

Dimension 3100 (D3100) SOP

Standard Operating Procedure for D3100 Tapping Mode Operation

General D3100 Procedure

- 1) Turn on the D3100 (Figure 1) using the power strip located at the bottom left corner of the D3100 vibration isolation table (Figure 2).

WARNING: Do not power cycle the computer with the AFM turned on, as the resulting voltage spike could cause severe damage to the piezos. *If the computer is turned off, turn on the computer before turning on the D3100 power strip.*

NOTE: Allow laser to warm up for ~30 minutes prior to imaging if your experiment requires very precise measurements.

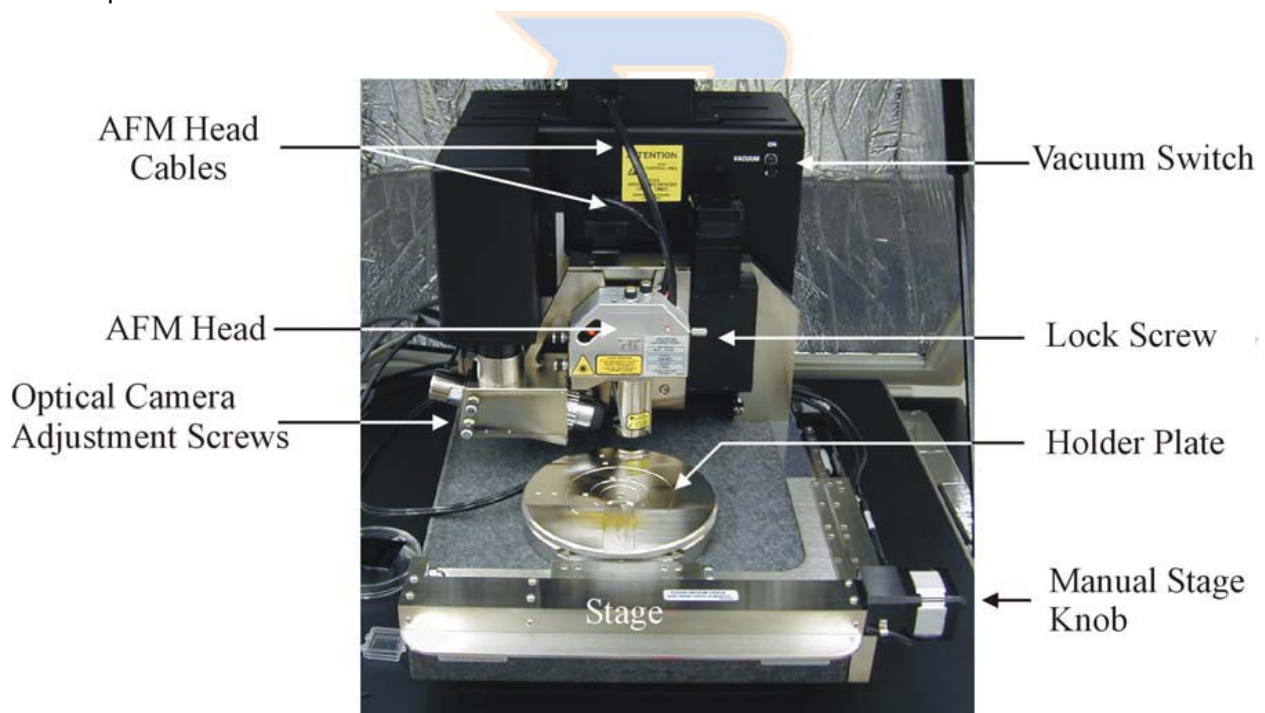


Figure 1. Basic components of the D3100 AFM.



Figure 2. Photo showing the location of the power strip for the D3100 AFM.

- 2) Log in on the Excel sign in spreadsheet located on the computer desktop.
- 3) Open the AFM control software by double-clicking the Nanoscope V6.13R1 icon on the computer desktop.
- 4) Once the software is open, click on the microscope icon (Figure 3) in the upper lefthand corner of the Nanoscope software window to begin Realtime setup.



Figure 3. Microscope icon for starting Realtime setup.

- 5) Once Realtime startup is completed, choose your imaging mode and initial parameters as described in the following steps:
 - a) On the menu bar select **Tools**→**Select Miroscope**→**D3100**→**OK**.
 - b) Select **File**→**Open Workspace** and open **default_tapping_d.wks**.
 - c) Select **Acquire**→**Meter**.
 - d) Select the Scan Parameter List in the left column of the toolbar.
 - e) Set initial (default) parameters:
 - Set initial **Scan Size** to zero to allow optimization of setpoint and gains prior to scanning, thereby increasing the longevity of the tip and minimizing damage to the sample.
 - Tip velocity is proportional to **Scan Rate** and **Scan Size**. Optimal **Scan Rate/Tip Velocity** will vary depending on the sample, scan size, and mode of operation, but 1 Hz is a good default starting point for the **Scan Rate**.
 - **X and Y offsets** should be set to zero prior to engaging to eliminate any applied voltage to the piezos and reduce the possibility of damage during the engage process.
 - Select the appropriate **Microscope Mode** (typically **Tapping**) based on the desired experiment type.

- Enabling **XY closed loop** engages a feedback loop that reduces the amount of drift between the anticipated engage point and the actual engage point. However, the closed loop X sensor on the D3100 doesn't work, so **XY Closed Loop** will need to be disabled.
- **Integral and Proportional gains** are initially set to 1 and 10, respectively. The optimal gain values to ensure the tip tracks the surface will vary based on the topography of the sample. If the gains are improperly set, the image quality will suffer (see Step 12).
- An **Engage Setpoint** of 1.0x is a good compromise between minimizing damage to the tip/sample and the probability of a false engage. This value can be increased to 1.1 for a "softer" engage, but it will increase the probability of a false engage. Decreasing the value to 0.9 will prevent a false engage, but may cause premature dulling of the probe.

Parameter	Initial Value
Scan Size	0 μm
Scan Rate	1 Hz
X Offset	0
Y Offset	0
Microscope Mode	Tapping, Contact, STM (choose mode you wish to use)
XY Closed Loop	OFF
Integral Gain	1
Proportional Gain	10
Engage Setpoint	1.10

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- 6) Mount a probe on the AFM head.
- a) Mount a probe holder on the probe holder mounting block as shown below in Figure 4. Be careful not to bend or break the contact pins on the probe holder mounting block!

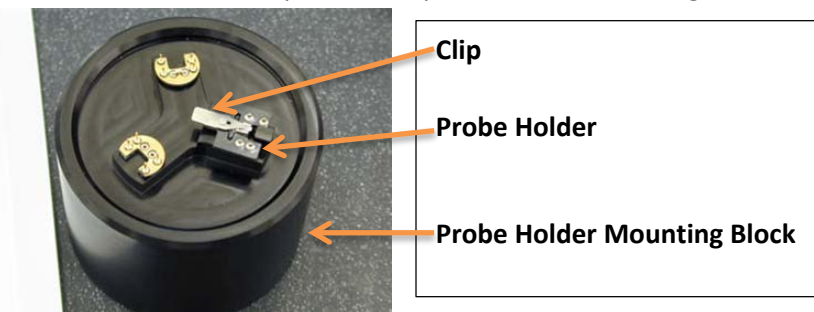


Figure 4. Probe holder mounting block with 3 probe mounting stations.

- b) Gently place a probe on the probe holder and secure the probe with the metal clip (Figure 4). Ensure the probe is parallel to all edges of the channel and not touching the rear of the holder by observing under the optical microscope in the SSL. Gently manipulate the probe with a pair of tweezers as necessary to achieve optimal alignment of the probe in the probe holder.
- c) Unlock the AFM head by turning the locking screw (Figure 5) **clockwise** (i.e., to the **right** – the **opposite** of a normal screw – the locking screw has a left hand thread).

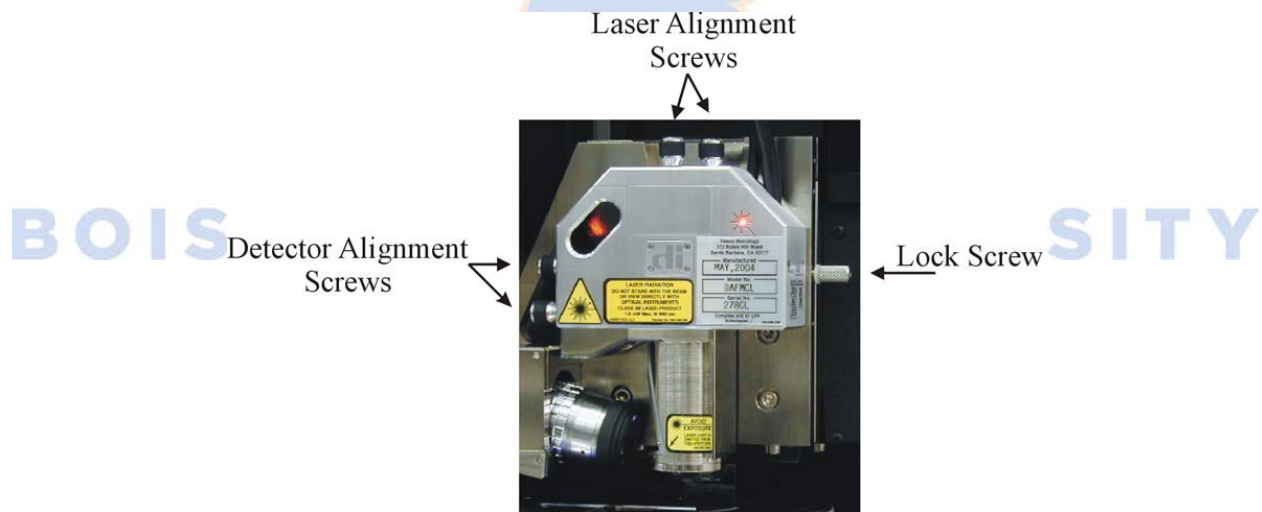


Figure 5. Photo of the D3100 AFM head showing the location of the locking screw and alignment knobs.

- d) Carefully remove the AFM head by gently sliding it up the dovetail rail holder. Install the probe and probe holder (Figure 6), taking great care to align the holes on the probe holder with the contact pins on the AFM head.

WARNING: Handle the AFM head with extreme care! Any impact to the head may cause severe damage to the piezo tube, which is **very** expensive to repair/replace. Be careful not to bend or break the contact pins!

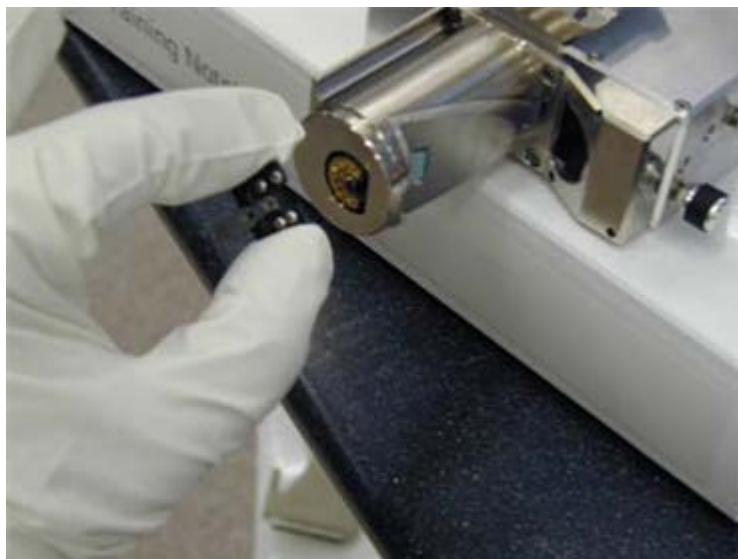


Figure 6. Installation of the probe holder on the D3100 AFM head.

- 7) Align the laser onto the probe cantilever and into the position sensitive quad detector.
 - a) Click on the photodiode meter icon to open the laser alignment window.



- b) While holding the AFM head, aim the laser onto a sheet of paper to view the beam (Figure 7).



Figure 7. Alignment of the laser onto the AFM probe cantilever.

- c) Use the laser alignment screws (Figure 5) to adjust the laser until it just hits the end of the probe cantilever (red laser spot will appear split on the paper), then maximize the photodiode SUM (Figure 7 & Figure 8) by moving it left/right and forward/back.

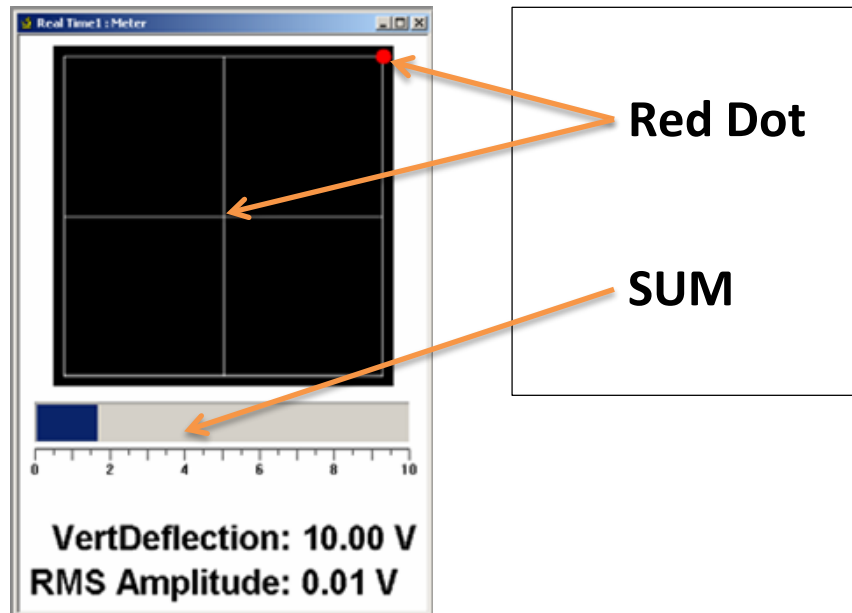


Figure 8. Position sensitive photodiode signal screen showing beam location and signal strength (SUM).

- d) Gently reinsert the AFM head on the dovetail rail holder and turn the lock screw to the **left** (opposite of a normal right hand thread screw) to secure the head (Figure 5).
 - e) Adjust the position of the red dot (Figure 8) using the detector alignment screws (Figure 5) until centered on the crosshairs. At this point the VertDeflection should be approximately zero (0.00 +/- 0.05 V) and the sum (bar graph) should be in the range of 4-5 Volts depending upon the type of probe used (probes with an aluminum backside coating are more reflective than bare silicon).
 - f) Iterate maximizing the sum and zeroing the VertDeflection as necessary to achieve optimal laser alignment.
- 8) Load your sample on the sample stage (wafer chuck).
- a) Load sample on position 1 (Figure 9). Note that the stage can be manually rotated for ease of loading the sample.

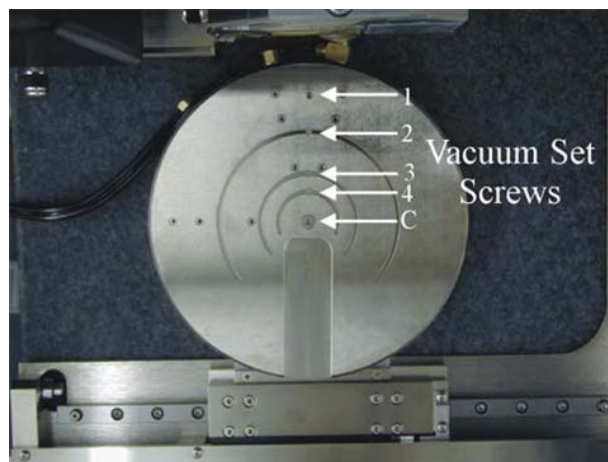


Figure 9. D3100 wafer chuck sample stage.

- b) Turn the vacuum switch to the ON position (Figure 1).
- c) The sample is now secured in place, and the stage can be rotated to position the sample under the AFM probe and head. If needed to ensure sample clearance, the probe tip height can be increased using the Z motor, Figure 10.

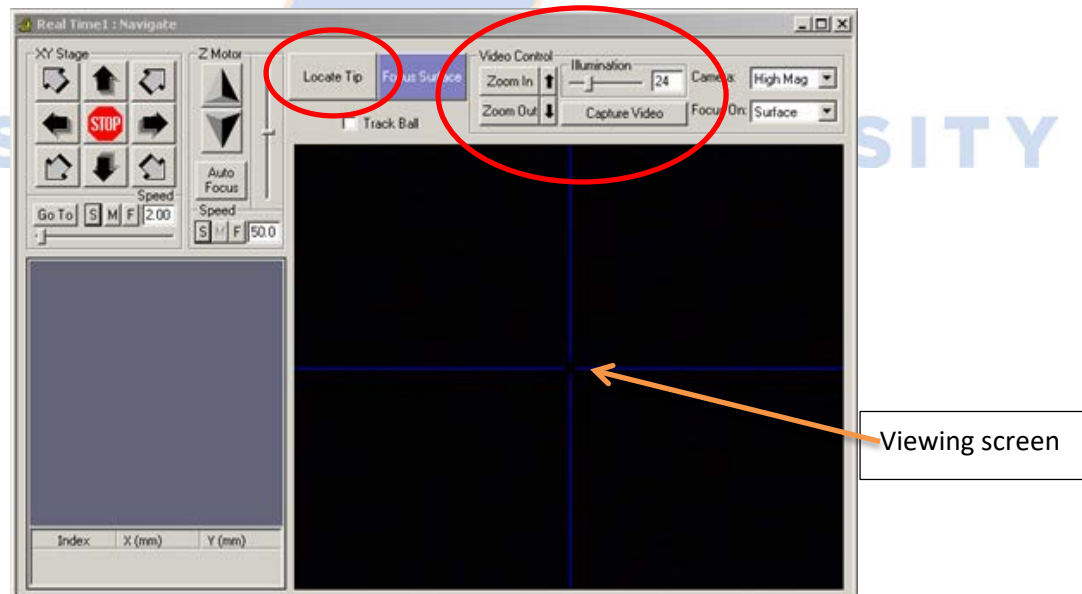
WARNING: Rotate the sample chuck slowly and visually check that the probe tip and head are well clear of the sample to ensure the tip and/or sample are not damaged. Use caution when adjusting tip height using the Z motor; set speed to "S" (slow) initially to reduce the risk of damage to the head, probe, and sample. Remember, the piezos located in the AFM head are fragile and expensive to replace!

- 9) Prepare the instrument for imaging.
 - a) Click on the Navigate icon to open the corresponding window (Figure 10).



- b) Click the Locate Tip button (Figure 10, upper panel). The cantilever and tip should now be visible in the viewing screen.

Note: Adjustments can be made to optimize the view by zooming in or out on the video control, as well as increasing or decreasing the illumination.



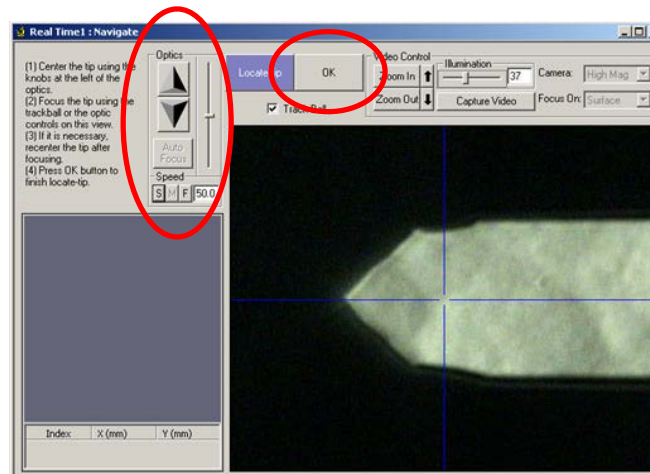


Figure 10. Nanoscope Realtime Navigate's "Focus Surface (top panel) and "Locate Tip" (bottom panel) viewing screens and controls.

- c) After the tip and cantilever are located (Figure 10, lower panel), center the tip on the crosshairs using the optics adjustment screws located on the left of the camera (Figure 1). Focus the optics on the tip using the Optics Up/Down arrows in the software (Figure 10, lower panel).
- d) When done, select OK (Figure 10, lower panel).
- e) Next click "Focus Surface" and move the Z motor (Figure 10, upper panel) until the surface is in focus.

Note: You may choose to focus on either the "Surface" or "Tip Reflection". Tip reflection mode should be used when the surface of the sample is reflective and/or highly transparent (such as mica on a metal puck). When opting for "Tip Reflection" mode, the surface will come into focus when the probe is ~ 2 mm away from the surface; continue to move the Z-motor down until the tip comes into focus. At this point, the probe will be ~ 1 mm away from the sample surface.

WARNING: Use caution when focusing on the surface using the Z motor. Always move the tip **away** from the surface initially and check the speed. Set speed to "S" (slow) and 5% initially to reduce the risk of damage to the head, probe, and sample.

Note: When the surface is in focus, the cantilever and tip will be ~ 1 mm from the surface.

- f) Use the XY Stage direction arrows (Figure 10, upper panel) to move the sample around until the area you want to scan is visible beneath the crosshairs.

10) Tune the tip (Tapping Mode only).

- a) Select the “Tune” icon on the tool bar located at the top of the screen.



- b) Set the “Start Frequency” and “End Frequency” (Figure 11) to the low and high ends, respectively, of the recommended frequency range for the selected tip (see back of probe box). Good defaults to start with are 500 mV Target Amplitude, 5% Peak Offset, and 400 mV Amplitude Setpoint
- c) Click the “Auto Tune” icon (Figure 11) at the bottom of the screen. The probe cantilever will now be automatically tuned. The resulting resonant peak should be a single, relatively narrow Gaussian-like peak, but may be somewhat asymmetric. Pick an Amplitude Setpoint value (pink line) that is slightly below the tuning curve’s amplitude (blue trace) at the auto-selected Drive Frequency (green line), but not so close that there will be a false engage. The lower the amplitude setpoint, the harder the probe will engage the surface.

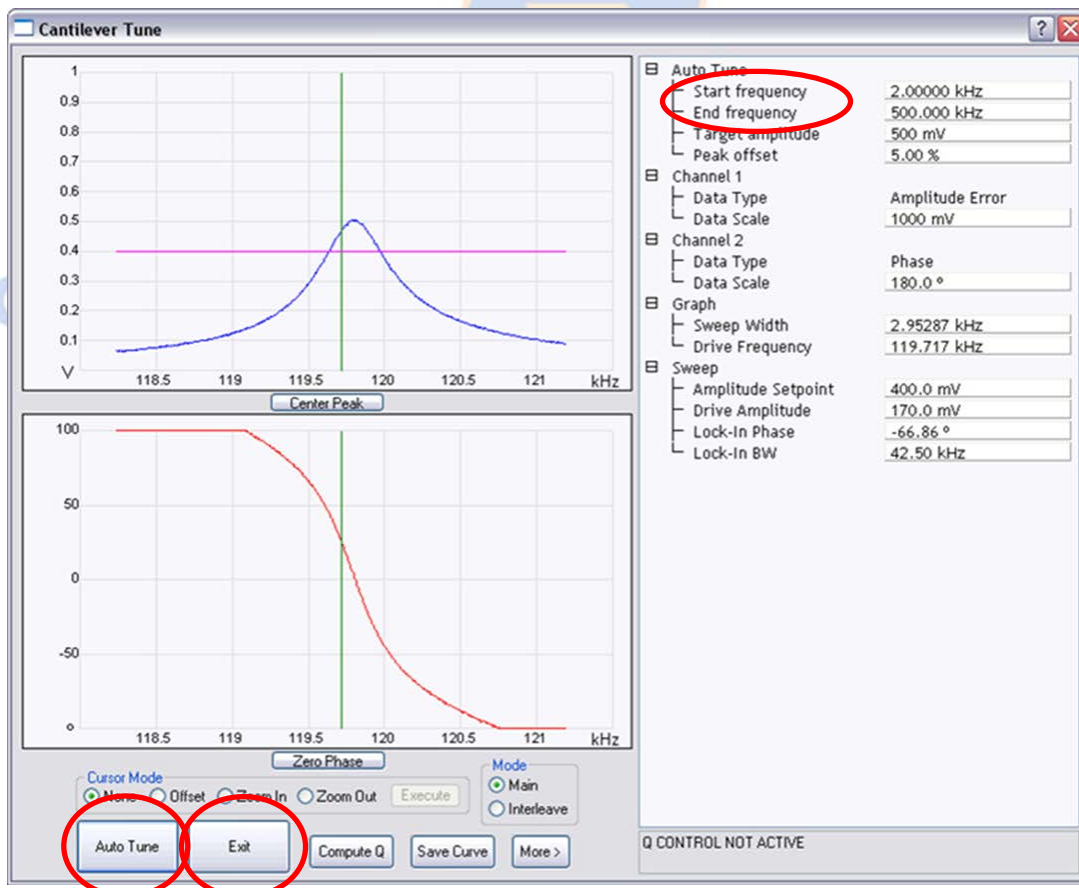


Figure 11. Probe cantilever tuning window with Start/End Frequency and AutoTune/Exit circled.

- d) If the Auto Tune doesn't return a reasonable looking peak (i.e., shape, amplitude, and frequency), increase the Sweep Width (range of frequencies visible in the tune window) until a peak appears. Then use the Offset function (located above the Auto Tune icon) to manually move the green line ~5% down the left side of the peak. (It may be necessary to lower the Sweep Width back down to enlarge the curve enough to set an appropriate Offset and Setpoint.)
- e) When tuning is complete, click the "Exit" button located at the bottom of the window.

Note: The Amplitude Setpoint should always be set to a reasonable value between 0.3-0.4 V if tuned at the recommended default Target Amplitude value of 500mV.

11) Engage the surface.

- a) Close and lock the acoustic hood enclosing the AFM by turning the latch clockwise.
- b) Check the Engage Settings. Click "Tools", then "Engage Settings". If running a tapping mode scan, select "Tapping" and ensure the parameters are set as shown in Figure 12 below ("Tapping Engage"). Select "OK". Next select "General" and ensure the parameters are set as shown in Figure 12 below ("SPM Parameters"), in particular the Sample Clearance (1 mm or 1000 μm) and SPM Safety (100 μm).

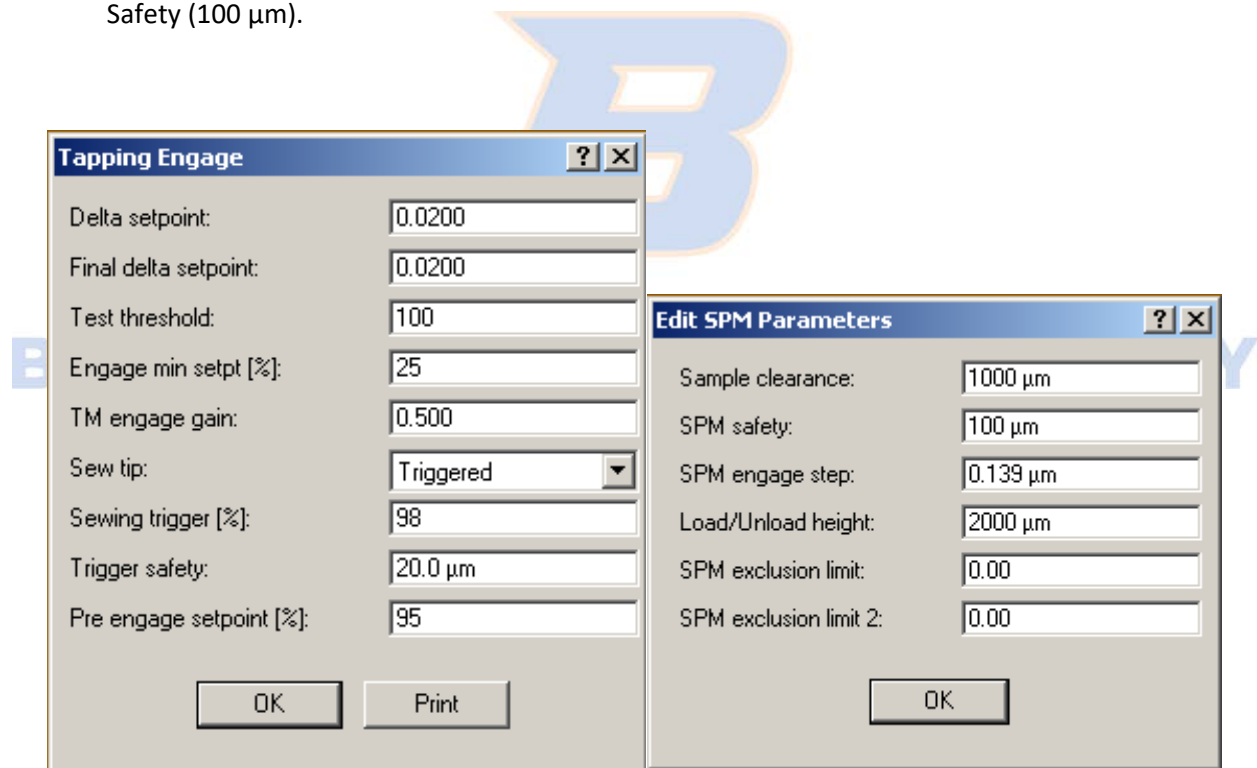


Figure 12. Tapping (left) and general SPM (right) engage settings.

- c) Click "Acquire", then "Scan-Single" to bring up the piezo monitor (Figure 13). Keep this in view at all times while imaging.
- d) Click the Engage icon shown below.



- e) Monitor the engage process to ensure the tip engages properly with the surface (i.e., make sure the tip doesn't crash into the surface or you experience a false engage above the surface).

Note: During the engage process, it is good practice to keep the navigation screen open to view the probe as it approaches the surface. If at any time the laser flashes or the tip appears to shatter, this is a good indication that the probe has crashed the surface. If this happens, the engage should be aborted **immediately** by clicking the Abort button.

- f) Once the probe has successfully engaged the surface, the system will beep. The piezo monitor (shown below in Figure 13) should be **GREEN**. It may instead turn **RED**, indicating that the Z-piezo is fully extended or retracted and the maximum voltage is being applied to it. This is indicative of a false engage (particularly likely with an Engage Setpoint of 1.10), and can be corrected by either decreasing the Amplitude Setpoint or retuning the cantilever at the surface as described next.

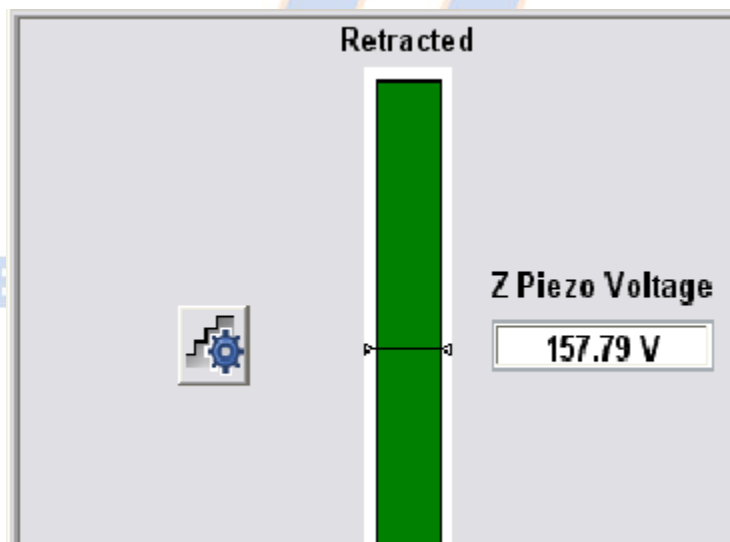


Figure 13. Z-piezo monitoring gauge (Extended/Retracted).

- g) To retune the probe at the surface, select the "Tune" icon. When the pop up window appears, ensure that the lift height is 300 nm or greater, then select "OK". Repeat Step 10) of this SOP and tune the cantilever again as directed in substeps a) through e).
- h) After retuning is complete, click the "Engage" icon as in part d) of this step. Upon engaging the surface, the D3100 will beep. The piezo monitor should now illuminate **GREEN**.

Note: It is important to keep the piezo monitor in the green (± 60 V). This will result in less wear/tear on the piezos due to excessive voltage being applied.

- i) After the probe tip has successfully engaged the surface, expand the scan size and ensure the trace and retrace curves are tracking one another. Adjust the gains and amplitude setpoint as necessary (see Step 12) for more details). Normal values for the Amplitude Setpoint range from 0.45 V (barely touching/interacting with the surface) to 0.30 V (much more vigorously tapping the surface). If the trace and retrace fail to track each other and are varying wildly, there may have been a false engage (i.e., the AFM thinks it has engaged with the surface due to an observed change in the laser intensity sum on the photodiode, when in fact the probe is still hovering somewhere above the surface). If this occurs, withdraw the probe and reengage, retuning if necessary. False engage is more common when imaging in fluid or if the Engage Setpoint is too high (the exact value that constitutes “too high” depends upon the imaging mode employed).

Note: As the Amplitude Setpoint voltage is decreased, the force of the tapping on the sample surface generally increases.



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12) Improve/optimize the image quality.

Note: The following pages are taken from the old Digital Instruments (DI) AFM Training Manual.

16.0 Real-time Operation

- The Real-time parameters are located in the control panels which are available in the Panels pull down menu. The main control panels for topographic imaging are “Scan controls,” “Feedback Controls,” “Other Controls,” and “Channel I.”
- There are three primary feedback parameters that need adjusting every time you engage the microscope to capture an image in TappingMode AFM or Contact Mode AFM: **Setpoint**, **Integral Gain**, and **Scan Rate**.
- To demonstrate how these parameters affect the image, examples of TappingMode operation on the $10\mu\text{m} \times 10\mu\text{m}$ pitch calibration grating will be shown. These examples were acquired by viewing the **Scope Mode** (in the **View** menu), which displays a two-dimensional view (X vs. Z) of the Trace (left-to-right) and Retrace (right-to-left) scan lines being acquired in Real-time.
- **Scope Mode** may be used to diagnose how well the tip is tracking the surface. If the feedback parameters are adjusted optimally, the trace and retrace scan lines should look very similar, but may not look completely identical.
- An optimized scope trace of a $40\mu\text{m}$ TappingMode scan across the pits of the grating is shown below.

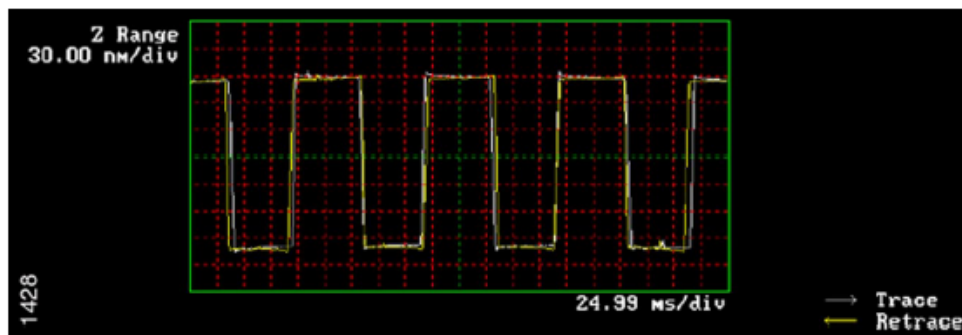


Figure 16.1 Scope Trace with Optimized Real-Time Parameters

16.2 Setpoint

- The Setpoint parameter tells the feedback loop what amplitude (TappingMode) or deflection (Contact Mode) to maintain during scanning.
- Set this parameter so that the smallest amount of force is applied during scanning while still maintaining a stable engagement on the surface.
- If the setpoint is too close to the free air amplitude (TappingMode) or free air deflection (Contact Mode) of the cantilever, the tip will not trace the topography properly.

- This problem will be more pronounced on downward slopes with respect to the scan direction. An example of this is shown below.

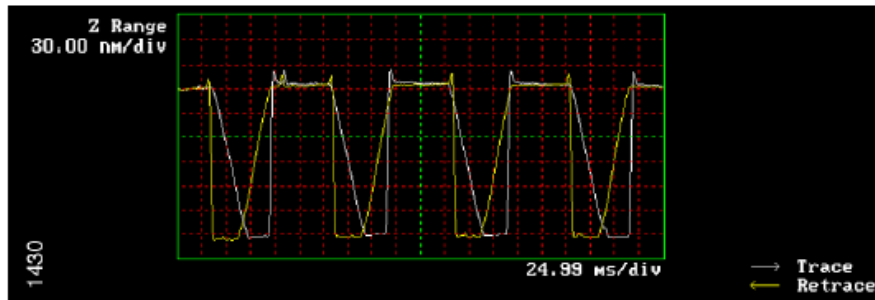


Figure 16.3 Scope Trace Demonstrating the Effect of the Setpoint Being Adjusted Too Close to the Free Air Amplitude During TappingMode AFM

- Notice that the tip tracks the surface up the sidewall properly but not down the sidewall with respect to the scan direction. This occurs on opposite sides of features for the Trace and Retrace scan directions.
- This problem can be remedied by decreasing the Setpoint in TappingMode, or increasing the Setpoint in Contact Mode.

16.4 Integral Gain

- The Integral Gain controls the amount of the integrated error signal used in the feedback calculation.
- The higher this parameter is set, the better the tip will track the sample topography.
 - However, if the integral gain is set too high, noise due to feedback oscillation will be introduced into the scan, as shown below.

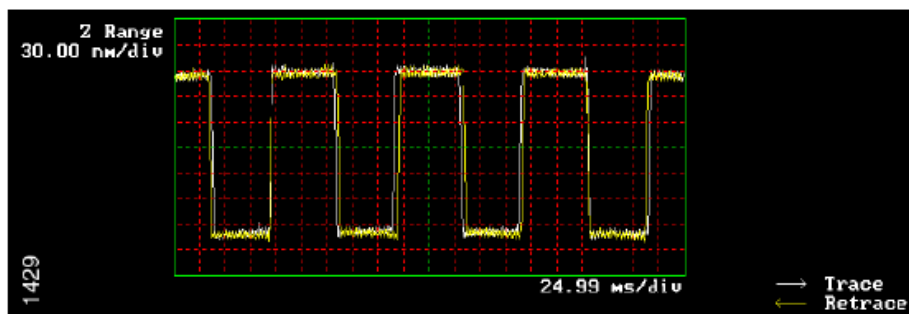


Figure 16.5 Scope Trace Demonstrating the High Frequency Noise that Occurs when the Integral Gain is Set Too High

- Setting the integral gain too low may result in the tip not tracking the surface properly, as shown below.

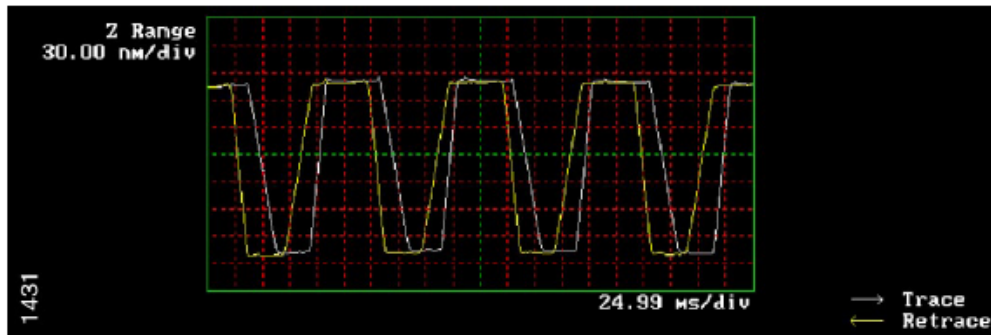


Figure 16.6 Scope Trace Demonstrating the Effect of Setting the Integral Gain Too Low

- Set the Integral Gain as high as possible by increasing the gain until noise is seen, then reduce the gain just lower than the value where the noise disappears.

16.7 Scan Rate

- The Scan Rate is the number of trace and retrace scan lines performed per second (Hz).
 - For example, with a scan rate set to 1Hz, the tip will scan forward and back (trace and retrace) in 1 second.
- Set this value so that the feedback loop has time to respond to changes in the sample topography.
- Setting the scan rate too high will result in poor tracking of the surface, as shown below.

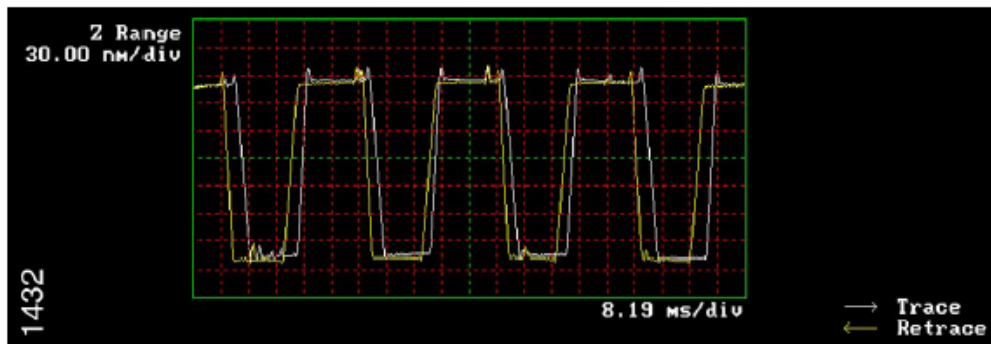


Figure 16.8 Scope Trace Demonstrating the Effect of Setting the Scan Rate Too High

- The actual *tip speed* or of the tip depends on two parameters: **Scan Rate** and **Scan Size**. Since the Scan Rate units are lines per second (Hz), increasing the Scan Size without changing the Scan Rate actually increases the scan velocity of the tip.

- For example, if the Scan Rate is 1Hz and the Scan Size is 1 μ m, the scan velocity is 2 μ m/sec (since it performs the trace and retrace scans in 1 sec). By increasing the Scan Size to 100 μ m, the scan velocity increases to 200 μ m/sec if the Scan Rate remains 1Hz.
- The Scan Rate setting will greatly depend on the scan size and the height of the features being imaged. In general, the taller the features and/or the larger the scan size, the slower the scan rate.
- Typical Scan Rate settings are 0.5 to 2.0Hz for TappingMode and 1 to 4Hz for Contact Mode.

16.9 Other Important Parameters

- **Scan Size:** Adjusts the size of the X,Y scan area
- **X Offset, Y Offset:** Translates the scan area in X and Y directions without changing scan size
 - *Note:* The **Zoom** and **Offset** commands found on the display monitor may also be used to change the scan size and X and Y offsets.
- **Scan Angle:** Controls the angle of the fast scan direction of the scanner with respect to the X axis
- **Samples/line:** Determines the number of data points or pixels in X and Y (128, 256, or 512)
- **Slow Scan Axis:** Enables and Disables movement of the scanner in the direction of the slow scan axis. (Y direction at 0° Scan Angle)
- **Proportional Gain:** Controls the amount of proportional error signal used in feedback calculation. Typically set 35 to 100% higher than the Integral Gain
- **Data Type:** Selects the signal viewed on the display monitor - this parameter is set to Height for topographical measurements
- **Line Direction:** Selects the direction of the fast scan during data collection: Trace (left to right) or Retrace (right to left).
- **Microscope Mode:** Sets mode of operation to Tapping, Contact, or Force Mod
- **Data Scale:** Controls the vertical scale of the full height of the display and color bar. This parameter does not effect the real-time operation of the microscope, only the expansion and contraction of the color scale.
- **Z-limit:** Allows the 16 bits of the digital-to-analog converters to be applied to a smaller vertical range. This has the effect of increasing the vertical sampling resolution and should be used for imaging very smooth surfaces (<5nm RMS) or performing atomic resolution.

For example, a Dimension or J MultiMode scanner may have a full vertical movement of 6 μ m over 440V. By reducing the Z-limit to 55V, the full vertical movement of the scanner is reduced to 0.75 μ m over the same 16 bits. Therefore, the vertical sampling size becomes smaller, which increases the Z resolution.

Warning: Do not reduce the Z-limit if there is a chance that a feature will exceed the reduced vertical range.

- **Z-Center Position:** The Z-center voltage scale is located to the right side of the image on the display monitor. It represents the current extension or retraction of the Z piezo electrode of the scanner during operation.

It is displayed from -220V to +220V (440V full range) which represents the entire vertical travel of the scanner (i.e. 5 to 6 μ m for a Dimension scanner or J MultiMode scanner).

It is recommended that the Z-Center Position be kept as close to 0V as possible when making height measurements. The Z-Center Position is adjusted by using Step Motor in the Motor menu.

13) Capture your image(s).

- a) Ensure the image is saved in the proper file directory by clicking on the “Realtime” tab, then selecting “Capture Filename”. Under “Directory”, select the location where the images should be saved.
- b) Select the image capture method desired. The following options are available in the drop down menu:
 - Capture: Will capture the next complete image, provided the scan parameters are not changed during the acquisition.
 - Capture Continuous: Will sequentially capture each complete image acquired until directed by the user to stop doing so.
 - Capture Now: Will capture the current image regardless of whether or not scan parameters were changed midway through (i.e., during) image acquisition.
 - Cancel Capture: Will cancel the currently selected capture image function.
 - Capture Withdraw: Will capture the next complete image and then withdraw the probe once complete. This feature is useful if you will not be in the lab when the scan finishes.

14) Withdraw the tip and shutdown the AFM.

- a) Once scanning is complete and images are captured and saved, select the Withdraw icon to stop imaging and move the tip well away from the sample surface.



- b) Open the AFM hood by rotating the latch on the front of the AFM enclosure counterclockwise.
- c) Slowly rotate the sample chuck to allow access to the sample, taking care to first ensure the probe has been withdrawn sufficiently far from the surface to prevent damage to the probe and/or AFM head piezos.
- d) Turn off the sample/stage vacuum (Figure 1) and remove the sample from the stage.
- e) Unlock the AFM head by turning the locking screw to the **right** (Figure 5).
- f) Carefully remove the AFM head from the rail dovetail holder. Remove the probe holder (Figure 6).

WARNING: Handle the AFM head with extreme care! Any impact to the head may cause severe damage to the piezo tube, which is **very** expensive to repair/replace. Be careful not to bend or break the contact pins!

- g) Place the probe and probe holder on the probe holder mounting block (Figure 4). Remove the probe and return it to the location/position in the box it came from, ensuring the probe is turned perpendicular to the remaining unused probes in the box to indicate that it has been used. Detail the usage and condition of the probe on the box label.

- h) Install the AFM head back into the dovetail rail holder and lock the head in place by turning the locking screw to the **left**.
- i) Turn the D3100 power strip off (Figure 2).
- j) Close the Nanoscope software.
- k) Complete the Excel log in spreadsheet on the computer desktop with the number of hours and new tips used, taking care to note any issues you encountered and the solutions to those problems.



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