Please note that printed notes and documents may not be taken into the clean room.

MNF documents will be printed on special clean room paper and provided to you when you enter the lab.
MICRO AND NANOFABRICATION LAB MODULE
Experiment 1: Photolithography
Location: 211 & 235 Micro and Nanotechnology (MNTL) Cleanroom Lab
Process Engineer: Michael R. Hansen, MNTL
Lab Assistant: Miki Takagi, Mechanical Science and Engineering

Purpose and Expected Outcome:
The purpose of this laboratory module is to provide an introduction to, and hands-on experience with, standard photolithographic techniques that are used to pattern 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 exposure by ultraviolet (UV) light. Areas that have been exposed to UV will exhibit selective solubility in a developing solution. There are two main types of photoresist – positive and negative. A positive resist will result in an exact copy of what is on the master pattern, while a negative resist will result in the inverse pattern. The lithography process presented in this lab module is capable of resolving features down to approximately 0.9 microns. The method of photolithography used in this module is called contact photolithography and a generalized process flow is presented in Figure 1.

Figure 1: Generalized contact photolithography process. When the exposure is completed, the wafer is submerged in a developer to wash away the cross-linked sections of photoresist. The end result is an exact copy (or the inverse, if using a negative PR) of what is opaque on the mask plate.
Module Outline and Workflow:

In this lab module, researchers will carry out all steps of a contact photolithography process. This includes spin-coating, exposing, developing, inspection 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 N₂ dry. All solvent work must in a fume hood be done into the solvent waste jug.
   1.2. Place the degreased wafer on the 110°C Al 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 lid. Careful, the lid will spring open if not held.
   1.5. Place the degreased silicon wafer polished side up on the spinner chuck, being careful to center it properly.
   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 good vacuum seal between the chuck and wafer. Check if the wafer is centered.
   1.8. Stop the spin by pressing the red button.
   1.9. Dispense 1mL of SPR220 positive PR onto the center of the wafer using the pipette. Be sure to not dispense bubbles. If there are bubbles, draw them back up into the pipette. Dispense excess PR off to the side inside spinner.
   1.10. Activate the spinner by pressing the green button twice and wait until it automatically stops. Check vacuum for good seal during spin.
   1.11. Open the spinner lid carefully by depressing the black button while holding the lid.
   1.12. Remove the PR coated wafer and transfer to the 110°C Al hotplate for 2 mins to perform a soft bake.
   1.13. Remove PR wafer from hotplate and place on cooling block for ~20secs. Place PR wafer into petri dish.

2. Exposure
   2.1. The Quintel aligner will already be ON. Verify the system is “Ready”.
   2.2. On the right-side control panel, press the MASK-LOAD button (LED should illuminate) and rotate the lamp housing out of the way of the mask chuck. Press MASK-LOAD again to lock it in place (LED should no longer be lit).
   2.3. Verify the mask chrome side is facing down (typically silver color facing up) and VACUUM LED is lit. Verify the mask is held firmly.
   2.4. Slide out the wafer tray and place the PR wafer on the chuck (PR facing up).
   2.5. Press the LOAD button and carefully slide the wafer tray in.
   2.6. Monitor the wafer contacting the mask and the aligner will enter into SEPARATION mode.
   2.7. On the right-side control panel, press the MASK-LOAD button (LED should illuminate) and rotate the lamp housing back over the mask chuck. Press LOAD again to lock it in place (LED should no longer be lit).
   2.8. Press the CONTACT/SEP. button so the CONTACT LED is lit.
   2.9. The exposure time is calculated by using the equation below
   Time(s) = Dose(mJ/cm²) / Intensity(mW/cm²)
   For our process, set the exposure time to 30-35secs.
   2.10. Press the EXPOSE button to start the exposure. DO NOT LOOK AT THE UV LIGHT!
   2.11. When the exposure is complete, wait for the ALIGN LED to be lit located next to the EXPOSE button. Carefully slide the wafer tray out and remove the sample. 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 the exposed areas will be where the PR is 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 N₂ gun.
   3.9. Inspect the developed wafer with the optical microscopes.
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: U Guen (Nick) Choi, Electrical and Computer Engineering

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 an ebeam 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
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. Make sure vent valve is shut. Open the roughing valve
10. When the pressure reaches 100 ~ 150 mTorr, close the roughing valve
11. Open the high-vacuum valve
12. Wait for desired vacuum
13. Turn on EVAP SUPPLY
14. Open the shutter and slowly increase the current for evaporation. The evaporation rate and thickness will be displayed on the controller (units in Angstrom)
15. Evaporate desired amount of Aluminum
16. Close the source shutter, set the current knob at 0, and turn off EVAP SUPPLY
17. Wait a few minutes to let the source to cool down
18. 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. Place the beaker in the ultrasonic bath and turn on the power (no heat).
3. Continue cleaning / agitating until all the unwanted metal is removed and the desired pattern is revealed.
4. Remove the wafers from the acetone and rinse with DI water.
5. Apply isopropyl alcohol and dry wafers with N2.
6. Dispose of the acetone in the solvent waste container.
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: Meng Tan, 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 anisotropicity (allowing high-aspect-ratio vertical structures).

Theory:
Typically, an RIE system has four main components: an etch-chamber, a 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. The etchant gas is selected to generate species that react chemically with the material to be etched, and whose reaction product is volatile, so different materials require different etchant gases. For example, halocarbon gases (CHF$_3$, CF$_4$) can be used to etch SiO$_2$ and Si$_3$N$_4$. Photoresist can be etched with 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 (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 SiO₂ Layer Growth**

**Photoresist Patterning**

**Reactive Ion Etching of SiO₂**

**RIE procedures:**

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

1. Press **Process** and 6 to select a CF₄ etch process.
2. Turn the key to the **Change Stored Parameter** position.
4. Turn the key back to **Manual Operation**.
5. Press **Manual** and **Vent**.
6. Wait for the vent cycle to finish (Vent light will come on.)
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 step 7; remove the wafer(s) from the chamber; repeat step 9.
MICRO AND NANOFABRICATION LAB MODULE
Experiment 4: Scanning Electron Microscope (SEM) & Atomic Force Microscope (AFM)
Location: 241 Micro and Nanotechnology (MNTL) Cleanroom Lab
Process Engineer: Edmond Chow, MNTL
Lab Assistant: Brian Dorvel, Electrical and Computer Engineering

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. A higher accelerating voltage produces more electrons, giving better signal to noise ratio, but also increase the probe area of the beam and therefore reduce the resolution.

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.

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.