Step-by-step operation instructions for DI Nanoscope III Multimode SPM Contact mode instructions followed by Tapping



Assembling the Multimode SPM

- 1. Power on the computer and the controller (switch in back right). The LCD displays should light up.
- 2. Mount a cantilever chip into the tip holder (either one) under the spring clip as shown in the picture. View under the benchtop optical microscope to make sure the chip is square with and fully tucked into the rectangular pocket of the cantilever holder, butted up against the wall of the pocket.
- 3. Mount a sample on a magnetic SPM puck with double-stick tape (clear or carbon, the latter stickier if needed; this may be better done >1 hour earlier, to avoid drift as the tape relaxes). Make sure the sample and tape do not extend beyond the perimeter of the puck. Mount the sample on the top of the scanner.
- 4. Holding it via the peg, put the tip holder (flipped over with tip pointing down) into the SPM head.
- 5. Lower the clamp via the knob on the back of the optical head to secure the holder (the screw should be finger tight, but not over torqued)
- 6. Having ensured that the scanner housing is elevated sufficiently (with UP motor control switch) to prevent crashing the tip into the sample, place the head on the scanner housing such that the ball contacts atop the latter are seated in the grooves of the former. Secure by connecting the retainer springs to the pins on the sides of the head while securing the head with one hand. Plug the head's electrical cord into the connector at back right of scanner support ring.
- 7. Visually verify that the chip and spring clip do not touch the sample. This could happen if there is a significant tilt of sample surface upward from left to right. (Always mount such that the surface tilt is upward right to left if present.)

8. Turn the head XY-translation knobs so the sample is centered within the hole in the bottom of the head.

Positioning the Multimode SPM

- 9. Place the base-scanner-head assembly on the Nano-k floating platform. Check the white indicator on the black vibration-isolation platform to verify that the platform is "freely floating". If not, adjust the platform via the crank (to account precisely for the SPM weight). Normally, only a small number of turns would be required. The need for adjustment derives from the variable weight of the Multimode depending on scanning size, head type (Triboscope transducer replaces Multimode head), or the use of a cover (to provide acoustic or electromagnetic shielding).
- 10. Bring the tip closer to the sample by holding the **motor control switch** DOWN. To view cantilever and sample while doing this, turn on the light source of the overhead optical microscope and the monitor. (There is a peg on the right side of the microscope to toggle between eye pieces and monitor viewing. You may need to find the black zoom knob on the left side, and zoom all the way out.) The focal plane of the microscope is shallow; initially the sample surface or the cantilever may be focused, but not both. Adjust the focus (use the biggest knob, of three, first if the microscope lens is far away from the optical head) to find the cantilever. Two long silver knobs (microactuators) on the translation stage where the base sits may be used to move the cantilever in the view field. The distance between the optical microscope lens and the knobs atop the head is usually less than 0.5 cm when cantilever is in focus (may depend on how low the sample surface is).
- 11. Turn the focusing knob to focus on the surface (look for some dust particles if you have a clean and flat surface). Turn the knob half way back so that the focal plane is in between the cantilever and the sample surface. Hold the **motor control switch** down until the cantilever comes into focus. Repeat this process until the cantilever is close to the surface, meaning nearly the same focal plane (say within ½ turn of the finest adjustment knob).
- 12. You may turn the **head X-axis and Y-axis stage adjust** knobs by roughly ± 1 mm to position the tip over a surface location of interest, but recognize that large offsets may result in the sample touching the head if not within the perimeter of the puck (i.e., hanging over).

Configuring the optics



- 13. Plug the cable from the SPM **head** into the back-right top of the **base**. The laser should be on. Find the laser spot in the optical view monitor. Use the **Laser X-axis adjust** and **Laser Y-axis adjust** knobs to move the laser spot position near the free end of the cantilever.
- 14. Slide a piece of paper in front of the detector window and adjust the Laser Y-axis adjust knob until the reflected laser spot is seen on the paper; you may need to also lower the mirror tilt with the lever at the back of the head. Further adjust the laser position along X and Y such that it is centered on the symmetric axis of the cantilever but spilling off the end (such that a symmetric pattern of laser light is seen on the paper, ideally a "V" or "X" for wedge-or V-shaped cantilevers, respectively, or a vertical fuzzy band for more purely rectangular cantilevers. Finally adjust the Laser X-axis adjust knob to move laser spot away from the free end of the cantilever (i.e., to the right) until only a pristine laser spot (oval shaped) is seen on the paper slip (and steepen the mirror tilt if adjusted earlier, such that the laser is clearly entering the detector window. The above laser spot positions are depicted below for V-shaped cantilevers.



- 15. Turn the top **photodiode adjust** knob until the elliptical bar graph **signal sum** is a maximum (white numbers, between 3 and 4 on SPM1 and around 6 on SPM2); further adjust the same knob such that the top digital indicator [(A-B)/(A+B), deflection] reads approximately 0 volts (within a couple tenths). If unsuccessful, coarse adjustment of the mirror angle may be required (via lever on backside of head). Adjust the back **photodiode adjust** knob such that the bottom digital indicator [(C-D)/(C+D), lateral force] reads approximately 0 volts. Some back and forth between knobs may be needed.
- 16. For <u>contact mode</u>: change the top LCD number to -0.3, then go to the next step. For <u>tapping mode</u>: flip the **mode selector switch** to "TM AFM" (left switch, forward position). Adjust the top **photodiode adjust** knob to zero the bottom LCD number (now the deflection signal, whereas the top LCD will be amplitude).

Start the control program

- 17. Log onto the controlling PC workstation
- 18. Double click **1** on the Windows desktop
- 19. Click \blacksquare menu (upper left corner of the window); choose Microscope select \rightarrow Extended Multimode.
- 20. Click the video button \blacksquare to go off line
- 21. Click (the capture directory)
- 22. Click 🗁 ᡰ 🔪
- 23. Hit the Delete key on your keyboard
- 24. Type *.* and click OK to clear the capture directory.
- 25. Click the microscope button \bowtie in the upper right corner to go to the real time operation window.
- 26. Click Microscope menu \rightarrow scanner \rightarrow match the scanner name from the list to the S/N number found on the label of the scanner.

- 27. Click Microscope menu \rightarrow Profile \rightarrow load the profile for the mode (contact AFM or tapping AFM).
- 28. Choose the **Capture** menu to select a root file name for all captured data.

Parameter settings and engaging surface in contact mode (alternatively skip to #25 for tapping)

- 29. Choose settings as shown in the following window snapshot. If imaging very smooth surfaces (like Si wafer), reduce the **Z** limit from its maximum value (6.072 microns in the shown example) by at least a factor of four to provide finer digitization of height.
- 30. Recheck the top LCD number and make sure it's still \approx -2 before approaching the tip, and then click the green arrow button $\boxed{\textcircled{}}$ to approach the tip.



After tip is engaged in contact mode

- 31. Sample starts scanning automatically after engagement. Click is to go to the force curve mode to adjust the imaging force. All XY movement ceases and feedback is disabled.
- 32. Adjust **ramp size** (Z approach-retract cycling range, e.g., 200 nm in graph below), **Z scan start** (offsets the Z cycle closer to or farther from the tip), and **Graph Scale** (range of deflection measurement) to obtain a force curve akin to that below. If <u>Z scan start is grayed out and thus not editable</u>, disable Triggering via the top setting in the rightmost panel. If only a white horizontal line at the bottom of the window appears, increase the Graph scale. If only a nearly horizontal white line appears but farther up into the window, increase the Z scan start until contact is achieved. Adjust **deflection setpoint** to set desired force for imaging; decreasing will result in a lesser vertical separation between the setpoint line (horizontal green dashes) and the horizontal (pre/post contact) part of the force curve; this provides for more delicate imaging.



33. Click icon to capture the force curve as data, as a record of initial tip-sample adhesion (tip sharpness) and setpoint during imaging. You will need to revisit the force curve more frequently to further offset the setpoint (to accommodate thermal drift) and record changes in tip sharpness or contamination. Click is to go back to imaging.

Tapping (AC) mode: cantilever drive settings

- 34. Click the turning fork button
- 35. Go to Sweep Controls panel, Set Sweep Width to 150kHz, Drive frequency 300kHz, Amplitude

setpoint 2.5. Set Data scale of Channel 1 window to 5V, and of Channel 2 to 180.

> Go to Nanoscope Image window (black), and click Offset at top of screen. Position the green vertical offset line exactly on the tallest peak of the white curve (the amplitude curve at resonance), and click Execute to move the resonance to the center of the Sweep window.

> Come back to Sweep controls panel, decrease the **Sweep width** to 1 kHz, and re-center the resonance using Offset. Adjust **Drive Amplitude** until the resonant peak has the same height as the Sweep window. This corresponds to roughly 75 nm (i.e., *amplitude sensitivity* \approx 15 nm/V).

> Go to Channel 2 window and click **Zero Phase** to offset the initial phase lag (yellow phase curve automatically offset by program). This removes extraneous contributions to the raw phase signal, such that a phase lag of 90° remains at resonance. (Proper phase lag scale is 0° at the top of the window and 180° at the bottom.)

> To save the cantilever sweep display as a "fingerprint" of your cantilever, double click A on the Windows desktop and follow instructions (alt-F12 to capture active window). Note one may capture to clipboard or save to disk.

> To work in the <u>repulsive regime</u>, place offset line \sim 4 divisions to the left of the resonant peak, click Execute. To work in the <u>attractive regime</u>, place offset line \sim 4 divisions to the right of the resonant peak, click Execute. Decrease Drive Amplitude such that the resonant peak is 4 divisions high (2 V = 30 nm).

Parameter settings and engaging surface in tapping (AC) mode

Click ^C icon to go back to the previous window. Copy parameters from the above window snapshot for contact mode, except use the following. If imaging very smooth surfaces (like Si wafer), reduce the Z limit from its maximum value (6.072 microns in the shown example) by at least a factor of four to provide finer digitization of height.

Scan controls:	Scan rate 1 Hz
Feedback controls:	Integral gain 0.2, Proportional gain 0.2
Other controls:	Engage setpoint 0.9
Channel 2:	Data type: Amplitude



- 37. Sample starts scanning automatically after engagement (sudden decrease of amplitude by 10% following slower decrease due to changed air damping). Increase **amplitude setpoint** until engagement is lost, and then decrease slightly to lightly re-engage with A/Afree ratio of 0.98-99. This is a lighter tip-sample interaction that the initial engagement setpoint, but tracking rugged topography will be more difficult.
- 38. You may alternatively click in to go to the force curve mode (amplitude vs Z and phase vs Z graphs) to adjust the **amplitude setpoint**. All XY movement ceases and feedback is disabled. The tapping mode profile has been configured such that one must click on the button with the zig-zag up/down icon to initiate Z cycling; before doing this, it is wise to decrease the Z scan start. The reason is that one must be very careful not to approach to the point that the cantilever oscillation is zero and the (very stiff) cantilever statically deflects, exerting huge forces on the sample and tip. Usually in this case the tip becomes contaminated, blunted, even bent or broken. Force curves in tapping mode can be rich in physical information and useful for assessing interaction regime (next section), but one must exercise great care by increasing Z scan start in small increments until the amplitude downturn is seen, and decreasing if the curve drifts rightward (sample upward, say due to tape relaxation).

Stabilizing repulsive or attractive regime under tapping (AC) mode

- 39. To determine which regime tip is being operated, go to scope trace $\neg \neg \neg$ set **Realtime planefit** under Phase Channel to "none", and set **Data scale** to 180°. If all phase signals are in the top (bottom) half of the window, one has stabilized the repulsive (attractive) regime.
- 40. To change from repulsive regime to attractive regime, decrease Drive amplitude (say by a factor of two), correspondingly decrease amplitude setpoint (by same factor) and/or increase drive frequency (monitor the phase signal change under scope trace when making adjustments).
- 41. Click ^[65] to go back to imaging.

Contact and tapping (AC) mode: optimizing tracking and capturing images

42. Click Scope trace icon Adjust gains under Feedback controls to minimize the deflection (contact mode) or amplitude (tapping mode) signal: increase both integral and proportional gains but not so far as to cause feedback oscillations. For surfaces that are very rough or containing ledges, also increase the

look-ahead gain. Click to go back to imaging. View preceding set of scan lines to see the effects of the above gain variations.

43. Click icon to start capturing an image. Click either of the icon icons to start a new scan at bottom or top. Changes of parameters during image capture will postpone the capture until the subsequent full raster scan, unless you click icon at least one more time to force the capture (also useful if you intend to save the changes in the image as a function of parameter settings like setpoint).