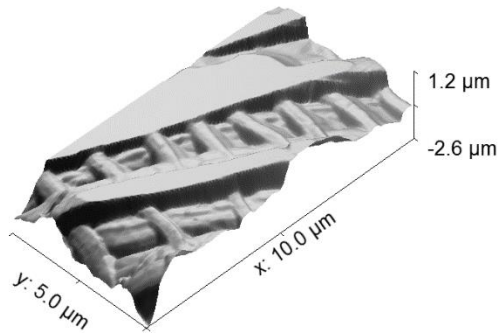


Atomic Force Microscope



Preparation

Before our first appointment to use the microscope, please watch the video tutorials at <https://www.afmworkshop.com/atomic-force-microscope-animated-tutorials/>. The geometry of our AFM stage is much different from the geometry in the tutorials, but the basic concepts are the same.

There are some scans that may take 20 minutes or more to complete. You can relax while the microscope is scanning, so I recommend bringing something to do. Or, you can work on the [force-curve simulation](#) whenever you like, regardless of whether you've taken any data. The simulation is not trivial, so please allow sufficient time to work on it.

Overview

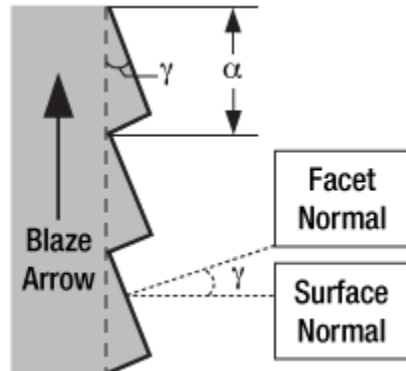
You will obtain images of the microstructures in [a DVD](#), [a CD](#), [a diffraction grating](#), and [a butterfly wing](#). You will also obtain [a force curve](#) (force between sample and tip as a function of the distance between the sample and the cantilever rest position) and [simulate the force curve](#).

DVD and CD

- Obtain AFM images of the data structures of a DVD and CD, following the [Imaging Procedure](#) and [Image Analysis](#) below.
- Determine the width, length, and height of the bumps. Determine the pitch (the distance between rows).
- From the density of bumps in the image, estimate the storage capacity of the disk.
- Shine a laser on the surface to determine the spacing of grooves acting as a diffraction grating. What distance in your image should you compare this to?
- Based on measured bump heights and http://www.hk-phy.org/articles/cdrom/cdrom_e.html, what wavelengths are used to read the DVD and CD? You should get a fairly accurate result for the DVD, though the CD may be too mangled. Explain how data is stored and read in non-recordable DVDs and CDs.

Diffraction Grating

- Obtain an AFM image of the grooves. Determine the groove spacing and the blaze angle, defined in the figure (from <https://www.thorlabs.com/tutorials.cfm?tabID=0ca9a8bd-2332-48f8-b01a-7f8bf0c03d4e>).



- Compare the groove spacing in your image with the spacing determined by shining a laser through the grating.
- If the grooves were simply flat notches, most of the light would go straight through. The grating is engineered to maximize the intensity of 500 nm light in the first order of diffraction ($m=1$). Specifically, consider incident light perpendicular to the un-grooved side of the grating. The light refracted by the grooved surface is parallel with the light in the first order of diffraction. Derive an equation to satisfy this condition. The blazing does not affect the angles (relative to the surface normal) at which constructive interference occurs. Use your equation and your measured groove spacing and blaze angle to determine the refractive index of the glass.

Butterfly Wing

- Obtain an image of the microstructure in a butterfly wing.
- There are some very sophisticated quantitative models of how the microstructure of the wing produces the colors we see. Briefly review a sampling of the literature.

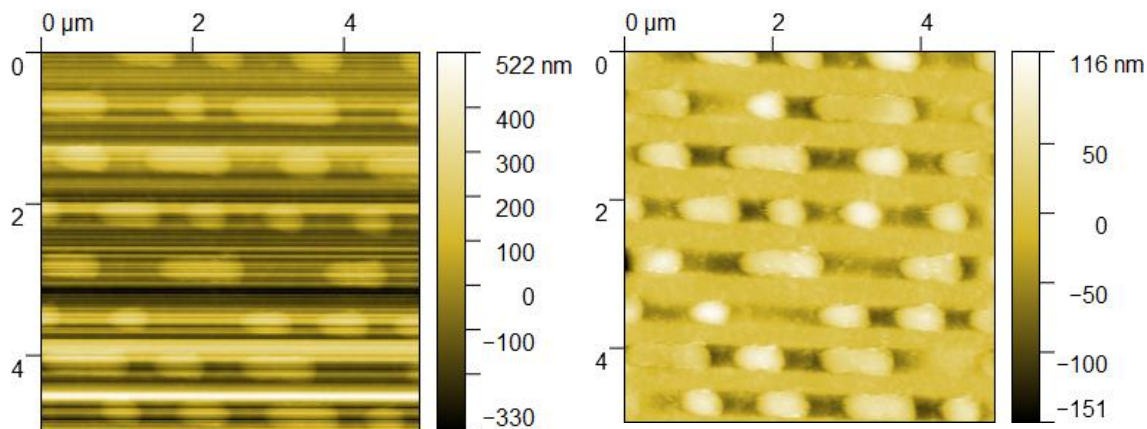
Imaging Procedure

- Turn on the two NanoScope electronics boxes.
- Turn on the light by turning the knob on the small box on top. You'll adjust it later.
- Open the Nanoscope software.
- Click the microscope icon in the upper left.
- Physically open the microscope lid. It will fall on your head unless you're very careful.
- Notice three pairs of knobs on the microscope. The central pair of knobs adjusts the position of the laser beam on the photodetector. The top knob in the central pair adjusts the vertical deflection. Set this close to 0 V for tapping mode (which we usually use).
- Click on the icon with a yellow triangular shape. This allows you to focus on the cantilever. You may need to adjust the illumination (the knob on the small, top box). If the cantilever is blurry, bring it into focus using the trackball. Hold down the Focus button (on the lower left) and roll the ball up or down. You can click Zoom In or Zoom Out for different magnification. Once the cantilever is in focus, click Ok.

- Click the icon with red rectangles and a magnifying glass. This allows you to move the stage and focus on your sample.
- To place your sample on the stage, you may need to move the stage toward you by rolling the trackball toward you. You can hold Lock (on the upper left) to keep the stage moving. Try not to touch the sample or the stage with your hands. Try to use tweezers if possible.
- Place your sample so that it covers as many holes in the stage as possible. Turn vacuum on by flipping the black switch up.
- Be extremely careful that the stage is low enough to get your sample safely under the cantilever. You can lower the stage by holding Focus and rolling the ball away from you.
- Use the trackball to place your sample under the cantilever. Carefully raise the stage to focus on your sample. It's probably easier to focus initially when zoomed out. You can move the stage with the trackball to view different parts of your sample. You can click AutoFocus when the focus already looks good.
- Under the Microscope menu, confirm that the profile is Tapping AFM.
- Click the tuning fork icon. Click Auto Tune to confirm the resonance frequency of the cantilever. Return to image mode.
- Adjust scan controls and other parameters as needed. The most commonly adjusted parameters are scan size, scan rate, and data scale.
- Click the green arrow at the top left to bring the tip into contact with your sample and begin taking an image.
- Once the image starts to appear, click the sine wave icon. This shows you the deflection signal as the microscope scans back and forth. Both signals should be the same. If they are not, the tip velocity may be too high such that the microscope can't accurately follow the topography of the sample. Alternatively, the scan size may be too small such that you're zoomed in beneath the scale of the structure.
- Under the Capture Menu, you can change the filename. A three digit number will automatically be added as an extension. Select capture to save the data from the next complete scan. At the very bottom of the screen, you will see Capture: Next. The scan is finished when you see Capture: Done.
- Click on the graph icon in the upper right. I've had to do this before I was able to successfully copy data.
- Copy your file onto a flash drive, CD, or floppy. The USB port hasn't been working.
- Click on the microscope icon to return to the microscope controls
- Click the red arrow at least once to disengage the tip from the sample. To move the stage laterally before removing your sample, click the Focus Sample icon (red rectangles and magnifying glass) and use the trackball.
- Turn off the vacuum and carefully remove your sample.
- If you're done for the day, close the Nanoscope software, and turn off the electronics boxes and the light.

Image Analysis

- Open the image file in Gwiddion. This program is on the computer connected to the microscope. If you like, you can download it for free on your own computer.
- The image file produced by the Nanoscope software can be opened only in Gwiddion or the Nanoscope software. Once you open a file in Gwiddion, you can Save As various types of graphics files under the File menu, so that you can include the image in your lab report.
- If your image has horrible streaks as seen on the left, you can hover over the icons under Data Process to find “align rows using various methods.” You can try different methods until you like the result.



- If you want to create a 3D graph of the image, click the appropriate icon under View. Click the gear with the “+” in it to show more options. Under the Light and Material tab, you can try displaying the image with “Lighting” or “Overlay.” This may improve the aesthetics. To save the 3D image, click the Save button along the bottom.
- To graph height along any straight line, click on the Extract Profile tool. Click on your image, hold down the button, and move the cursor to create a line along which to graph the height. Use the mouse to adjust and move the line you create.
- Once you like the graph in the Profile window, click Apply. To save the graph, select Export Bitmap under the Graph menu.

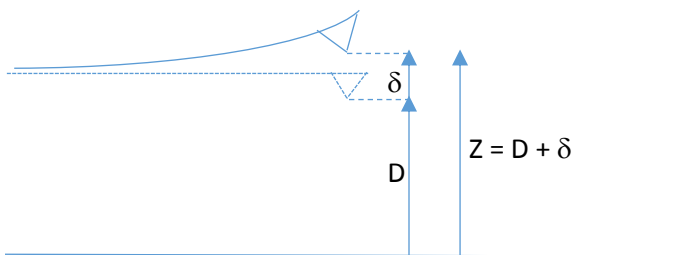
Force curve

- To make sure you don’t damage a delicate part of the sample, obtain the force curve on a surface we don’t plan to image, such as the glass that the butterfly wing is mounted on, or the silicon that the CD is mounted on.
- We will use Contact mode, not Tapping mode, because the manufacturer warns that the tip is easily damaged when acquiring a force curve in Tapping mode.
- Set the vertical deflection to -2 V.
- Under the Microscope menu, choose the Contact AFM profile. Make sure Microscope mode is set to Contact under Other Controls.
- When the probe is positioned where you want it, click the green arrow to begin taking an image. You don’t need to save this image.

- While the microscope is scanning, select Force Mode → Calibrate under the view menu.
- Change the Ramp size to adjust the scale of the horizontal axis.
- Change the Data scale to adjust the scale of the vertical axis.
- Raising/lowering “Z start scan” moves the curve to the right/left.
- Raising/lowering “Data center” moves the curve vertically.
- When you’re satisfied with the curve, Capture it.
- Click the eye icon in the upper right to return to image mode.
- Click the graph icon where the eye icon just was.
- Gwiddion can’t open the force curve. To export the image from the Nanoscope software, highlight the file you just saved. Under Utility, select TIFF Export.
- [Simulate a force curve as described below](#), and qualitatively explain the features of the force curve.

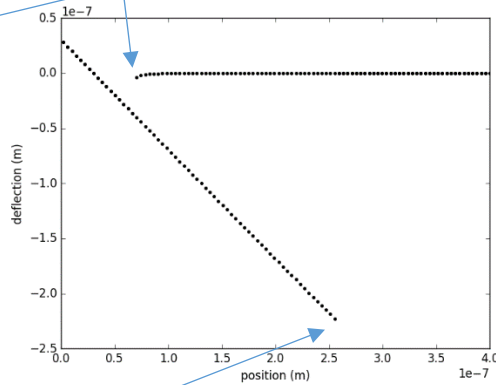
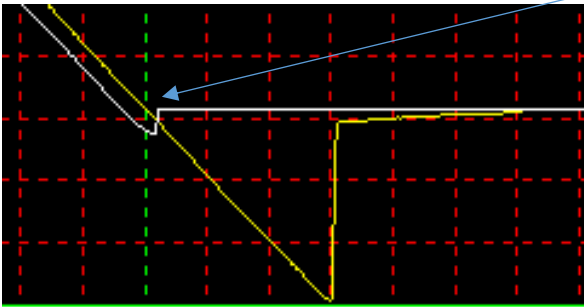
Simulation of the Force Curve

- The force curve indicates cantilever deflection, δ , as a function of D , the distance between the surface and the undeflected tip position. δ is usually negative: the tip is attracted toward the surface unless D is extremely small, in which case the surface repels the tip.



- We would like to reproduce the qualitative features of the force curve: the “snap on” as the probe approaches the surface and becomes attracted to it, and the “snap off” as the probe withdraws from the surface and overcomes the attractive force.
- Below, the figure on the left shows measured data, and the figure on the right shows a simulation. In both cases, the figures plot δ vs. D . The simulation doesn’t show the vertical lines that indicate discontinuities in deflection (the “snaps”).

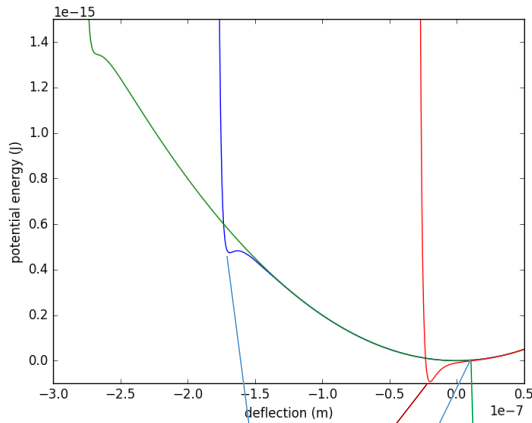
“snap on” during approach due to attractive forces



“snap off” (deflection jumps up to 0) during withdrawal

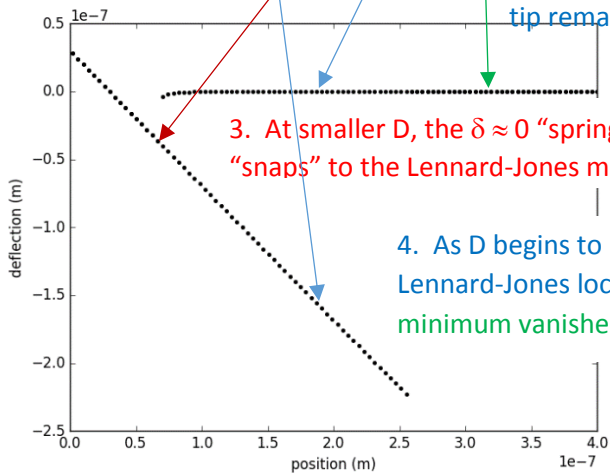
- To understand these curves, we need a model for the forces acting on the tip, or, more conveniently, the total potential energy of the system.
- One form of potential energy is the “spring” energy stored in the cantilever as it deflects: $\frac{1}{2}k\delta^2$, where k is the spring constant. In the absence of other forces, the equilibrium position of cantilever is at the minimum energy of this potential well, $\delta = 0$ (no deflection).
- The other form of energy is due to the interaction between the tip and the surface. At very small distances, the tip is repelled from the surface because it can’t pass through the surface. At larger distances, the tip is attracted to the surface by van der Waals forces (forces between neutral atoms due to spontaneous polarization into dipoles, oriented to attract each other).
- For simplicity, we will use the Lennard-Jones model for the potential energy between the tip and the surface: $\epsilon \left[\left(\frac{Z_m}{Z} \right)^{12} - 2 \left(\frac{Z_m}{Z} \right)^6 \right]$, where Z_m is the equilibrium distance between the tip and surface in the absence of other forces, and ϵ is the depth of this potential well.
- Now we can plot total potential energy as a function of tip deflection δ , for different values of probe position D . We see that sometimes there are two stable equilibrium positions (local minima) for δ , and sometimes only one. In all cases, we see the Lennard-Jones “dip” superimposed on the “spring” parabola ($\frac{1}{2}k\delta^2$). **The force curve shows us the stable equilibrium positions of δ as a function of D .**

The graph to the left shows potential energy as a function of deflection (δ) for three values of probe position (D):



- Green is for large D : the only minimum is at $\delta = 0$ (cantilever undeflected, away from surface).
- Red is for small D : the only minimum is in the Lennard-Jones “dip” (tip attracted to surface).
- Blue is for intermediate D : there are two local minima, one at $\delta = 0$ (cantilever undeflected, away from surface), and one in the Lennard-Jones “dip” (tip attracted to surface).

1. At large D , the only minimum of potential energy (in the green curve above) is due to the “spring” potential energy of the cantilever. The tip is too far from the surface to be significantly attracted to it. 2. As D decreases, a second local minimum appears (in the blue curve), but the tip remains for now in the $\delta \approx 0$ well.



3. At smaller D , the $\delta \approx 0$ “spring” minimum vanishes, and the tip “snaps” to the Lennard-Jones minimum (in the red curve above).

4. As D begins to increase again, the tip remains attracted to the surface in the Lennard-Jones local minimum until D is so large that the Lennard-Jones minimum vanishes and the tip “snaps” off the surface to $\delta \approx 0$.

- So, the mostly horizontal line in the force curve, at $\delta = 0$, represents an undeflected cantilever, largely unaffected by the surface. The diagonal line in the force curve represents a tip trapped in the Lennard-Jones potential well near the surface. For large D , there’s only one stable equilibrium, an undeflected cantilever. For small D , there’s only one stable equilibrium, a tip in the Lennard-Jones potential well. At intermediate D , there are two stable equilibria. This permits the hysteresis in the force curve. The tip stays in one potential well until that well vanishes (as D changes), and then the tip “snaps” to the only potential well that remains
- Simulate the force curve by plotting the equilibrium value(s) of δ for different values of D . I used $k = 0.04 \text{ N/m}$, $\epsilon = 10^{-16} \text{ J}$, and $Z_m = 30 \text{ nm}$. These are not intended to be realistic; the model is too simplistic to fit to data.