

University of Toronto
ADVANCED PHYSICS LABORATORY

AFM

Atomic Force Microscopy

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Please send any corrections, comments, or suggestions to the professor currently supervising this experiment, the author of the most recent revision above, or the Advanced Physics Lab Coordinator.

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1. OVERVIEW

The Atomic Force Microscope (AFM) allows measurement and manipulation of atomic surfaces, and was invented by Gerd Binnig, Calvin Quate, and Christoph Gerber in 1986.¹ The AFM is one of a family of instruments developed after the invention of the Scanning Tunnelling Microscope (STM) in 1981, for which Binnig and Heinrich Rohrer won the Nobel Prize in 1986. Unlike the STM, the AFM does not require a dry, clean, conducting surface, and so can be used to measure insulators or biological samples.

The goal of this experiment is to familiarize the student with mesoscale and nanoscale AFM measurements used in fields such as condensed matter physics. As feature sizes of the objects of study become smaller, optical techniques become unable to resolve these features. While electron microscopes can have the required resolution, they do not provide height information. AFM and STM technology has become the standard methods used in probing atomic, molecular and domain features, with resolutions reaching down to a few angstroms in the most advanced instruments. This experiment will make use of a low-resolution AFM by Digital Instruments to investigate topographic features at the micron scale.

¹ G. Binnig, C.F. Quate, Ch. Gerber, *Atomic Force Microscope*, Phys. Rev. Letters, 56 (1986) 930-933; <http://dx.doi.org/10.1103/PhysRevLett.56.930>.

2. ATOMIC FORCE MICROSCOPE

2.1 Introduction

The AFM technique is a subcategory of scanning probe microscopy (SPM). SPMs are instruments that use a raster-scanning tip to measure surface properties such as the local height, friction, electronic or magnetic properties, and construct a map of this data to form an image. The first SPMs constructed were STMs, where the tunnelling current or conductance between a tip and the sample is used as the measurement of the local electronic density of states.

2.2 Theory of Operation

A standard AFM instrument consists of a cantilever and sample assembly, the laser and photodiode detector, and the feedback and measurement electronics; seen in Figure 1.

Atomic Force Microscopy (AFM) : General Components and Their Functions

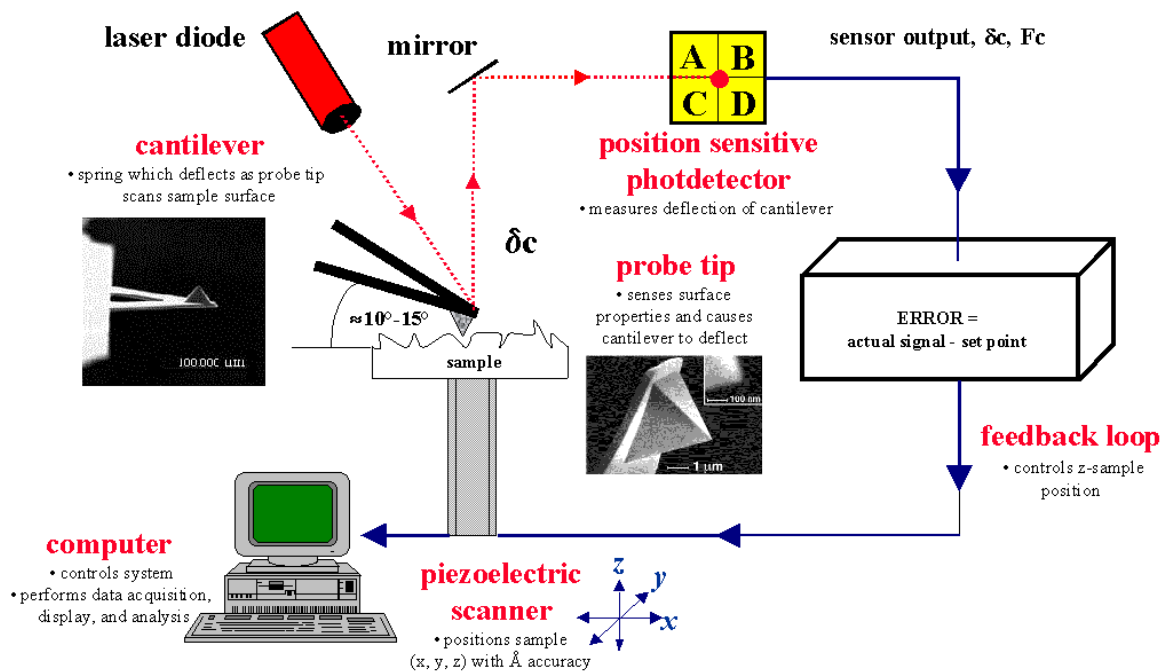


Figure 1: Overview of the AFM system. (Image from <http://web.mit.edu/cortiz/www/afm.gif>.)

An AFM operates by measuring the minute force between a sharp tip and the sample surface. Typical AFM tips have an end radius $< 15\text{nm}$ and a height of 3 to $20\text{ }\mu\text{m}$, mounted at the end of a cantilever. Usually the tip material is silicon (Si), since Si etching is well understood from integrated circuits. If conductive AFM operation is desired, then the tip can be coated with a conductive material. It is possible for higher specification tips, but the instrument is a limiting factor in this experiment.

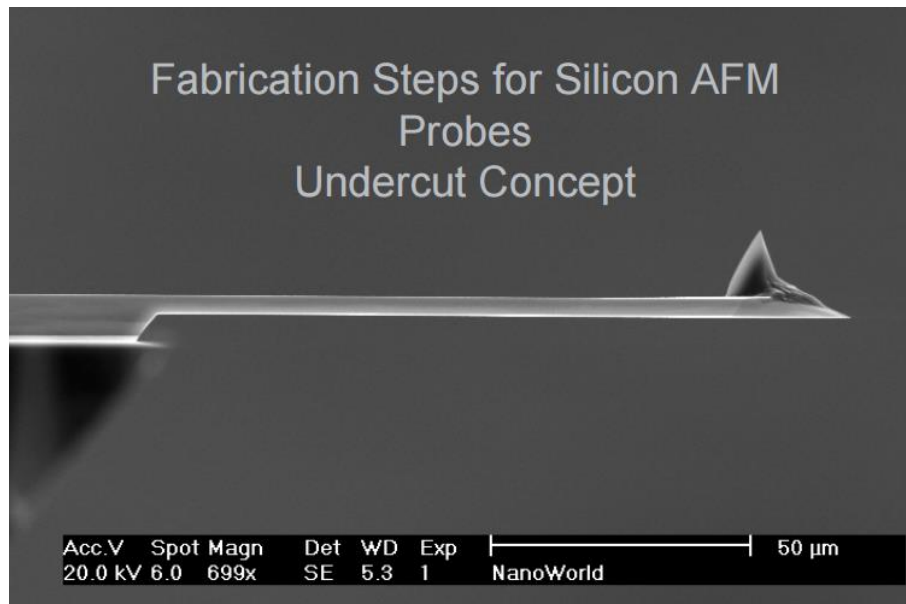


Figure 2: Typical shape of the AFM tip. (Image from Oliver Krause, NanoWorld Services GmbH; from https://www.agilent.com/cs/library/slidepresentation/Public/AFM%20Probe%20ManufacturingNanoworld_tip_technologyPRussell07.pdf)

In modern AFMs, a laser beam is reflected from the cantilever towards a 2- or 4-segment photodiode. The 4-segment setup is able to measure lateral force and vertical force, while the 2-segment photodiode used in this experiment can only measure the latter. The AFM records the deflection of the cantilever by measuring the position and intensity of the reflected beam on the different segments of the detector.

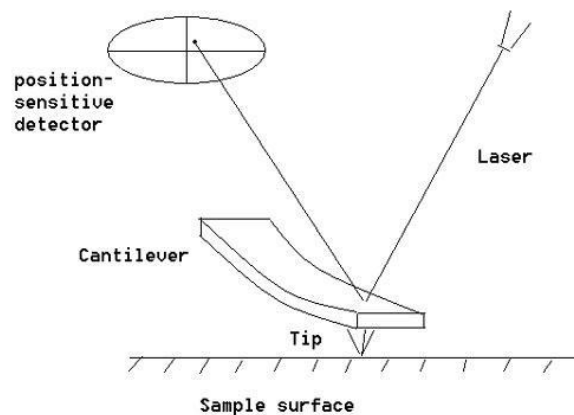


Figure 3: Schematic of the AFM setup. (Image from Wenjie Mai, *Fundamental Theory of Atomic Force Microscopy*, <http://www.nanoscience.gatech.edu/zlwang/research/afm.html>)

Because the tip needs to scan over very small features, precise control on the position of the tip is required. Scan heads are generally built using piezoelectric components to achieve nanometre level resolution. Piezoelectric materials change their physical dimensions in response to an applied potential. When constructed in a hollow cylindrical shape, with the voltages applied to 4 different quadrants, lateral movement in the x-y plane can be achieved in the small angular deviation limit.

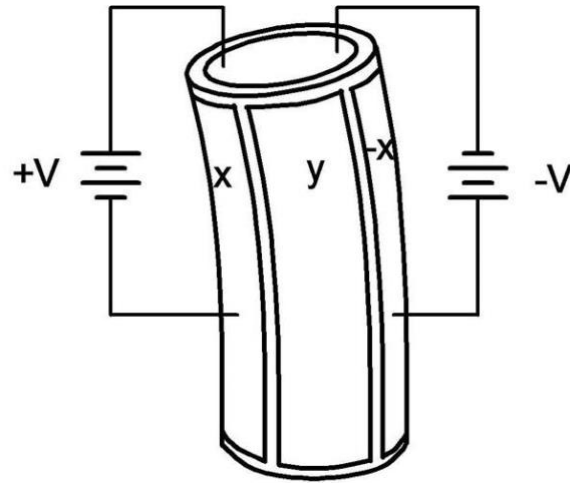


Figure 4: Division of the quadrants in a tube scanner. Motion in the x-y plane can be achieved with a combination of the 2 voltages with the inner Z voltage. (Image from Park Systems, <http://www.parkafm.com/index.php/applications/data-storage/202-afm-metrology-considerations-of-hard-disk-manufacturing>)

2.3 Modes of Operation

There are 3 main modes of operation: Contact mode, non-contact mode, and tapping mode. In contact mode, the tip is brought into physical contact with the sample, and measures a repulsive force. Because the force-displacement curve is much steeper in this regime, the signal is much larger than the non-contact mode. Therefore, this mode doesn't require sensitive instrumentation and can be measured directly using DC coupling. The contact mode can be further broken down into two additional modes: constant height mode and constant force mode.

- Constant height mode locks the z height of the scanner and measures the deflection of the cantilever. This has a higher risk of crashing, and therefore is typically used on samples that are known to be approximately atomically flat. The advantage of this mode is that it has lower thermal noise, and can see atoms easier.
- Constant force mode keeps the tip at the same force, and therefore distance with respect to the surface. The z piezo voltage is recorded and converted to a signal. This mode has a lower chance of crashing and is typically used to image samples with protruding structures.

In the non-contact mode, the tip is situated at a farther distance away from the sample, and experiences an attractive force. While the magnitude of the force is several orders of magnitude lower than that of the contact mode, it has the advantage that the tip is less likely to be damaged from contacting the sample. In this mode, a very stiff cantilever is used, which resonates at a characteristic frequency. AC coupling is used to measure small deviations in vibration frequency due to the small force experienced by the tip. Typically, soft samples such as tissues are imaged using non-contact mode. The device used in this laboratory is limited to contact mode only.

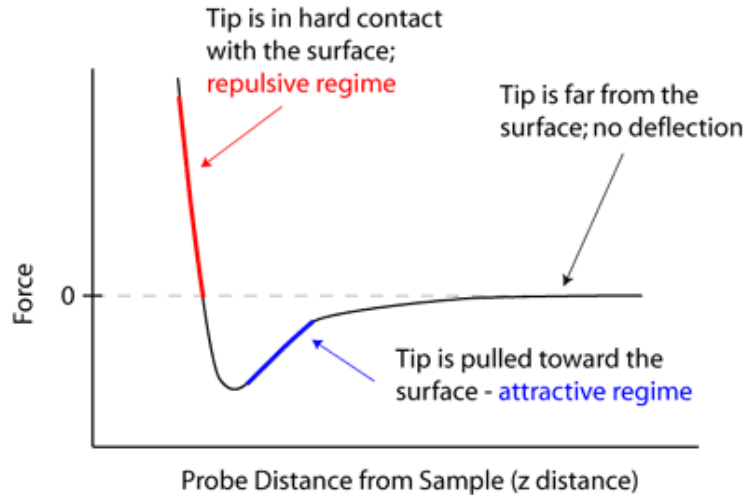


Figure 5: Different regimes within the force curve. (Image from nanoScience Instruments, Atomic Force Microscopy, <http://www.teachnano.com/education/AFM.html>)

2.4 Tip-sample Interactions

In a force curve such as the one shown in Figure 5, the distance between the tip and sample is not what the z piezo measures, due to cantilever deflection and sample surface deformation. The two types of force curves will be distinguished using: force-distance curve for the true atomic potential; force-displacement curve for the measured z piezo vs. photodiode signal.

The deflection of the cantilever can be modelled by the simple Hookean relationship

$$f = kz \quad (1)$$

where the k here is the effective spring constant of the cantilever. This value can be theoretically calculated from the shape of the cantilever by finding the moment of inertia; however, the thickness of the cantilever is very difficult to measure, therefore the k value can be taken as the value written on the box containing the tips. There are usually several cantilevers on a tip substrate, with the smallest cantilever having the highest spring constant.

We can assume a Lennard-Jones force for the interatomic interaction, so that

$$F(D) = -\frac{a}{D^7} + \frac{b}{D^{13}} \quad (2)$$

where D is the actual tip to sample distance, and a and b are constants. We can plot on the same graph the force curves for the cantilever (see Figure 6), which are simply straight lines of slope k . At each D value, the cantilever deflects until the elastic force equals the tip-sample interaction, resulting in an equilibrium. However, the force value at each intersection must not be associated with the D value at that point, because that is the actual tip-sample distance and is not directly measurable. We can assign the force values to the rest position of the cantilever, i.e. the horizontal zero intersect of the k lines. For example, the force f_a is assigned to α . We can do this going from right to left on the $F(D)$ curve, representing an approach, or left to right, which represent a withdrawal. In the region between the 2 and 3 lines, going different directions lead to different forces for the equilibrium positions, and this results in the so-called force-displacement hysteresis.

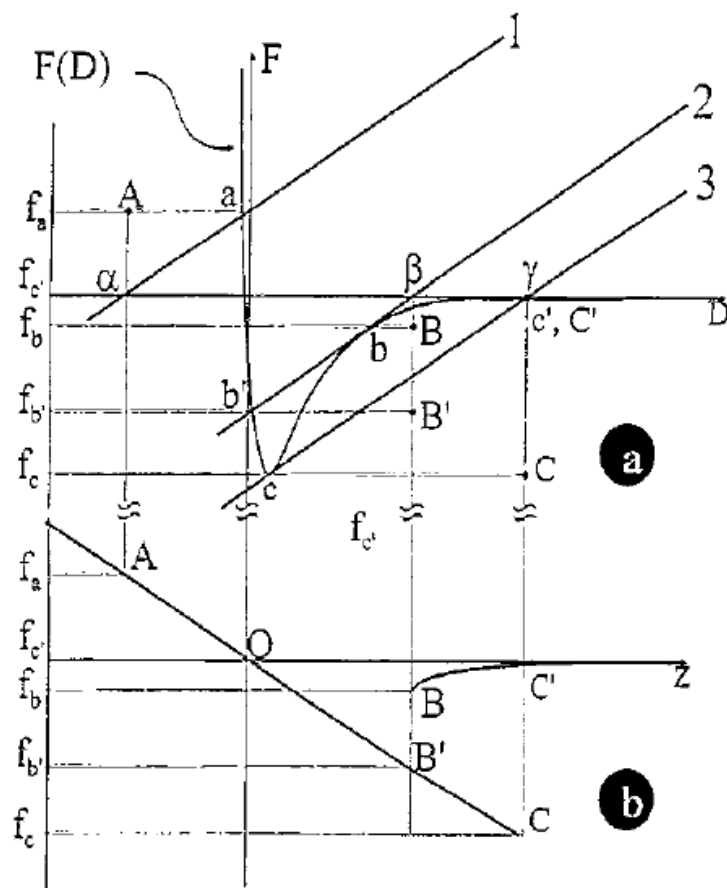


Figure 6: (a) force distance curve of interatomic interactions and cantilever deflection. (b) force displacement curve construction from corresponding points in (a). Image from “force distance curves by atomic force microscopy” (Image from Cappella, p.9).

Because the interatomic forces are dependent on the tip and sample materials, as well as moisture content in the air, it is very difficult to produce exact relationships for specific cases. For a full derivation of the general relationship between force-distance and force-displacement, refer to “force distance curves by atomic force microscopy”, see Cappella.

2.5 Safety

Safety Reminders

- The piezo voltages can reach as high as 150V, and 300V peak-to-peak. These lines are located in the bus wire that connects the controller to the base of AFM, where the scanner is located. If any part of the wires is exposed, immediately contact the laboratory technician. Do not attempt to open the controller by yourself as there are high voltage sources inside as well.
- Chemical used in this experiment may include solvents such as acetone and ethanol, and sample materials such as graphite and polymers. It is advisable to read the Material Safety Data Sheet (MSDS) for these materials before the experiment. (For MSDS information, see <http://www.ehs.utoronto.ca/resources/info.htm>.)
- Acetone is an irritant to the skin and eyes, and while it's not very toxic – it is a common component of nail polish remover and paint thinners – direct exposure to it should be avoided. Read the MSDS sheet at <http://bit.ly/1UzvQhq> (Let Supervisor know if link is broken.)
- The AFM stage has a 2mW diode laser built in. When the scan head connector is connected, the laser is on. This classifies the laser as Class 3R, meaning that although no eye protective equipment is needed as the source laser is unexposed, but it is not recommended to look directly into the laser or any reflections from mirrors. It is safe to look at the diffuse scattered light from rough surfaces.
- The tweezers are quite sharp and should be handled with care.

NOTE: This is not a complete list of every hazard you may encounter. We cannot warn against all possible creative stupidities, e.g. juggling cryostats. Experimenters must use common sense to assess and avoid risks, e.g. never open plugged-in electrical equipment, watch for sharp edges, don't lift too-heavy objects, If you are unsure whether something is safe, ask the supervising professor, the lab technologist, or the lab coordinator. When in doubt, ask! If an accident or incident happens, you must let us know. More safety information is available at <http://www.ehs.utoronto.ca/resources.htm>.

3. OPERATION INSTRUCTIONS

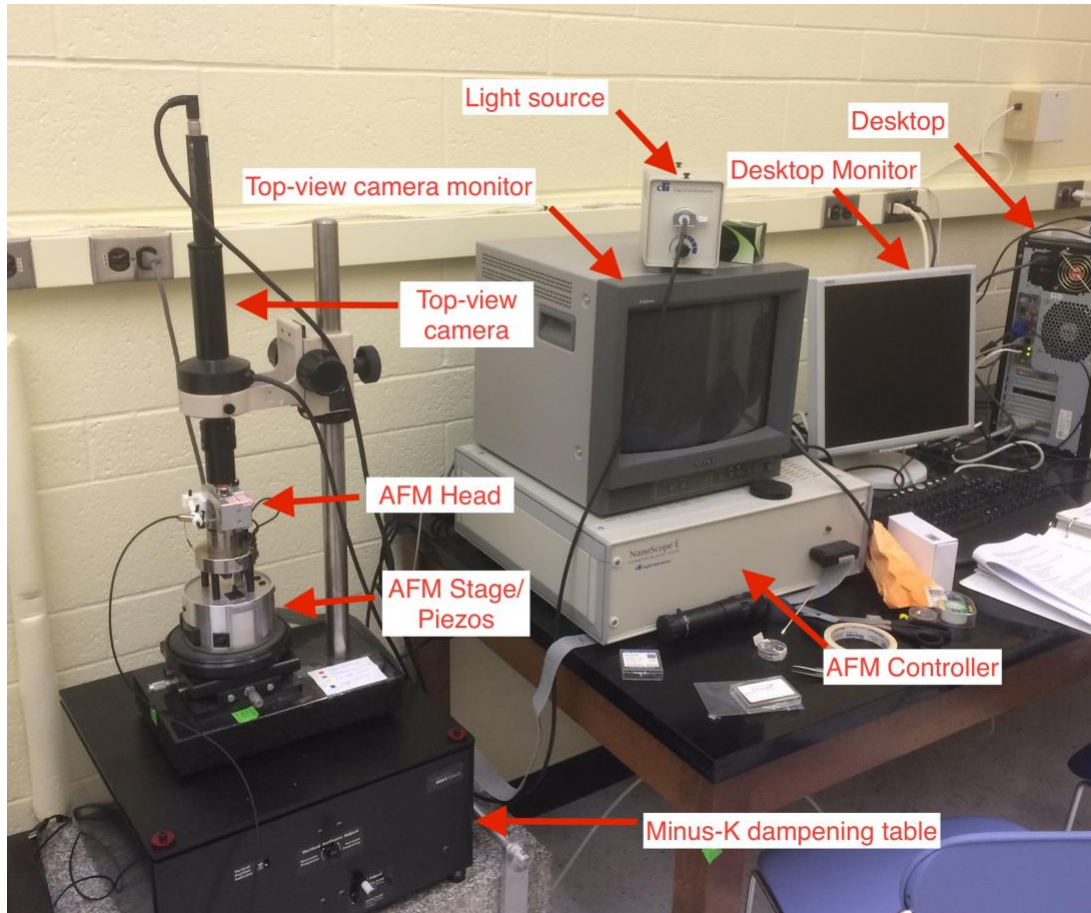


Figure 7: Equipment for this experiment.

3.1 Equipment preparation

1. Turn on the Nanoscope E controller with the switch at the right-back of the main controller
2. Turn on the TV monitor
3. Turn on the computer and sign in using the student account, "AFM USER"
4. Double click the AFM icon on the desktop
5. Click on the microscope icon in the top left, select *scan-single* (or multiple scan windows for simultaneous taking multiple types of data), and click *ok*

3.2 Setting up the Scanner Stage

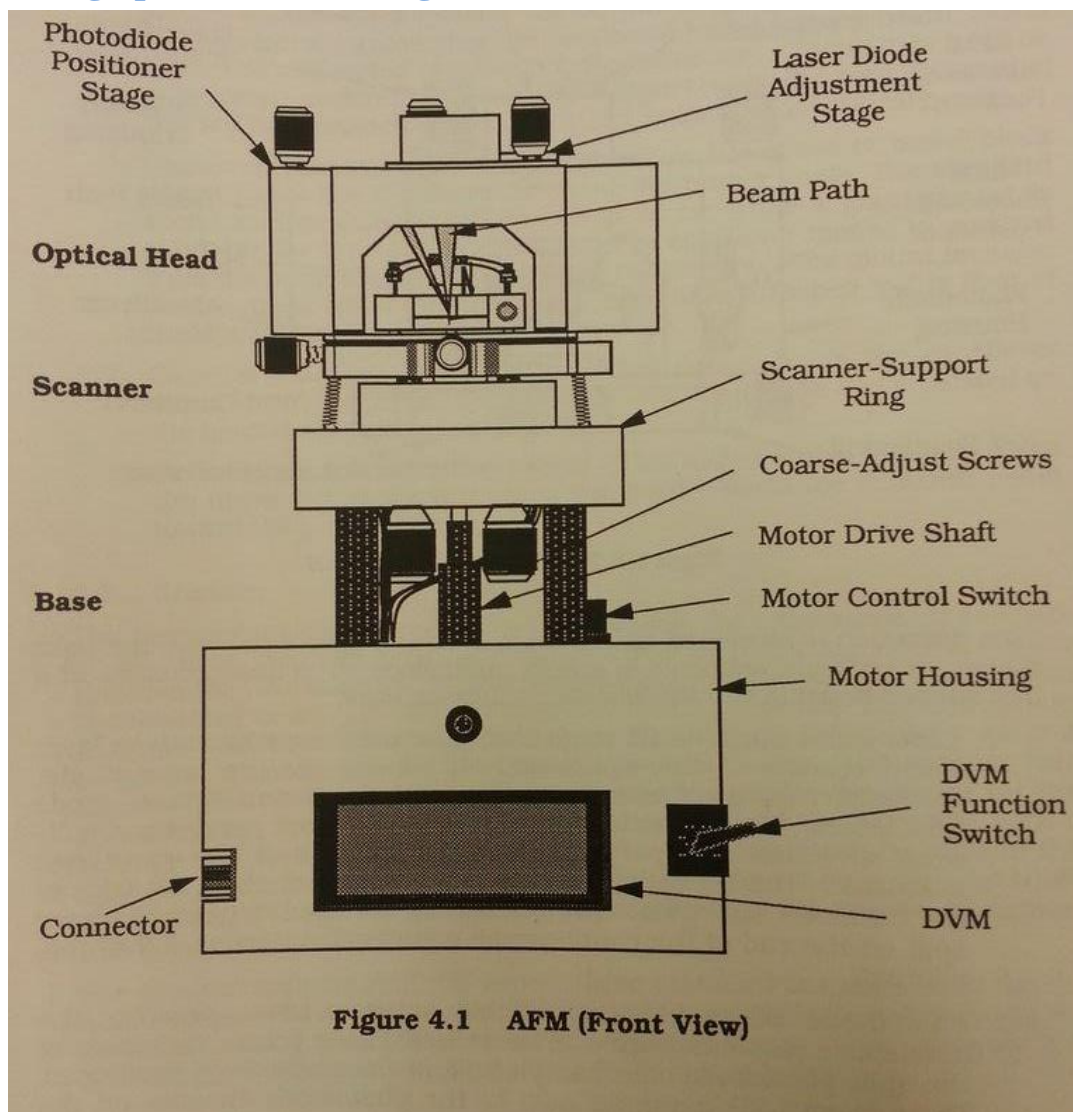


Figure 8: Schematic of the AFM body. (Image from *Digital Instruments NanoScope® III Atomic Force Microscope Instruction Manual*, Version 2.2, 8 July 1992.)

1. If a new sample needs to be mounted, take the AFM head off by first unplugging the 9-pin connector on the side of the scanner head. Then hold the head down firmly with one hand, and unhook the springs on both sides of the head (beneath the head itself), before taking the scanner off. Note that the stage is magnetic.
2. Use silver paint or epoxy to glue the sample onto the sample holding puck, then place it on the scanner. (see details in the experiment section for sample preparation instructions)
3. Make sure the scanner head block is mounted back correctly – the legs are seated inside the grooves and the springs are attached to the side of the head before letting go of the head. Plug the 9-pin connector back in.
 - *Note:* Use caution during this step as if the sample is large and hits the tip, it may break before you even begin!

4. Ensure that the tip holder - aluminium block holding the tip substrate - is placed inside the AFM head and clamped down with screw in the back of the head (Marked in yellow). Be careful about tilting the head as the holder may fall out if not properly clamped.
5. Turn on the light source, and focus the image on the sample surface by moving the top-view camera. Most times it needs to be moved down until it is about ~1cm above the head lens.
6. Align the screen to find the cantilever (triangular shaped) using the camera adjust knobs at the very base of the AFM body. You have a choice between a smaller or larger cantilever; which to use depends on application and desired effects.
7. Use a combination of the “UP – DOWN” on the Motor Control Switch (MCS), seen in Figure 8 and Figure 9, and the two coarse screws under the head (marked in blue) so that the tip is very closely to the sample (within 1mm). Note that the coarse screws, when looking up from the bottom, raises the tip when turned clockwise, and lowers the tip when turned counter-clockwise.

There are two ways to do coarse approach:

- a) Use the side-view camera and visually lower the tip until it's close to the surface. Issues with this method are that with too much light it may be difficult to see the approach. To use this camera, plug it in, and go to Start->My Computer->USB Camera.
- b) Use the top-view camera. First change the focus of the camera to make sure the tip and sample are in the field of view (usually they should be visible at difference focusing at this point). With the tip in focus, slowly lower the head by using a combination of the two coarse screws and the UP - DOWN DVM display switch. When both the tip and the sample are in focus, that means the tip is very close to the sample.

Warning:

1. There are 2 tips on one edge of the cantilever holder. When using the smaller tip, always be mindful of the larger tip as it dips lower than the smaller tip. When approaching with the smaller tip, it is very easy to break the larger tip if you don't pay attention. In order to use the smaller tip, the sample head must be tilted at an angle (when viewed from the front), this way the smaller tip can approach the surface before the larger tip crashes.
2. Always monitor the cantilever when approaching. Whenever a cantilever turns gold/white, that means it is about to break!

3.3 Aligning the laser and photodiode

1. Locate the laser spot on the screen. If you have difficulty doing this, see Section 3.7.
2. Move the laser spot using the two laser adjust knobs (should be labelled) so that the spot is near the tip of the triangle. Use the visual feed from the top-view camera to confirm the laser spot is at the tip.
3. Check the mirror inside the chamber of the head to see that the laser spot is reflecting off of it. If the mirror is crooked, then it can be adjusted by using a 1/16in Allen key at the back of the AFM head.
4. Flip the vertical switch on the side of the AFM (the display switch) body to UP, so that the digital display reads the A+B photodiode signal
5. Adjust the laser positioning knobs such that the total signal is locally maximized. This can be an iterative process.

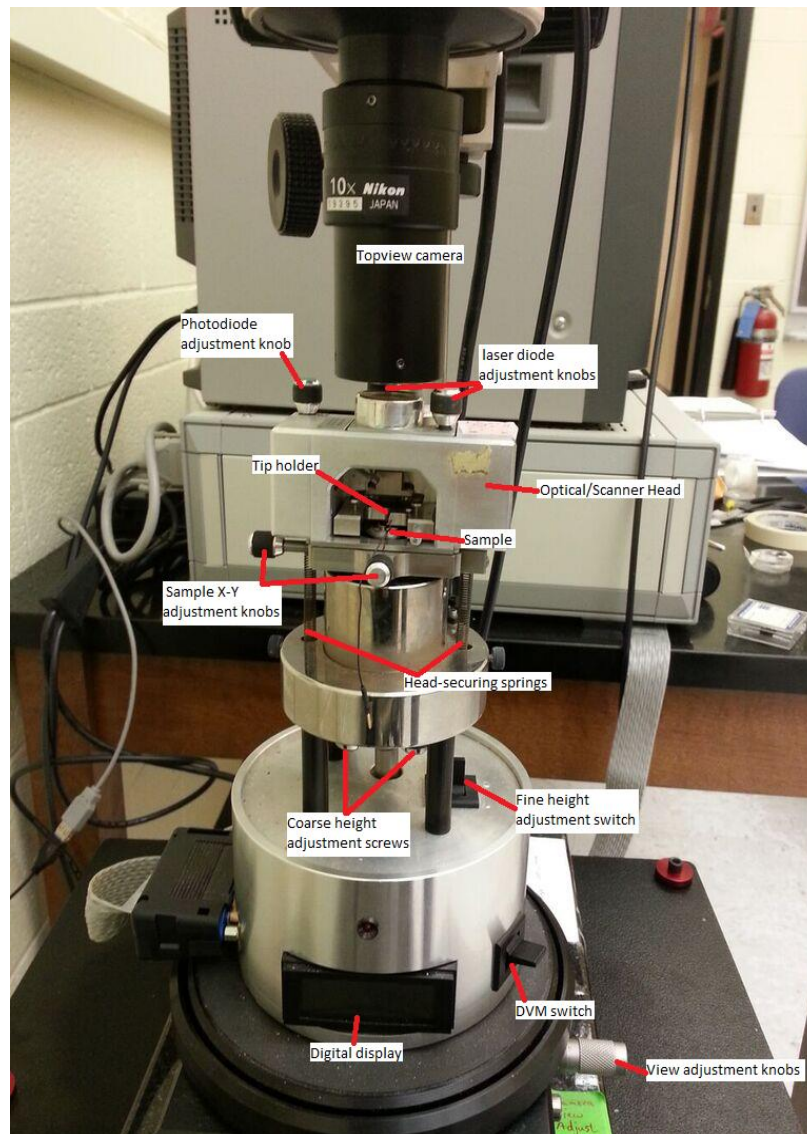


Figure 9: AFM body (front)

6. Adjust the photodiode positioning knob (which moves the photodetectors up or down) such that the total signal is maximized. The maximum total signal should be around 5-8 V. If the maximum is significantly lower than that, it means that the laser spot is not reflecting off of the cantilever, and step 4 needs to be repeated to achieve proper reflection.
7. Flip the DVM function switch to DOWN so that the display reads the A-B signal. The initial set point for this value is approximately -4V.
8. Adjust the photodiode knob until the display reading is between -3 and -4 volts. A more negative voltage means the tip will be closer to the sample and will be more sensitive to the features, however it would be more susceptible to crashes.

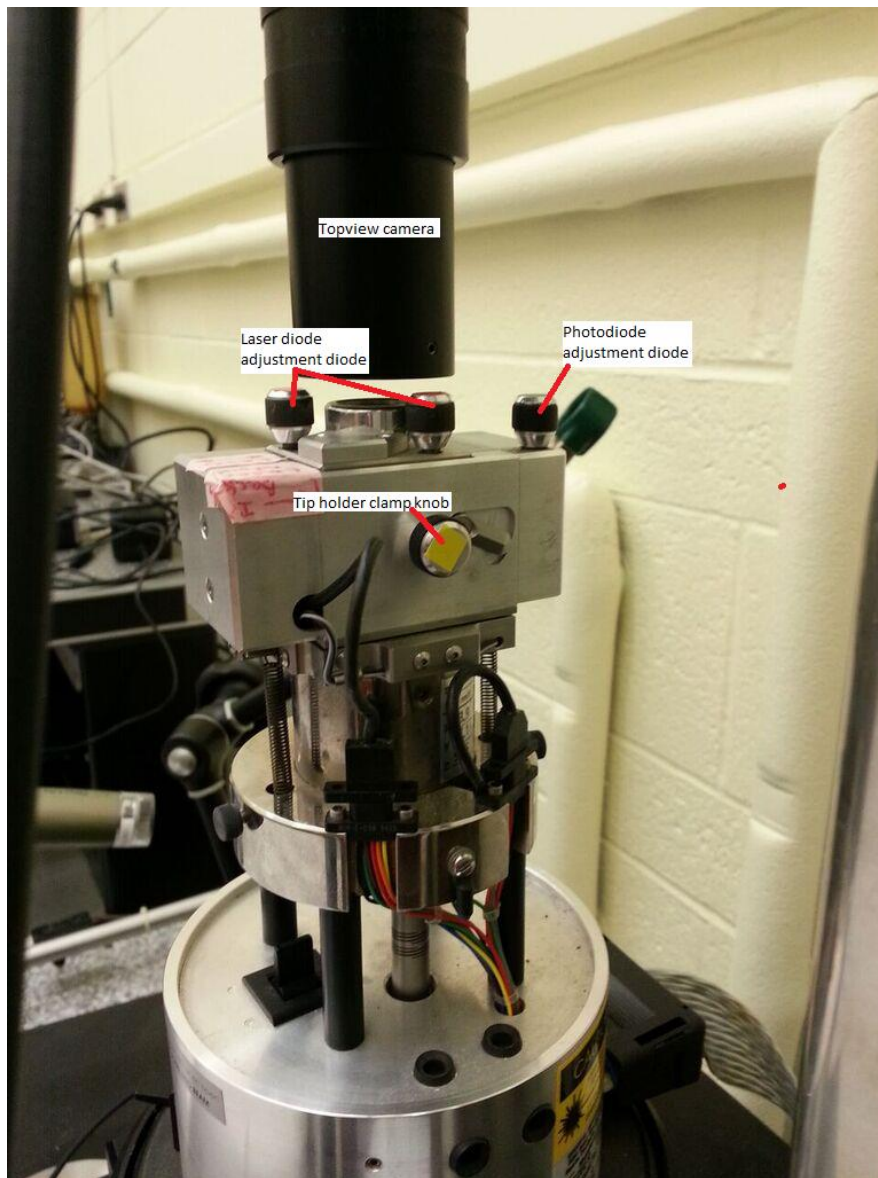


Figure 10: AFM body (back)

3.4 Scanning and capturing an image

Suggested starting values (software parameters, as seen in Figure 14):

- Scan size: 10 μ m
- Scan rate: 2Hz (the program will slightly adjust the rate)
- Samples/line: 512
- Lines: 512
- Aspect ratio: 1
- Integral gain: 2
- Proportional gain: 4
- Deflection setpoint: 0V
- Initial A-B setpoint: -4V (set in the previous section)
- The data scale value depends on sample feature sizes and should be adjusted

1. Move the cantilever to a location of interest by using the translation knobs just below the removable head.
2. Unlock the vibration isolation locks by unscrewing the three red knobs if they are screwed in. Check that the isolation table is freely rotating. Check that the Minus-K table is properly adjusted; see the Minus-K manual for instructions.
3. Engage the tip by clicking the engage button in the scan window. The approach and scan should be automatic.
4. During engage, monitor the A-B signal. A good approach should have the A-B voltage jump from the initial setpoint to the deflection setpoint immediately. See section on false feedback if the voltage is drifting slowly.
5. The scan location can be moved with 2 screws that are located near the bottom of scanner. They can be used to move the sample with respect to the tip so that the area of interest can be scanned.
6. To capture an image, go to *Realtime -> Capture Filename*, and select the desired directory. Make sure to click “Capture” instead of “ok”, or the scan will not be captured.
7. The scan will continue until the full screen is done, then the image file will be saved as an .001 file, which is what the AFM program reads. Note that the AFM will scan indefinitely unless stopped, but the capture is complete when “Done” is displayed at the bottom of the window.
8. To obtain an ASCII file of the data, open the image file and go to Export -> ASCII

When adjusting these values, keep in mind that for best results more than one may need to be changed. For example, if you change the scan rate, the PID values should also be adjusted.

A zoom function is available for obtaining smaller sized scans in a chosen area of the current scan. The button is located just below the real time figure. Once pressed, simply create a square on the scanned area by dragging and a new scan will start on the selected area. Due to software limitations this may not work perfectly on the first try, so zooming and unzooming by resetting the scan size may be necessary to zoom in on the intended region.

3.5 Obtaining Force-Displacement Curves

1. Move the tip to the desired location by selecting the point and shoot function and click the spot on the scan window.
2. Select the *acquire -> ramp plot* function.
3. The force-displacement curve is taken automatically. Bring the plot feature into view by changing the plot start voltage and the plot range. Typically, begin with a starting scan voltage of around 200V, and a scan range of 400V. At this point it should look like just one single sloped line. Make any scan size adjustments to obtain a proper window size.
4. The proper force-displacement curve can be obtained by changing the setpoint. It should look like Figure 11.
5. Again the data can be saved by first capturing as a .001 file, and then exporting as an ASCII file.

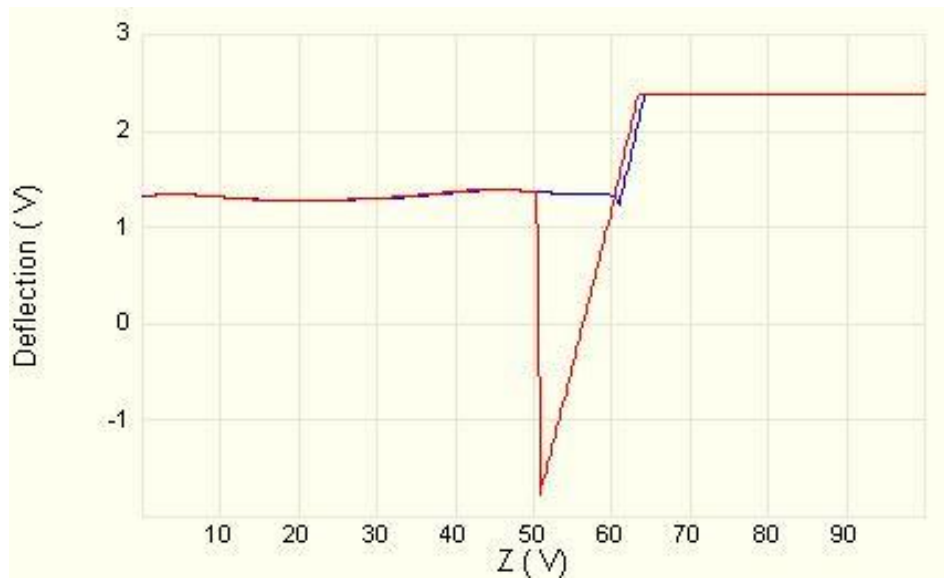


Figure 11: Force Distance Curve Obtained with the AFM

Define the zero line by extending the lower horizontal line (this is the line that represents no force on the cantilever), and the contact line by extending the sloped straight line for both approach and withdrawal (this is the line representing the contact deflection). The origin can be taken as the intersection between the zero line and the contact line.

The contact line's slope is a combination of the sample stiffness and tip stiffness, and the relationship is simply parallel combination of the two stiffnesses.

Knowing the cantilever stiffness will allow the sample stiffness to be found.

Looking at the c point of Figure 6, we can see that it is very close to the global minimum of the interatomic force curve. By finding the D position of point c relative to the origin, we can estimate the true equilibrium distance between the tip atom and the surface atom (without influences from bending of the cantilever). The B point is the lowest point of the dip in the blue trace line, and the C point is the lowest point in dip of the red retrace line. We know what the force difference is between the C and C' point (approximate C' to be at 0 force) from the measured force distance curve, so we can calculate the distance from the constant slope k line for the tip stiffness that was used.

3.6 Shutdown

1. Raise the tip by clicking the Withdraw button
2. If the tip won't be used for a long time, raise the tip a bit more by using the UP toggle switch
3. Remove the tip holder and the sample if necessary
4. Turn off the controller and the light source
5. Lock the vibration isolation table
6. Save your files on a USB drive
7. Log out of the computer

3.7 Troubleshooting of common problems

1. Head does not engage

The tip may be initially too far away from the sample. Use the UP – DOWN DVM display switch and the coarse adjustment knobs to lower the tip. If the alignment is good, then the cantilever is probably broken and need to be replaced.

2. False Engagement

If the voltage slowly drifts instead of jumping to the setpoint, then the tip is probably in false engagement. This happens due to light reflecting back from the sample triggering the feedback. The approach needs to be aborted if this happens. The laser point should be moved visually to be on top of the tip, instead of relying on maximizing the A+B signal. The photodiode can be adjusted to a more negative initial A-B voltage so that the tip is pushed down more.

3. Tip Dragging

If on the scanning image, extra lines appear, similar to noise, this could be due to tip dragging. If this is observed, it can interfere with data collection by skewing the topography imaged as well as spontaneously deposit dragged flakes. To resolve this, it can be attempted to reduce the deflection set point.

4. Locating laser spot

If upon setting up the AFM as outlined, with a sample, and ensuring that the laser is on, you cannot find the laser spot with the top-view camera, there are a few key things that can be done to address this. The first and most common fix is to use the stage adjustment knobs (at the base of the device) to see the area surrounding the cantilever. Note that this does not adjust the laser or mirror but just pans the visible area. Sometimes, if the laser spot is on the tip substrate it will be faint. In the case that no laser scatter, reflection, or faint mark can be seen, use the side view camera or by human eye, to see if the laser is over the aluminium components. If this is the case, the laser is significantly off and can be carefully adjusted by inspection until it is in view in the top-view camera.

5. Image Hysteresis

If the trace-retrace plot is showing hysteresis, it is likely that the sample is slanted. This can occur due to the x-z scan coupling and the response time in z. To minimize this effect, try rotating the scan in the software. This can be done by altering the Scan Angle in the Scan tab shown in Figure 13. At an appropriate angle, the hysteresis effect should be minimized and the trace-retrace curves should match.

For more specific problems, refer to section 5.1 of the AFM manual from Digital Instruments.

3.8 Analysing the image via Gwyddion

To analyse the collected data, we will use the Gwyddion software² found on the desktop. Simply open the software, and open the .000 or .001 file under *File -> Open* or Ctrl + O hotkey. Once in the file browser, navigate to the data file directory and open the file with the file type set to *Automatically detected*. This will automatically open the file with all the relevant parameters already set, such as scan size and height units.

² <http://gwyddion.net>

The *Data Process* -> *Level* submenu has various methods for getting rid of nonphysical trends in the image by flattening the background. You can also zero the lowest height measured with the *Data Process* -> *Level* -> *Fix Zero* utility.

To obtain the surface roughness parameters of a flat surface, click the *Obtain Roughness Parameters* button under tools. Then, drag a line across the image and various measures of roughness will be calculated such as RMS and Kurtosis.

Next, to obtain a 1D surface profile, click on *Extract Profile* button under *Tools*, highlighted in red in the Figure 12 below.

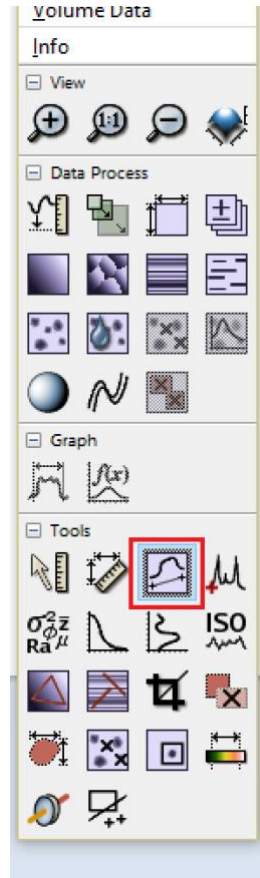


Figure 12: Gwyddion2 Main Menu.

After click this button will allow you to drag a line on the image of sample and the height profile along the line will pop up. Then, click the *Apply* button located on the bottom right to open an interactive plot of the profile.

From here, you can measure the step height by clicking *Measure Distance in Graph* button highlighted in green in Figure 13 below and creating and dragging two vertical lines to either side of the step and make sure the method is set to intersections, highlighted in red in Figure 13.

After aligning the two vertical lines, step height can be read off of the height parameter, and the step width can be read off of the length parameter from the section highlighted in blue in Figure 13. For measuring with more than two vertical lines, the length and height parameter of a vertical line is always measured with respect to the previous created vertical line.

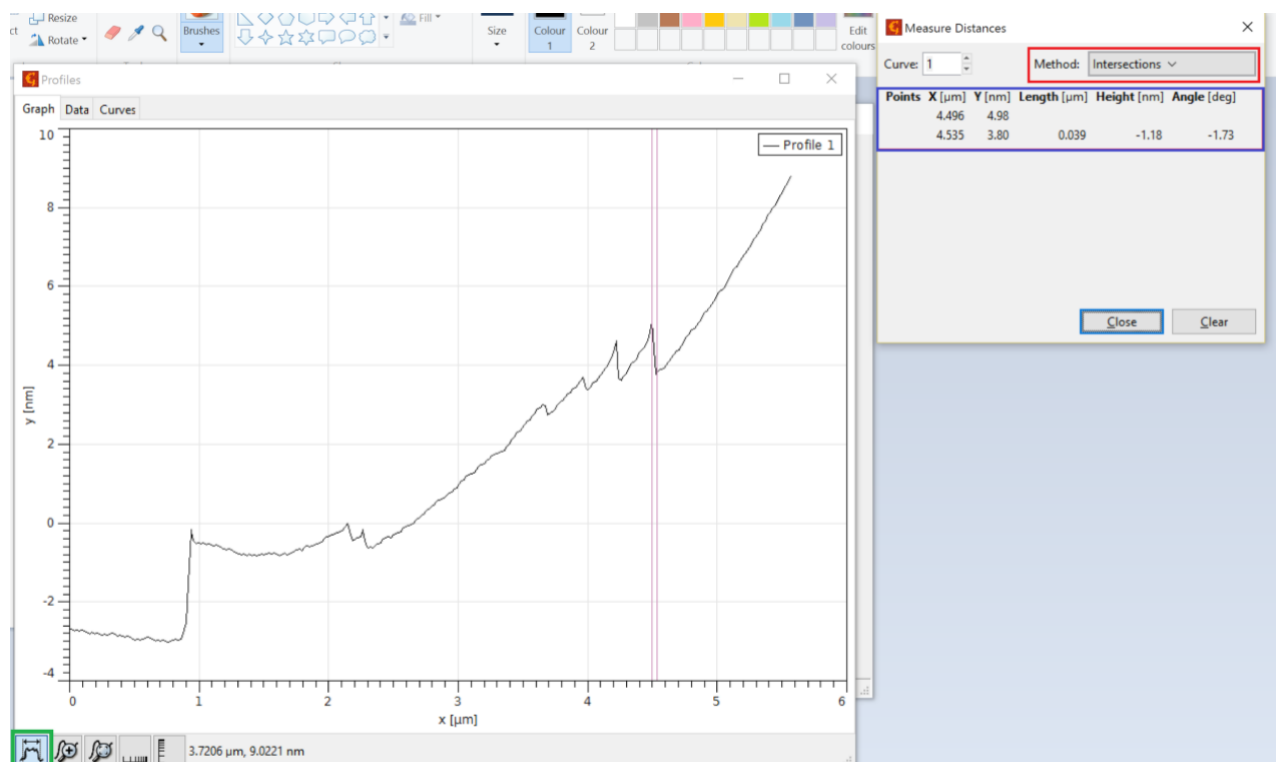


Figure 13: 1D Profile Measurement Interface.

For additional exploration into the collected data, a profile of the AFM tip can also be estimated with the software; this can also bring meaning to the non-zero width of step edges. To do this in Gwyddion, use the *Data Process -> Tip -> Blind Estimation* function.

4. EXPERIMENTS

4.1 Measuring the Calibration Grid

The first experiment is to measure a calibration sample to verify the accuracy of the piezos. The grid is inside the box that is labelled as “AFM calibration grid NS32400”. The feature sizes should be 4μm by 4μm, with a step height of 25nm. Explore different scan parameters based off of the values in Section 3.4, to see if you can obtain a better contrast image. If the scan appears unclear or irregular, see Section 3.6 for troubleshooting help. The obtained AFM image should be similar to Figure 14 below. Note the blue arrow on the left side of the scan image is the current scan location. You may want to scan in several directions in order to better calibrate both the X and Y directions.

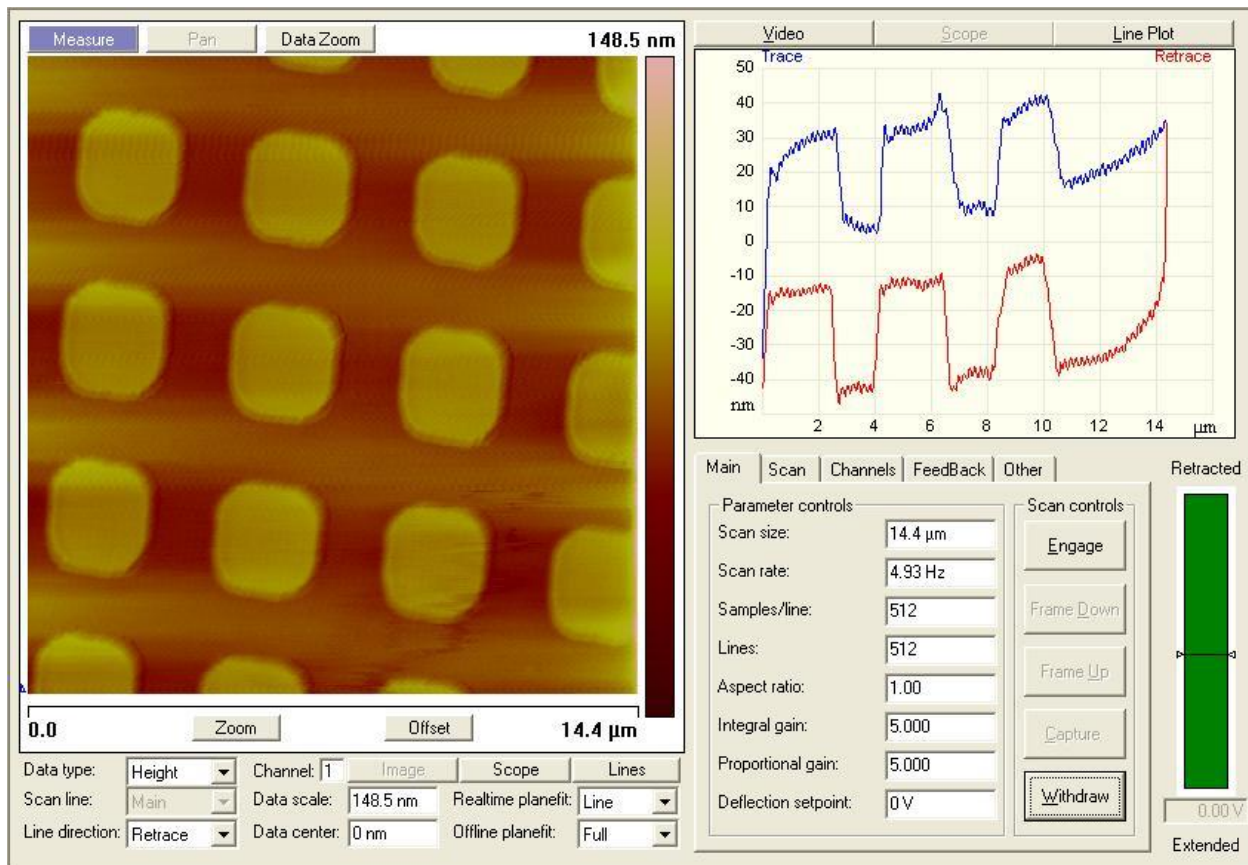


Figure 14: Sample Image for the Calibration Grid

After a satisfactory image is obtained, use the Gwyddion software on the desktop to open the .001 file - see section 3.4 on how to obtain this file. Carefully construct a line measure across one of the steps in a parallel direction and record all relevant dimensions (length & height of the step, width of the step edge), see section 3.7 for how to do this. Do this several times.

Guiding questions:

1. What is the mean and standard deviation of the periodicity of the patterns? Note that the given 4 micron dimension is the periodicity, and not the width of a square
2. What is the mean and standard deviation of the step height of the patterns?
3. How much is the measurement off from the nominal lengths, in 0 degree and 90 degree scan directions? If the lengths are significantly different from the calibration grid nominal values, calculate scale factors in order to convert any length measurements obtained in later experiments.
4. What is the mean and standard deviation of the width of the step edge, in 0 and 90 degree scan directions? This will give you information about the size and shape of the tip (How?).

4.2 Measuring the Bare Silicon Substrate

Many measurements that are not made on bulk samples require a substrate to hold the sample of interest. Usually, Silicon (Si) wafers prepared in the 001 crystal orientation are used because they can be polished down to a very flat surface and are easy to obtain as they are used for electronics. Here, the bare substrate is scanned to establish a base for the surface roughness.

First clean the sample with acetone and ethanol. If this is the first time you are handling a particular sample, place the sample in a beaker with acetone and clean it in the sonicating bath inside Rob's office for about 10 minutes. If you know the sample is relatively clean, simply wipe it with an acetone-soaked Q-tip. After any acetone cleaning, wash the sample with ethanol to rid the surface of acetone residue. Be careful as to not touch the surface with anything, except for holding the sample at the edge with tweezers.

Scan the Si substrate using the method outlined in the previous sections and export the data, see section 3.4 for instructions. Then use Gwyddion to analyse the roughness of the data collected, see section 3.8 for details.

Take force distance curves with several different tips of varying stiffness. Be careful when using the smaller tip, as you may break the bigger tip (which dips lower than the smaller tip) if you don't pay attention. Refer to

Guiding questions:

1. What is the roughness of the surface from the Gwyddion roughness measurement feature? If you have time you may want to calculate this yourself by finding the standard deviation of the data. How does this compare to the industry standards for silicon wafers?
2. Find the true equilibrium distance between the tip and the surface. The tip is usually made of Silicon as well. How does this value compare to the interatomic distance between Silicon atoms? Why might it be different?

4.3 Measuring Step Edges in Graphite

Graphite is a material made up of many layers weakly bonded to each other. There are step edges between these layers on the surface of a graphite sample. Imaging graphite and resolving the step edges is a challenging exercise. Resolving a step edge provides an upper bound on the noise floor of the instrument. The noise in the signal on either side of a step edge gives a rough estimate of the noise floor. Since the graphite step edges are perfectly sheer, the lateral extent of the step edge in the AFM signal is indicative of the lateral noise of the instrument and the size and shape of the tip.

Obtain the Highly Ordered Pyrolytic Graphite (HOPG) sample and exfoliate it with a piece of adhesive tape. This removes a few layers off of the sample surface and ensures the image will be taken on a clean surface. Again, note to carefully switch samples as a sample of a different thickness can hit the tip before approach.

Perform the coarse adjustment of the sample height in the usual way as described in section 3.2. To get a good image, it is recommended to do the more elaborate alignment procedure outlined below to position the laser diode at the vertex of the cantilever. Adjust the photodiode to get the maximum A+B signal. If the laser diode is positioned at the vertex this should be 4-5 V. To check that the laser diode is reflecting only off of the vertex, adjust the spot position in the transverse direction (parallel to the substrate). The A+B signal should change from 0 to a single maximum and back to 0 in around a quarter of a turn of the adjustment knob. If this is not the case adjust the spot position further away from the substrate. If there are two maxima the spot is reflecting off of both cantilever legs and it must be adjusted closer to the vertex. Once the spot is located properly, move the DVM display switch to the DOWN position to see the A-B voltage. Adjust the photodiode until this is between -3 and -4 V and engage the tip.

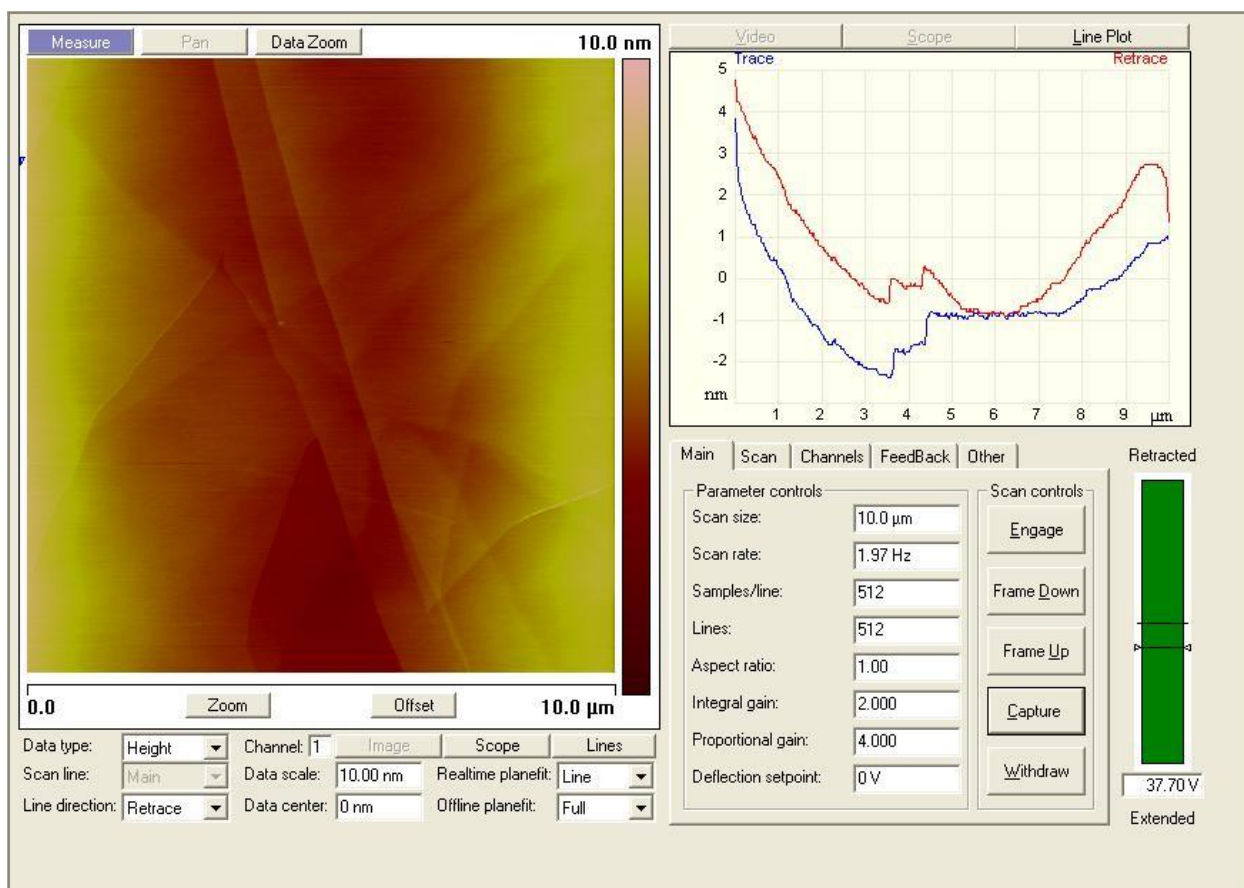


Figure 15: Sample Image for the graphite

As the AFM scans, observe the signal trace in the Nanoscope software to get an idea of the height of any step edges that are apparent. The amplitude of the fluctuations in the regions between these step edges is indicative of the noise of the instrument. Export the scan and measure the step edges with Gwyddion.

Perform force distance curve scans over several spots. You may have to change the setpoint to see more of the curve.

Guiding Questions:

1. What is the height of the step edges? How does this compare to the interplanar distance of graphite in the literature?
2. What is the width of the step edges? Are they different in 0 and 90 degree scans? Change the scan angle by both electronically (via the software) and physically (via rotating the sample). Why would the two methods be different?
3. Why would there be hysteresis in the trace-retrace curves? Why is there a “bowling” effect, where the sides of the scan are higher?
4. How does the F-D curves compare to that on Silicon? Can you conclude if graphite is softer or harder? Quantitatively compare them by calculating the spring constant of the sample.

4.4 Scanning Exfoliated Graphite Sheets

An AFM can measure the thickness of a layer of material deposited on a substrate and take force distance curves to measure elastic properties. Therefore it is a good instrument to study the

elastic modulus of graphite flakes as a function of the number of layers. However, this experiment is extremely challenging and luck-dependent, as it is difficult to find good candidate flakes of the desired size and thickness.

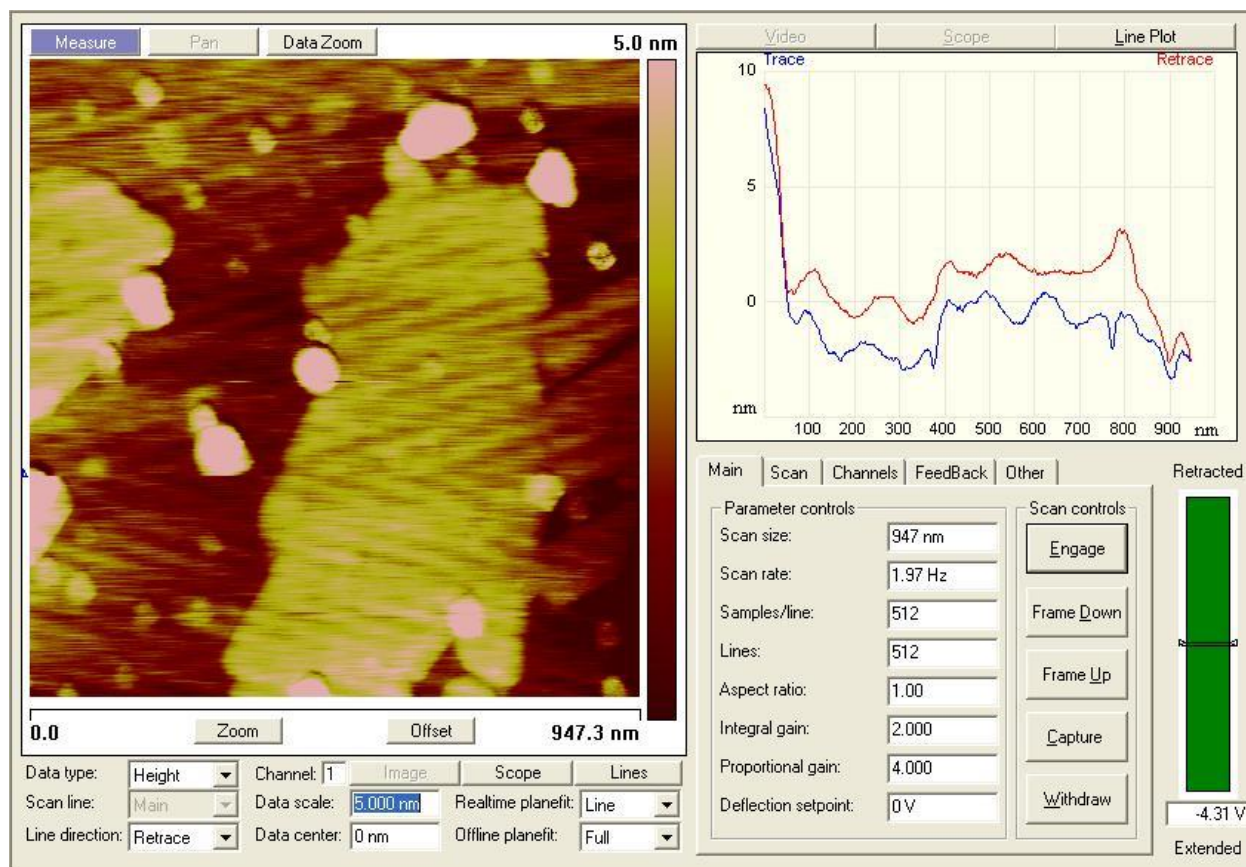


Figure 16: Sample Image for a thin graphite flake. The thickness of the centre piece is approximately 2-3 nm.

To deposit graphite flakes of various thicknesses on a silicon substrate, first use a piece of scotch tape to exfoliate some layers from a HOPG sample. This is done by attaching a piece of tape to the HOPG sample and removing it. Then, transfer some of the layers from the original piece of tape to a new piece. Do this repeatedly until the graphite on the original piece of tape is no longer shiny. Put a drop of acetone on the tape to dissolve the adhesive and then press the tape onto a Silicon substrate. Remove the silicon and visually inspect the surface. There should be very faint marks, indicating that some graphite material has been transferred. If under several angles of light you do not see anything, try the transfer again. Usually it is a good idea to do this several time to transfer as many flakes as possible

Using the TV monitor and the sample translation knobs, identify pieces of candidate graphite sheets and Image them with the steps mentioned in Section 3.3. Start with a large scan size of around 10 μm and use the zoom function to magnify the thinnest flakes found. Then perform force distance curves on the thin flakes. Repeat this for flakes of varying thicknesses. The zoom function does not work perfectly, so a few tries may be necessary correctly zoom in on the right region. Then capture the file and analyse it in Gwyddion to make sure the step seen is in fact a single graphite step edge from the substrate.

Perform F-D curve scans over different pieces of varying thickness.

Guiding questions:

1. Label all the images you print out with the step edge heights. What is the distribution of flake thicknesses you see?
2. Calculate the spring constant of the samples with different thicknesses. How does the stiffness change with thickness? Would this be different if you had a different substrate?

5. FURTHER INVESTIGATION

5.1 Other layered materials

HOPG is not the only layered material that is suitable for AFM studies. Other materials include mica, MoS₂, TaS₂ and much more. If the APL has obtained some of these materials, you may want to repeat the experiment for graphite on these samples as well. Confirm that the step edges correspond to the inter-layer distances, and comment on any effect of the material structure on the surface stiffness.

5.2 Carbon Nanotubes

Two bottles of carbon nanotube samples have been donated to the experiment by a group in condensed matter physics. The nanotubes are suspended in a toxic organic solvent; therefore you are required to wear gloves and work with caution when dealing with this experiment.

First shake the bottle well to ensure the suspended nanotubes are dispersed. To prepare the nanotubes for scanning, simply use a pipette and take up a very small amount (<1mL). Drop a small droplet on a clean Silicon substrate and let the solvent dry. Eventually only the nanotubes will be left on the surface of the substrate.

Identify potential candidates on the top-view camera screen, and perform scans with the AFM. Measure the height and width of the nanotubes. Perform F-D curve scans over different regions of the nanotube.

5.3 Silicon polymers

Further investigation for this laboratory can be to compare these results with other materials such as silicon polymers that can be made in the lab. As can be seen in Figure 17, the Young's modulus is expected to vary with the amount of crosslinker agent used. The process in making such extra samples is easy: mix Sylgard 182 Silicone Elastomer Base (e.g. 2g) with the desired ratio of Sylgard 182 Silicone Elastomer Curing Agent (e.g. 0.5g), then heat at 150 C for 15 minutes.

Perform F-D curve scans for several different mixture ratios.

Warning: The crosslinker breaks down into formaldehyde vapour when above 180C. Do not exceed this temperature!

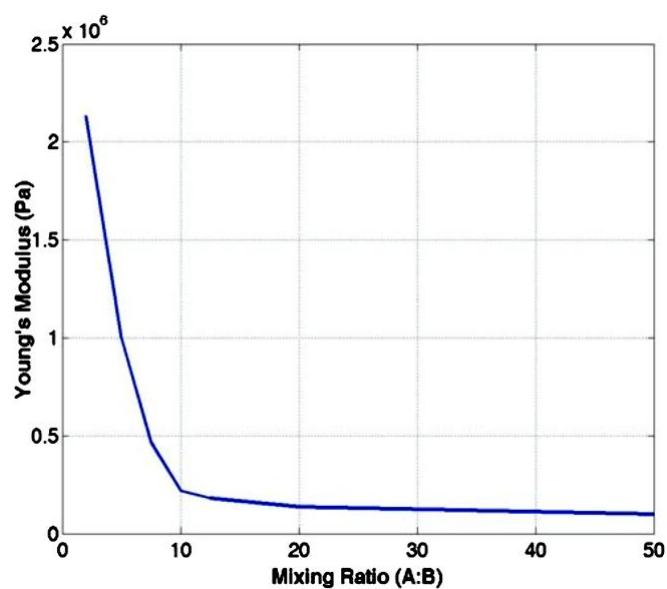


Figure 17: Young's Modulus vs Crosslinker Silicon Base mixing ratio. (Image from M. Liu and Q. Chen, *Characterization study of bonded and unbonded polydimethylsiloxane aimed for bio-micro-electromechanical systems-related applications*, Journal of Micro/Nanolithography, MEM, and MOEMS, 6 (2007) 023008; <http://dx.doi.org/10.1117/1.2731381>).

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