EXPERIMENT # 3: Oxidation and Etching

Experiment # 3: Oxidation of silicon - Oxide etching and Resist stripping
Measurement of oxide thickness using different methods
KOH etching, SEM characterization

The purpose of this experiment is to study the oxidation of silicon and to measure the resulting oxide thickness using the color chart, the ellipsometer, the Dektak profilometer and the Watson interference microscope. The second part of this experiment is aimed at the study the anisotropic etching of silicon. The etched structures are analyzed with the SEM which provides the necessary depth of field and spatial resolution to observe the details of the etched features.

Reading: Jaeger Sections 3.1, 3.2, 3.3, Section 3.9 & ellipsometer handout/interference microscope handout, Campbell 259-264. For an overview of crystal structures and crystal planes, see "Introduction to Solid State Physics", C. Kittel, 5th edition, Wiley & Sons (not required)

3.1 You are given a silicon (100) p-type wafer.
The wafer is a 2 inch wafer which is 11 mils (1 mil = 10⁻³ inches = 25.4 micron) thick, doped with boron, having a resistivity of 10⁻¹⁵ Ω cm.
The wafers are placed in the quartz oxidation boat and oxidized at 1050° C in a water vapor / oxygen atmosphere.
How long should the wafer be oxidized to form a 0.4 μm thick oxide?
What is the expected color of the wafer? (use the oxidation charts and the color chart)

3.2 After the oxidation write down the color of the wafer:
a) as seen under normal incidence (in a white light environment)
b) as seen under large angle
c) as seen under the microscope (lowest magnification, also turn down the light intensity to see the color)

On the basis of the color chart what is your estimate of the oxide thickness?

3.3 If experiment #2 was performed on an oxidized wafer skip to 3.4.
Pre-dry the wafer on a hotplate at 120C for 2min.
Spin positive resist on the wafer for 30 sec @ 6000 rpm.
Soft bake the resist @ 100° C for 90 sec on the hotplate.
Expose the resist using the S10 etch mask for 35 sec.
Develop in 3:1 AZ400 developer/DI water, rinse in the rinse tank and blow dry

Inspect your wafer under the microscope. Develop some more if necessary. Take a picture of a specific feature for future reference.
Check for dirt on the wafer, sharpness of the pattern, color of resist-on-oxide-on-silicon as opposed to resist-on-silicon.

3.4 Postbake @ 120º C for 90sec on the hotplate.
The postbaking hardens the resist, making it more resistant to the etchant.
It also improves resist adhesion, which prevents the etchant from penetrating between the resist and the oxide layer. Poor adhesion typically yields poor edge definition after etching.

3.5 Buffer Oxide Etching (BOE) of silicon dioxide.
**CAUTION: BOE is a dangerous chemical.**
Make sure you wear gloves as well as safety glasses.
Keep a safe distance between the beaker and your head.
Do not move the beakers.
Ask for assistance if your wafer slips out of the wafer holder.
Before starting, estimate the minimum etch time based on a nominal etch rate of 100nm/min for.
The etch rate of SiO$_2$ in BOE ranges from 10 to 100 nm/min at 25º C, depending on the density of the oxide and the concentration of the BOE.
Place the wafer in the plastic wafer holder and secure it in place with the sliding part.
Put the wafer in the beaker and observe (from a safe distance) the etch wetting the oxide layer at first and being repelled by the bare silicon wafer as the oxide is removed.
This can most easily be observed on the back of the wafer and in large area windows in the resist. Etch 10-20 sec longer to make sure that small features are also fully etched.
Write down the actual etch time.
Rinse the wafer in the DI water tank, starting with the right section, moving on to the middle section and finally the left-hand section.
Blow the wafer dry with the nitrogen gun.
This works best by blowing dry a section of the wafer while it is still in the wafer holder.
Then take out the wafer from the holder, while picking up the dry part wafer with tweezers.
Blow dry the front as well as the back of the wafer.
Inspect your wafer under the microscope to make sure that all the oxide is etched. At this point the resist edges look thicker and darker in part due to the rounding of the resist due to the postbake and the undercut of the etched oxide.
If some oxide remained, repeat the etch and rinse procedure. Get help if you are not sure.
Take a picture of a well-developed area; one after the BOE and another one after stripping the resist.
Look for dirt particles and other types of debris on the wafer.
Take a picture of one and identify what type of contamination it is and during what part of the process it landed on the wafer.
Also how would you prevent this from happening in the future.

3.6a Resist stripping using acetone/AZ400T. **When using the asher or RIE skip to 3.6c**
Place the wafer securely in the wafer holder.
Strip the photo resist by soaking the wafer for 3 min. in acetone/AZ400T resist.
Rinse the wafer in DI water for about 1 min and blow dry.
Check that all of the resist is removed from the wafer, if not repeat the process.
Inspect your wafer under the microscope for any resist residue.

3.6b Resist stripping using the plasma etcher (asher) or the March RIE system. **Skip to 3.7 when stripping using acetone/AZ400T.**

A plasma etcher can be used instead of the resist stripping liquid to remove the resist. The process uses an oxygen plasma generated in a vacuum chamber which reacts with the resist. Oxygen radicals and ions which are present in the plasma attack the organic material which consists primarily of carbon and hydrogen, yielding CO$_2$ and H$_2$O which are pumped away with the roughing pump. It can be looked upon as a controlled burning of the organic material hence the name ashing, eventhough no ashes remain.

Use the instruction sheet provided in the lab to operate the equipment.

Use gloves when loading your wafer to avoid contamination of the vacuum system.

3.7 Dektak measurement. Use the instructions on the website, also available at the instrument. **CAUTION: the Dektak is a sensitive piece of equipment.**

No force should be needed when operating this equipment. Ask for help if needed.

Position your wafer on the stage and rotate it under the Dektak stylus. Locate the A-A’ region on the wafer.

Identify some 50-100 $\mu$m size lines on your wafer so that you can run the stylus up and down the oxide and turn the wafer until these features appear as vertical lines on the screen. Lower the stylus until the reflection of the stylus touches the stylus which means that the stylus touches the wafer.

The stylus and its reflection should touch each other only right at the tip.

An increase/decrease in thickness reading on a flat surface indicates a tilt of the chuck which can be compensated by turning the larger bottom wheel.

Check the size of the feature you used and compare it with the mask lay-out in the handout.

Measure both the oxide thickness on your wafer and the oxide thickness on the wafer provided (note that this wafer has a different color).

3.8 Gold sputtering: This part is only required when measuring the oxide thickness with the interference microscope. This is only performed when time permits.

The interference microscope requires a highly reflective surface. To achieve this we coat half the wafer with a thin (20 nm) layer of gold. This is done in a gold sputtering system (a DC magnetron), masking half of the wafer with a microscope slide.

**USE THE FOLLOWING PROCEDURE:**

Lift the black top of the sputter system and place it face down on the wipe to the left.

If the top is stuck to the glass cylinder, separate it by rotating it rather than trying to break the two pieces apart using any other technique.

Then lift up the glass cylinder and place it against the metal brace on the right.

Place your wafer on the holder and cover half with a microscope slide.

Carefully put the glass cylinder back in place as well as the black top.

Start the sputtering system by turning on the main power.

Close the vent.

Briefly open and close the valve on the Argon bottle to fill the line.

Do not touch the needle valve which controls the sputtering rate.

As the pressure reduces below 100 Torr and stabilizes, turn on the high voltage.
A plasma should now be visible in the vacuum chamber (It helps to turn off the room lights to see the blue glow).
Sputter gold onto your wafer for 3 minutes.
The wall of the chamber should now be opaque.
To turn off the plasma, turn off the high voltage switch.
Turn off the main power to turn off the pump.
Then open the vent and wait until the pressure has reached the ambient pressure before trying to take out the wafer.
Take out the wafer using the same procedure as for loading the wafer.
Clean off the gold from the glass cylinder with a paper tissue.
Put the chamber together again but do not pump down.

3.9 Watson Interference microscope.
NOTE: the adjustment of the Watson interference microscope can at times be difficult, if not frustrating.
The procedure below should work independent of how the settings were left by the previous user. However once the instrument is grossly misadjusted, it becomes next to impossible to obtain the fringes and the whole procedure must be repeated. Get help if no fringes are obtained after two tries.
Measure the thickness of the oxide with the Watson interference microscope.
Place the wafer underneath the microscope and make sure it is level.
Adjust the focus so that you clearly see the features on your wafer.
Make sure you are looking at the part of the wafer which is gold coated.
Adjust the fine focus slowly and/or look around on your wafer until you find the interference fringes. (Note: this can sometimes be tricky, especially if you start turning the three set screws which control the mirror. If you do not find the fringes, adjust the horizontal position of the mirror using the left set screw until a "<" shaped feature located on the mirror surface comes into focus. Again adjust the fine focus on the microscope.)
Once you find the fringes adjust the set screw to the left to keep the fringes within view while refocussing and returning to the feature of interest.
Adjust the other two set screws so that the fringes are perpendicular to the oxide step of interest while spreading them out over the full field of view.
Write down the color sequence starting from the black line in the middle and check whether the color sequence is the same on each side of the black line.
Write down the approximate shift of the fringes when crossing the oxide step.
Use the green filter (555 nm) to get a more accurate measurement.
Take a picture which shows the pattern across a step, placing the pointer at one of the central black lines.
What is the thickness of the oxide?
What happens to the fringe pattern if the wafer is not coated with gold?
Carefully observe the color sequences and write them down.

3.10 Manual ellipsometer measurement. Use the automated ellipsometer if available.
CAUTION: The ellipsometer contains a laser. Do not look directly into the laserbeam.
Measure the oxide thickness on the wafer using the ellipsometer (see also separate handout). First use the color chart to estimate the wafer thickness.
Mark the corresponding point on the $\Psi$–$\Delta$ curve as well as on the $P_1$ versus $A_1$ chart. Put the wafer on the vacuum chuck and turn on the vacuum. Open the laser beam shutter and adjust the stage height so that the spot on the wafer coincides with the crosshairs (It helps to make the laser beam hit a dust particle on the wafer to more easily see the position of the beam. Make sure you move the laser beam away from the dust particle before starting the measurement procedure). **Note:** the vacuum will cause the wafer to bend in the vicinity of the vacuum suction holes, therefore avoid those areas.

Starting from the approximate values for $P_1$ and $A_1$, adjust the polarizer (left dial) and analyzer (right dial) until a minimal signal is detected. The first value for $P$ should be in the "red" range, i.e. the range of angles which are listed on the polarizer in red numbers. If a minimum is not readily found, start with both the analyzer and polarizer set to 45 degrees. Adjust the gain to the middle of the scale.

Read of the values for $P_1$ and $A_1$ and measure a second set of values, $P_2$ and $A_2$. Use the vernier to obtain the values accurate to 0.1 degree. The expected values for $P_2$ and $A_2$ are given by:

$$P_2 = 90 + P_1 \quad A_2 = 180 - A_1$$

Verify that your measurement is close to the predicted ellipsometer curve contained in the ellipsometer hand-out.

Write down the values for $P$, $A$, $\Psi$ and $\Delta$ corresponding to the measured thickness and the refractive index of the silicon dioxide.

Use the ellipsometer spreadsheet to find the corresponding values of the refractive index and the oxide thickness.

3.11 Remove any remaining oxide with a short BOE dip if the wafer was not stripped the same day. Etch the wafer in KOH at a temperature of approximately 80° C for 6 min. Agitate gently during the etching. The KOH mix we use does contain some alcohol to reduce surface tension. Write down the actual temperature and etch time.

If gold was sputtered onto the wafer as part of the interference microscope measurement, it will start to peel off and should be completely removed after 6 min. After the etch transport the wafer in a beaker with DI water to the cleanroom. Rinse the wafer in DI water and blow dry.

The etch rate of silicon (100) wafers in KOH is approximately 0.6 $\mu$m/min at 60° C and 8 $\mu$m/min at 100° C.

Measure the depth of the etch based on the 54.7 degree angle ($54.7 \degree = \arccos(1/\sqrt{3})$) between the (100) and (111) planes. Take a picture of the 24 and 12 mil squares, using the highest possible magnification. For each size, take a picture with the oxide layer in focus and one with the bottom of the etchpit in focus.
Determine the depth of the etch and calculate how much longer needs to be etched to etch 12 microns deep into the silicon.
Etch the silicon again.
Check the color of the oxide and compare it to the color before etching.
Measure the thickness of the oxide using the ellipsometer.

3.12 After etching check the depth of the etch (taking the appropriate pictures as evidence).
Check the etched pattern and draw carefully and to scale the crosssection between points A and A' and between B and B' as indicated on the mask layout in the handout.
Make sure you also draw the oxide layer.
Take a picture of both areas and attach to your report.

3.13 Find oxide bridges and cantilevers on your wafer.
Indicate their position on the mask layout provided in the handout and add it to your report.

3.14 Measure the flatness of the bottom of the etched regions with the Watson interference microscope.
Describe the features you see and their height.
Take a picture of the bottom surface with the interference fringes.
If applicable, compare the area which was initially covered with gold to the area which was not covered with gold. How do you explain the difference?

3.15 Break off a 1 cm² piece for inspection in the Scanning Electron Microscope (SEM).
Cleave one edge through the A-A’ section so that it can be used to determine the angle between the (100) and (111) plane.
To do this use a diamond scriber to scribe the front side of the wafer in a direction which is either parallel or perpendicular to the wafer flat.
Scribe a line on the wafer so that when cleaving along that direction, the cleave runs through an appropriate etch pit.
Do not scribe across, but rather adjacent to the etch pit of interest.
Place the sample vertically into the SEM sample holder, with the cleaved edge facing upwards. Follow the SEM instruction sheet available on the website or at the instrument.

3.16 Inspect your etched structure with the SEM and take pictures of the areas A-A' and B-B' indicated on the mask layout.
Look at the cleaved edge and measure the etch depth and the angle between the (100) and (111) surface.
Look for the overhanging bridge structures and draw a crosssection of the bridge, indicating the length and thickness of the overhanging oxide, the depth of the edge and the distance between the middle of the bridge and the silicon surface.

3.17 This experiment is only performed if there is enough time. Look at thin soap membranes under the microscope and take a picture of the thinnest (darkest) region of the membrane.
CAUTION: Keep a safe distance between the soap film and the objective lens; once you have a soap film on the lens you will get blurry images.
Form the membranes by dipping a perforated piece of metal (or some specially prepared silicon samples) into a soap solution.
Look at the soap film and observe how the features move and change color.
Wait for the structure to settle.
You now should have black areas as well as several bright colors.
If you observe primarily pink and green colors and do not get a good focus the soap film is too thick. Repeat the process and hold the metal vertical for a while to thin the membrane.
Write down the color sequence in order of increasing film thickness. How do these colors differ from those observed with the interference microscope?
Note that the colors vary in steps rather than continuously. How do you explain this?
Carefully count the number of steps between the darkest region and the first white region.
Find out the thickness of the thinnest region.
Also take a color picture.
Report questions:

a) Describe the different steps performed in the lab, adding observations and numeric results as requested in the handout. Attach photographs, printouts and Dektak traces where requested.

b) Using figure 4.8 in Chapter 4, determine the doping concentration of the wafer. What is the corresponding mobility?

c) What is the oxidation time to obtain a 0.4 μm thick oxide when oxidizing at 1100°C in a water vapor atmosphere? Calculate using the equations listed in Chapter 3. What is the expected color? Assuming the actual thickness to be 80% of the predicted value what is the oxide thickness and color? Compare both values and colors to the experimental value and color.

d) How long did you etch the oxide in BOE? Based on the best value of the actual oxide thickness, what is the etchrate? We will use this etch rate in the future to more accurately estimate the required etch time.

e) Paste a picture of dirt and/or debris onto your report. Indicate the approximate size as well as the possible cause of the defect.

f) How does the wafer look after stripping as compared to before? Can you see that the resist is removed? How? Are there any other observations?

g) Add the Dektak trace to your report. Indicate the width and height of the feature you measured. Also add a copy of the mask layout to your report indicating the feature you measured. Is the measured width consistent with the mask layout?

h) This part is only required if gold was sputtered on the wafer. Why do we coat the wafer with a 20 nm gold layer? Why would 5 nm not be sufficient for this purpose? Explain how the gold is transported from the target to the wafer. What gas is used to form the plasma?

i) This part is only required if gold was sputtered on the wafer. List the color sequence of the interference fringes as observed on the gold coated region. Carefully check whether the pattern is symmetric around the central dark fringe. Why is the central line dark, corresponding to destructive interference, as opposed to being bright due to constructive interference?

k) Write down the color sequence in a region where the oxide layer is not coated with gold. Carefully check whether the pattern is symmetric.

l) Provide the ellipsometer measurement. Attach the printout corresponding to your ellipsometer measurement. Indicate which thickness is the actual thickness. Do the values of A and P satisfy the relations in the handout? Check this by indicating the values A₁, P₁,
A2, and P2 on the attached chart. Is the value of the refractive index close to the expected value? Is the measured thickness consistent with the color chart?

m) List the colors and thicknesses for wafers 1 through 3. If the color chart is inconclusive use the ellipsometer (only if there is enough time - optional). Tilt the wafer to obtain the next color as additional information (this does not always work as the reflection coefficients depend on angle). Extra credit (required for the graduate students): list the color and thicknesses for the wafers labeled 4, 5 and 6.

n) Compare the different ways of measuring the oxide thickness based on accuracy, ease of measurement and destructive/non-destructive nature. What thickness measurement(s) require or benefit from a gold coating and why?

o) Extra credit (required for graduate students): using the values for the oxide thickness and refractive index obtained from the ellipsometer measurement calculate all the parameters described in the ellipsometer handout: r01, r12, φ1, φ2, rTM, rTE, Ψ, Δ, A1, A2, P1, P2. Use n2 = 3.875 - i 0.018 and compare A1, A2, P1, and P2 to the measured values and the values obtained with the ellipsometer spreadsheet.

p) What devices require anisotropic silicon etching? Refer to the corresponding page number in the book. (Chapter 9 and 10)
q) What was the actual temperature during KOH etching? How long was the total etch time?

r) Determine the etch depth based on the shape of the etch pits and a 54.7 degree angle between the (100) and (111) planes. Is the total depth close to 12 μm? What is the corresponding etch rate? Paste in the pictures you used to determine the depth, indicating the size of the squares.

s) Why would one want to reduce the surface tension of the KOH solution?

t) What was the color of the oxide before and after etching in KOH? What are the corresponding thicknesses? How do those thicknesses compare to those obtained with the ellipsometer. Based on the change in thickness find the etch rate of the SiO₂ and the etch selectivity between silicon and silicon dioxide.

u) Draw carefully and to scale the crosssection between point A-A' and between B-B'. Make sure you also draw the oxide. Attach a picture of each region.

v) Identify some oxide bridges and cantilever structures. Indicate their position on the attached mask layout.

w) Describe the appearance of the etched silicon surface. What is the height of the surface roughness as measured with the interference microscope? Does the gold (if gold was deposited) affect the surface roughness? If so, how do you explain this and how large is the effect?

x) Attach pictures of areas A-A' and B-B' to your report. Indicate the angle on your picture of the cleaved edge and measure that angle.

y) Draw a crosssection of an overhanging bridge structure indicating the length and thickness of the overhanging oxide, the depth of the edge and the distance between the middle of the bridge and the silicon surface.

z) Do the colors of a soapfilm correspond to those observed in the interference microscope? Why do the colors of the soap membrane vary in steps rather than varying continuously? How many steps did you count up to the first white region? What is the thickness corresponding to the first white region? What is the thickness of the thinnest region and how did you obtain this value? (this part is optional)
Etch mask indicating the position of the A-A’ and B-B’ locations