



July 29 - August 9, 2013

University of Illinois at Urbana-Champaign

## MICRO AND NANOFABRICATION LAB MODULE

### Experiment 1: Photolithography

Location: 211 & 235 Micro and Nanotechnology (MNTL) Cleanroom Lab

Process Engineer: Glennys Mensing, MNTL

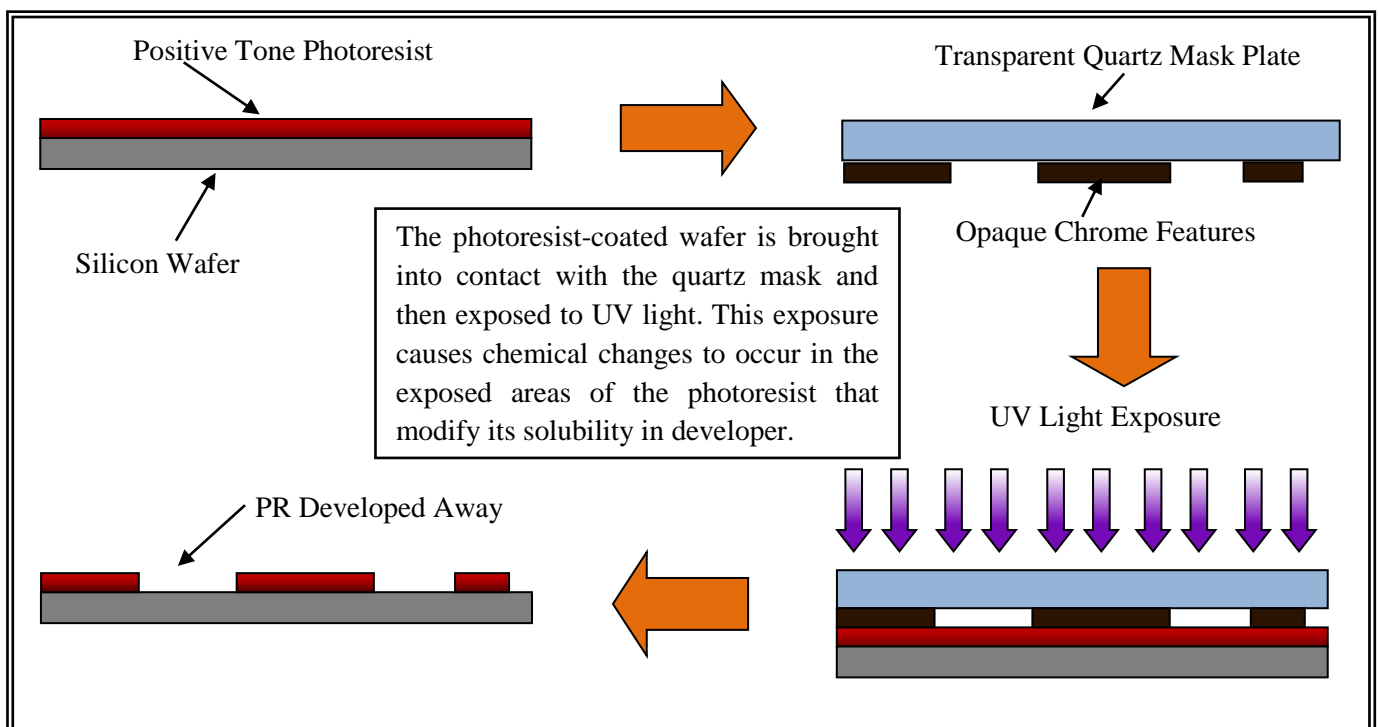
Lab Assistant: Katrina Keller, Bioengineering

#### Purpose and Expected Outcome:

The purpose of this laboratory module is to provide an introduction to standard photolithographic techniques and hands-on experience by patterning a silicon wafer. Upon completion of this module, the researcher will be able to understand and perform all the steps in a conventional optical lithography process. This will be followed by an examination of the patterned wafer under a microscope.

#### Overview of Photolithography:

Photolithography is a common and basic step used in the creation of bioMEMS and biosensors. This fabrication technique has been used for many years in the fabrication of integrated circuits, transistors, and MEMS devices. The underlying principal of photolithography relies on the way certain polymeric compounds, called photoresists (PR), respond to ultraviolet (UV) light exposure. Areas that have been exposed to UV through a mask will exhibit selective solubility in a developing solution. There are two main types of photoresist – positive and negative tone. A positive tone resist will dissolve after exposure to UV light, while a negative tone resist will be insoluble after exposure to UV light. The lithography process presented in this lab module is capable of resolving features that are less than 1.0 micron. The method of photolithography used in this module is called contact photolithography, and a generalized process flow is presented in Figure 1.



**Figure 1:** Contact photolithography process: When the exposure is completed, the wafer is submerged in a developer to wash away the exposed sections of the positive tone photoresist. The end result is an exact copy (or the inverse, if using a negative tone PR) of the opaque chrome features on the mask plate.

## Module Outline and Workflow:

In this lab module, researchers will carry out all steps of a contact photolithography process. This process includes spin-coating, exposing, developing, inspecting and hard-baking of a 2" Si wafer.

### Procedures:

#### 1. Spin-coating

- 1.1. Degrease a 2" silicon wafer using acetone, IPA, DI water, IPA and N<sub>2</sub> dry. All solvent work must be done in a fume hood and waste needs to be put into the solvent waste jug.
- 1.2. Place the degreased wafer on the 270°C hotplate for 1min to perform a dehydration bake.
- 1.3. An appropriate spinner chuck for a 2" wafer has already been installed.
- 1.4. Open the spinner lid carefully by depressing the black button while holding the spring loaded lid.
- 1.5. Place the degreased silicon wafer, polished side up, on the spinner chuck and center the wafer to the chuck.
- 1.6. Close the lid and verify recipe #7 is selected. This recipe will spin the wafer at 4000rpm for 30secs.
- 1.7. Turn on the spinner by pressing the green button. Check to ensure there is a good vacuum seal between the chuck and wafer. Check if the wafer is centered.
- 1.8. Stop the spin by pressing the red button, press the green button to clear any errors.
- 1.9. Dispense 1mL of SPR220 positive tone PR onto the center of the wafer using the pipette. Be sure to minimize bubbles. If there are bubbles, draw them back up into the pipette. Dispense excess PR off to the side but still inside the spinner.
- 1.10. Activate the spinner by pressing the green button and wait until it automatically stops.
- 1.11. Open the spinner lid carefully by depressing the black button and remove the PR coated wafer. Transfer the wafer to the 110°C Al hotplate for 2 mins to perform a soft bake.
- 1.12. Remove the PR wafer from the hotplate and place on the cooling block for ~20secs. Place PR wafer into petri dish.

#### 2. Exposure

- 2.1. The EVG 620 mask aligner will already be ON, a mask will be loaded, and the tray will be out and ready to load.
- 2.2. Center your wafer on the 2-in wafer chuck. Press the continue button to indicate the substrate is loaded and move tray in when prompted to do so.
- 2.3. No alignment is needed so press continue when prompted to expose the wafer (the dose will be set and the aligner will adjust the time accordingly).
- 2.4. For our process, the dose used is 300 mJ/cm<sup>2</sup>. The exposure time is calculated by using the equation below:  
$$\text{Dose(mJ/cm}^2\text{)} = \text{Intensity(mW/cm}^2\text{)} * \text{Time(s)}$$
- 2.5. The aligner will be set up for contact mode, which means the mask and wafer will come into contact with each other during UV exposure.
- 2.6. When the exposure is complete, move tray out when prompted to do so. Carefully remove the wafer from the wafer chuck. Place the exposed wafer into your petri dish.

#### 3. Develop/Inspect

- 3.1. Transfer the exposed wafer to the acid hood.
- 3.2. Rinse a Pyrex dish cover with DI water
- 3.3. In the acid hood, a pre-measured solution 5:1 ratio of DI water and AZ400K developer has already been prepared in a large beaker.
- 3.4. Pour the developer solution into the clean Pyrex dish about ¾ full.
- 3.5. Submerge the wafer fully in the developer solution and lightly agitate for ~30-40 secs. Notice that any exposed areas will have the PR removed.
- 3.6. Place the developed wafer into the DI water beaker for 1min (called a DI quench).
- 3.7. Remove the developed wafer and rinse with copious amounts of DI water.
- 3.8. Carefully dry the developed wafer using an N<sub>2</sub> gun.
- 3.9. Inspect the developed wafer with the optical microscopes.



# 2013 GEM4 BioNanotechnology Summer Institute

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## MICRO AND NANOFABRICATION LAB MODULE

### Experiment 2: Metallization by Thermal Evaporation & Metal Liftoff

Location: 209 & 235 Micro and Nanotechnology (MNTL) Cleanroom Lab

Process Engineer: J. Hal Romans, MNTL

Lab Assistant: Chen Zhang, MNTL

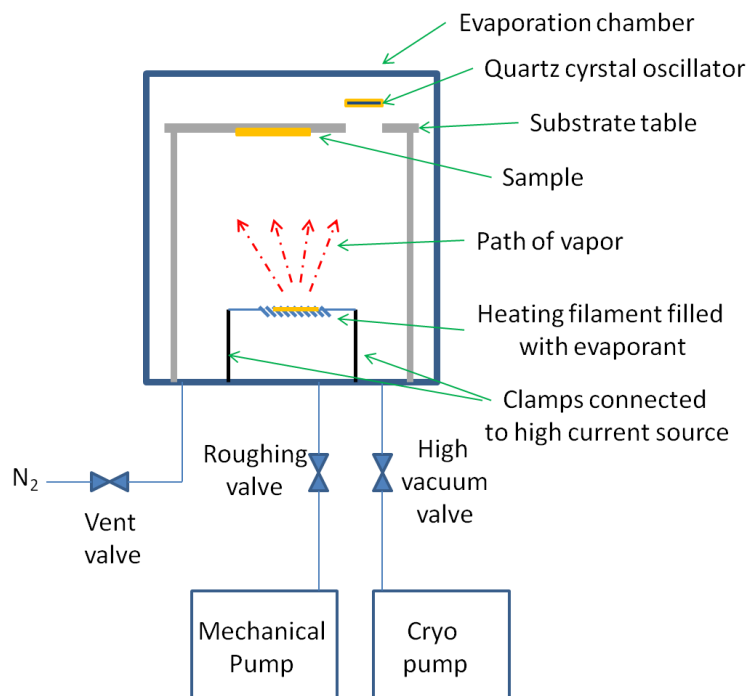
#### Purpose:

The purpose of this laboratory module is to provide an introduction and a hands-on demonstration of the metallization process and subsequent liftoff process.

#### Overview of the Thermal Evaporation Process:

In this experiment, a 2" wafer which has been previously patterned with photoresist, will be loaded into a thermal evaporator. The vacuum chamber will be pumped down and 100 nm of Aluminum will be deposited on the wafer using a thermal source.

#### Schematic of a Basic Thermal Evaporator System:



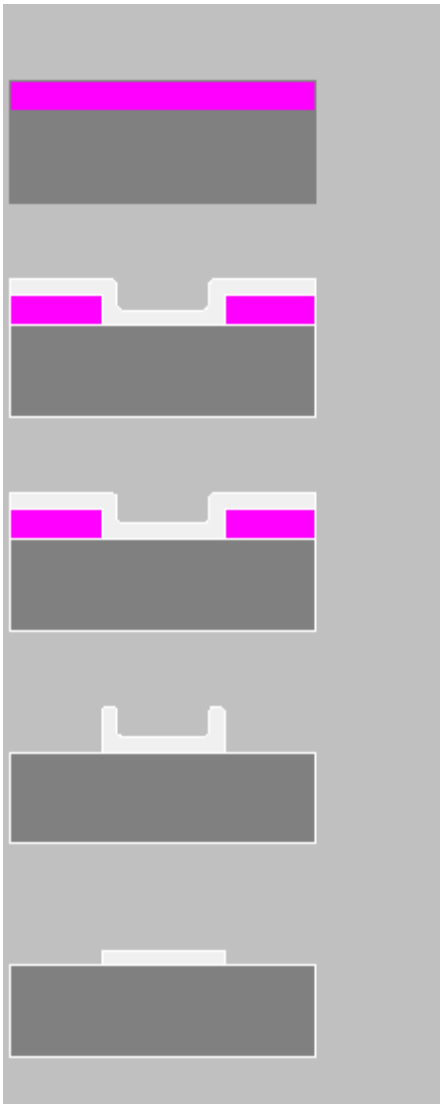
After venting and loading samples, the deposition chamber is first pumped with a mechanical roughing pump then a cryo pump to a final vacuum of about  $1 \times 10^{-6}$  Torr.

A heating element containing the evaporation source is heated using a current regulated power supply. The voltage is <30VAC and the current is adjustable from 0 to 300amps.

## Evaporation Procedure:

1. Turn off the ion gauge
2. Close the high vacuum valve if it is open
3. Open vent valve
4. When atmospheric pressure (~760 Torr) is reached, raise the chamber.
5. Close the vent valve
6. Insert source material
7. Load sample
8. Lower the chamber
9. Shut the fore line valve
10. Make sure vent valve is shut. Open the roughing valve
11. When the pressure reaches 100 ~ 150 mTorr, close the roughing valve
12. Open the fore line valve
13. Open the high-vacuum valve
14. Wait for desired vacuum
15. Turn on EVAP SUPPLY
16. Slowly increase the current for evaporation. The evaporation rate and thickness will be displayed on the controller (units in Angstrom)
17. Evaporate desired amount of Aluminum
18. Set the current knob at 0, and turn off EVAP SUPPLY
19. Wait a few minutes to let the source to cool down
20. Repeat #1 – #10 to remove sample and pump down the chamber

## Lift Off Procedure:



1. Place the wafer in a beaker and cover with acetone.
2. Agitate until all the unwanted metal is removed and the desired pattern is revealed.
3. If needed, the beaker can be placed in the ultrasonic bath.
4. Continue cleaning / Remove the wafers from the acetone and rinse with DI water.
5. Apply isopropyl alcohol and dry wafers with N<sub>2</sub>.
6. Dispose of the acetone in the solvent waste container.



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## MICRO AND NANOFABRICATION LAB MODULE

### Experiment 3: Reactive Ion Etching (RIE)

Location: 232 Micro and Nanotechnology (MNTL) Cleanroom Lab

Process Engineer: Yaguang Lian, MNTL

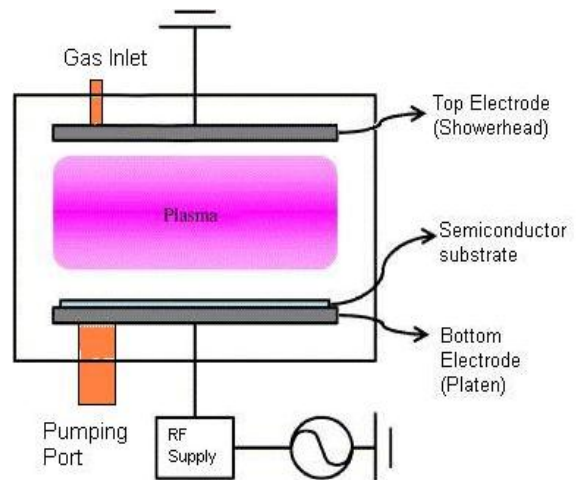
Lab Assistant: Shiul Khadka, Electrical and Computer Engineering

#### Purpose:

The purpose of this experiment is to provide an introduction and a hands-on demonstration of RIE technology. RIE is a “dry” etching process. It is widely used in micro-fabrication. Compared with wet etching, dry etching has two key advantages: less undercutting (allowing smaller lines to be patterned) and higher anisotropy (allowing high-aspect-ratio vertical structures).

#### Theory:

Typically, an RIE system has four main components: an etch-chamber, a 13.56 MHz radio frequency (RF) power supply, a pumping system, and a gas handling system. The applied RF power produces ionized gas plasma containing reactive species (atoms, radicals, and ions). Plasma dry etching incorporates a physical component (similar to glow-discharge sputtering or ion milling) and a chemical component (volatile reaction). The etchant gases are selected to generate species that react chemically with the materials to be etched, and whose reaction products are volatile, so different materials require different etchant gases. For example, halocarbon gases ( $\text{CHF}_3$  and  $\text{CF}_4$ ) can be used to etch Si,  $\text{SiO}_2$ , and  $\text{Si}_3\text{N}_4$ . Photoresist can be etched with  $\text{O}_2$ .



#### Overview of the RIE experiment:

For our demonstration of an RIE process, we will be starting with a silicon wafer onto which a thermal oxide layer ( $\text{SiO}_2$ ) has been grown. On top of the oxide you will see patterned photoresist on the wafer, the creation of which is detailed in Lab Module 1. The patterned wafer is inserted into the RIE chamber where the oxide layer will be etched. The photoresist acts as an etch mask to prevent etching in those areas.

The main steps of the process are shown below:

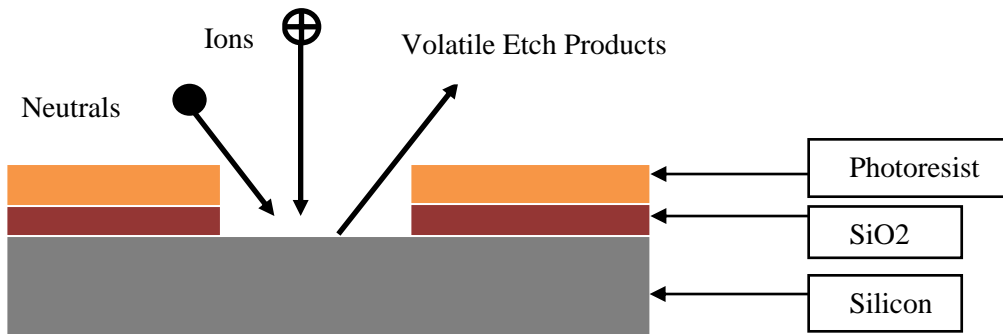
### Thermal SiO<sub>2</sub> Layer Growth



### Photoresist Patterning



### Reactive Ion Etching of SiO<sub>2</sub>



### RIE procedures:

We will use a PlasmaLab® Freon RIE system in our experiment.

1. Press **Process** and **4** to select a CF<sub>4</sub> etch process.
2. Turn the key to the **Change Stored Parameter** position.
3. Set in the parameters for CF<sub>4</sub> Flow Rate, RF Power, Process Pressure, and Time.
4. Turn the key back to **Manual Operation**.
5. Press **Manual** and **Vent**.
6. After vent cycle finishes (**Vent** light will come on), wait for 15 to 20seconds. Turn off **Vent** button.
7. Open the chamber by pushing the **Joystick** to the right and pushing down on the **Large Black Button** at the top right corner of the cabinet.
8. Put the wafer(s) into the chamber.
9. Close the chamber by pushing the **Joystick** to the left and again pushing the **Large Black Button** down.
10. Open the gas cylinder.
11. Press **Run**.
12. Wait for the process finish until **Vent** light is on.
13. Repeat the steps 6 and 7; remove the wafer(s) from the chamber; repeat step 9.
14. Press **Manual** and **Rough** to pump down the chamber and close gas cylinder.



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## MICRO AND NANOFABRICATION MODULE

### Experiment 4: Scanning Electron Microscope (SEM) & Atomic Force Microscope (AFM)

Location: 241 Micro and Nanotechnology (MNTL) Cleanroom Lab

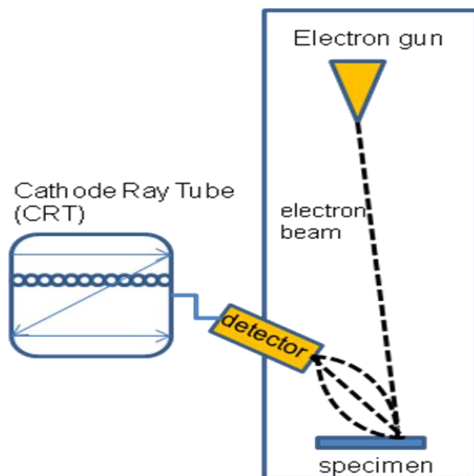
Instructor: Edmond Chow, MNTL

#### Purpose and Expected Outcome:

The purpose of this laboratory module is to provide an introduction and a hands-on demonstration of the two most common imaging techniques that have nanometer-scale resolution (much better than optical microscopy): SEM and AFM.

#### Theory:

Basic principles of the scanning electron microscope (SEM):

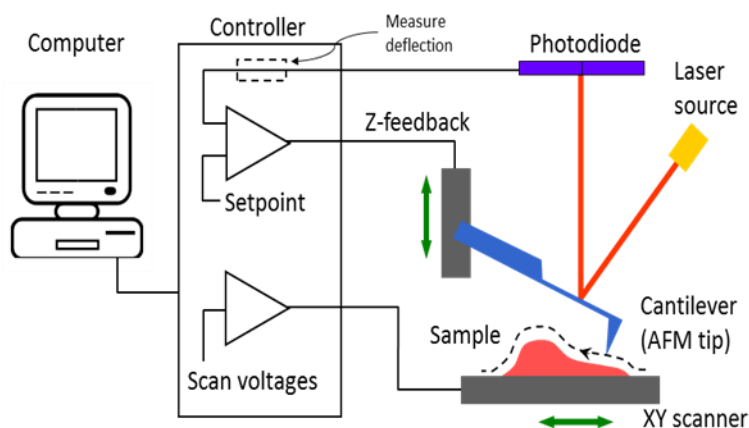


An electron beam is scanned over the specimen. An electron detector is used to record the number of electrons scattered from each point on the specimen. Measured electrons intensity will be displayed on the CRT at the corresponding pixel location to reconstruct the image.

Image contrast is obtained due to the difference in electron scattering efficiency from different topology and materials.

The typical accelerating voltage of electron beam is 1kV-30kV. Higher accelerating voltage electron beam has a smaller spot size and therefore provides higher resolution image, but also increases the chance of specimen damage and charge-up.

Basic principles of the atomic force microscope (AFM):



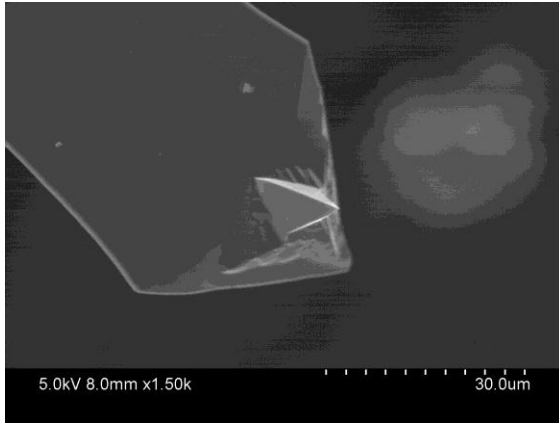
A laser source and photodiode are used to monitor the deflection of the AFM tip. The feedback controller is used to maintain the deflection at a specific set point by moving the tip up and down depends on the topology of the specimen. The recording of the z-motion of the tip is used to reconstruct the image of the specimen.

The proper choice of feedback parameters and set point is crucial in obtaining an accurate image of the specimen.

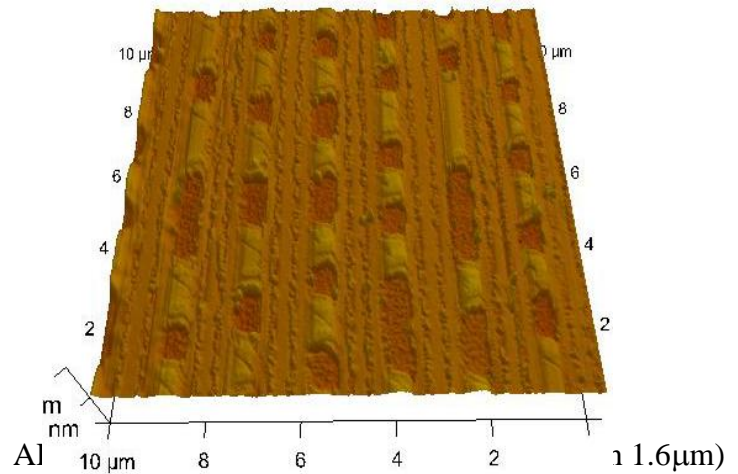


## Experiment:

An SEM will be used to image an AFM tip, with tip radius of curvature around 10-20nm. Then we will use AFM to image the track of a compact disk (CD) to see the data bit recorded in it.



SEM image of an AFM tip



## Equipments and materials

- 1) Hitachi S4800 SEM, 2) Veeco Dimension 3000 AFM, 3) Veeco Si AFM tip, 4) CD

## SEM imaging procedure

1. Mount the AFM tip onto the SEM sample holder.
2. Put the sample holder into the specimen exchange chamber and evacuate the specimen exchange chamber.
3. Open the gate valve and transfer the sample holder to the specimen chamber.
4. Move the sample holder to the "HOME" position.
5. Select 5keV accelerating voltage and turn on the high voltage.
6. Record an image at 500 X magnification by adjusting the focus
7. Measure the cantilever width and length
8. Tilt the sample holder at 20 degree and record an image at 1.5 kX magnification.
9. Tilt the sample holder at 40 degree and record an image at 80 kX magnification.
10. Estimate the radius of curvature of the AFM tip.
11. Save all the images in a folder D:\Images\BSBA\sectionXX\

## AFM imaging procedure

- 1) Mount an AFM tip onto the AFM tip holder
- 2) Put the AFM tip holder to the AFM scanner head (be careful, it costs \$30K)
- 3) Align the laser to the AFM tip
- 4) Align the detector to the reflection of the laser spot.
- 5) Put the CD under the AFM tip and adjust the height of the stage to bring the CD in focus.
- 6) Choose the scanning width to be 10µm, height scale to be 40nm, P-gain to be 0.6. and I-gain to be 0.4
- 7) Engage the tip and start scanning
- 8) Adjust the setpoint to be around 1.2-1.4 to bring the trace and re-trace to align together.
- 9) Save the image in a folder C:\capture\BSBA\sectionXX\
- 10) Measure the CD track pitch from the capture image with data analysis software.