Furnace Oxidation Lab (60 points)

Objective: The objective of this online lab is to demonstrate and overview the procedures necessary to operate the Lindberg tube furnace.

The student will watch a series of videos pertaining to each step of the thermal oxidation process. These videos will provide detailed insight onto each step of the oxidation process. After watching the videos and analyzing the provided data the student will be required to answer review questions on ANGEL.

Background:

Furnace oxidation can be used to grow two types of oxides, dry and wet. Dry oxidation is a slow reaction between oxygen and silicon that creates a dense, high quality oxide layer that can be used as a dielectric gate material. The temperatures used for dry oxidation typically range from 800°C-1100°C. The typical thickness of a thermally grown dry oxide is in the range of 20-2500 Å. The reaction sequence below is associated with dry oxidation.

$$Si\ (s) + O_2\ (g) \leftrightarrow SiO_2\ (s)$$

Wet oxidation is a relatively fast reaction between oxygen, water vapor, and silicon that creates a less dense, fast growing oxide layer that can be used as a field oxide. These field oxide layers are typically employed as a diffusion barrier for a subsequent dopant diffusion process. Similar to dry oxidation, the temperatures used for wet oxidation typically range from 800°C-1100°C. However, due to the faster growing and lower quality oxide, the typical thickness of a thermally grown wet oxide is in the range of 2,500 Å-25,000 Å. The reaction sequence below is associated wet oxidation.

$$Si\ (s) + 2H_2O\ (g) \leftrightarrow SiO_2\ (s) + 2H_2\ (g)$$

The faster growth rate for wet oxidation can be justified by comparing the sizes of the two molecules. The smaller H_2O molecules will diffuse faster through the SiO_2 to the Si surface than the larger O_2 molecules will; this results in a faster deposition rate for the wet oxidation. The drawback to this is that the resultant film grown using wet oxidation will have an open SiOH structure, affording non-stoichiometric SiO_x. Therefore, when higher quality oxides, such as the gate oxide for a transistor, are required dry oxidation is the preferred method since it can be used to obtain stoichiometric SiO_2.

Experiment: During this exercise 23 two inch silicon wafers will be oxidized in a furnace using a dry oxidation procedure. The resultant oxide thickness for each wafer will then be gauged with the ellipsometer. Read through the following steps and watch the clips corresponding to each stage of the oxidation process in order to answer the lab questions on ANGEL.

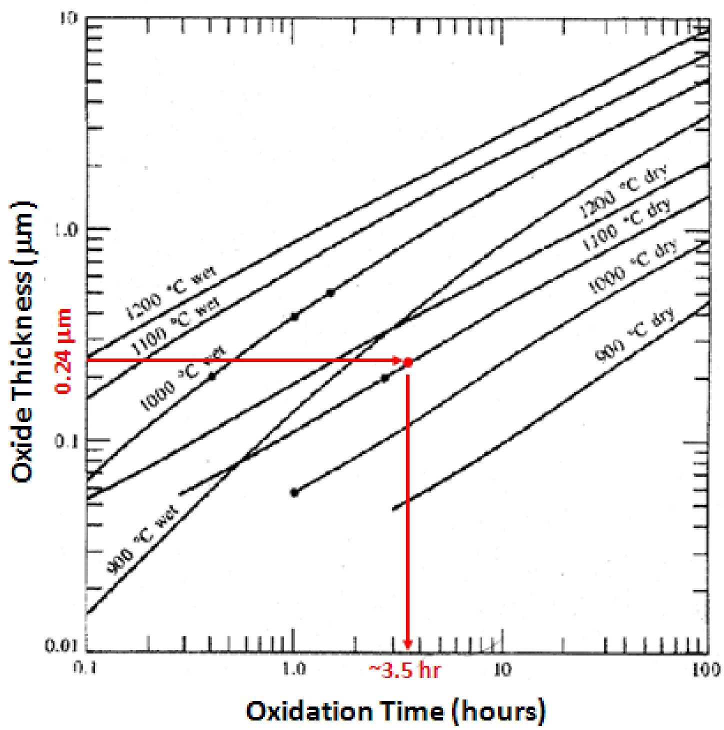
Step 1: Wafer Clean

In industry, it is necessary to conduct a rugged clean on all wafers in order to ensure a quality baseline. Many types of contamination can accumulate on the wafer's surface during the shipping and handling processes of the wafers. Therefore, a standard cleaning sequence may be used to remove any residual materials and native oxide on the wafers. This will afford a pristine and high quality crystalline surface to grow the oxide on. The table below shows a cleaning process that may be used prior to growing an oxide:

Cleaning Process for Wafers				
Step	**Solution**	**Time**	**Temp**	**Notes**
1	Acetone	10 min	40°C	Physical Removal of organics
2	Isopropyl Alcohol	10 min	40°C	Physical Removal of organics
3	SC1) H_2O:H_2O_2:NH_4OH	10 min	85°C	Chemical Removal of organics
4	SC2) H_2O:H_2O_2:HCl	10 min	85°C	Chemical Removal of metallics
5	Diluted HF in DI Water	10 sec	Room Temp	Remove Native Oxide
6	DI Water Rinse	10-30 sec	Room Temp	Removes dilute HF

Step 2: Design the Recipe

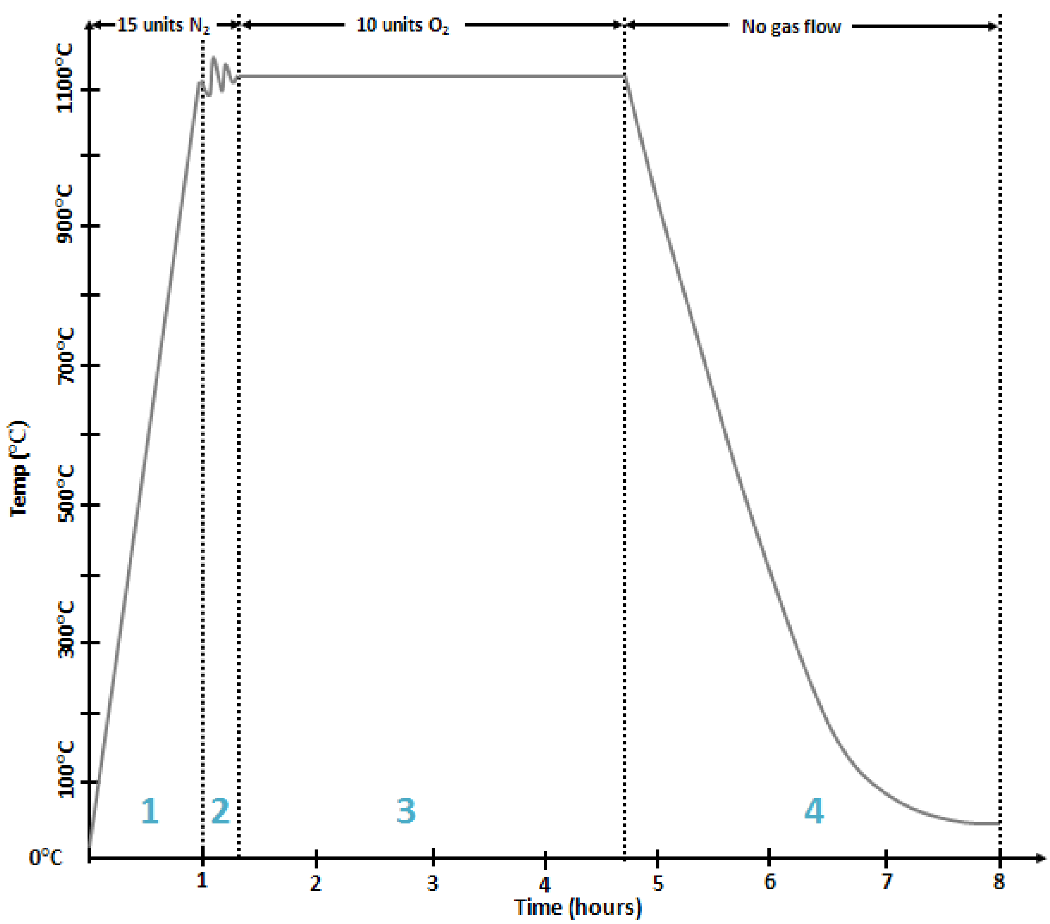
The thickness of a thermally grown oxide is dictated by the time, temperature, concentration, and energy used during the process. All of these variables are related to the thickness of the thermally grown oxide through Fick's Law. In this lab, our goal is to grow 2400 Å of SiO_2 using dry oxidation and a temperature of 1100°C. In order to determine the time necessary for this growth we consult the following Fick's law chart. The red lines on this chart indicate how our desired oxidation time for a 2400 Å film is derived based on dry oxide growth conducted at 1100°C.



From the Fick's law chart it is shown that the desired oxidation time is ~3.5 hours. Thus, the samples will all be exposed to oxygen at 1100°C for 3.5 hours. In addition to this step, it is also necessary to gradually warm the furnace up to this processing temperature. Then, following oxidation, it is necessary to gradually cool the furnace back down to room temperature in order to remove the samples. These steps are very important for minimizing the thermal stress of the wafers. Drastic temperature changes can cause a great deal of damage to the wafers including defect formation such as slip.

The oxidation process chart of time versus temperature throughout the entire process is shown in the following figure. Step 1 (teal) in this plot represents the 1 hour ramp up time from room temperature (25°C) to the desired processing pressure of 1100°C. Step 2 is a short 15 minute interval in which the temperature is allowed to stabilize at 1100°C. During steps 1 and 2 fifteen units of nitrogen gas is flown through the furnace tube.

After stabilizing the temperature at 1100°C the nitrogen gas is turned off and ten units of oxygen are then flown through the system for the next 3.5 hours to perform the thermal oxidation. During step 3 the oxide grows in accordance to the previously illustrated Fick's law chart. After the oxidation time has elapsed the oxygen gas is turned off and the system is cooled back down to room temperature through natural radiation convection. This takes several hours due to the exponential nature of the cool down.



Step 3: Load the samples

Each of the 23 two inch silicon wafers to be oxidized are loaded into a quartz boat with the primary flat facing down. This quartz boat is then loaded into the furnace. Watch the complete sample loading process at the link below:

http://www.engr.psu.edu/mediaportal/flvplayer.aspx?FileID=53003f7e-551b-4869-bef9-f

Step 4: Program and Run the recipe

After the user designs the recipe (Step 2) and determines the time necessary for their oxidation conditions they must program the desired operating temperature(s) and corresponding hold time(s) into the furnace. When operating the furnace it is the operator's job to open up and close the gas valves at the appropriate times throughout the process. Thus, it is imperative that the operator pays special attention to the precise times that they begin to run the recipe and when they are required to open and close the gas valves. Watch the recipe being programmed and ran at the link below:

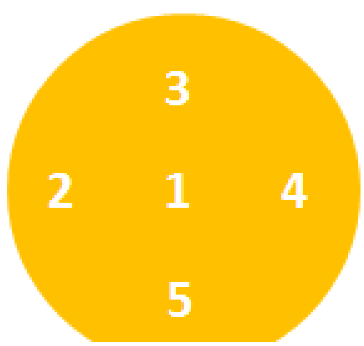
http://www.engr.psu.edu/mediaportal/flvplayer.aspx?FileID=e7f0722a-f00c-4385-896a-9

Step 5: Remove the Samples

Once the oxidation process is complete and the samples have cooled to room temperature they are removed from the system using the same quartz rod that was used to center them during the loading procedure. Special care should be taken to keep track of the samples' locations during the unloading procedure in order to properly characterize and gauge the uniformity of the wafer thicknesses across the quartz boat.

Step 6: Characterize samples with the Ellipsometer

Next, each of the 24 oxidized samples was characterized using a 5 point ellipsometer measurement. The locations of each of the 5 measurements are shown in the figure below. Recall that the primary flat was loaded down (facing the bottom of the quartz boat) for all 23 wafers.



For a recap on the operation of the ellipsometer feel free to review the link below:

http://www.engr.psu.edu/mediaportal/flvplayer.aspx?FileID=409bbc03-1c3a-4107-be2e-5

The following table shows the results of this characterization:

Wafer	Position					
	1	2	3	4	5	Avg.
1	2092.4	2194.2	2106.4	2220.3	2291.5	2181.0
2	2263.7	2355.9	2281.7	2294.0	2374.5	2314.0
3	2373.4	2398.0	2348.6	2412.2	2428.8	2392.2
4	2455.5	2501.7	2419.1	2526.8	2546.7	2490.0
5	2465.2	2503.7	2413.7	2512.1	2500.1	2479.0
6	2448.3	2536.1	2429.4	2462.6	2526.0	2480.5
7	2455.2	2478.2	2414.4	2471.0	2514.2	2466.6
8	2489.6	2600.5	2420.7	2484.6	2547.5	2508.6
9	2535.8	2633.0	2472.6	2512.6	2570.3	2544.8
10	2503.5	2609.3	2451.3	2479.4	2558.1	2520.3
11	2462.5	2525.8	2435.4	2450.8	2503.2	2475.5
12	2458.4	2512.7	2402.0	2515.7	2500.9	2477.9
13	2440.2	2466.9	2390.7	2462.6	2485.7	2449.2
14	2423.7	2493.3	2381.7	2410.0	2488.4	2439.4
15	2480.2	2501.8	2389.1	2445.1	2709.2	2505.1
16	2431.5	2548.8	2388.3	2419.0	2489.7	2455.5
17	2410.2	2431.1	2347.0	2426.9	2460.9	2415.2
18	2406.9	2434.1	2344.0	2400.9	2440.8	2405.4
19	2379.4	2390.7	2317.2	2398.8	2419.4	2381.1
20	2348.0	2400.3	2300.9	2379.1	2398.4	2365.4
21	2285.2	2325.0	2246.7	2346.1	2355.3	2311.7
22	2189.6	2293.9	2175.6	2213.9	2309.4	2236.5
23	2026.3	2038.5	1984.8	2055.8	2210.3	2063.1

It should be noted that wafer #1 was loaded at the far end of the boat (closest to the incoming gas) and wafer #23 was loaded at the opposite end of the boat (furthest from the gas source). The entire boat was loaded within the furnace tube as close to center as possible. Recall that the goal is for all 23 wafers to have an oxide with a thickness of about 2400 Å. Students will need to manipulate the data above in answering some of the ANGEL review questions.

An oxide color chart is a common way to qualitatively gauge the thickness of a thermally grown oxide. This chart is shown in the following figure:

Color Chart for Thermally Grown SiO_2

Color Chart Table for thermally grown silicon dioxide films observed perpendicularly under daylight fluorescent lighting. Copyright 1964 by International Business Machines Corporation; reprinted from Volume V INTRODUCTION TO MICROELECTRONIC FABRICATION

Film Thickness (µm)	Color and Comments
0.05	Tan
0.07	Brown
0.10	Dark violet to red violet
0.12	Royal Blue
0.15	Light blue to metallic blue
0.17	Metallic to very light yellow green
0.20	Light gold to yellow; slightly metallic
0.22	Gold with slight yellow orange
0.25	Orange to melon
0.27	Red violet
0.30	Blue to violet blue
0.31	Blue
0.32	Blue to blue green
0.34	Light green
0.35	Green to yellow green
0.36	Yellow green
0.37	Green yellow
0.39	Yellow
0.41	Light orange
0.42	Carnation pink
0.44	Violet red
0.46	Red violet
0.47	Violet
0.48	Blue violet
0.49	Blue
0.50	Blue green
0.52	Green (broad)
0.54	Yellow green
0.56	Green yellow
0.57	Yellow to "yellowish" (not yellow but is in the position where yellow is to be expected; at times appears to be light creamy gray or metallic)
0.58	Light orange or yellow to pink borderline
0.60	Carnation pink
0.63	Violet red
0.68	Bluish" (not blue but borderline between violet and blue green; appears more

Students should be familiar with using this oxide color chart, the Fick's law chart, and the oxidation process chart prior to answering the lab questions on ANGEL.

Questions to be answered on ANGEL (NO HARD COPY REQUIRED)

1. What is the typical temperature range for thermal oxidation? (2)

2. Compare the quality of the films and the deposition rates for wet and dry oxides: (3)

3. What type of oxidation should be used to grow the field and gate oxide? (2)

4. Why is the diluted HF etch performed prior to growing the oxide on the wafers? (2)

5. What was the average ramp rate to the desired furnace oxidation temperature? (3)

6. What gas was flowed during the warm up and stabilization steps? How long in total was this gas flown for? (3)

7. How many units of nitrogen and oxygen were used during the warm up and oxidation processes respectively? (3)

8. How was the oxidation time determined? (2)

9. What was the average ramp down rate of the furnace from 1100C to room temperature? Is this rate linear? (3)

10. Using the oxide color chart predict the color of the wafers grown in this lab. (2)

11. Using the oxide color chart predict the thickness of an oxide that is dark to red violet (3)

12. Using the Fick's Law chart estimate the time to grow a 1200 Å (royal blue) oxide layer using dry oxidation at 1000ºC. (3)

13. Using the Fick's Law chart estimate the time to grow a 4400 Å (carnation pink) oxide layer using wet oxidation at 1100ºC. (3)

14. What percentage of the oxide thickness will lie above the original surface? (2)

15. If 200 Å of silicon is consumed during an oxidation process, what will be the total thickness of the thermally grown oxide? (4)

16. In this lab, roughly how deep into the silicon wafer did our thermally grown oxide penetrate in position 1 on wafer 22? (4)

17. In this lab, roughly how much of our thermally grown oxide grew above the original surface in position 4 on wafer 2? (4)

18. How did the average thickness across each of the 23 wafers vary as a function of the wafer's positioning within the quartz boat? (4)

19. How did the thickness vary as a function of position on each of the 23 wafers? (Hint: average the thickness for each of the 5 positions) (4)

20. How much did the overall average oxide thickness vary from the target value of 0.24 um? (4)