EXPERIMENT # 3: Oxidation and Etching
Week of 2/5/01 and 2/12/01

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

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.

Reading: Sections 3.1, 3.2, 3.3 (week 1), Section 3.9 & ellipsometer handout/interference microscope handout (week 2)

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 1100º 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 Spin positive resist on your wafer for 30 sec @ 6000 rpm (using the same procedure as for experiment #1).
   Prebake the resist @ 80º C for 10 min.
   Expose the resist using the S94 etch mask for 35 sec.
   Develop in 4:1 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 20 min.
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.

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.
- Write down the actual etch time.
- Rinse the wafer in the DI water tank, starting with the left section, moving on to the middle section and finally the right-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, applying the wafer tweezers to the dry part.
- Holding the wafer with the tweezers one can the 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.
- If some oxide remained, repeat the etch and rinse procedure.
- Get help if you are not sure.
- 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.

3.5a Resist stripping using 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 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.

3.5b Resist stripping using the plasma etcher (asher) or the March RIE system. **Skip to 3.7 when stripping using 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\textsubscript{2} and H\textsubscript{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.

**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 vacuum chuck and turn on the vacuum (If the gauge does not indicate a vacuum of 15-20 psi, turn on the switch on the north wall next to the sink).

Identify some 50-100 µm 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 horizontal lines when looking through the ocular.

Move the wafer up or down by using the manual speed control and left or right by turning the metallic blue wheel in front of the chuck.

Lower the stylus (which also changes the focus) 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.

A broad area contact between the two indicates that the stylus is damaged and should be replaced.

Further lower the stylus slowly until the pen on the chart recorder moves.

You'll find that the instrument is very sensitive to small variations especially when the smallest range (100Å) has been selected.

Use the 10K scale (1 µm full scale).

Scan a flat area and check that the recorder shows a straight line.

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

Turn the wheel so that the pen moves in the opposite direction.

Tear off the chart paper and keep it for your report.

Write down the range, the setting on the 1x/2x box, the Dektak speed and the chart recorder speed.

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)

**CAUTION: make sure that the stylus is raised when using the manual speed control, or when removing the wafer. Failure to do so could result in a broken stylus.**
3.8 Gold sputtering: 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 Ellipsometer measurement.

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:

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

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.
Report # 3  Due week of 2/17/01

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 1100ºC in a water vapor atmosphere? 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) Paste the Dektak trace in the space below or attach to the report. Indicate the width (obtained from the stylus speed and the width on the Dektak trace) 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) 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) 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?

j) Why do we use the green color filter to measure the oxide thickness? Why don't we use the color filter all the time, i.e. is there any additional information when using white light?

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. Explain why the colors are different compared to those on the gold coated region.
l) 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 $A_1$, $P_1$, $A_2$, and $P_2$ 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: $r_{01}$, $r_{12}$, $\phi_1$, $\phi_2$, $r_{TM}$, $r_{TE}$, $\Psi$, $\Delta$, $A_1$, $A_2$, $P_1$, $P_2$ Use $n_2 = 3.875 - i \times 0.018$ and compare $A_1$, $A_2$, $P_1$, and $P_2$ to the measured values and the values obtained with the ellipsometer spreadsheet.

Fig.A.7.7  $A_1$-$P_1$ and $A_2$-$P_2$ curves for silicon dioxide on silicon. Thickness increases counter clock wise from 0 (at the square marker on the left) for $A_1$ versus $P_1$ and counter clock wise from 0 (square marker on the right) for $A_2$ versus $P_2$, both in steps of 10 nm (black diamonds)