

Laboratory Report 1

For this Lab Report, you will learn some of the basic MEMS fabrication processes, namely wafer cleaning, oxidation, photolithography and etching both oxide and silicon. **DO NOT ATTEMPT ANY PROCEDURE BEFORE BEING PROPERLY TRAINED.** Follow the procedures outlined in this document, and write the lab report following guidelines posted on the web (and distributed on the first day of class), making sure to answer all the questions in the reporting section for each step.

Process Outline for Laboratory 1:

1. Step 1: Oxidation
 - a. Sample cleaving
 - b. Sample cleaning
 - c. Thermal oxidation
2. Step 2: Photolithography and Oxide Etching
 - a. Spinning photoresist
 - b. Contact lithography
 - c. Development
 - d. Wet etching Silicon Dioxide with Hydrofluoric Acid (HF)
3. Step 3: Silicon Etching
 - a. Wet etching Silicon with Potassium Hydroxide (KOH)

STEP 1: Growing an Oxide

Overview:

Start with half of a 4in <100> Silicon wafer. Use a diamond tipped scribe to make a small scratch on the rough side of the wafer where you want to start the cleave. Flip the sample over and press the scribe down on the polished side where you made the scratch. This should crack the wafer along a crystal plane enabling you to cut the wafer (in a controlled manner) into smaller rectangular pieces. Thermal oxidation requires 2 steps of cleaning: a solvent based clean to remove the majority of contaminants and a Piranha acid clean to further remove organics to prevent contamination of the oxidation furnace. Finally, grow 300nm of “wet” thermal oxide on top of your silicon sample. This is done in an oxidation furnace at 900C with controlled gas conditions (H_2O vapor and N_2).

Procedure:

Sample Cleaving

1. Always wear latex or nitrile gloves while handling the chips. Do not touch samples with your bare hands; the oils from your skin are full of hydrocarbons which are unwanted impurities for all processes.
2. Take a <100> Silicon sample (half of a 4in wafer or so) and place it shiny side down on a cleanroom wipe.
3. Using the diamond tipped scribe, make a small scratch on the unpolished side of the wafer (perpendicular to a flat edge of the sample) where you want to start the cleave (try to get three ~1in x 2in rectangular samples out of the half wafer).

4. Flip the sample over and put pressure on where you scratch the sample. The wafer should begin to crack along a crystal plane. Keep applying pressure until the crack cuts the sample in two.
5. Continue cleaving until you have the desired chips (you should have 2 chips 1in x 2in or (slightly) greater.
6. Scratch meaningful labels on the unpolished side of your samples.
7. Place them into sample carriers.

Solvent Cleaning

1. Using tweezers, place your samples into the wafer boats designated for solvents.
2. Place the wafer boat into the acetone beaker then put the acetone beakers into the sonicator and run for **3 minutes**.
3. Move the wafer boat into the isopropyl alcohol beaker and place in the sonicator for **3 minutes**.
4. Fill a beaker with deionized water and put your wafer boat into the DI water and rinse with DI for **3 minutes**
5. Remove boat from the DI water, place samples onto a new cleanroom wipe and blow dry your samples with the filtered N₂ gun.
6. Place samples back into the wafer carriers.

Note:

1. The cleaning solutions can be reused several times. All the groups will share the same set of cleaning solvents, so be careful not to contaminate the solutions.
2. In case the existing cleaning solvents need to be replaced, dispose them in the appropriate container- **Do not pour solvents down the drain.**
3. All beakers in the lab must be labeled correctly. Beakers are color coded to prevent cross-contamination, please do not use for other purposes.

Acid Cleaning

1. Very slowly add **5 parts of H₂SO₄ to 1 part of H₂O₂**. The mixture is self heating and attains a temperature of 80 degrees centigrade. When cool, the mixture may be re-used by adding 5 ml of fresh H₂O₂. Heat to 80-100C
2. Place samples into a Teflon boat designated for Piranha.
3. **Carefully** place boat into the hot Piranha solution and leave for 10 minutes.
4. Remove boat and place in DI water for 3 minutes
5. Remove samples from boat, blow dry with N₂ and place in your sample carrier
6. Take samples to the HF hood.
7. **Put on the appropriate protective equipment (Nitrile (purple) or acid gloves, apron, and face shield).**
8. Place samples in the Teflon boat for HF.
9. Dip in HF solution for 15 seconds to remove any native oxide.
10. Remove boat from HF and rinse with DI water for 3 minutes.
11. Blow dry with N₂ and place samples in wafer carrier.

Thermal Oxidation

1. Open the N₂ valve at the back of the furnace till the flow meter reads **15** let the furnace purge for **10 minutes**. Reduce gas flow to load or unload furnace.
2. Load the wafers on to the boat
3. Place the boat at the mouth of the furnace for **5 minutes**.
4. Slowly push the boat in over a period of **3 minutes** till you reach the flat zone.
5. Allow **5 minutes** for the wafers to heat up to the furnace temperature. Nitrogen flow should be 15 for the duration of soak time.
6. Adjust gas flows for wet oxidation and leave the wafers for the **desired time**.
7. Slowly pull the wafers out of the furnace over a span of **3 minutes**.

- Let the boat sit at the mouth of the furnace for **5 minutes**.
- Remove the boat from the furnace. Gently remove your samples from the boat. Let the boat cool for at least **20 minutes** before moving the samples.

Reporting:

- Describe, in your own words, the procedure you used to clean the chips. Write down all the steps you performed, including the details of the procedure, including details like the time you kept the chips in the ultrasonic cleaner, the exact temperature of the oven, the length of time the chip was blown dry and kept in the oven, etc...
- Take a picture of your blank starting substrate showing the scribed alphabet. Write/label visible defects, if any. (Don't forget scale bars on the image!!)

STEP 2: OPTICAL LITHOGRAPHY and OXIDE ETCHING

Overview:

You will prepare the wafer for spinning on the positive photoresist (AZ 4110) including the dehydration bake and HMDS treatment. In brief, you will spin on AZ 4110 and then softbake the resist for 1 min. @ 85 deg C. Next, you will expose for the appropriate time using Mask-1. There is no alignment at this stage. Use the exposure times suggested by your TA. (20 sec. @ 7.5 mW/cm²) [The lamp is preset for 7.5mW/cm²]. Next, you will develop the patterns in 4:1 AZ 400K developer. Again use the develop time of 60 seconds. Inspect the photoresist under the optical microscope and ensure that the patterns have been transferred accurately. Etch through the quartz in 50:1 H₂O:HF. Etch rate of 50:1 H₂O:HF for SiO₂ is between 45-65 Å/min. at 21 deg C. Using this value as a starting estimate determine the etch rate for quartz. Immerse the chip in DI for 1 minute before you place it in HF. This prevents bubble formation and ensures uniform etching in HF. Your etch depth should be approximately 50nm. Strip off the photoresist in acetone and then rinse the samples in isopropyl alcohol and DI H₂O.

NOTE: the procedures given here are for optical lithography using AZ 4110, which is a positive photoresist.

Procedure:

Spin on Photoresist

- Solvent clean your samples.
- Place cleaned chips on the aluminum tray, place the tray on the aluminum block in the furnace. Dehydration bake for 3 minutes at 120 °C.
- Place all chips but one under the HMDS vapor deposition hood for 1-3 minutes.
NOTE: For the reporting section, we will ask you to perform lithography (BUT NOT ETCHING) on one substrate without this step to understand the effect of HDMS on resist adhesion. (This chip will be cleaned without being etched, so that it will be good as new and can be used as the second substrate in subsequent steps.)
- Place a dummy chip or substrate on the spinner and set the spinner speed at 4000 RPM. This is to check if the spinner vacuum is on and other things are working as expected.
- Place the actual sample on the spinner and put drops of the resist onto the substrate until all of the chip is covered.
- Spin the chip at 4000 RPM.
- Pre-bake for 1 minute at 85 °C on the hot plate.

Exposure using MJB-3 mask aligner

1. Turn on <POWER>
2. Slide out mask holder and put the correct mask in with the chrome side facing the chip.
3. Put your chip on the chuck and slide tray under mask
4. Rotate lever to raise chuck. Make sure you actually are in <CONTACT>.
5. Put chip in <SEPERATION>and align mask to your pattern, (if needed)
6. Set exposure <TIMER> (UV=7.5 mW/cm²) recommended for resist in use (17-25 seconds). For AZ 4110, this time is 20 seconds.
7. Bring chip and mask in contact and press <EXPOSE>
8. Lower chuck and remove your chip.

Developing (AZ 400K)

1. Place the chip in the holder and immerse into a beaker containing 4:1 H₂O: AZ400K with mild agitation for 60 seconds.
2. Rinse in DI H₂O for 60 seconds.
3. Blow dry with N₂ gas.
4. Inspect for photoresist residue under the optical microscope and insure that the patterns have been transferred accurately.

Etching (50:1 HF)

1. **Read the instructions on working with HF and be very careful when working with it. DO NOT PERFORM ETCHING BEFORE APPROPRIATE TRAINING**
2. Immerse the chip in DI for 1 minute before you place it in HF. This prevents bubble formation and ensures uniform etching in HF.
3. Dip the chip in 50:1 H₂O:HF for the calculated time. (You have to etch down by 50 nm. Etch rate of 50:1 H₂O:HF is between 45-65 Å/min. at 21 deg C.)
4. Strip off the photoresist in acetone and then rinse the samples in isopropyl alcohol and DI water.

Caution:

- * Spinner will not start unless the vacuum chuck is turned ON.
- * Always **wear eye protection** in the lithography room, UV light will seriously damage your eyes.
- * Do not touch any parts of the MJB-3 Aligner with bare hands.
- * Do not use plastic tweezers while handling hot chips.
- * Do not throw any Developer down the drain. Put it in the container marked "Waste Developer".

Reporting:

1. Describe, in your own words, the procedure you used for lithography and etching. Write down all the steps you preformed, including the details of the procedure, including all the exact times that you preformed each step, temperatures of ovens, etc... Show schematics when appropriate.

2. Discuss HMDS application- is it required? Why? Draw conclusions by going through the process once without HMDS and comparing the developed photoresist profile in the two cases. Provide pictures if applicable.
3. Discuss photoresist application:
 - a. Did it work the first time? What were issues if so or if not?
 - b. How even was the coverage? Draw a schematic of the coverage, fringes, etc... of each substrate that was coated. Label each substrate so that you can refer to them in subsequent parts of your processing and lab reporting.
4. Discuss exposure/development
 - a. Why do we turn away from the mask aligner during exposure?
 - b. What is the purpose of the pre-bake? Discuss other baking steps commonly used in optical lithography.
 - c. Take pictures from different regions of the substrate after development and comment. Discuss each substrate that was processed, and the good parts and bad parts of each substrate. Discuss whether you had to re-do any substrates, or whether they were all ready for etching. Take as many pictures as possible of the substrates, and label them and discuss the difference between them.
5. Discuss Etching
 - a. Take a picture of the etched structures. Comment on the quality of the etch- what do you think about the surface roughness on the bottom of your channel? How can you improve the surface quality?
 - b. Accurately write down the measured depth of each substrate at 3 different places on the substrate. Measure this depth with the dektak, and provide a schematic of each processed substrate and the depths of each substrate at the various positions you measured. Be as accurate as humanly possible.
 - c. What is the etch rate of 50:1HF that you used? What reasons do you have for why it varied?
 - d. Comment on the quality of the channels – are the sidewalls straight and perfect? If not, why not? Was it in the lithography or etching step? How would you improve your channels? Do you feel it necessary to repeat the steps in this lab to ensure a better end device? (remember, the processing only gets harder and less accurate). If so, please do so, but make SURE you write down everything in the report as well.
6. Discuss alternate designs and fabrication methods for what you have accomplished so far. (e.g. using negative resist, using a different kind of lithography, etching, substrate, etc...) What is the most optimal technique? Why or why not?

STEP 3: KOH Etching

Overview:

In this step, you will use the patterned oxide as a mask for etching silicon using Potassium Hydroxide (KOH). KOH etches 100 Silicon anisotropically with an angle of 54.7 degrees (Figure 1) with a very high selectivity to SiO₂ (about 1000:1 at 20%, 80C) so we will use it to etch the silicon master for your PDMS microchannel device.

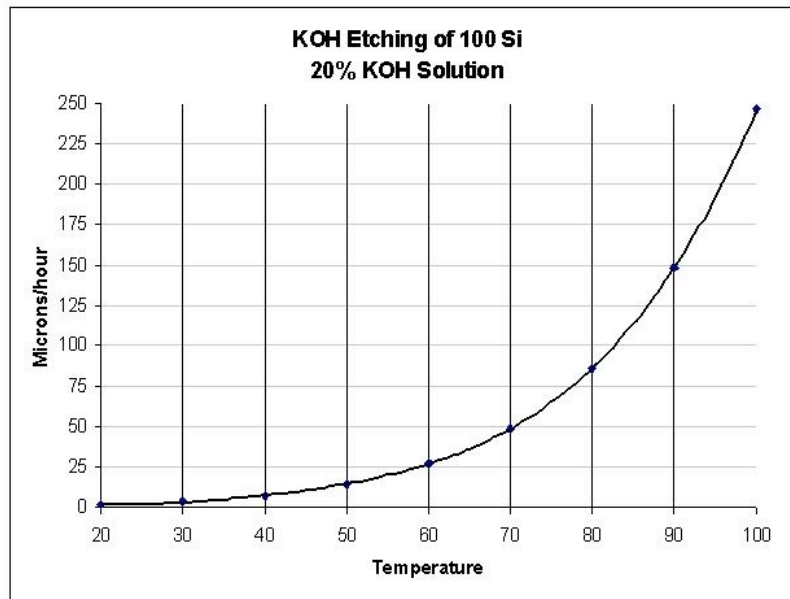


Figure 1: Beige is SiO₂ and Grey is <100> Si. (left) etched SiO₂ pattern on Si substrate before etching. (right) after etching, KOH etches anisotropically at a 54.7 degree angle.

Procedure:

KOH Etching

1. Prepare 300-400 mL, 20% KOH solution by diluting 45% KOH solution with DI water. Always pour base into water (slowly).
2. Place small stirring magnet into the 20% KOH solution and heat 80 degrees Celsius on the stirring hot plate.
3. Once at 80 degrees, place sample in Teflon boat then into the KOH solution and use Figure 2 to estimate the time required to etch the desired depth. If your chip is A, then you want to etch 1 micron. If your design is B, C, or D, your target depth is 10 microns.
4. Remove from KOH solution and rinse with DI water and clean up. Figure 2: 20% KOH etch rates for 100 Si as a function of temperature. Etch rate curves for different KOH concentrations can be found here <http://cleanroom.byu.edu/KOH.phtml>.



Questions:

1. Calculate the final shape of your silicon mold and sketch to scale. How much wider will the top of the channel become due to anisotropic etching?
2. Take a picture of the etched structures. Comment on the quality of the etch- what do you think about the surface roughness on the bottom of your wafer? How can you improve the surface quality?
3. Accurately write down the measured depth of each substrate at 3 different places on the substrate. Measure this depth with the dektak, and provide a schematic of each processed substrate and the depths of each substrate at the various positions you measured. Be as accurate as humanly possible.
4. What concentration of KOH did you use? What problems did you encounter while making the solution? What etch rate did you use? Did it yield accurate results?
5. Compare the calculated final shape to the actual final shape using your dektak measurements.
6. Comment on the quality of the wafer surface – are the sidewalls straight and perfect? If not, why not? Was it in the lithography or etching step? How would you improve your channels?