Atomic Force Microscopy (I)

- Optical Grating AFM and the thermal noise measurement

2.674 Lab 10

Spring 2016

Pappalardo II Micro/Nano Laboratories

AFM Imaging (with home-made AFMs)

I. Safety Notes

This lab involves the use of a laser (5 mW at 635 nm) as part of the atomic force microscope apparatus. Remember to not stare into the beam, deliberately or for extended amounts of time, i.e. several seconds! . It is normal that you will see some diffuse reflections of the red laser spot as you look through the microscope, and this is all that you should experience unless the apparatus is set up incorrectly. It is not normal for those reflections to feel at all uncomfortable. **If your eyes feel uncomfortable at any point, look away!** Inform an instructor, who will help you adjust the apparatus to remove the offending reflection. According to safety standards, this will provide adequate protection against this kind of laser, since it takes longer exposures than the momentary exposure described above to harm your eye. The Maximum Permissible Exposure (MPE) to the eye from a visible, continuous wave light source is 2.55 mW/cm^2, according to the ANSI Z136.1 standard.

This lab also involves a 40 V power supply that supplies voltage to the piezo tube that positions your sample in the AFM. The 40 V terminals are covered up, and you do not need to access them. **Do not attempt to get underneath the covering and access the terminals**.

II. Introduction

Atomic force microscopy (AFM) is one of a range of different imaging techniques that are used to gain information about structures at the micro- and nanoscale. These techniques have been developed over the years to obtain different kinds of information about small-scale structures. We will briefly describe several approaches to imaging to put AFM in its proper context.

Optical microscopy: The original technique for imaging small structures was the optical microscope. Optical microscopes continue to provide useful information about the structure of small objects, but only for structures that are greater than the wavelength of the light being used in the microscope; smaller structures cannot be resolved in a conventional optical microscope because of the diffraction limit.

Electron microscopy: Electron microscopes shoot a beam of electrons at a surface and then capture the electrons that are either backscattered or emitted from the surface in order to obtain information about underlying structures. Because electrons effectively have a shorter wavelength than the wavelengths of light, they provide higher resolution. Scanning electron microscopes (SEMs) provide images of sample topography, while transmission electron microscopes (TEMs) provide images of the internal structure of a sample.

Scanning tunneling microscopy: Historically, scanning tunneling microscopes (STMs) provided yet another way of obtaining information about a microscale or nanoscale sample's structure. In scanning tunneling microscopy, a conducting probe with a sharp tip is raster-scanned over a surface. A voltage is applied between the sample and the tip; if the tip and the sample surface are in close proximity (on the order of several ångström; 1Å = 0.1nm), electrons will tunnel from one to the other and a current will flow. This provides an extremely sensitive measurement of the sample's topography, since the electron tunneling is exquisitely sensitive to the distance between the tip and the sample.

Atomic force microscopy: In AFM, a probe is scanned across the surface in order to obtain information about its topography (or in variations of the AFM technique about other properties, such as elastic modulus or chemical composition). In general, AFMs drag a tip across the substrate much like an old record player used to pull its tip through the grooves in an old vinyl record, though non-contact and tapping modes are also used. However, unlike both the STM and the record player, the AFM contains at its heart a MEMS device that makes its operation possible. This MEMS device is a micromachined cantilever beam with a sharp tip at its end. Because the cantilever is long and thin, it is quite flexible. Among other benefits, this allows the AFM's cantilever beam to interact with the underlying surface with very low forces so as not to damage the sample that is being imaged or dull the sharp tip that is doing the imaging.

One interesting aspect of these different imaging techniques is the fact that they often reveal specific types of information that might not be visible using other techniques. You may find that there are features on some of your samples that are not at all visible under the optical microscope.

Atomic force microscopes were first reported in 1986 [1]. Since then, a wide variety of variations of the AFM concept have appeared. These include a large diversity of cantilever designs, different methods of reading out how much a cantilever has deflected when it interacts with a surface, various distinct AFM operating modes, and a variety of ways of interacting with the surface. For example, conventional AFMs have three main operational modes. In contact mode, the cantilever tip is "pressed" against the sample surface. Repulsive forces between the sample and the cantilever cause the cantilever to bend, and the bending is recorded optically (more on that below). In non-contact mode, the cantilever tip is held a short distance away from the sample surface. At this distance, attractive forces between the cantilever and the sample cause the cantilever to bend, and the bending is once again recorded optically. In the third major mode, tapping mode, the cantilever oscillates back and forth above the sample. The amplitude of the cantilever's oscillation varies with the topography encountered by its tip, and once again the information is recorded optically. Beyond these three main AFM operational modes, there are other potential uses for systems that resemble AFMs; more broadly, such systems are referred to as scanning probe microscopes, or SPMs. For example, a specialized AFM cantilever could be made that records the magnetic or electrostatic interaction between the cantilever tip and the surface of the sample. Such data may then be correlated with topographical information to obtain a complete picture of the sample and its behavior.

Although there is a MEMS device at the heart of an AFM, it is important to keep in mind that there are a great many other aspects of the AFM system as well, without which it could not work. For example, the cantilever and the sample surface must be correctly positioned with respect to each other. This is conventionally accomplished with a piezoelectric tube scanner. (Piezoelectric materials undergo a strain when an electric field is applied across them. This strain is usually quite small. This renders piezoelectrics sutiable for positioning objects with fine resolution.) Piezoelectric scanners are used in our AFM system. For the AFM technique to work, the cantilever tip must not be pressed too hard onto the sample, and it should also not be too far away from the surface; therefore, there is often a feedback system associated with atomic force microscopes to maintain the appropriate tip – sample surface spacing. (Our AFMs do not have such a feedback system; it's up to you to bias the microscopes correctly in order to make sure that the cantilevers are positioned appropriately with respect to the sample. More on that later.) A third non-MEMS aspect of the AFM system is the method by which the cantilever bending (and hence the sample

topography) is measured and recorded. In conventional AFMs and in the course AFMs, this readout is usually accomplished optically.

There is a great deal that can be said about the MEMS devices that serve as the AFM cantilevers in the class AFMs. We will not address the MEMS devices in detail during this week's lab, but rather will focus on the "whole-system" issues of imaging, and on what we see when we image our samples.

III. Laboratory Objectives

In this laboratory, you will:

1) **Understand** how the atomic force microscope operates, and learn to operate it.
2) **Calibrate** the output signals from the atomic force microscope to enable quantitative results.
3) **Image** several different samples using the atomic force microscope and compare the results with imaging results obtained by other means.

IV. Details of the Atomic Force Microscope System Hardware

Different AFMs employ slightly different systems and structures in order to obtain their information. Conventional AFMs, like the ones we discussed in class, are sensitive to ambient vibration and must be carefully isolated from the environment. The AFMs that we will use, have been designed to minimize the systems' sensitivity to ambient vibration. The AFM design in the teaching labs is the result of work by Prof. Scott Manalis and Maxim Shusteff, who pioneered these teaching AFMs in the context of the MIT Bioengineering class 20.309 (Biological Instrumentation and Measurement Laboratory, Fall) [2]. Parts of the manual that your are reading right now are from the instructions created for the 20.309 lab. The teaching AFMs were conceived based on earlier research work on AFMs [3, 4]. Before you begin to work with this tool, it is important to understand how this particular system is structured, and how its components work together to allow you to obtain AFM images. The following section describes the various components of the AFM you will use in the lab, and particularly how they differ in their operation from a commercial AFM. A photo of the AFM setup is provided in Figure 1 for you to refer to as you learn about the instrument.

Motion control system

To acquire an image of nanoscale surface features, an AFM needs to scan its probe tip over the sample surface. Our microscopes are designed with a fixed probe and a moveable sample (also true of some, but not all, commercial AFM systems). For our experiments, we will always move the sample rather than the tip in order to obtain relative motion between the tip and the sample. The sample is actuated for scanning and z-modulation measurements by a simple piezo disk, shown in Figure 2. The piezo disk is controlled from the computer using scanning software implemented in MATLAB$^{\text{È}}$. The software is described below.

For vertical motion (positioning along the z-axis), there are two regimes of motion:

1. *Manual (**medium to coarse**):* turning the adjustment knob with your hand (clockwise moves the stage up). The knob is located where the picomotor is indicated in the photograph above. (We do not have a picomotor, but the knob is located in the same place.)

2. *Piezo-disk (**fine**):* actuating the piezo disk over a few hundred nanometers using the MATLAB® software.

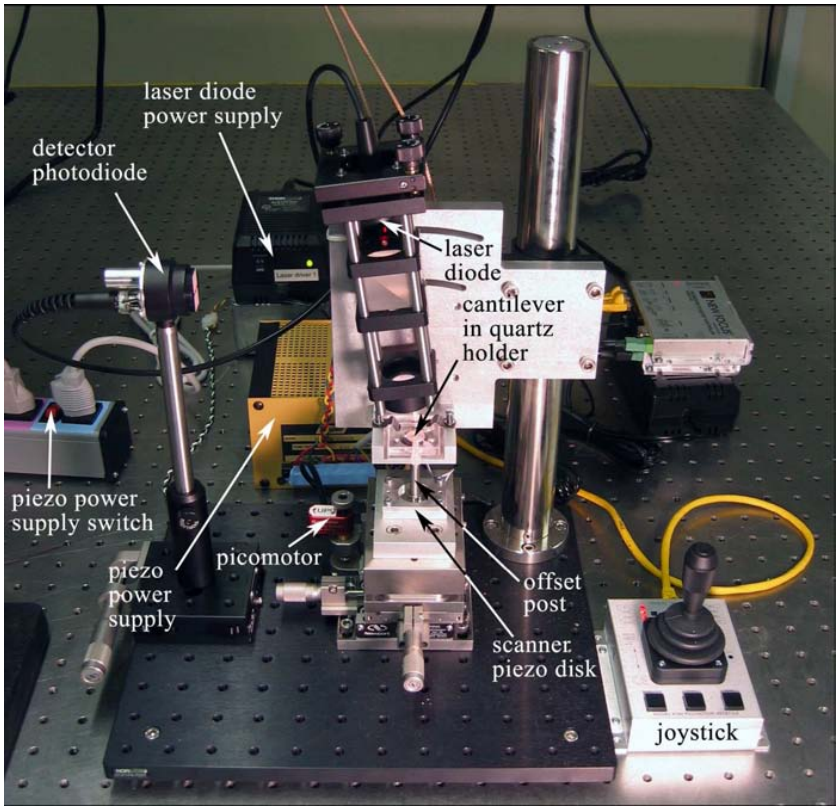


Figure 1: The AFM setup, with major components indicated.

For x-and y-axis positioning (in the sample plane), coarse movements are accomplished with the stage micrometers, and fine (several micron) movements are attained using the piezo disk.

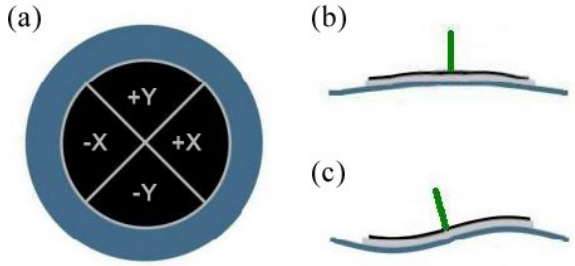


Figure 2: Schematic of the piezo disk used to actuate the AFM's sample stage. The circular electrode is divided into quadrants as shown in (a) to enable 3-axis actuation. When the same voltage is applied to all quadrants, the disk flexes as shown in (b), giving z-axis motion. Differential voltages applied to opposite quadrants produce the flexing shown in (c), which moves the stage along the x-and y-axes, with the help of the offset post, represented here by the vertical green line.

Optical system

These microscopes use a somewhat different optical readout from a conventional AFM to sense cantilever deflection. A standard AFM bounces a laser beam off the reflecting back surface of a cantilever, like the ones described in lecture. When the cantilever interacts with the surface of the sample, it bends either up (from repulsive forces) or down (from attractive forces). As the

cantilever bends, the angle of its back surface of which the laser beam bounces varies, and the angle of reflection of the laser light changes. By the time the laser beam reaches the position-sensitive detector at some distance from the cantilever, this change in angle has resulted in a change in laser spot position on the detector. The position-sensitive detector is composed of either two or four photodiodes that are measured together to quantify the position of the incident laser spot. This allows determining the cantilever angle with high precision and therefore quantifying the variation of tip height above the scanned surface. This is how a conventional AFM setup uses optical effects to detect deflection.

The AFMs that we will use in this class use a different optical readout mechanism that includes not only a laser but also diffracting cantilever structures that are designed for this particular purpose. Instead of the conventional AFM tip geometries including just a single cantilever beam for scanning, this structure includes a) a cantilever beam for scanning and b) two shorter beams on either side of the scanning beam. Both the main cantilever beam and the two shorter beams have narrow finger structures sticking out on the side. These fingers are what we call interdigitated (ID); they interleave with each other in an alternating pattern, as is shown in Figure 3.

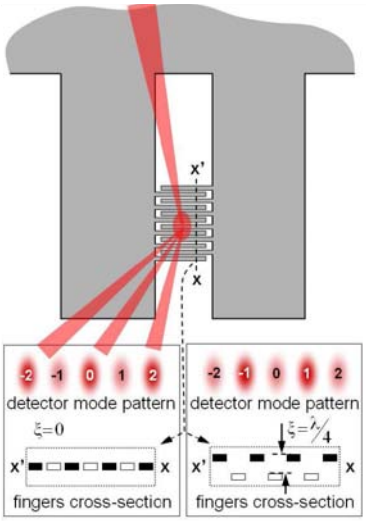


Figure 3: A sketch of the interdigitated (ID) interferometric fingers, with the detection laser shown incident from the top of the figure. When the finger sets are aligned, as in the left box above, the even-numbered modes are brightest, and odd modes are darkest. When they displace relative to each other by a distance of a quarter of the laser wavelength, the situation reverses, shown on the right. This repeats every $\lambda/4$*, in either direction.*

As in the case of a conventional AFM, changes in the position of the cantilever are detected by capturing the reflection of laser light from the cantilever structure using a photo detector. The difference lies in the mechanism by which the intensity changes on the photodetector. In conventional AFM systems the laser reflects of the backside of the tip-bearing cantilever. In our AFMs, we exploit the diffraction of light from the interdigitated fingers (IDs) to obtain quantitative information about the cantilever bending. Therefore, the laser spot must be lined up so that it bounces off the interdigitated (ID) fingers, which act as diffraction grating. If the cantilever beam deflects upwards, the fingers connected to it will also deflect upwards. The net result is that every other finger moves upwards (the ones connected to the scanning beam), while the other fingers (the ones connected to the side beams) stay put. This results in a predictable variation in the distribution of intensity in the gratings diffraction orders. Consequently, as the cantilever moves up and down,

resulting in a change in the diffraction grating's geometry, a characteristic intensity variation can be observed in the 0^{th} order of the diffraction grating and in any other order as well. It is the variation in intensity in the 0^{th} diffraction order that is measured by the photodetector.

If you shine a coherent light beam (for instance resulting from a laser, where the light is all in phase) onto a flat surface, it will do exactly what you expect: it will bounce off at a reflection angle that is equal in magnitude and opposite in sign to the light's angle of incidence. If you shine a coherent light onto a diffraction grating (in our case the ID fingers, as shown in Figure 3), the light will instead form a diffraction pattern. The individual spots in the gratings diffraction patterns are called diffraction orders, and are also sometimes referred to as "modes". The light that bounces off of the high fingers and the light that bounces off of the low fingers has to travel different distances before it joins up in the far distance. If the "high finger" light is out of phase with the "low finger" light in the far distance, there is a dark spot in the center of the diffraction pattern (the 0^{th} order). If the "high finger" light is in phase with the "low finger" light in the far distance, there is a bright spot in the diffraction pattern center. This effect is also occurring in any of the other diffraction spots. The net result is a variation of the intensity in all diffraction orders as a function of a change in the vertical distance between adjacent ID fingers

In this AFM, a diode laser with a wavelength λ = 635 nm is focused onto the ID finger structure, and we observe the brightness of the 0^{th} diffraction order using a photodetector. The photodetector is mostly covered up, so that light can only enter it through a narrow slit, which allows us to collect light exclusively from a single diffraction order. We choose to collect the grating's 0^{th} diffraction order as it has the highest intensity (this is true for all square gratings such as the ID fingers). Consequently, a deflection of the tip from topographic features of the sample results in a bending of the cantilever, entraining a variation of the diffraction grating geometry, which causes a variation in the intensity of the grating's 0^{th} diffraction order that translates into a proportional variation in the voltage signal provided by the photodetector. This variation in voltage signal as a function of tip height allows us to deduce topographic information about the sample's surface.

Qualitatively, it can be observed that the brightness will vary as the out-of-plane spacing between the ID fingers changes. However, the intensity I in the grating's 0^{th} diffraction order does not vary linearly with cantilever position. In fact, the variation is not even monotonic. Instead, the dependence of intensity I on the deflection ΔZ of the ID fingers is described by the following relation, which relates the wavelength of the laser light λ to the deflection:

$$I(\Delta Z) \propto \sin^2\left(\frac{2\pi}{\lambda}\Delta Z\right).$$

The intensity varies periodically, and its variation as a function of a specific deflection depends strongly on the ratio of ΔZ and λ (Fig. 4, left). If the intensity is on a sharply increasing slope (Fig. 4, right), the variations with deflection will look locally monotonic and almost linear. On a peak or in a valley, the intensity variation with deflection will be almost zero. This nonlinearity makes the sensor's sensitivity depend critically on the operating point along this curve at which a measurement is done. To make useful measurements, the deflection-induced vertical spacing between the ID fingers therefore needs to be biased to a spot on the $\sin^2$ curve where the function's slope is greatest, midway between the minimum and the maximum, as sketched in Figure 4 on the right.

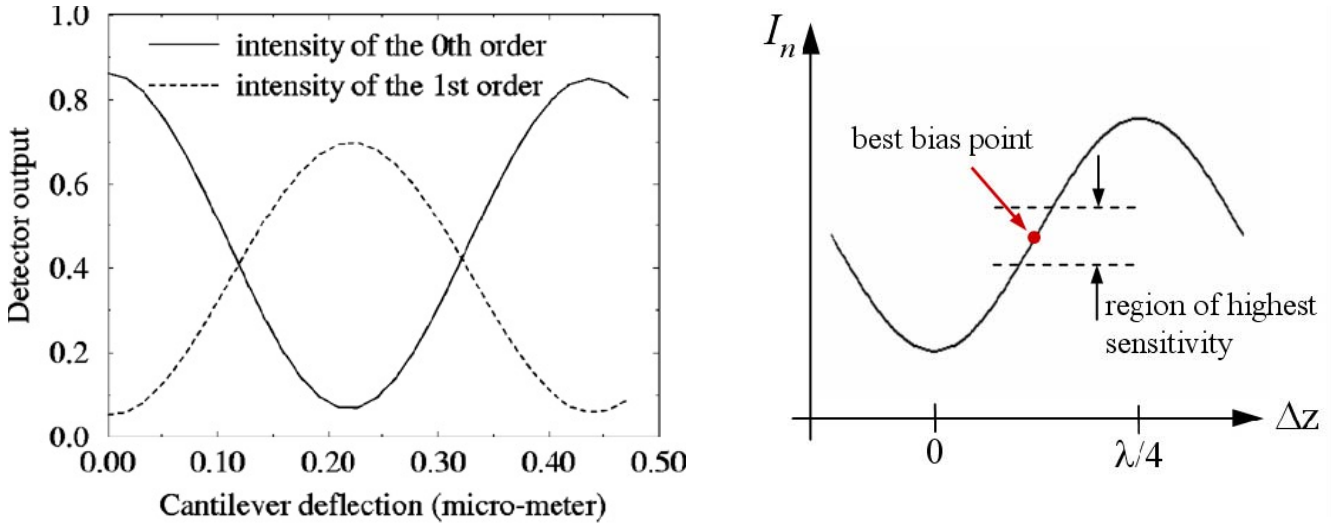
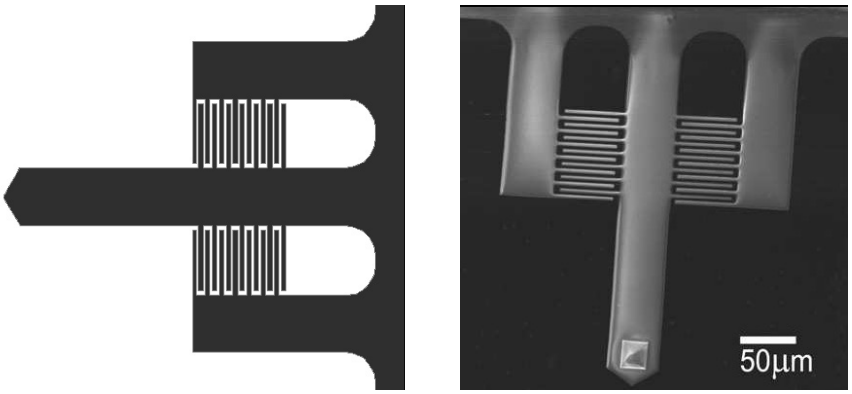


Figure 4: The characteristic output of the ID interferometric sensor. Left: the non-linear intensities of the 0^{th} and 1^{st} order modes as a function of cantilever displacement (from [4]). Right: the desired operating point for maximum deflection sensitivity is sketched here on the $\sin^2$ output characteristic of the ID fingers.

Due to residual strain in the silicon nitride from which the cantilevers are fabricated, the relative planar alignment of the two finger sets varies slightly over the area of the grating. (One set of fingers is higher than the second set of fingers on one side, and the second set of fingers is higher than the first set of fingers on the other side.) This variation is typically a few hundred nanometers in the lateral direction. Therefore, the bias point of the detector's output can be adjusted along the $\sin^2$ curve by moving the incident laser spot side to side on the diffraction grating.

Cantilever probes

The probes are shown in Figure 5 with relevant dimensions. The central beam has a tip at its end, which scans the surface. The shorter side beams to either side have no tips and remain out of contact. The side beams provide a reference against which the deflection of the central beam is measured; the ID grating on either side may be used.



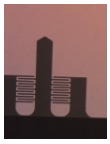
Application	Long Imaging	Thermal Noise	Modulus Measurement	Modulus Measurement
Length (±5 μm)	400 μm	350 μm	325 μm	250 μm
Width (±2 μm)	50 μm	60 μm	60 μm	60 μm
Resonance Frequency	~6000 Hz	~7000 Hz	~9000 Hz	~16,000 Hz
Force Constant	~0.02 N/m	~0.02 N/m	0.035 N/m	0.08 N/m
Schematic				

Figure 5: (upper) Plan view and (Lower) SEM images of the various cantilevers. We will use the long imaging beam (the first one). Its beam dimensions are length L = 400μm, and width b = 50μm. The finger gratings begin 118μm and end 200μm from the base. Thickness of all cantilevers is 800 nm.

Can you see why this structure might be less sensitive to vibration than a system with a single cantilever would be?

The cantilevers are designed to be flexible in the up and down direction so that a) they will be useful for scanning and b) it will be relatively more difficult to break the cantilevers by bringing them down onto the surface. Because they're flexible in that direction, they have a significant ability to bend out of the way as you bring them in contact with the surface. This up and down flexibility comes from the fact that the cantilevers are long and skinny. However, this is the only direction in which the cantilevers are flexible. If you try to bend them side to side, they will break because they are much wider than they are thick, and their sideway stiffness is therefore much higher. Similarly, they are also prone to break if you load them axially.

From a practical point of view, this has several implications. First, you should be careful about moving the sample up and down, but you should not be afraid to move it. We expect that some cantilever probes may be broken. If you break a cantilever, we do have replacements; the main consequence of breaking one is that you will then have to get a replacement probe in there and realign the laser. Second, you should be very careful not to drive an edge of the sample sideways into the probe, because that will definitely break it!

Detailed instructions on maneuvering the cantilever, the sample, and the laser with respect to each other are provided below in the instructions section of the lab manual.

Signal connections and data flow

The system should already be set up for data acquisition when you arrive to use the AFM. The following information describes how the data acquisition system is structured.

The AFM itself requires two signal inputs (X_{in} and Y_{in}) to drive the piezo actuator, which connect to the electronics board on the back of the head plate. They are provided by the computer's digital signal outputs (DAC0OUT and DAC1OUT). The computer also needs to read these signals in, together with the AFM signal output, so these become the three DAQ inputs.

The output from the AFM's photodetector is a current signal proportional to the brightness of the laser spot, which needs to be converted to a voltage (a 100 kΩ resistor to ground is used for this purpose). It's good to be able to amplify and offset this voltage at our convenience, so this signal runs through a Tektronix AM502 amplifier before it enters the DAQ board. Finally, during calibration, it's very useful to watch the detector signal as a function of stage movement in real time, on the oscilloscope screen, so we run these signals to the scope as well. All the described connections are illustrated in Figure 6.

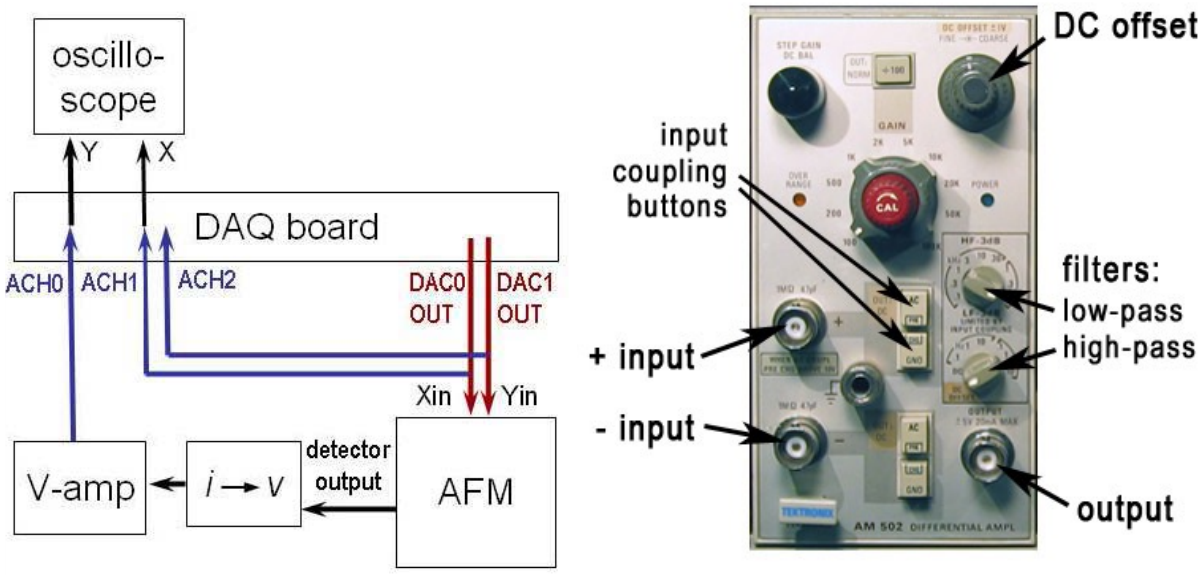


Figure 6: Left: Schematic of signal connections. Right: An AM502 differential amplifier. The gain is determined by the central red knob, together with the ÷100 button in the center above it.

The Tektronix AM502 is a differential amplifier, so it amplifies the voltage difference between the two input signals. Both inputs have DC, AC, and GND input coupling, like the scopes. The amplifier can be used single-ended if the (-) input is left unconnected and ground-coupled. The gain (amplification factor) ranges from 1 to 10,000, and is set by the red knob together with the "÷100" button above it. The instrument also has high-and low-pass filters. These are somewhat confusingly labeled "HF-3dB" and "LF-3dB" (see Figure 6). This does not mean "high-pass" and "low-pass," but refers to the "high (cutoff) frequency" of a LPF and the "low (cutoff) frequency" of a HPF. Therefore, HF-3dB is the low-pass filter, and LF-3dB is the high-pass filter.

Both filters are considered "off" when the low-pass is turned all the way up, and the high-pass all the way down. Finally, the lower (high-pass) filter knob also has two settings for controlling output signal DC level: dc and dc offset. When set to dc, the amplifier outputs the *actual* DC level of the input signal, multiplied by the gain. Set to dc offset, you can manually adjust the DC level using the knob at the upper right of the AM502. In both cases, the AC component is simply added on top.

V. Details of the AFM Software

The software that interfaces with the AFM is an application that runs in MATLAB. Its graphical user interface (GUI) is launched by typing `AFMControlPanel' in the MATLAB command

window. Its main function is to systematically scan the probe tip back-and-forth across the sample, recording the cantilever deflection information at each point, line by line, and assembling that data into an image. Figure 7 shows a screen capture of the scanner control window, and an overview of its operation is provided below.

Controls overview

Many of these are self-explanatory, such as the start imaging and stop buttons, as well as the image area in the lower right, which displays the image currently being scanned. Some notes are given below on features that are not immediately obvious. To begin with, it's easiest to simply use the default settings on all these controls, and to experiment with changing them as you become more familiar with the tool.

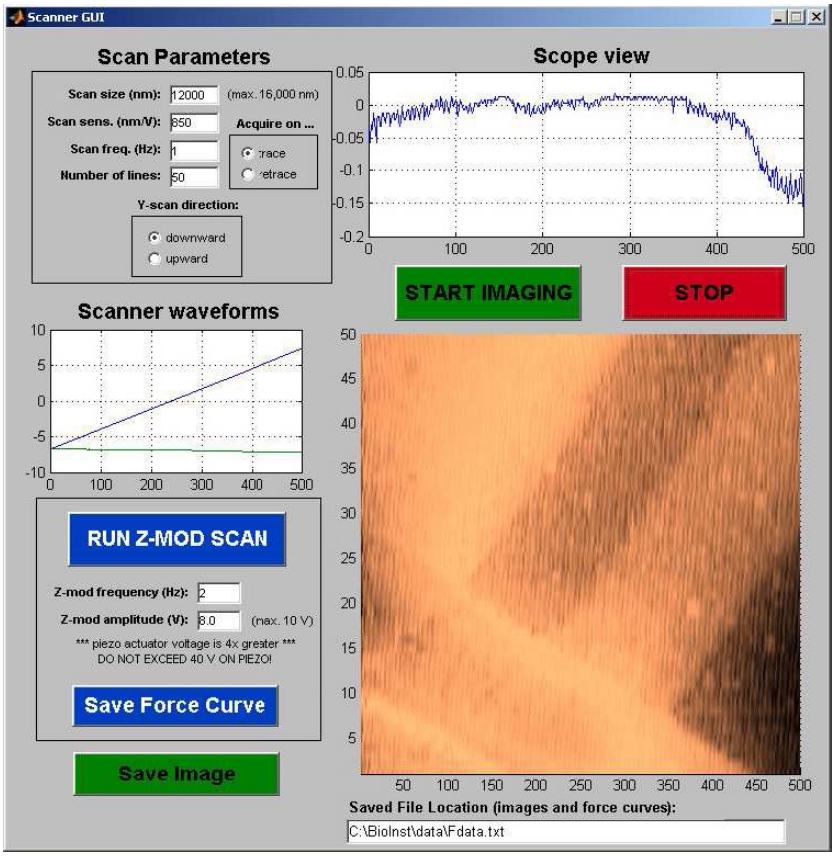


Figure 7: The Scanner GUI window. The AFM is scanning a 12 μm × 12μm area, at a rate of one line per second, and is currently near the bottom of the image.

Scan Parameters – The Scan size sets the length and width of the image in nanometers (always a square shape), but its accuracy depends on having the correct value for Scan sensitivity (which should already be set for you, but may require calibration). The Scan frequency (lines per second) sets the speed of the tip across the surface, and together with the Number of lines affects the amount of detail you will see in the image. Setting the Y-scan direction tells the scanner whether to start at the top or the bottom of an image, and the trace/retrace selector determines whether each line is recorded as the tip scans to the left or to the right.

Scope View - As the tip scans back and forth, this plots the tip deflection data for each line. It is useful for quantitative feature height measurements.
Scanner Waveforms - Shows the voltage waveforms driving the piezo scanner, for each scan line that is taken. Helpful for knowing where in the image the current scan line is located, and the output level of the waveforms driving the scanner.
Z-mod Controls - These are only active during a z-mod scan, and have no effect when taking an image. These will be used during set up of the AFM for imaging, and more information is provided below.

Image mode operation

This is the primary operating regime of the AFM, and provides a continuous display of the surface being scanned, as the probe is raster-scanned up and down the image area. To use this mode, **the switch** on the back of the AFM must be flipped **upward**. Remember that the maximum scan area is only about 9 μm square, and adjusting the position of the sample under the tip requires only the smallest movements of the stage micrometers. Finally, keep in mind that there is always a delay after pressing start imaging before the scan begins, as the actuator drive signals are buffered to the I/O hardware.

Z-mod operation

In this mode, the piezo moves the sample only along the z-axis – i.e. straight up and down (hence z-mod, short for z-modulation). To use this mode, the **switch** on the back of the AFM must be flipped **downward**. The default frequency and amplitude of 2 Hz and 8 V provide a nice force curve. Besides being critical for calibrating and biasing the readout, this mode is used to perform force spectroscopy experiments, in which tip-sample forces can be measured as the tip comes into and out of contact with the sample.
(Note that the red stop button is also used to stop a z-mod scan).

Saving AFM data

The software allows you to save the raw data of both images as well as force curves. Unsurprisingly, the "Save Image" and "Save Force Curve" buttons do this. In both cases, an instantaneous "snapshot" of the current image or force curve is written to the file location specified in the entry box at the bottom of the window.

An image is written to a file as a square matrix (interpolated to have the same number of rows and columns), with the value at each point representing height data. Force curve data is saved as two columns: x-axis (stage deflection) data in column one, y-axis (mode intensity) in column two.

If you intend to save an image, it is best to set the filename before starting the scan – the filename box behaves . . . elusively while the scanner is running due to some peculiarities of the software.

As a final note, a "quick and dirty" way to save an image is by simply doing a screen capture while the AFM is scanning (press "Print Screen" on the keyboard). The captured image can then be pasted into MS Paint (Programs → Accessories → Paint) and cropped to leave only the scanned AFM image. Alternatively, ALT+PrintScreen will capture just the active window so there is no need for cropping. You can paste directly into MS Word so it is easy to write notes and keep the different scans in order.

Part I: Experimental Instructions for Imaging

A. Setting up the AFM

First, you must turn on the AFM. This includes turning on three things: (1) the laser, (2) the photodetector, and (3) the piezo-driver power supply. The photodetector has a battery that provides reverse bias, and the others have dedicated power supplies (refer to Figure 1 for where these switches are located). **Do not "hot swap" any components while the AFM is turned on, or you will blow them out!**

When you finish using the AFM, do not forget to turn off the three switches you turned on at the beginning.

Next, you must align the laser and get a good diffraction signal going into the photodetector. To get a readout of the cantilever position, the laser needs to be well focused on the interdigitated fingers of the cantilever. Use the white light source and stereo-microscope to look at the cantilever in its holder. The laser spot should be visible as a red dot. (There may be other reflections or scattered laser light, but the spot itself is a small bright dot.) Adjust its position using the knobs on the kinematic laser mount (not the one at the center of the L pattern, but the ones on the outside), until the laser spot hits the interdigitated fingers (use the cantilever schematics in Figures 5 as a reference). **Record** approximately how far the laser spot is down the cantilever. Recording this value as an estimated fraction is fine: for example, "the spot is about x% of the way down the length of the cantilever".

When the laser is focused in approximately the right position, the white "screen" around the slit on the photodetector will allow you to see the diffraction pattern reflected of the grating. Observe the spot pattern on this screen while adjusting the laser position until you see several evenly spaced "modes." Make sure you aren't misled by reflections from other parts of the apparatus — some may look similar to the diffraction pattern, but they aren't what you're looking for.

When you see that the proper diffraction pattern is on the detector, adjust the detector's position such that only one mode passes through the slit. Typically the 0^{th} mode gives the largest difference between bright and dark. **Sketch or photograph the final diffraction pattern and detector position in your notes.**

B. Loading and positioning the sample

First, choose a sample to image. Before you load it into the AFM, you should look at it under the optical microscope to get an idea of what you might be seeing in the AFM. **Record** your observations, and make a quick prediction or sketch of what you expect to see in the AFM.

Correctly mounting a sample in the AFM is a key part of obtaining quality images. Our samples are always mounted on disks, which are magnetically held to the offset post that in turn connects magnetically to the piezo actuator. The AFM can image only a small area near the center of the opening in the metal cantilever holder, so be sure that the area of interest for imaging ends up there. The way to do this is by adjusting the position of the sample disk on the offset post, not by adjusting the position of the offset post on the piezo actuator. The offset post must always be positioned at the center of the piezo actuator so that the motion of the actuator approximates the x-y scan that you want it to be.

To look at a sample, you must first mount it on the sample disk. This is done using the "sticky dots" provided in the lab. **You will look at two different samples** from among the various samples available. Choose a sample disk, and transfer a sticky dot to the disk as best you can. (This isn't entirely easy, but fortunately, you don't have to do it perfectly.) Then, using tweezers, place a sample on top of the adhesive and press it in place. Then mount the sample disk onto the offset post.

Now you are ready to mount the sample into the AFM. When changing or inserting a sample disk, the 3-axis stage must be lowered far enough for the disk to clear the bottom opening of the cantilever mount, as shown in Figure 8. This requires a large travel distance, so exercise caution when bringing the sample back up to the cantilever, and take care not to crash the tip. In addition, as you change samples, it is critical to reposition the offset post as nearly centered as possible on the actuator disk, to ensure true horizontal motion in the x-y plane. (Centering the sample disk at the top of the offset post is not critical; rather, it's the position of the bottom end of the post on the piezo scanner disk. For instance, in Figure 8 on the right, the sample disk is visibly off-center, which is not a problem).

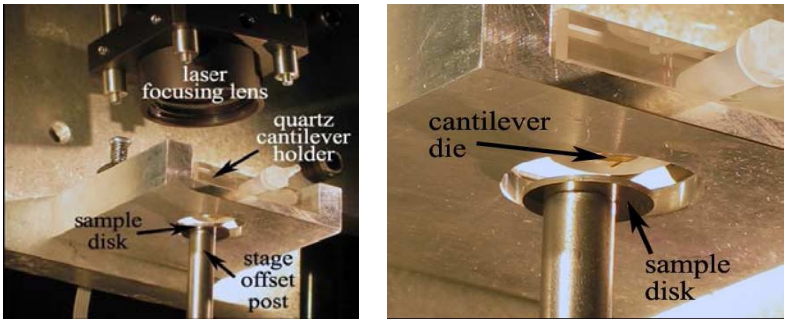


Figure 8. Left: photograph of the underside of the cantilever mount, with the sample disk lowered for changing samples. Right: A close up view of the opening through which the sample rises, showing the cantilever die and the sample disk.

C. Setting Up to Image the Sample

Now that the sample is loaded, it's time to bring the cantilever into position so that it can measure the topography of the sample. The process of bringing the probe tip to the sample surface so that we can scan images and measure forces is called "engaging". The aim is to get the tip in close proximity to the sample so it is just barely coming into contact, and bending only slightly. If the probe does not touch the surface, it is obviously useless, but if it's bent too much against the surface, it is equally useless.

Before engaging the tip, start the piezo z-modulation scan in the MATLAB software (see the software instructions above). This will start the piezo tube oscillating very slightly up and down. Be sure the mode switch on the AFM electronics board (behind the apparatus) is flipped down to "force spec. mode," and make sure to turn on the piezo power supply. Carefully bring the tip near the surface, turning the height adjustment knob by hand in appropriately small increments. When you make contact, you will see the modes on the photodetector fluctuate in brightness. This is because the cantilever is bending and unbending due to the rising and falling of the sample surface.

Because of the device geometry, only the central long cantilever with the tip will make contact with the sample surface.

Now that the tip is engaged with the sample, we could switch over to imaging mode and start to image; but the problem with that approach is that we wouldn't know what our data meant. The computer will tell us that the difference between point 1 and point 2 is a certain number of volts as measured at the data acquisition board, but we don't actually know what height on the sample surface that corresponds to. In order to find that out, we must calibrate our system. Each microscope must be calibrated individually, and you must do the calibration each time you use the scope. The calibration will vary from microscope to microscope and from specific set up to set up. We will calibrate by comparing the measured voltage with a known distance; when we are done, we will know how many nanometers of height are represented by one volt of output measured on the data acquisition card. In the process, we will also bias (set) the AFM setup so that it is at its point of maximum sensitivity just when the cantilever comes into contact with the sample.

With the AFM still set in z-modulation mode, look at the AFM signal on the oscilloscope in x-y mode and/or on the z-mod output display of the AFM controller program. You should see something like the plots shown in Figure 9, in which a flat line breaks into a $\sin^2$ function at a certain x-value. The flat line is the cantilever out of contact with the oscillating surface, and the oscillating section is the cantilever bending because it is in contact with the oscillating surface. If your $\sin^2$ trace isn't centered around zero (you can check it on the computer display), you can use the offset on the voltage amplifier to position the $\sin^2$ so that it is centered around zero. Then, set the out-of-contact bias point by moving the position of the laser focus on the fingers until the flat section of your force spec. curve is approximately at zero volts, halfway between the maximum and minimum, as in the rightmost part of Figure 9. You will want to move the laser side to side, towards one cantilever or the other, but not along the cantilever's length. If you do move the laser spot along the cantilever's length, you will need to re-record the estimated location of the laser spot along the cantilever's length. **Capture** a representative trace of the z-mod scan once when the system is properly biased for out-of contact sensitivity. If you have difficulty obtaining a flat line as shown in Figure 9, you can still obtain the offset – it is simply the voltage reading when the cantilever is disengaged after moving the stage down.

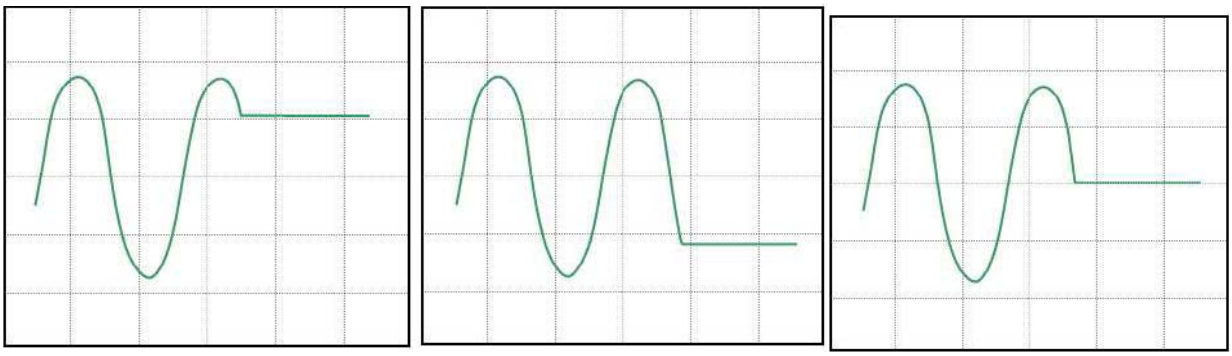


Figure 9: Proper setting of the bias point for out-of-contact sensitivity. Left: Bias too high. Center: Bias too low. Right: Bias set to optimal sensitivity for out-of-contact.

As we stated before, the goal of this calibration process is to relate the detector's voltage signal to physical tip deflection – i.e. how many nm is the cantilever tip bending for every volt of signal. These curves give us the data necessary to do so. You can take advantage of the fact that a mode's

brightness goes from fully bright to fully dim (from peak to trough on the $\sin^2$) as the fingers deflect through a distance of $\lambda/4$ ($\approx$ 160 nm). This should allow you to quantify the horizontal scale and calculate the relationship between deflection and y-axis voltage in nm/V. **Record** the measured values that you obtained in the process of the calibration, along with your calibration.

The result that you get will need to be corrected for the fact that the interdigitated finger detector is located partway along the length of the cantilever rather than at the tip. The tip is certainly deflecting more than the central part of the cantilever. To correct for this fact, you will need to multiply the calibration by a correction factor A_{corr} to account for the location of the diffraction fingers with respect to the tip of the cantilever. A_{corr} can be estimated most simply by assuming a quadratic shape for the bent cantilever. Then $A_{corr} = 1/m_{ID}^2$, where $m_{ID} = L_{ID}/L_T$ is the ratio of the distance of the ID fingers from the cantilever base to the total cantilever length. A more precise expression is $A_{corr} = 2/\left(3m_{ID}^2 - m_{ID}^3\right)$ (derived from the equation for the shape of a simple rectangular beam, with an applied end-load).

D. Imaging Samples

Now you get to image your sample! You'll have just a little more set up to do en route to actually getting good images. The general approach to imaging is to (1) set the overall output signal range and offset while in the z-mod regime (we already did this), (2) stop the z-mod scan and engage the probe with the surface, (3) carefully adjust the cantilever deflection to give the desired bias point, and (4) start the image scan.

You might ask why we need to engage the probe with the surface if we already engaged it with the surface before. The answer is that the probe was only engaged with the surface while the piezo was driving the sample up and down. If you biased the system correctly in the previous step, once the piezo stops driving up and down, your tip will no longer be in contact with the surface; it will be on the flat part of the z-mod curve, where the tip is not yet in contact with the sample.

You also might ask why we need to adjust the bias point, since we adjusted it before. We will keep the same placement of the laser on the ID fingers (so that's one part of the set up that remains the same), but we will need to bring the tip into light contact with the sample. The reason is that before we biased the sample so that we had maximum sensitivity just when the tip came out of contact with the sample. That bias point was great for calculating the sensitivity, but it's not great for imaging, because half of your sensitivity is wasted on the z-range that corresponds to the tip not contacting the sample. It is better to have the point of maximum sensitivity closer in z to where the features are.

You might then wonder if we should bring the cantilever and the surface into really solid contact, so that there's a lot of cantilever deflection. The answer to this question is no! A lot of cantilever deflection means a lot of force, and the more force the probe applies to the surface, the more wear and damage to the probe and surface can result. In addition, we calibrated the sensitivity of the cantilever for small deflections, and we will not get the same sensitivity if the cantilever is heavily deflected.

Regardless of which sample you are imaging, you may find that you have to navigate around the surface of the sample to find interesting features to image. Unfortunately, unlike commercial instruments, these AFMs do not have an integrated optical viewing system, so when positioning the sample it is difficult to determine exactly what spot on the sample the probe tip will scan. Use the

stereo-microscope (at moderate magnification) and a fiber-light to observe the tip and position it as well as you can. Temporarily turning off the sensing laser will make it easier to see. You can move the sample "on the fly", as you're imaging, but it takes some practice to make small enough stage movements. You will also likely have to readjust the bias point by moving the sample (also fine to do on the fly). **If you move the laser spot, you will have to re-do the calibration!** And please be careful about where you are going; try not to drive off the edge of the sample and then back onto it, because that will break the cantilever probe.

You can measure in-plane image dimensions approximately by knowing the size of the scan (from the Scan Size control). This gives the size of the image square, which can then be related to the imaged feature dimensions. Finally, feature height (z-axis) measurements can be made by observing the Scope View of the AFM software. The waveform display shows the feature height dimensions as voltages – the calculated calibration factor then gives corresponding feature sizes.

Now that you are up and running with the AFM, you should choose a feature of interest (or a few features of interest) on each sample. Take scans of these, and save them (with the program's save utility, or with a screen capture). Make sure to use the Scope View feature of the software to get a profile of your features, and be sure to save it (either because you saved the whole file, or through a screen capture). The profiles often give information that a simple color scale image cannot. Do the features look exactly like what you expected? What do you think accounts for any discrepancies between your expectations and what you observed?

Working from those data, determine the lateral and vertical dimensions of the imaged features.

Lab write-up questions:

1. (*2 points*) Show your work as to how you determined the calibration (i.e. calculated the sensitivity of your AFM in nm/V), and report your final result. Include all the data on which you based your calibration and show the captured trace of your z-mod scan.

2. (*1 points*) Present representative scans of your two samples, both the full scans and the line scans. Present the lateral and vertical feature sizes, along with a discussion of how you obtained these feature sizes (measure one lateral and one vertical feature for each sample). Discuss how the features that you observed align (or do not align) with your expectations, and suggest causes for any discrepancies.

3. (*1 point*) Briefly state what do you think is the reason why this AFM architecture is less sensitive to external vibration than conventional AFMs.

Part II. Characterization of AFM Cantilevers Using Thermal Noise

In this part, you will again use the entire apparatus, but your goal will be different: to characterize the cantilevers themselves, and to gain insights into the process by which they are fabricated.

In this part, you will characterize the properties (in particular, the stiffness) of the AFM cantilevers by measuring the noise. To do that, you will observe the behavior of the cantilevers when they are not in contact with the surface. When the cantilevers are not in contact with the surface, the cantilever motions that you observe are primarily caused by the cantilever's response to thermal noise. We must therefore consider both the properties of an AFM cantilever and the behavior of thermal noise in order to fully understand what will be done in this lab.

A. Cantilever properties

An important property of an AFM cantilever is its spring constant, which relates the force applied at the cantilever tip to the cantilever's deflection. You will recall that these cantilevers are almost rectangular beams, but each cantilever has a set of interdigitated fingers protruding from the side of the cantilever in about the second quarter of its length. Because the cantilevers are asymmetric and have a profile that varies rapidly along their length, the most accurate way to predict their spring constant would be numerically. However, a reasonable starting point for estimating the spring constant of a given cantilever is to adopt the analytical formula for the spring constant of a tip loaded cantilever beam with a rectangular cross-section that does not vary along its length. The resulting predicted spring constant is given by

$$k = \frac{EWh^3}{4L^3},$$

where E is the Young's modulus, W is the cantilever width, h is the cantilever thickness, and L is the cantilever length. Since the cantilever is almost entirely made of silicon nitride, it is an appropriate approximation to assume that the cantilever is entirely composed of silicon nitride. The Young's modulus of silicon nitride films can vary considerably as a function of the film deposition parameters; for our purposes, we will assume that the Young's modulus of the silicon nitride is about 250 GPa.

A second important parameter in describing the behavior of a cantilever is its resonant frequency, which is given by

$$\omega_0 = \sqrt{\frac{k}{m_{eff}}},$$

where m_{eff} is the effective mass of the cantilever. Hopefully, you will have taken dynamics and learned about resonance. If not, that's okay; everything that you need to know is included here. The cantilever's effective mass is less than its actual mass; this has to do with the fact that the mass at the tip moves a lot when the cantilever moves, but the mass near the root of the cantilever does not. The effective mass can be approximated as

$$m_{eff} = \frac{33}{140}\rho hWL,$$

where ρ is the density of silicon nitride. ρ usually also depends on the silicon nitride deposition conditions. We will assume that the density is 3.4 g/cm^3).

B. Thermomechanical noise

The final ingredient for this lab is thermomechanical noise. Without going into an extensive mathematical derivation of thermomechanical noise, we can appreciate its origins and its approximate functional dependence.

Statistical mechanics tells us that if a system is in thermal equilibrium, each mode of energy storage in this system has an average energy equal to $\frac{k_B T}{2}$, where k_B is Boltzmann's constant (k_B = 1.38 x 10^{-23} J/K) and T is the temperature (in Kelvin). Dynamics in turn tells us something about the behavior of AFM cantilevers, which are second order systems (damped simple harmonic oscillators). A mechanical second order system is typically described by the following differential equation:

$$F = m\ddot{x} + b\dot{x} + kx$$

where F is the applied force. This mathematical expression signifies that if zero force is applied to a simple harmonic oscillator system in motion, the damping represented by b will gradually slow the system's motion until it comes to a stop at its zero-deflection position. At this point, the system will have no kinetic energy and no potential energy, so its stored energy will be equal to zero. According to the statistical mechanics this can only be possible for a system at absolute zero, which is unattainable.

The solution to this conundrum is given by the Fluctuation-Dissipation theorem, which states that anything that can dissipate energy (i.e. any system in which the damping b is not equal to zero) must also be subject to fluctuations that inject energy into the system. This seems counterintuitive from our macroscopic point of view; we are not aware of fluctuations randomly transferring energy to the pens on our desks, for example. At the microscopic scale, however, these fluctuations can play a very large, dominant role in what happens to the system. This is the case for the cantilever when it is out of contact with the surface.

For a mechanical system, the fluctuations that the system is exposed to take the form of what we call a "noise force". The noise force is noise in the sense that it is sometimes positive, sometimes negative, has a varying amplitude and frequency, and averages out to zero. (Its mean square value, the average value of the noise squared, is not zero, however.) We will not prove this, but this noise force is a form of what we call "white noise", meaning that the noise fluctuations occur equally often at all frequencies. If you could feed the noise force into a spectrum analyzer (which analyzes a time-varying signal to determine what frequencies are represented in it), you would observe a flat line vs. frequency. This noise force is proportional to the temperature; this makes sense, because more energy must be put back into the system at higher temperatures to maintain the statistically required $\frac{k_B T}{2}$ than is necessary at lower temperatures. The noise force is also proportional to the damping, which also makes intuitive sense; more damping means more energy to replace in a given time. The equation for noise force is

$$\left\langle f_n^2 \right\rangle = 4k_B T b,$$

where $\left\langle f_n^2 \right\rangle$ is the spectral density of the noise force, measured in units of N^2/Hz. These are peculiar units if you are not used to spectral densities; if you want to know the total mean square

noise force acting on an object that is subject to a particular range of frequencies, you would simply integrate $\left\langle f_n^2 \right\rangle$ over the frequency range of interest.

When a noise force acts on a system, it can make it move in ways that are determined by the characteristics of the system. Large, stiff objects don't move much, because noise forces tend to be small; smaller, more flexible objects move more. A second order system like the AFM cantilever will have an interesting response to the noise force. The noise force excites the cantilever over a wide range of frequencies, and it causes the cantilever to vibrate back and forth. These vibrations also take place over a wide range of frequencies, but the amplitude of the vibration at a given frequency depends on the cantilever's properties. Below the cantilever's resonant frequency, the vibration amplitude is proportional to the amplitude of the force noise; the proportionality constant is simply k. At the cantilever's resonant frequency, the vibration is amplified; above the resonant frequency, the vibration is increasingly suppressed.

In this lab, the displacement of the cantilever when it is out of contact with the surface will be measured, and its frequency dependence will be determined. The amplitude vs. frequency data will be used to extract the cantilever's resonant frequency and spring constant. The remaining relevant physics are described below.

A resonance may be described by its Q value, which reflects the ratio of energy that is stored in the resonant motion to the energy dissipated in one cycle (in a time period of $1/\omega_0$). A high Q means that if you start the resonator going (for example if you give a swing a push), it will keep oscillating back and forth for a long time. A low Q means the opposite; an excitation dies out very quickly. The Q value is inversely proportional to the damping b, and the relationship is described by

$$Q = \frac{k}{b\omega_0}.$$

If you have measured the spectral density of the displacement, you can use the shape of the resonant peak to determine Q, and you can determine the frequency of the resonant peak. You can also read off (or fit to determine) the value of the mean square displacement in nm^2/Hz at frequencies below the resonant frequency. When you combine that information with the spectral density of the noise force, you can solve for the spring constant of the cantilever.

C. Laboratory objectives

In this part of the lab, you will:

1) **Calibrate** the output signals from the atomic force microscope to enable quantitative results.
2) **Measure** the spectral density of the cantilever displacement under the effects of thermomechanical noise.
3) **Determine** the spring constant of the cantilever, and draw conclusions about how the structure of the cantilever and its manufacturing process lead to the values of the spring constant that you obtain here.
4) **Repeat** the process for a second cantilever, as cantilever supplies permit**.**

D. Experimental instructions

The completion of this lab requires that you recall how to operate the AFM system from the AFM imaging lab. Please refer to that lab's handout as needed to remind yourself about the proper operation of the AFM.

Calibration of the system

The first step is to calibrate the sensitivity of the system as you did during the previous lab (bringing the cantilever to the surface and executing a z-mod scan to determine the sensitivity of the set up in V/nm). Remember to include the correction factor that accounts for the fact that the ID fingers are located partway along the cantilever length rather than at its tip. This will give you a calibration that is correct for the gain settings (presumably gain = 1) at which you do the calibration.

Measure the cantilever deflection

Next, bring the cantilever all the way out of contact with the surface so that you can measure its deflection under the influence of noise. (It should be noted that the cantilever may also deflect due to ambient vibration and other extraneous effects, along with the vibration induced by thermal noise. Our advantage against the other noise sources stems from the fact that a) the thermomechanical noise is white noise, rather than noise at a particular frequency as you might find from other ambient noise, and b) the cantilever's resonance behavior is expected to be distinctive. You can expect to see some 1/f noise, though. Your lab instructors can help you sort out what is what.)

In order to see the noise deflections, you will need to greatly increase the gain of your system. First, press the ÷100 button on the amplifier so that it no longer divides the signal by 100. Then, increase your gain with the gain knob (don't turn the calibration inset) until you have reached the highest gain value that you can without sending the amplifier into overload. **Record** the new gain value; this, combined with your old calibration values, will determine the new calibration of the system under the new gain.

The cantilever's deflection will be measured with a LabView program that emulates a spectrum analyzer. The main controls are the sampling rate (200 kHz is a good choice) and the number of averages (which is off the right hand side of the screen … scroll over). You can also adjust the frequency range that's shown on the display by typing in new values for the minimum and maximum frequencies on the display's x-axis. About 50 Hz to 100 kHz is a good choice. Whenever you change a parameter or the gain of the system, you must press "Restart Avg." on the LabView interface so that you're not averaging data from your old settings with your new settings.

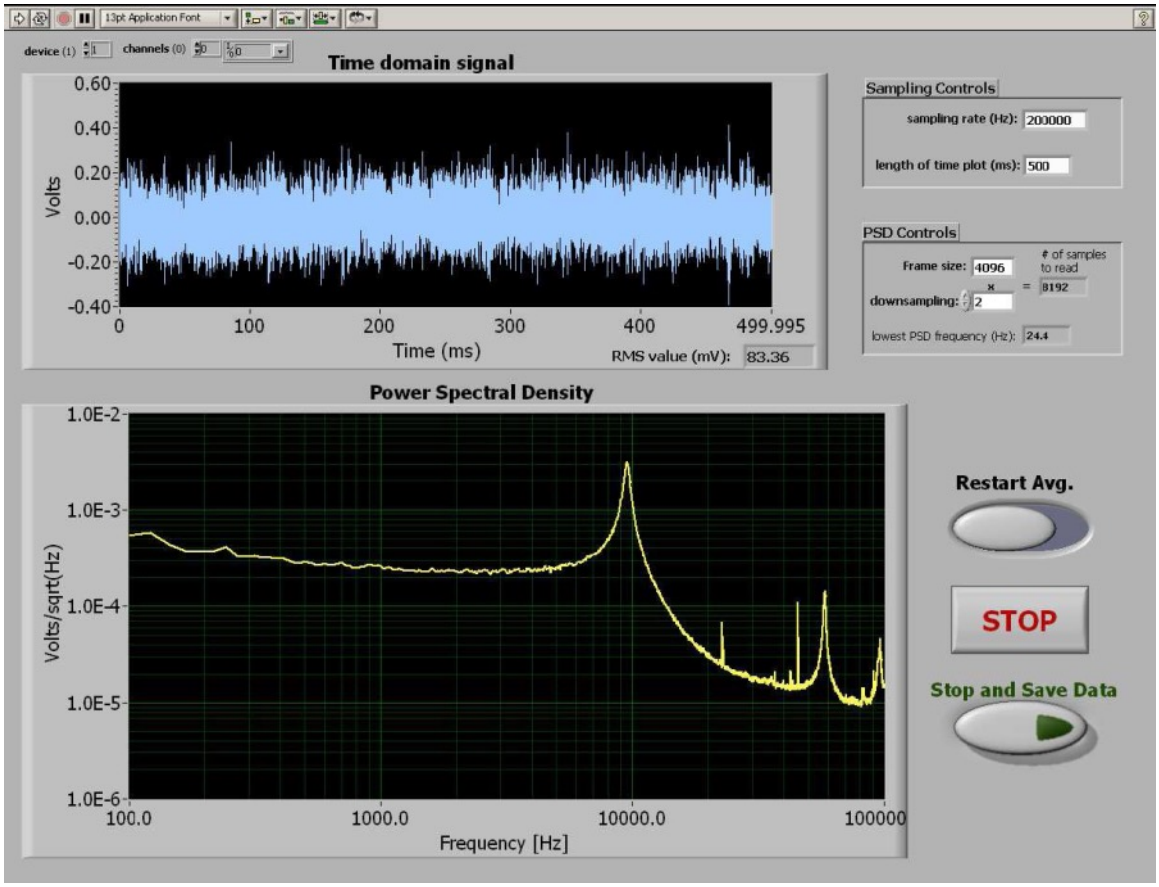


Figure 12: The LabVIEW Spectrum Analyzer VI runs continuously until the stop button is pressed. Each Power spectral density (PSD) dataset is averaged with all the data that precedes it, so the spectrum's signal to noise ratio improves the longer you acquire. Note that the "PSD controls" (Nfft, downsampling) can be changed on the fly, but to change the sampling rate you must stop and re-start the VI. Pressing "Stop and Save Data" pops up a window in which you can choose a file name to store the spectrum currently displayed.

Get a good spectrum of the cantilever's deflection, averaging until you are happy with the level of the noise. Then **save the data file** of the spectrum; you will need to be able to access the data in MATLAB or Excel in order to get the quantitative results that you need. You can also capture a screen shot in order to have a convenient image of the spectrum to paste into your lab report.

Parameter extraction

First, read off and **record** the cantilever's resonant frequency; this is easiest from the data file.

Next, you will determine the spectral density of the cantilever deflection. This you can read off from the appropriate part of the data file; make sure that you pay attention to your units, so you know whether this represents a deflection or a squared deflection. **Record** the value that you obtain.

Now you will determine the Q value for your cantilever's resonance. You can estimate Q by looking at the shape of the resonant peak; this will be easiest in Excel or MATLAB. Although Q has the physical meaning described above, it can also be calculated by

$$Q = \frac{f_0}{\Delta f},$$

where f_0 is the resonant frequency of the cantilever and Δf is the width of the peak at the point at which it contains half as much **energy** as at the peak; think carefully. This should give you enough information, combined with the introduction above, to calculate an experimental value for the spring constant.

Lab write-up questions: (1 point each except Q4 which has 2 points, extra point for Q5, if well elaborated.)

1. Report the sensitivity of your AFM in nm/V. It is not necessary to show your calibration work since you already did this calculation for Part I. What is the new gain at which you set the system for thermal noise-based characterization? Calculate the new calibration corresponding to this new gain.

2. Show the spectrum that you obtained for the cantilever under thermal equilibrium. Label the primary features, in particular the roughly white noise region and the resonant peak.

3. What are the measured resonant frequency, the spectral density of the deflection in the white noise region, and the quality factor Q?

4. Estimate the spring constant from your measurements of the cantilever under thermal equilibrium. Show your work and the equations used.

5. Calculate the expected resonant frequency and the expected spring constant from the used cantilever's nominal geometry. Do the theoretically calculated values of the resonant frequency and spring constant agree with experimentally measured values? If not, suggest possible causes of the difference. *You may base your answer on the following questions (these are not to be answered one by one):* Do you think the error stems from uncertainties in your measurement, or do you think that the cantilever geometry and thickness does not match its nominal dimensions? Does the same error mechanism explain both the discrepancy in stiffness and in the resonant frequency (i.e. is the discrepancy consistent with error in a single parameter that affects both values?).

References

[1] G. Binnig, C. F. Quate, Ch. Gerber, "Atomic Force Microscope", Phys. Rev. Lett. 56 (3):930-933, 1986.

[2] M. Shusteff, T. P. Burg, and S. R. Manalis, “Measuring Boltzmann’s constant with a low-cost atomic force microscope: an undergraduate experiment”, Am. J. Phys. 74 (10), 2006.

[3] S. R. Manalis, S. C. Minne, A. Atalar, C. F. Quate, “Interdigitatal cantilevers for atomic force microscopy,” Appl. Phys. Lett. 69 (25):3944-3946, 1996.

[4] G. G. Yaralioglu, S. R. Manalis, A. Atalar, C. F. Quate, “Analysis and design of an interdigital cantilever as a displacement sensor,” J. Appl. Phys., 83 (12):7405-7415, 1998.

2.674 / 2.675 Micro/Nano Engineering Laboratory

Spring 2016