

AFM - Atomic Force Microscope
Physics 111B: Advanced Experimentation Laboratory
University of California, Berkeley

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1 Summary

Atomic Force Microscopy (AFM) is a relatively new method of imaging objects from sub-nanometer to micron scale. It can scan surfaces in 3D and provides a quick and easy way to measure dimensions, roughness, topography, and many other material characteristics. Unlike Scanning Tunneling Microscopy (STM), AFM can measure both conductive and insulating materials in ambient condition with little to no sample preparation, and is thus widely used in scientific and engineering fields across the board.

In this experiment, you will learn the working principles of AFM and get trained to use a commercial AFM by [AFMworkshop](#). You will perform different measurements to get a taste of its wide-ranging applications. You will gain experience in making extremely precise measurements and in improving data quality by optimizing environmental and instrument parameters. You will also use the AFM to estimate Boltzmann's constant.

1. Pre-requisites: Physics 110A, 112
2. Days allotted for the experiment: 8

This lab will be graded 40% on theory, 40% on technique, and 20% on analysis. For more information, see the [Advanced Lab Syllabus](#)

Revised: 12/2021. Comments: winthrop@berkeley.edu

2 The Atomic Force Microscope Experiment Photos



Figure 1: AFM Ebox
[Click here to see larger picture](#)



Figure 2: Illuminator
[Click here to see larger picture](#)

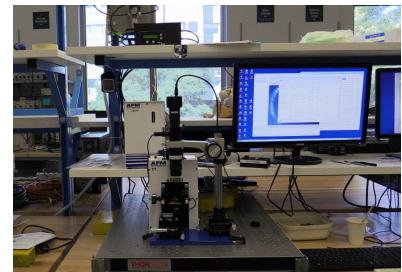


Figure 3: AFM on level stage
[Click here to see larger picture](#)

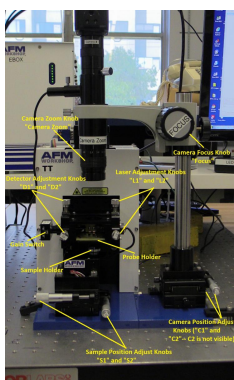


Figure 4: AFM apparatus
[Click here to see larger picture](#)

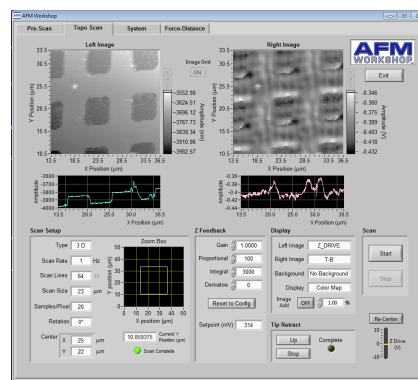


Figure 5: Topographic scan window
[Click here to see larger picture](#)

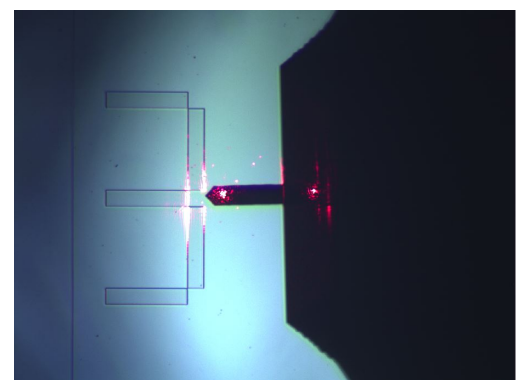


Figure 6: Laser focused on cantilever
[Click here to see larger picture](#)

3 Before the 1st Day of Lab

Complete the AFM Pre Lab found in the [Signature Sheet](#) for this lab. Print the signature sheet, discuss the experiment and pre-lab questions and answers with any faculty member or GSI, and receive their signature. In the course of the lab there will be examination points where you must STOP and get a GSI or professor to verify your understanding and/or verify proper experimental setup. You cannot skip these checkpoints, and must receive signatures demonstrating that you've consulted the staff. Some experiments may have mid lab questions that must be completed by specific days of the experiment. The completed [Signature Sheet](#) MUST be submitted as the first page of your lab report. Quick links to the checkpoint questions are found here: [1](#) [2](#) [3](#) [4](#) [5](#)

1. Watch the [AFM Basics](#) introductory video
2. Review [these slides](#) for a brief overview of AFM history, background, and basic operation.
3. Read **Chapter 1** in [Atomic Force Microscopy](#). Skim through **Chapter 2** and **3**.
4. Read through this entire experiment lab manual. **Watch all the videos herein** – they are extremely helpful and will make your experiment much smoother.
5. On the first day of lab, ask a staff member for your allotted probe tips.
6. Last day of the experiment please fill out the [Experiment Evaluation](#)

Suggested Reading

- AFM Workshop [TT-AFM Users Guide](#). Comprehensive reference manual of the AFM.
- See [this paper](#) for an in-depth discussion on Force-Distance measurements.
- Other references located in the C:/AFM Experiment/Articles PDFs

4 Objectives

- Understand the working principles of AFM
- Learn how to align, calibrate, and operate an AFM, and how to visualize, process and analyze AFM data with Gwyddion and other software tools
- Gain experience in and understand the limitations of improving data quality in extremely delicate and sensitive measurements by optimizing environmental and instrument parameters
- Conduct a wide variety of specific AFM measurements.

5 Introduction

The basic mechanism of the AFM is shown in Fig. 7. A sharp tip attached to a cantilever interacts with the surface of a sample. Light from a laser is bounced off from the cantilever into a [position-sensitive photodetector](#) that measures the deflection of the cantilever. The sample is placed on a [piezoelectric scanning stage](#) that moves the relative tip-sample position in 3D with nanometer precision. A feedback loop maintains constant tip-sample interaction during scanning and thus traces out the topography of the sample.

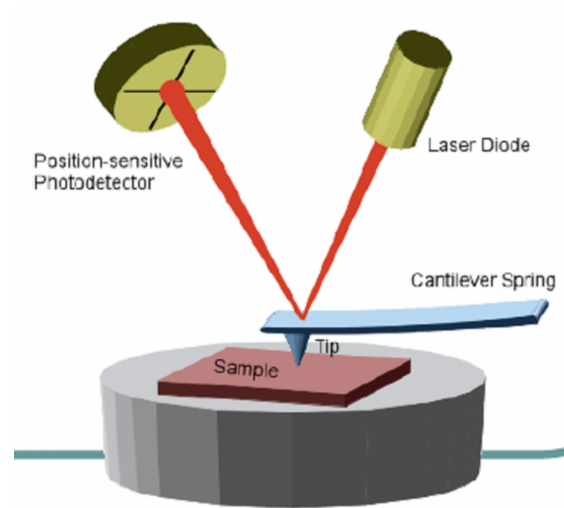


Figure 7: Basic components of an AFM. The deflection of the cantilever as the tip moves across the surface of the sample is measured by the position-sensitive photodetector. This deflection is used by the feedback loop to adjust the relative position between the cantilever and the sample with a piezoelectric scanning stage.

The following features make AFM a versatile tool and allows it to become the basis of many other [Scanning Probe Microscopy \(SPM\)](#) techniques.

- Works in diverse environments (air, liquid, vacuum).
- Physical interaction with the surface allows for direct measurements of mechanical, chemical, electrical and magnetic properties of a surface with appropriate probes.
- Works for both conductive and insulating samples (unlike STM).
- Wide length scale range from sub-nanometer to micron.
- Very high resolution and accuracy in the vertical direction.

5.1 Apparatus

Read page 5-7 of the [TT-AFM Users Guide](#) to understand the major components of our AFM (Fig. 8).

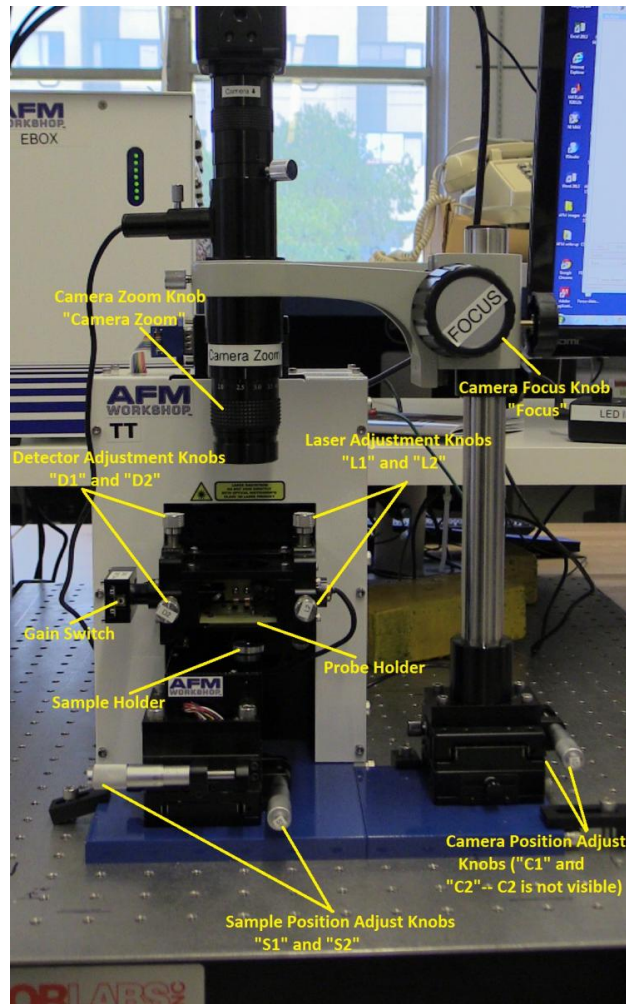


Figure 8: Overview of major AFM components

5.1.1 AFM Probes

An AFM probe, often made of silicon, contains a sharp tip attached to the end of a cantilever, which is in turn attached to a carrier chip (Fig. 9, 10). We mainly use [ACLA probes from AppNano](#) that are optimized for **Vibrating** mode. See animations of: [the probe interacting with the sample surface](#), [the effect of tip shape](#), [the probe interacting with contamination](#) present on samples, and [the probe interacting with small features on the sample](#).

AFM probes are extremely fragile, so you must handle them with extreme care throughout this experiment. **Each probe costs about \$30**, so keep this in mind when you do this experiment.

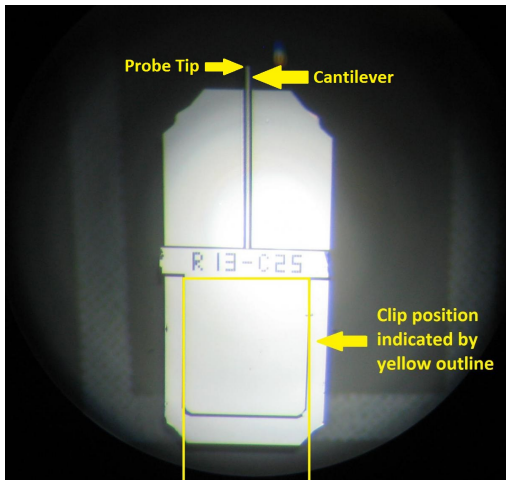


Figure 9: AFM probe bottom view (i.e. the tip is facing towards the viewer). The cantilever is hardly visible to the naked eye and the base of the tip is barely visible under a good optical microscope.

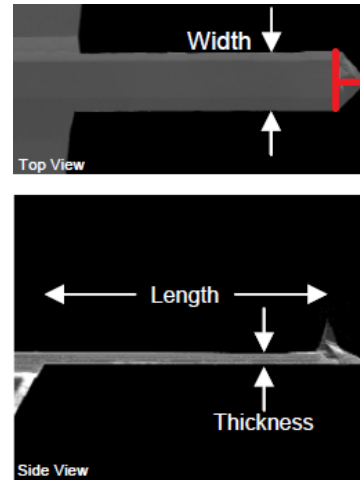


Figure 10: AFM probe top and side view taken with an electron microscope. Imagine a cross-hair at the triangle point of the cantilever. That is roughly where the tip is located.

5.1.2 Piezoelectricity and Scanning Stages

The **piezoelectric effect** is the ability of certain materials to generate an electric charge in response to applied mechanical stress. It also works in reverse, meaning that piezoelectric materials exhibit stress, and in turn strain, when an electric field is applied. Therefore, applying a voltage across a piezoelectric material **stretches or compresses** the piezoelectric material, by a precisely controllable amount.

This is the basis of many precision scanning stages, including those used in STM and AFM (animation for the **Y-** and **Z-axis** motion). The piezo stages in our AFM have a lateral range of $50\ \mu\text{m}$ and a vertical range of $17\ \mu\text{m}$ at maximum high-voltage gain.

5.1.3 Light Lever and Feedback Loop

A light lever is created by reflecting the light off of the cantilever and onto a **position-sensitive photodetector**. The signal from the top half (T) minus the signal from the bottom half (B), i.e. T-B, gives the relative vertical deflection of the cantilever (**animation**).

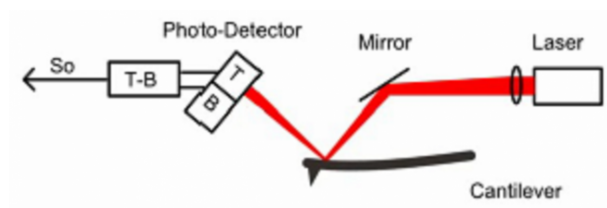


Figure 11: Laser-cantilever-detector setup

In our AFM, the entire light lever module can be moved up and down by a **coarse stepper motor** (Z motor) in order to engage the probe with the sample.

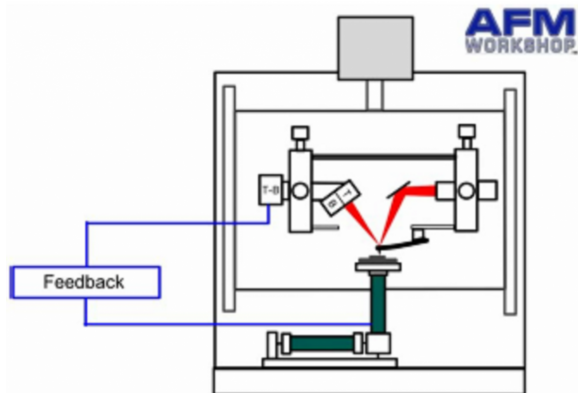


Figure 12: Simplified feedback loop schematic

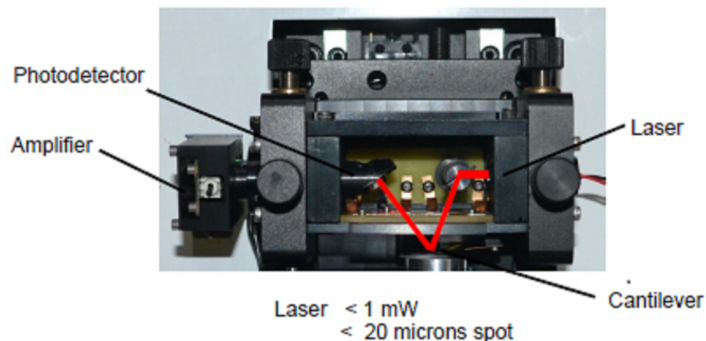


Figure 13: Laser beam path

The piezoelectric sample stages perform raster scans. When the probe encounters a surface feature (e.g. a bump), the cantilever bends and thus changes the T-B signal. Depending on the operating mode (vibrating or non-vibrating), the feedback loop tries to keep either the AC or DC component of T-B at a constant "set point" by moving the Z piezo.

The feedback signal (drive voltage to the Z piezo, or **Z_DRIVE**) thus gives the local height of the surface. Other simultaneously measured signals, such as the phase of the cantilever oscillation (**Z_PHASE**) or the twist of the cantilever (**L-R**) contain information about the chemical or mechanical properties of the surface. See page 16 of the [TT-AFM Users Guide](#) for the definition of all available signals.

5.1.4 Electronics

The Ebox contains most control electronics and is located on the shelf behind the AFM. The Ebox houses the micro-controller unit (MCU) and the National Instrument (NI) data acquisition device (DAQ), and interfaces the AFM hardware with the software on the computer. Additional electronics for e.g. voltage amplification are located in the main AFM body.

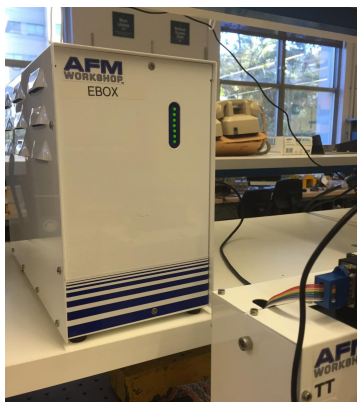


Figure 14: AFM Ebox

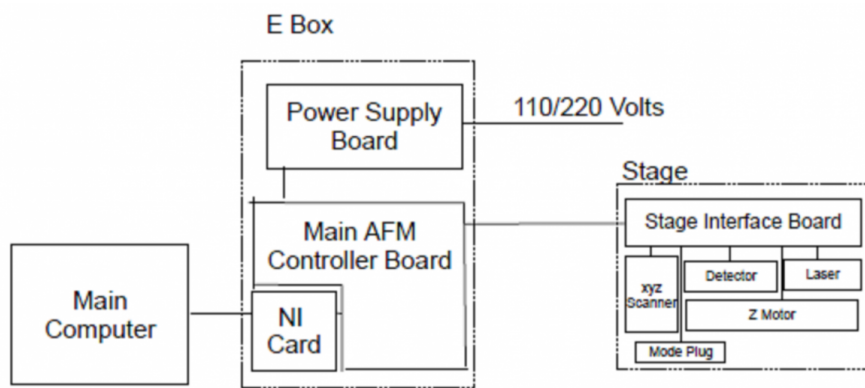


Figure 15: AFM Electronics block diagram

5.2 Control Software

The LabVIEW-based **AFM Workshop** software controls the AFM operation and exports collected data. It controls the motion of the sample stage, automates the tip approach, measures the resonance frequency

of the cantilever for **Vibrating** mode (see below), collects signals from the photodetector, and controls the feedback loop.

Read page 8-20 of the **TT-AFM Users Guide** to get familiarized with the **Pre-Scan**, **Topo Scan** and **System** Tabs.

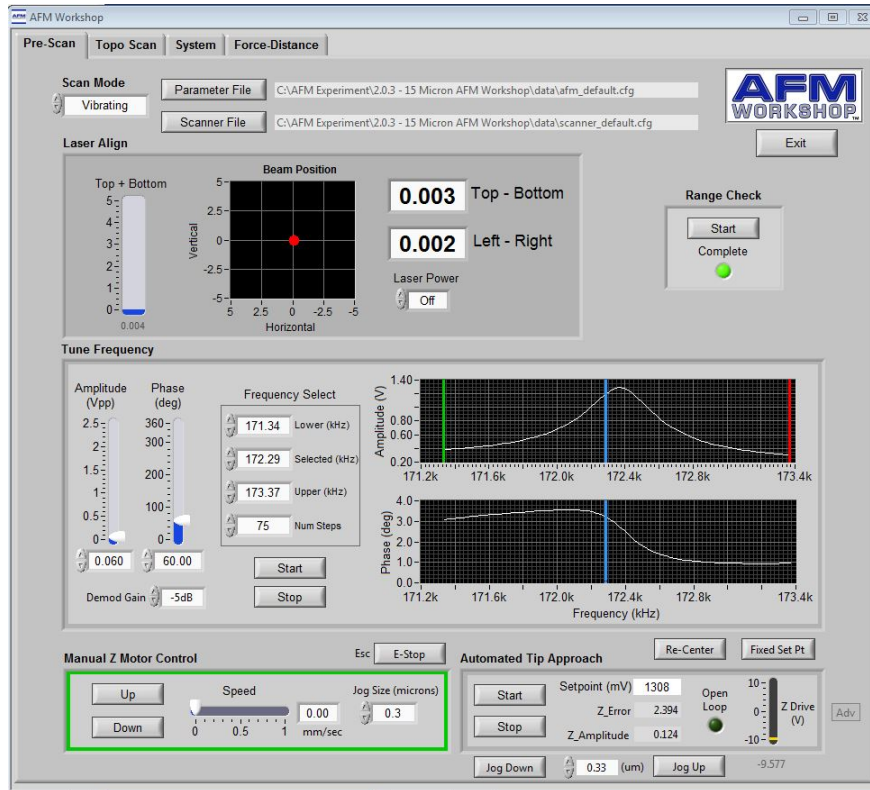


Figure 16: Pre-Scan Tab for Vibrating mode with a well-tuned resonance. This tab is used for laser alignment, range check, cantilever frequency tuning, manual Z motor control, automated tip approach, and switching between Vibrating and Non-Vibrating mode.

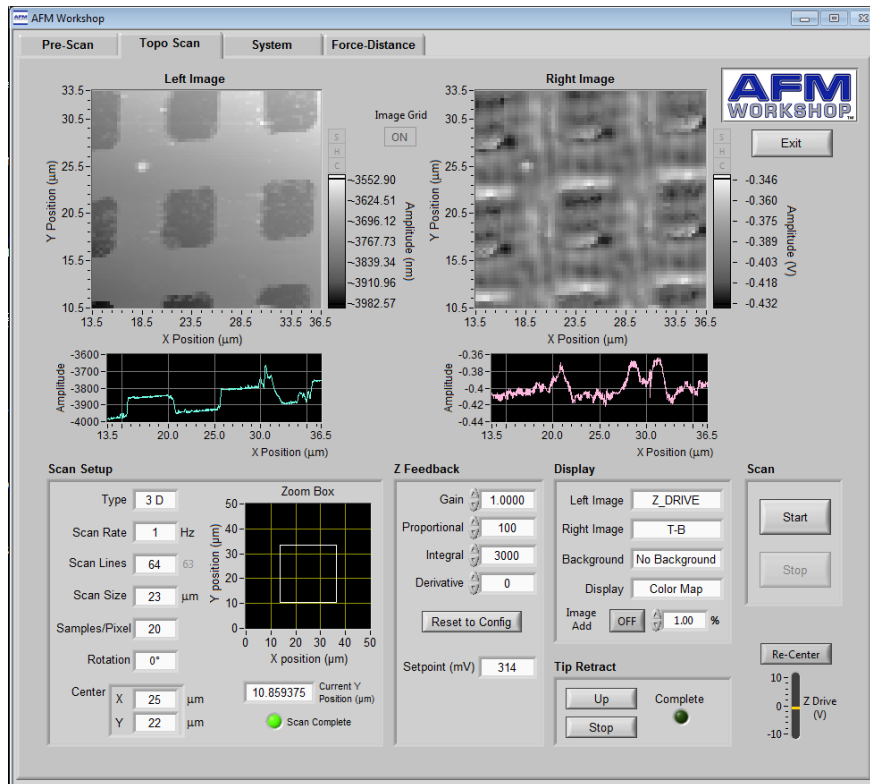


Figure 17: Topo Scan Tab with a completed scan of the calibration sample. This tab is used after a successful tip approach to set scan parameters including Z feedback and display signals, and to monitor the results during scanning. It is also used to perform a Tip Retract.

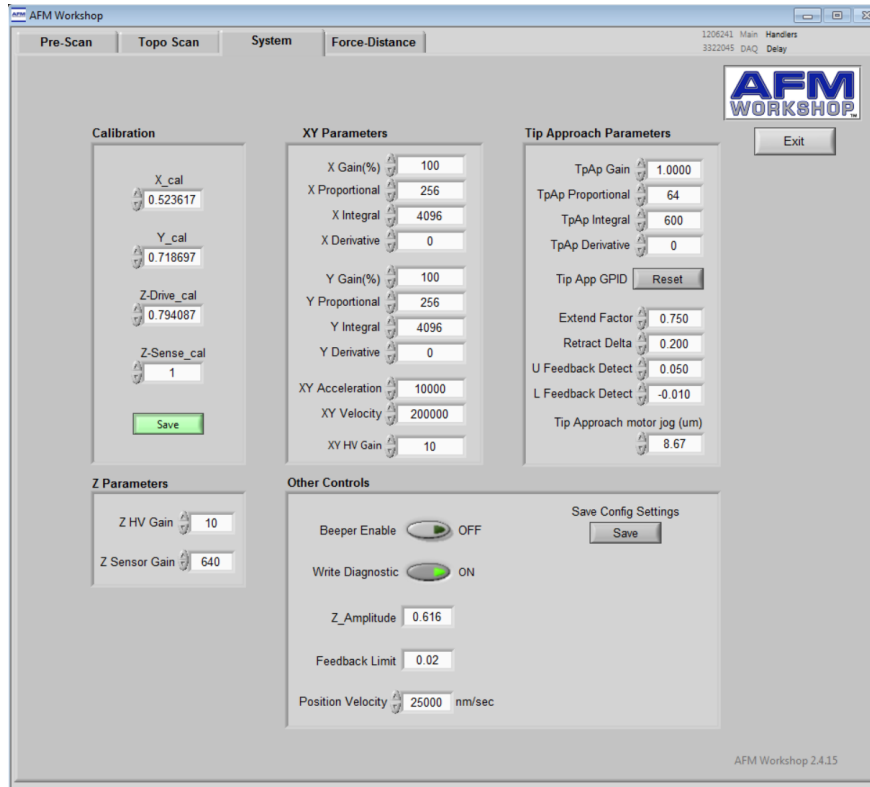


Figure 18: System Tab. This tab is used to change other system parameters including the XYZ calibration factors and the high-voltage gains (XY/Z HV Gain, default 10).

Note: If you are experiencing bugs in the software, refer to the [Troubleshooting](#).

6 Experimental Procedures

This section describes the standard operating procedures for common AFM operations. As you follow along with this lab write-up, you will find the referenced instructional videos to be **VERY helpful**.

6.1 Starting Up

Follow steps in the instructional video [Getting Started and Probe Exchange](#) (start to 2:00). Briefly:

1. Power on the Ebox via the switch in the lower rear. Series of green LED lights will turn on.

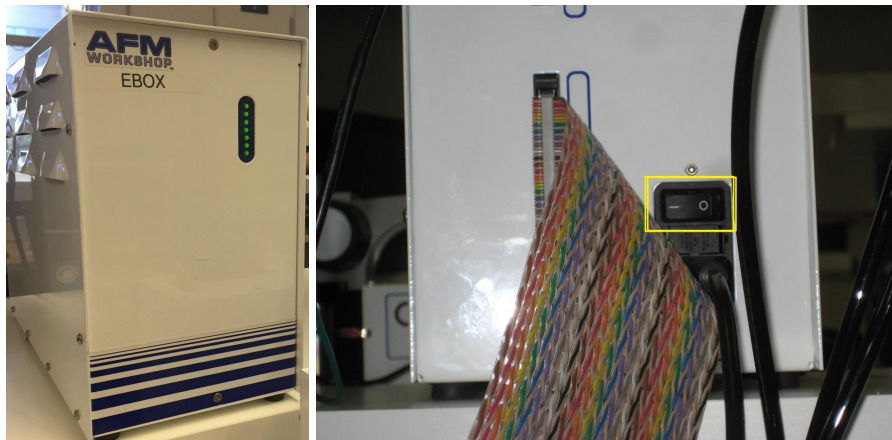


Figure 19: Ebox power switch

2. Turn on the LED illuminator. Adjust the LED power as needed.



Figure 20: LED illuminator

3. From the Desktop, start **AFM Workshop 2.4.13** and **USB2.0-Camera** (click the play button on the top left of the program window to start the camera; adjust the exposure time as needed).

After turning everything on, check that the red laser is on. Go to the **Manual Z Motor Control** in the **Pre-Scan** tab and click and hold **UP** to ensure that the Z motor moves the probe upward. When you are moving the Z motor UP you should be able to hear the motor and physically see the probe moving up and away from the sample. If you are experiencing bugs, please refer to [Troubleshooting](#).

6.2 Exchanging Probes

Follow the steps in [Getting Started and Probe Exchange](#) (starting 2:00) **CAREFULLY** and those on page 23 of the [TT-AFM Users Guide](#) to install or exchange a probe. You can practice with a damaged probe first for a few times before using a new probe.

PROCEED WITH CAUTION – THE PROBES ARE VERY DELICATE

- NEVER PICK UP THE PROBE WITH YOUR HANDS
- NEVER PICK UP THE PROBE ALONG THE LONG LENGTH
- NEVER TOUCH THE CANTILEVER SIDE OF THE CHIP WITH ANYTHING

6.3 Alignments

In this step, you will perform three sequential alignments: (1) Align the center of the sample holder, the end of the cantilever, and the center of the camera's field-of-view, (2) Align the laser spot to the back of the cantilever (Fig. 21), and (3) Align the reflected laser spot to the center of the positive-sensitive photodetector (Fig. 22). Refer to the video tutorial [Alignment: Camera, Laser, and Detector](#) and page 24 of the [TT-AFM Users Guide](#).

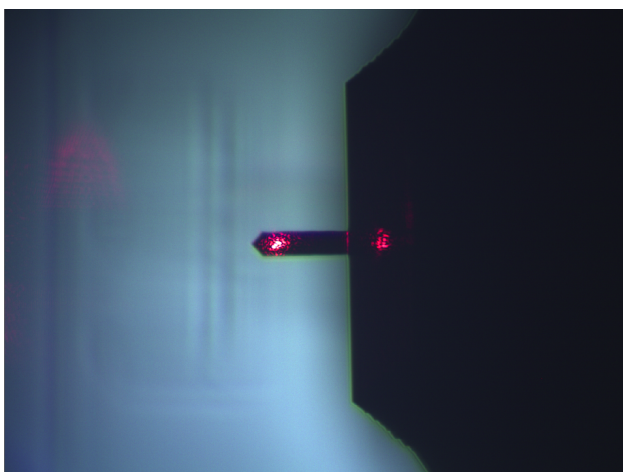


Figure 21: Laser aligned to the back of the cantilever

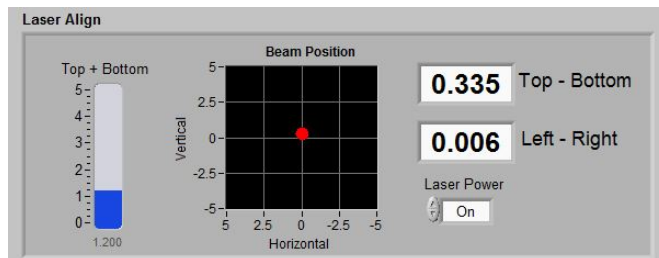


Figure 22: Laser aligned to the near-center of the detector. For technical reasons you shall aim for a small positive T-B value (0.2-0.4) instead of zero.

Checkpoint Alignments:[†] Show a GSI that you have completed all three alignment steps. Identify any debris or dirt on the sample holder or the probe.

6.4 Preparing and Loading a Sample

6.4.1 How to Prepare a Sample

If the samples that you are going to use are already prepared, you can [skip to the next step](#).

1. The small sample to be imaged must be less than 8mm thick, and must be able to fit on the adhesive tab on the 15 mm diameter metal sample disk.
2. It is critical for the sample to be kept as clean as possible. Avoid touching it with your fingers.
3. Use the provided double-sided adhesive tabs and tweezers. Peel off one side of the adhesive protection backing and carefully attach it to the sample disk. Next, peel off the other side, and use tweezers to place your small sample to be imaged firmly on the sticky tab. Gently press the corners of the sample until secured.
4. Make sure that the area of interest is near the center of the disk, the sample is as flat as possible and that it does not protrude off the edge of the disk.

6.4.2 How to Load a Sample

Watch [Pre-Scan: Tune Frequency, Tip Approach, and Scanning](#) carefully (5:00 to 7:00) and page 22 of the [TT-AFM Users Guide](#).

Load the calibration sample (Sample 7, [AppNano Silicon Step Height Reference, Part# SHS-0.1-1](#)) and find **Feature B** if it is the first time and/or if you are about to perform a calibration.

6.5 Setting up Pre-Scan Parameters

Watch [Pre-Scan: Tune Frequency, Tip Approach, and Scanning](#) (0:00 to 5:00) and page 9-12 of the [TT-AFM Users Guide](#).

1. On the **Pre-Scan** tap, set **Scan Mode** to **Vibrating** for tapping mode (default) or **Non-Vibrating** for contact mode. The **Tune Frequency** window becomes enabled only in **Vibrating** mode.
2. Focus the camera on the sample surface. You can adjust the magnification/zoom of the camera by turning the knob labeled “**Camera Zoom**”. The focus of the camera is adjusted by the large black knob to the right of the black tube labeled “**Focus**”.
 - Clockwise rotation of the **Focus** knob raises the focus.
 - Counter-clockwise rotation of the **Focus** knob lowers the focus.

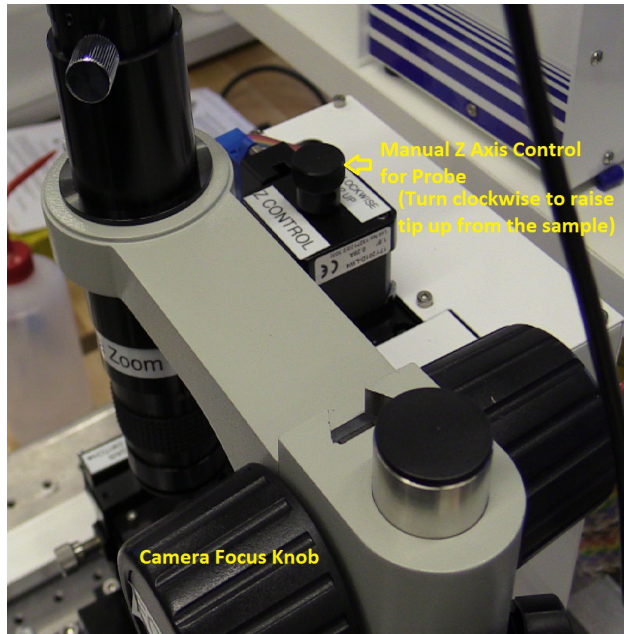


Figure 23: Z-motor and camera focus knobs

3. **MAKE SURE THE TIP IS NOT TOO CLOSE TO THE SAMPLE.** If the sample is in focus, the cantilever should be out-of-focus – refer to Fig. 24 below. Make minimal adjustments to the X-Y micrometers labeled S1 and S2 to move the area of interest of the sample under the tip.

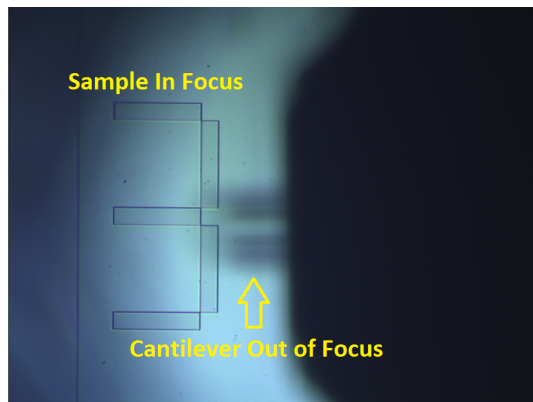


Figure 24: The sample is in focus while the cantilever is not, when there is a safe distance between the two.

6.5.1 Range Check

Make sure the tip is a safe distance away from the sample and the **Range Check** indicator light is **off**. Click the **Range Check** button and wait for it to finish. The **Complete** light will turn on.

During range check, the system ramps up and down the voltages to the XYZ piezos, and captures the strain gauge output so that it can scan linearly. You have to do this, or you'll get bizarre scans and most likely **break the tip**. If you do not see the sample move relative to the tip, check [Troubleshooting](#).

6.5.2 Tuning the Cantilever Resonance

This part is only relevant for **Vibrating** mode.

1. In the **Tune Frequency** window, under **Frequency Select**, set Lower and Upper Frequencies to the range specified on the probe box. Leave the **Selected** frequency anywhere in between this range
2. Set the number of steps to 100 and the Demodulation Gain to 4dB
3. Click the **Start** button to begin the frequency sweep. Once a coarse sweep is finished and the fundamental resonance can be identified in the **Amplitude** window, drag the green and red markers therein to be closer to the resonant frequency for finer sweeps. Adjust the excitation **Amplitude (Vpp)** and if necessary the Demod Gain to get a smooth curve with a peak amplitude of 0.5 - 2 V.
4. Adjust **Phase (deg)** such that the phase profile is relatively flat away from the resonance, and is anti-symmetric across the resonance.
5. The software will generally find a suitable operating frequency automatically on the low-frequency shoulder of the resonance. If not, drag the blue marker slightly and redo a frequency sweep. See Fig. 16 for an example of a well-tuned resonance.

Checkpoint Pre-Scan Settings:[†] Show a GSI the probe resonance and the settings you have selected prior to performing a tip approach. Discuss what these settings do, and why you chose them.

6.6 Tip Approach

For **Vibrating** (Tapping) mode, read page 25, 30 and 31 of the **TT-AFM Users Guide** and follow the steps in **Pre-Scan: Tune Frequency, Tip Approach, and Scanning** (7:00 to 12:30) closely.

For **Non-Vibrating** (Contact) mode, follow the steps in page 25-26 of the **TT-AFM Users Guide**. This mode is only used for Force-Distance curves and Boltzmann's constant experiments.

A brief summary of the steps:

1. Move the probe module down by turning the Z-motor knob (Fig. 23) while **physically** monitoring the tip-sample distance, until the tip is few mm away from the sample surface.
2. Focus the camera on the sample surface. Use the **Down** button in the **Manual Z Motor Control** section of the **Pre-Scan** tab to **slowly** lower the tip to the sample until the cantilever is just slightly out of focus (Fig. 25).

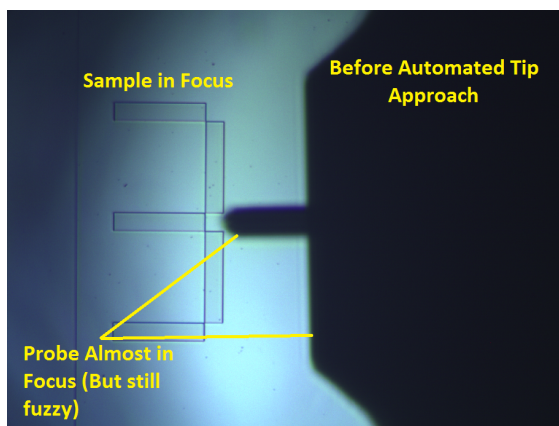


Figure 25: Approximately how focused the cantilever should be before starting an automated tip approach (laser turned off for clarity)

3. Start the **Automated Tip Approach**. If you start when the cantilever is too far away from the sample, this step can take hours.
4. Once the tip engages the sample, the **In-Feedback** indicator will turn on. Both the sample and the cantilever will be in focus (Fig. 26). If the **Z Drive (V)** is very close to the ± 10 V limit, do a **Re-Center** (be careful with the feedback parameters, see details in the video tutorial, including on **False Feedback**).

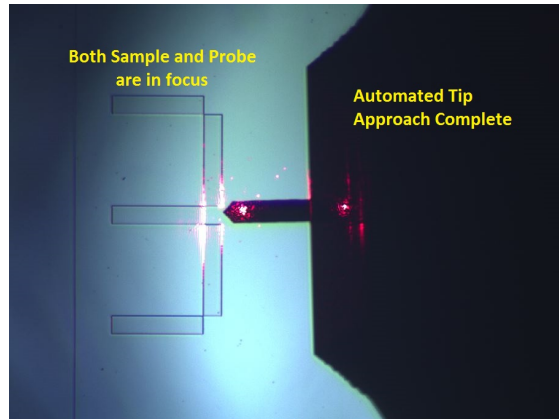


Figure 26: Tip Approach completed

6.7 Topography Scans

Once the tip is in stable feedback, we can start topography scans. The first scan shall be performed on the calibration sample as noted [above](#). Follow **Pre-Scan: Tune Frequency, Tip Approach, and Scanning** (12:30 to end) closely and page 28-29 (**Non-Vibrating** mode) and 32-33 (**Vibrating** mode) of the **TT-AFM Users Guide**. A brief summary below:

1. Set up the scan parameters at the bottom of the Topo Scan tab according to the feature of interest. See Fig. 17 for an example. It is often helpful to start with a relatively slow (0.3-0.5 Hz), large-area (25-40 μm) and coarse (64 lines) scan, before doing finer scans of smaller areas (Table. 1).

In general you can set the Left image to be **Z_DRIVE** which represents the topography of the surface, and the Right image to be **Z_ERR** or **Z_PHASE** for feedback error signal or phase contrast, respectively.

Scan Lines	Scan Frequency	Scan Time
128 Lines	1 Hz	2 minutes
128 Lines	0.5 Hz	4 minutes
256 Lines	1 Hz	4 minutes
256 Lines	0.5 Hz	8 minutes
512 Lines	1 Hz	8 minutes
512 Lines	0.5 Hz	16 minutes

Table 1: Scanning Times

2. Start the Scan. The sample will move in a raster pattern with the fast axis determined by Scan Rotation. The sample moves forwards and backwards along the fast axis at the Scan Rate, but data is only taken during the forward movement.
3. You will most likely need to tune the **Z Feedback** generalized PID (GPID) parameters iteratively to achieve optimal feedback performance and thus accurate topography measurements. Refer to Appendix

G on page 56 of the [TT-AFM Users Guide](#). **Include in your report line profiles of the calibration sample with too much, too little and optimal feedback gain.** Discuss why the derivative gain (D) is zero and why we mostly change the integral (I) gain.

4. When the scan is finished or **Stop** is pressed, a window will pop up that lets you change the file name and/or directory if needed, and save the data.
5. If you want to scan another region close by, click **Tip Retract** one or two times, gently move the sample with the X/Y micrometers, and redo a **Tip Approach**. If you need to move a large distance or to change samples, use **Tip Retract FIRST** followed by **Manual Z Motor Control** to move the tip a safe distance away.

6.8 Data Processing and Analysis

AFM images are 2D arrays of numbers which contain information about the surface properties of the sample. There are three types of processes that one can do with this data: (1) Display: changes how the numbers are viewed, such as changing the color scale, (2) Process: changes the numbers in the array, such as subtracting a background, (3) Analysis: extract numbers from the data array, such as surface roughness.

Use the program **Gwyddion** to view, process, and analyze your data:

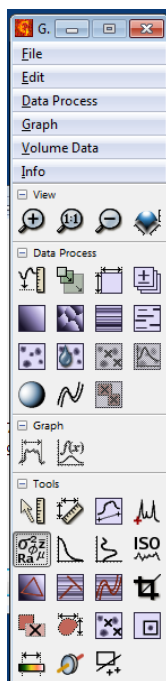


Figure 27: Gwyddion main window

Watch the [video tutorial for Gwyddion](#) and browse [the Gwyddion guide](#) online. Make sure you understand how to: **subtract a background plane, do line-leveling, extract line profiles, measure in-plane and vertical distances between features, and extract statistical parameters like root-mean-square (RMS) roughness.** If you wish to do some of your data processing on another computer, you may download it for free [here](#).

One of the most important skills for AFM data analysis is to identify and mitigate artifacts. Include some examples of artifacts and debris present in your scans in your report and explain what caused these to occur and how they affected your measurements. Refer to page 46-49 of the [reference slides](#) and [AFM Image Artefacts](#).

6.9 AFM Calibration (Complete on 1st Day)

For your first scan, you will use the calibration sample (Sample 7, [AppNano Silicon Step Height Reference, Part# SHS-0.1-1](#)) and measure **Feature B** therein so you can calibrate the piezo stages of the AFM. See Appendix C, page 47 in the [TT-AFM User Guide](#) for more details. Below is a brief summary of the steps:

1. Perform a large enough topography scan of a clean area in Feature B region of the calibration sample. This region contains square pits with $10\ \mu\text{m}$ lateral pitch and $102\ \text{nm}$ vertical depth (Fig. 28), so the scan needs to be $> 20\ \mu\text{m}$ to cover multiple periods (See Fig. 17 for an example). It is helpful to rotate the sample such that the grid is aligned with the scan X/Y direction. Pay attention to Scan Rotation.

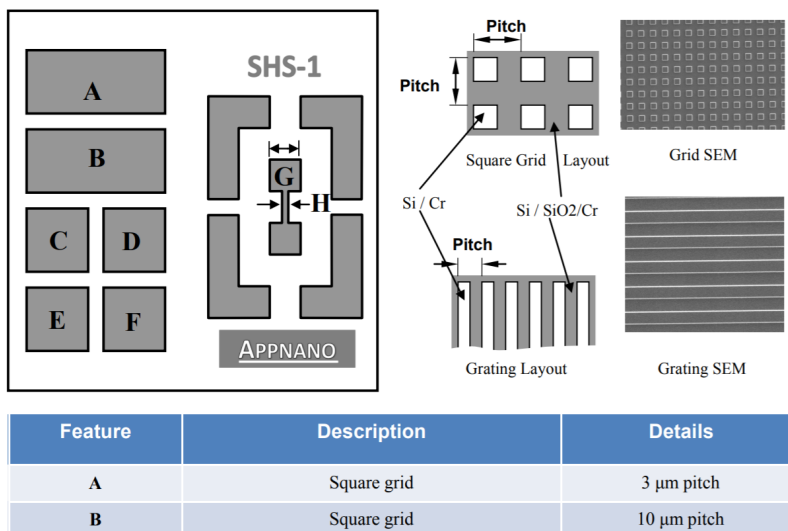


Figure 28: Calibration sample schematic

2. Load the **Z_DRIVE** image into **Gwyddion** and perform plane/line fitting and/or background subtraction if needed. Make **multiple** measurements of the lateral pitches along X and Y directions, and the depths of the pits. Follow Appendix C in [TT-AFM User Guide](#).
3. Open **Calibration XYZ.html** from the Desktop, populate the form with the current X/Y/Z calibration factors from the **System** Tab (upper left, see Fig. 18), the measured and actual values of the X/Y pitches and Z depths, and calculate the new X/Y/Z calibration factors.
How different are the new values from the current ones? Write down the current values as a backup. Input the new values to the **System** Tab and click **Save**.
4. Do another topography scan to confirm that you are getting more accurate results. Now the AFM is properly calibrated, you are ready to perform the [Experiments](#).

Checkpoint Calibration:[†] Show a GSI the processed topography scans that you used to generate the new calibration values. Discuss any differences from the current values. Demonstrate that the AFM is now well calibrated.

6.10 Shutting Down

At the end of each day, turn off the LED illuminator and exit the control software. At the end of your multi-day experiment session, also turn off the Ebox.

7 Experiments

You will perform the following **FIVE** experiments. We recommend that you do them in the order as listed.

1. [Noise Floor Experiment](#)
2. [CD/DVD Experiment](#)
3. [Glue Stick Polymer Experiment](#)
4. [Force-Distance Curve Experiment](#)
5. [Boltzmann's Constant Experiment](#)

For any of these experiments, you may use the stereoscope to the left of the AFM to get an overview of the samples. It is particularly helpful for experiments that require scanning over a specific area of the sample.

7.1 Noise Floor Experiment

This short experiment is designed to measure the noise in an AFM measurement from both internal and environmental sources. This will allow you to distinguish actual features from noise. You will also demonstrate ways to mitigate internal and environmental noise. **Use Sample 8, Blank Silicon Wafer**

1. With the tip a safe distance away from the sample, change the **XY HV Gain** to 0 and **Z HV Gain** to 15 in the **System** Tab.
2. Perform a Vibrating Mode Tip Approach in a clean region on the sample.
3. Perform **TWO** topography scans with the following parameters, one in a very quiet environment (box closed, no talking or touching the bench) and the other in a noisy environment (box open, talking to partner, light typing on the bench). No need to wait for the scan to finish, 50 lines is plenty.
 - Scan Size = 0.5 microns
 - Scan Rate = 1 Hz
 - Scan Lines = 512
 - X, Y Center = (2, 2)
 - Left, Right Image = Z-DRIVE, Z-ERR
4. Retract the tip, set **Z HV Gain** to 5 and repeat the previous two steps.
5. Retract the tip and set both **XY HV Gain** and **Z HV Gain** back to 10.
6. Follow Appendix D of the [TT-AFM User Guide](#) to process the **FOUR** scans (combinations of 2 binary conditions) and extract the RMS values in height.
7. Perform Fourier transform on a few line profiles in the four scans and plot the noise spectra. Clearly label the axes. Note that one line takes 0.5 s for a Scan Rate of 1 Hz.
8. Discuss in your report:
 - How does the Z drive voltage noise (roughly proportional to **Z HV Gain**) and environmental noise (air flow, sound and vibration) affect the RMS noise floor?
 - What about the noise spectra? What are the most common noise frequencies and how are they affected by the voltage and environmental noises?
 - Can you identify and explain the different measures that the lab has taken to reduce the noise for the AFM experiment?
 - What are other ways and precautions that you can take to lower the noise floor?

7.2 CD/DVD Experiment

In this experiment you will scan the information-carrying structures in a CD and a DVD, to gain a deep understanding of the working principles and capacity limitations of these optical disks. You will also learn to prepare your own AFM samples.

1. Explain how a CD/DVD works and what we expect to see (Ref. [1](#), [2](#), [3](#), [4](#)).
2. You will begin by preparing your own CD samples from the provided disks. You need a fresh CD sample because the information-carrying foil will quickly oxidize (in about a week) after it is removed from the protective plastic disk. The DVD samples are more robust and are already mounted, but you will have to remove the blue plastic covering before scanning.
 - (a) Lay out a paper towel on the table to the left of the AFM and work exclusively on it to catch any flakes that come off the CD and to protect the table.
 - (b) Place a large piece of double-sided tape on a magnetic sample plates and use the X-Acto knife to cut away the excess tape so the tape is entirely contained on the plate.
 - (c) Take a silicon wafer piece and attach it firmly to the magnetic plate such that the shiny side faces up, then repeat the step above to put tape on the silicon piece.



Figure 29: Supplies needed



Figure 30: Cutting the CD

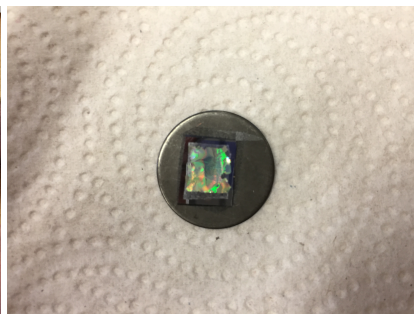


Figure 31: Final prepared sample

- (d) Take the "CD with data" and lay it on the paper towel shiny side DOWN (The underside of a CD is a thick plastic disk that we will NOT be cutting through). Using the X-Acto knife, cut a small (smaller than the silicon wafer) square out of the top of the CD, and use the tweezers to peel and attach it to the silicon wafer shiny side UP. Keep the foil as flat and contamination-free as possible.
 - (e) *At the end of your lab time with AFM, please remove the silicon, tape, and CD foil from the sample plates so the plates can be reused by the next group.*
3. Find a clean region on the CD sample and obtain a topography scan with multiple pits and tracks. You may select **No Background** in the Display window to avoid artifacts from line leveling. Are the pitch depth and track pitch consistent with expectation?
 4. Remove the cover from the DVD sample. Obtain a topography scan using the same scan size as the CD sample. Replace the cover once you are done.
 5. Include in your report:
 - How is information stored on a CD? What wavelength of light is used? Why? How is it different for a DVD?
 - Explain constructive and destructive interference, find the expected pit depth for CD/DVD.

- Processed topography scans of CD and DVD with the same scan size. Does it make sense to use line-by-line leveling or plane subtraction for these samples?
- Compare the expected pit depth values to your measurements.
- From the measured pit density and track pitch, estimate the information density and the total capacity of a single-layer CD/DVD. Compare with standard values.

Checkpoint CD/DVD:↑ Show a GSI scans of the CD and DVD samples, describe how you have processed or plan to process them and discuss whether the pit depth and track pitch are roughly consistent with expectations.

7.3 Polymer Glue Experiment

In this experiment you will attempt to use phase contrast to distinguish different materials on an otherwise smooth surface. Read about the physics behind an AFM phase measurement on page 40-45 in [these slides](#). Use **Sample 12, Hot Glue**.

1. Use **Vibrating** mode, set your Scan Size to $\leq 15 \mu\text{m}$.
2. Scan a clean region on the sample, with the left image set to Z_DRIVE and the right image set to Z_PHASE. You may need to move around to find a smooth region with good phase contrast.
3. Include in your report:
 - Side-by-side comparison of the processed topography image and the phase image.
 - Do all the features in the phase image correlate with those in the topography image? Why?
 - Does an increase in phase in the absence of topographic features correlate to a relatively harder or softer material? Why?
 - How many different polymers do you think you can identify in this hot glue sample?

7.4 Force-Distance Curve Experiment

Force-Distance (F-D) curves are created by measuring the deflection of the cantilever as the sample is moved towards or away from the tip without active feedback (animation [1](#), [2](#)). The shape of a F-D curve depends primarily on the length and stiffness of the cantilever, the thickness of the surface contamination layer and the hardness of the sample. See page 17-21 [here](#) for more details.

F-D curves also allow us to translate raw **T-B photodetector output** (often in V) to **cantilever deflection** (in nm), so we can quantitatively measure e.g. the thermal motion of the cantilever by monitoring the T-B signal.

A brief summary of the steps below:

1. In a clean region on the blank silicon wafer or calibration sample, do a Tip Approach in **Non-Vibrating** mode. **Re-Center** if needed.
2. In the **Force-Distance** tab, set the **Reverse Trigger** to **T-B Signal** with a value a few hundred mV higher than the current set point. The AFM will keep moving the tip into the sample until T-B is increased by this value, before retracting.
3. Set **Vertical Axis** to **T-B** (because we do not know the proportionality constant between T-B and deflection, or the cantilever spring constant). Test with **Rate** set at **Fast** first and take the final data with **Slow** (Fig. [32](#)).

- Click the large **Start** button to the right of the plot window and wait for it to finish. A window will pop up to allow you to save the data. Take multiple curves, check their consistency, and average. Estimate the proportionality constant between T-B signal and cantilever deflection.

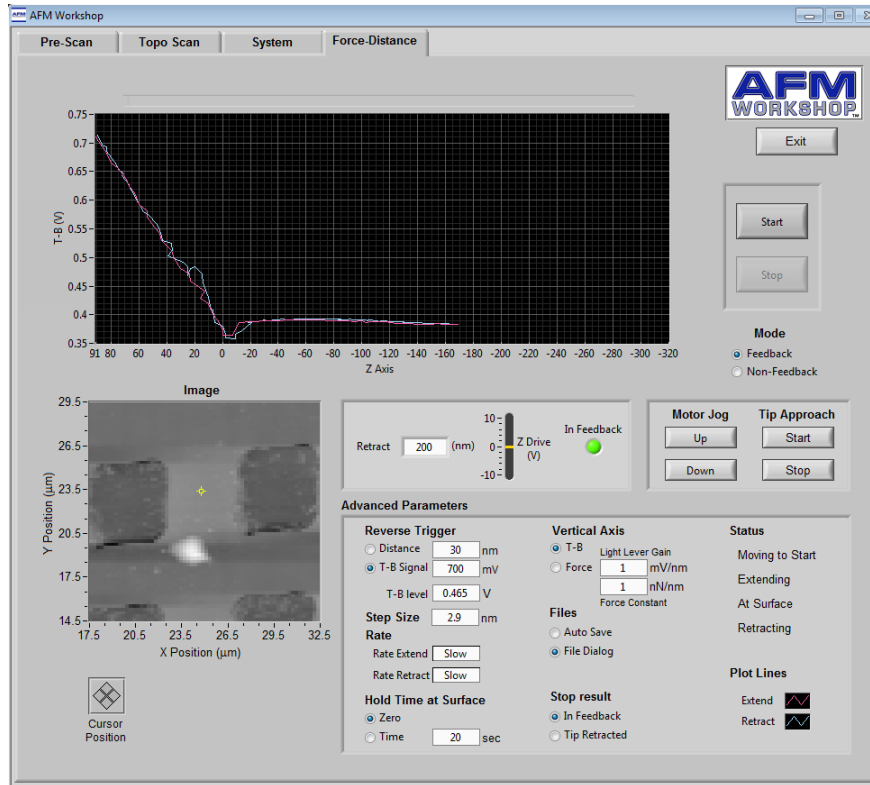


Figure 32: Example Force-Distance curve settings and result

- Estimate the spring constant k of the cantilever. The manufacturer, AppNano, specifies a range of resonant frequencies and spring constants for their probes on the box. Modeling the cantilever as a simple harmonic oscillator gives a relation between spring constant k and resonant frequency ω_r . Using the end points of the frequency and spring constant range gives you two points along this curve. Using the point (0,0) will give you a third point. Interpolating these three points with a Lagrange polynomial yields the exact relation predicted by the model. The **Pre-Scan** tab allows an accurate measurement of ω_r . Plugging this into your polynomial to estimate the spring constant k of your probe.
- Include in your report:
 - High-quality F-D curves with the vertical axis in T-B. Explain the various features in the curves, including any "snapping" and "adhesion" of the tip to the sample.
 - Describe how you calculated the proportionality constant between T-B signal and cantilever deflection.
 - Describe how you estimated the cantilever spring constant k .
 - Using results from the previous two steps, re-plot the F-D curve, this time with the vertical axis in force (e.g. nN).
 - Discuss: Is T-B mostly due to the rigid vertical movement of the cantilever, or the change in its inclination (angle)?

7.5 Boltzmann's Constant Experiment

The equipartition theorem relates the temperature of a system to the thermal energy in its various degrees of freedom. In the case of the cantilever, we can model it as a 1D harmonic oscillator bobbing up and down. In this case, there is one degree of freedom z (no translational motion, no twisting, etc.). The energy associated with z is $\frac{1}{2}kz^2$, and thus we have $\frac{1}{2}k\langle z^2 \rangle = \frac{1}{2}k_B T$, where k is the spring constant of the cantilever and k_B is Boltzmann's constant. You just estimated k in the F-D curve experiment. For T , check the thermometer in the acoustic box. This leaves you to measure $\langle z^2 \rangle$ in order to estimate k_B .

It is up to you to design an experiment to measure $\langle z^2 \rangle$ with the best possible accuracy and precision. You can use any internal AFM signals under any scan conditions. You can also directly access the raw T-B signal using the BNC cable connected to the back of the Ebox. You can use any combinations of a [low-noise voltage amplifier](#) with tunable bandwidth and gain, a spectrum analyzer ([SSA3021X Plus](#)), and a digital oscilloscope to measure T-B.

Consider the following:

- What is the expected value of $\langle z^2 \rangle$? Can we measure it given the noise floor of the AFM?
- Recall that the cantilever is a mechanical resonator with the lowest resonant frequency at tens to hundreds of kHz – **Is most thermal energy concentrated at this frequency?** If not, why? If yes, does it make sense to use the [internal analog-to-digital converter](#)?
- Does T-B measure z or dz/dx of the cantilever? What is the implication to our measurements?
- Very helpful references: [1](#), [2](#).
- Tutorial on [spectrum analyzers](#). A key message is that the spectrum analyzer reading is by default the **power** within the **resolution bandwidth** (RBW).

Include in your report the design of the experiment, the rationale, the measurement result and the estimated k_B . Discuss any approximations made and potential sources of errors.

Checkpoint Boltzmann's Constant:[†] Show a GSI the theoretically estimated $\langle z^2 \rangle$, the spring constant k , the experiment plan to measure $\langle z^2 \rangle$ and any preliminary measurements, if available.

8 Troubleshooting

Below are solutions to some common problems you might run in to while operating the AFM. For a wider range of solutions, refer to [Chapter 4: Measuring AFM Images](#) and [Chapter 6: Image Artefacts](#), and page 34-35 of the [TT-AFM Users Guide](#).

8.1 Software Bugs

If you are experiencing problems with the AFM software such as:

- Laser does not turn on.
- Photodetector is non-responsive to L/D knob adjustments.
- Probe does not move during Range check.
- Cannot perform a tip approach properly (excessive tip approach timeouts).
- Z-Drive will not zero after Re-Center.
- Program still says the tip is in feedback even though the tip is clearly raised above the sample.

Try the following: Exit the program, power cycle the Ebox, restart the program. If the bug persists, also restart the computer after turning off the Ebox. If the AFM is still not functioning properly, get assistance.

8.2 Broken Probes

Throughout this experiment, it is very likely that you will break one or more probes.

- Common ways to break a tip:
 - If you jam a tip into the sample using Z motor, the tip will most likely become too dull to be usable.
 - If you perform a range check, or move the sample while the tip is actively engaged with the surface of the sample, your tip will be destroyed.
 - If your probe slides off of the exchange tool, off of the alignment grooves on your probe holder, is dropped, is flipped, or is picked up incorrectly during probe exchange, the tip (and often the cantilever) will likely be destroyed.
- Evidence of broken or damaged tips:
 - The cantilever is visually broken or missing in the camera view.
 - A dull or blunt tip will result in blurry and/or fuzzy appearing images, especially noticeable on surface features with known sharp edges.
 - A fractured tip will produce "double edges" or "ghosting" around sharp features.

8.3 Other Issues

- If the total DC photodetector signal (T+B) is saturated, try changing the photodetector gain switch to low gain (flip down).

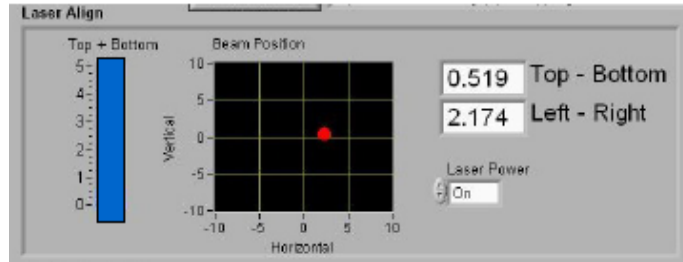


Figure 33: Saturated photodetector

- Highly tilted or unstable scans: Try attaching the sample to the sample plate more securely.
- Probe not seated in holder correctly: You will want to re-adjust the probe as a *LAST RESORT*.
- Cantilever resonance signal is saturated. Reduce **Amplitude (Vpp)** and/or **Demod Gain**.

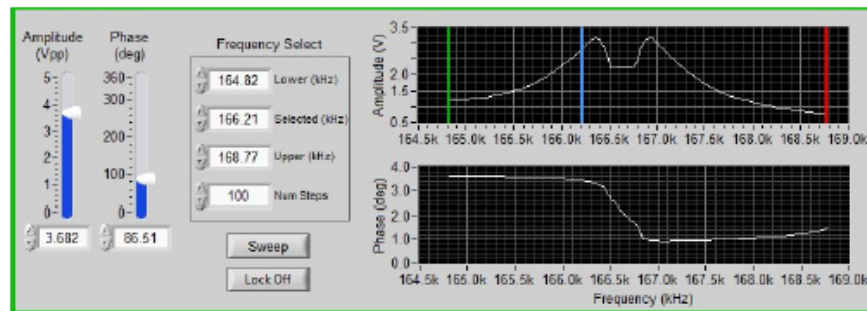


Figure 34: Peak amplitude is too high, resulting in saturation.

- **False Feedback:** If the scan line profile is smooth but random, you are probably in False Feedback as the tip interacts with surface contamination. Try fine-tuning the set point and/or selecting a different spot on the sample. Refer to the instructional video.