive amplitude, repeat the auto tuning. Click on the “**Back to Image Mode**” button to go back to the Multimode Tapping AFM window.



- Set up the scan parameters: use the following starting values and change as needed. Make sure the X and Y offsets and scan angles are set to 0.

Scan Controls:



Scan size:	start with 1-5 $\mu$ m;
Scan rate:	start with 1-2Hz;
Samples/lines (resolution):	start with 256
Slow scan:	Enabled

Feedback Controls

Integral Gain:	~0.4147
Proportional Gain:	~2.144

Channels 1-3

Data Type:	at least height in Ch.1
Data Scale (Z- range):	select for good contrast

- Double check if the Horizontal and Vertical Differences remain close to zero and that the laser intensity is at its maximum before you engage the tip. Engage tip by clicking the engage tip icon (). The AFM will go through some precautionary procedures and then it will engage on its own. Wait until scanning image will appear on the right screen. Make sure the Vertical difference (as adjusted in II.10) remains as close to zero as possible. If not, disengage the cantilever and repeat procedures II.9-10. And then engage again.
- Optimize all parameters. Click on the scope mode icon () on the top bar of NanoScope control window. You'll see a graph of z-position versus X – Y positions. The white and yellow lines represent the trace and retrace of the tip as it is rastered across the sample. Since the difference in space between the trace and retrace is small, these two



should closely agree, so the white and yellow lines should overlap to get a valid AFM image. You can switch between image mode () and scope mode () to check how the change in parameter is affecting the resulting image. If the white and yellow lines do not overlap, follow this procedure (refer the Table for typical range of values for scan parameters):

Table. A quick reference to typical values for Scan Parameters  
(Ref. <http://nanonet.rice.edu/manuals/afm.html#opt>)

Parameter	Definition	Typical Value	Range of Values	Effect on Image Quality
Setpoint	The value of the RMS of the cantilever vibration amplitude that the feedback loop maintains. (Setpoint is thus proportional to force applied to surface)	2.00 V	1.00 V - 5.00 V	Reducing setpoint often leads to better quality images
Drive Amplitude	The amplitude of the force at which the cantilever is driven ( $F_0$ in Eq. 6)	50 mV	30 mV - 1.00 V	Increasing drive amplitude often gives better phase data, up to a point
Proportional and Integral Gains	Determine how sensitive the feedback loop is to variations in the tip's amplitude of oscillation	0.400/ 4.00	0-0.600/0- 6.00	Increasing gains often helps obtain better images (especially height) but only up to a point, above which high frequency noise is observed
Scan Rate	Controls the rate at which the cantilever scans across the sample area	2.00 Hz	1.00 Hz - 4.00 Hz	A slower scan rate generally leads to better images, but not always. Decrease until you get the best image
Number of Samples	The number of pixels used to create the image	256	128-512	Increasing leads to better image quality, but there is a trade-off with time

**Decrease the Set Point.** Do this until the image improves or until you reach 1.0 V using the left arrow key (←).

**Increase the Drive Amplitude.** Often times there will be a significant amount of strong noise. Increasing the Drive Amplitude will take care of this. Once you have a good picture, back off on Drive Amplitude as much as you can without coming off the surface.

**Change the gains.** High values of gain may cause high frequency noise, so it is often necessary to reduce their value, keeping them in approximately a 1:10 integral to proportional ratio. However, the gains should be kept as high as possible, since they control how fast the cantilever will respond to changes in topography.

**Change the scan rate.** If reducing the gains does not eliminate noise, this can help. Scan rate values that are too low as well as too high will cause poor images. You should try a range of values while in image mode to obtain the best one.

**Adjust the Z Range.** Although this can be changed later (e.g., in Flatten), it is often important to have the most contrast available while you are imaging. Thus, you should set the Z Range in such a way that the scope mode picture just fits within the Z Range.

**Note.** *If you have an especially rough sample, your data scale for height (on one of the channels) should be fairly large. However, if it needs to be bigger than 1  $\mu\text{m}$ , you may try a profilometer first.*

#### **IV. Procedure for capturing image**

To capture an image, first go to Capture option on the top bar of Nanoscope Control window and choose Capture. From the same column choose Capture “file name” and give your image some name (choose your folder in drive d). Start a clean scan from top or bottom of the scan area using the “up” or “down” icons. Then immediately press the camera icon to start capturing an image. On the bottom bar of the Nanoscope control window you will see the message “**Capture: On**”. When it changes to the message “**Capture: Done**”, you will be able to find your image in off-line mode files. To change windows setting to off-line mode, click the multi-colored icon at the top right corner of the NanoScope control window.

#### **V. Analyzing Captured Image**

1. Click on the offline icon (wavy rainbow all the way on the right corner).
2. All captured images need to be flattened. To do this, select a channel; Image Menu  $\Rightarrow$  Select First/Second/Third image  $\Rightarrow$  click on the Flatten icon (rolling pin)
3. For More details on the many options of image analysis, please refer the “Image Analysis” folder.
4. Images will only be viewable using the Nanoscope software. To view using a graphics program, you must export your file to a .tiff file. To do this,  
select desired file  $\Rightarrow$  select Utility Menu  $\Rightarrow$  .tiff export (save it in reverse colors to get rid of the black background).

#### **VI. Ending your session**

1. Disengage the tip by clicking on the red up arrow button.
2. Raise the tip some more by using the “**UP**” toggle switch that is located below the tube scanner on the right side.
3. Turn off the light source and the laser and scanner- switch located at the back of the controller.
4. Remove the cantilever holder.
5. Remove your sample.
6. Copy all your files and sign the look sheet. Include any notes on how the AFM performed, especially if anything was not working properly.
7. Close the NanoScope SPM program.

## Scanning in Contact Mode

- (a) Start with procedures I and II above – they are the same for the Tapping mode and Contact mode.
- (b) Check if the SPM is on AFM & LFM mode.
- (c) Start NanoScope SPM program. Wait for 20-30 sec. Nanoscope control and NanoScope image windows will show up on the left and right monitors.
- (d) Click real time icon. And then click Microscope  $\Rightarrow$  Profile, select Multimode Contact AFM.
- (e) Click Microscope  $\Rightarrow$  Scanner, select the exact scanner you are using (3417jV).
- (f) Set the Deflection set point  $\sim$ 1-2V.
- (g) Then adjust the feed back parameters as in the Tapping mode.

**Note:** \* Scan angle of  $90^0$  is recommended so that to avoid twisting the tip

\* It is also good idea to choose at least one of the channels to show friction image as it can provide some information about the surface of the sample.

## Trouble-Shooting

(Ref. <http://nanonet.rice.edu/manuals/afm.html#opt>)

Here are a few problems you may encounter in the course of doing AFM.

1) *AutoTune will not work.*

- Make sure that the tip holder has been secured in the AFM.
- Make sure that your tip is not in contact with the sample. If so, do a tip up, and the tip may still be OK.

2) *Sample seems to move when lowering the tip.*

This probably means that you have placed the sample in such a way that it is coming in contact with the tip holder. Rotate the sample so that any part of it that is hanging over the piezo-tube are coming towards you (out of the microscope).

3) *Getting poor images.*

- Try optimizing parameters again.
- Realign the optics, making sure that you get the laser as close to the end of the cantilever as possible and are getting as much light to the photodiode as possible
- Be sure that the laser is as close to the end of the cantilever as possible and that you have a clear spot.
- Try another area of the sample or change samples, especially if you are not seeing the kind of image you expected to see. Do not waste your time with a bad sample.
- Change tips, but don't throw away old tip. The tip might be salvageable.

4) *Tip will not engage.*

- Make sure that something is not interfering with the tip reaching the sample. This impediment may be as small as a big ridge on your sample
- Check that you still have a cantilever. If you try to engage with no cantilever the AFM will simply drive what's left of your tip into the sample