

# Group 4 Lab Report

## Metals/Semiconductors/Oxides



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## Introduction

This is the lab report for group 4. This provides information on our experiences during our first seven weeks in the lab. It provides a summary of each lab, along with graphs and relevant measured data.

We also provide a flow chart at the end which outlines the steps necessary to create and test our final “product.” For this first lab report our final product is a TLM patterned metal on a semiconductor.

## Week 2: Intro to Clean Room

This lab was our first experience in the clean room. We learned about the classification of clean rooms. A class 10 clean room means that the particle count is less than 10 particles per cubic foot (measuring particles .5um and larger).

We measured the particles counts of our room and also the hallway. Inside the clean room were 3 particles. In the hall there were 73,000. Our clean room would be considered a Class 10 clean room.

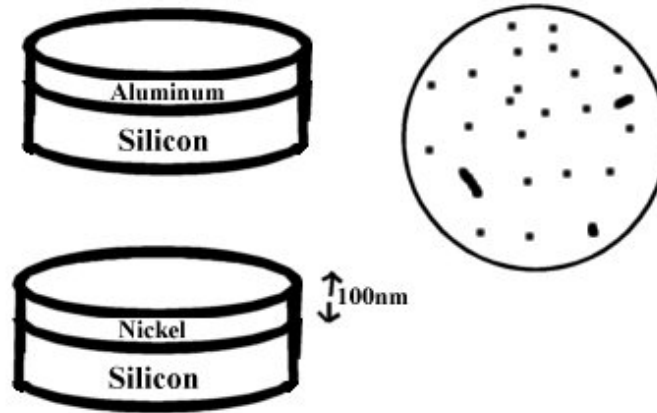
Next, we used the E-beam evaporator to evaporate 100nm of Nickel onto 4 inch silicon wafers. The wafer was placed on an oil diffusion bell with an ionization level of  $5.5 \times 10^{-5}$ . In this pump hot oil vapor is jetted toward the walls of the bell capturing air molecules, and dragging them to the bottom as the oil vapor sinks. At the bottom it is pumped out mechanically. After the oil is pumped out, an electron beam vaporized the Nickel and caused it to deposit onto our wafer.

After doing Nickel Evaporation, we performed Aluminum Evaporation. Aluminum vaporization takes place in the cryo filter. In a cryo liquid helium is used to freeze the air and particles in it. This forms an ice ball, which is melted and pumped out the bottom. The aluminum is placed on the mount. The mount heats up, and the aluminum heats and vaporizes. During depressurization the aluminum cools and coats the wafer.

Finally we measured the resistivity of the wafers using a 4 point probe.

Wafer Type	Resistivity
Silicon wafer	.046 Ohms
Al coated wafer	.06 Ohms
Ni coated wafer	.7 Ohms

Finally we examined the features of metal deposited on the surface it had a grainy semi-pitted look. The figure below illustrates how the nickel, aluminum is deposited and also how the wafers looked under a microscope.



### Week 3: Lithography

Photoresists have three basic components a photosensitive compound, a base resin, and an organic solvent. In positive photoresists the pattern absorbs radiation and becomes soluble. After development the exposed areas are removed. Patterns formed in positive resist will appear the same as the mask. The opposite is true for negative photoresist.

Since the banding of light around the mask pattern places a limit on the resolution of the mask, UV rays are used in photolithography since the angle of refraction is less with waves with a shorter wave length.

The dose is the amount of time that UV light is shine through the mask. Varying the dose affects the thickness of the photoresist. Overexposure results in rounding of edges and decrease in feature size. If we underexpose the pattern might not be transferred at all.

We chose  $155\text{mJ}/\text{cm}^2$  as the exposure latitude. So, since  $1\text{J} = 1\text{Ws}$ . The time in seconds for exposure is simply  $155\text{mJ}/\text{cm}^2$  divided by  $3.0\text{mW}/\text{cm}$  (Lithography machine). This gives the result of 52 seconds. We found upon experimentation this was too large and caused overexposure.

At the longer time there were rounded corners and features were smaller. We tried Lithography doses at 30, 40, 50 seconds. Both 30 and 40 adequately represented the features. 50 seconds caused overexposure. The figure below illustrates the appearance of the text on the wafer under a microscope.



During the Lithography process we settled on a recipe for applying photoresist:

- Dehydrate bake the wafer for 10min at 110°C
- Place wafer on spinner
- Drop 2 or 3 drops HMDS (adhesive) onto wafer and spin at 5000 RPM
- Apply 30-40 drops of AZ3330 (positive resist) and spin at 5000 RPM for 30 seconds
- Soft bake wafers on hotplate (90°C) for 60 seconds.

We also learned how to clean the wafers if the photoresist was not evenly coated:

- Place wafer on spinner at solvent bench, and start it spinning
- Squirt Acetone onto the wafer
- Squirt Isoproponal onto the wafer
- Rinse the wafers with water
- Blow dry with Nitrogen gas, and restart the photoresist process with dehydration bake

To get the pattern onto the wafer:

- Place the resist coated wafer into the mask aligner
- Expose for 30 seconds
- Develop in chemical developer for 35-60 seconds

## **Week 4: Wet Etching**

This lab focused on the relationship between temperature and etch rate and how different metals react to etching. It also taught that after metal has been deposited on to a semiconductor, an annealing step is done. This helps increase the adhesion of the metal to the semiconductor.

As the temperature increases, the etching rate also increases. For instance, at 300°C it pits to .2  $\mu\text{m}$  and at 450°C it pits to 2  $\mu\text{m}$ . It is also important to get the photoresist mask to adhere to the layer being etched because if it is loose the etching will occur beneath it where the semiconductor is supposed to stay. With the mask firmly attached, there will be some "bleeding" but not enough we really have to worry about. To get an optimal anneal, it is best to set the furnace to 450°C and leave the wafer in for 5 minutes. If we were to anneal for a longer time and at a higher temperature this would cause pits to form and shorts or other problems that would make the resistance higher than what other methods would produce would occur.

Using the methods found from week 3, we used photolithography to transfer a pattern of lines onto our two different wafers, one with aluminum and one with nickel. In order to now put this pattern onto our wafer we etched them separately and we found for aluminum it is best to etch at 50°C for 4 minutes and for nickel at room temperature for 2.5 minutes. However, as we learned in a later lab, we actually over-etched our wafers, especially the aluminum-coated wafer. We think we did not have enough aluminum on the wafer to begin with.

To make sure the metal is sticking to the wafer below it a "Scotch tape test" can be used. We simply took a small piece of Scotch tape and stuck it on our wafer. Since nothing came off with the tape when we removed it we knew we were in good shape. Then, we broke our wafers

apart into 6 samples by making a good nick and then carefully hitting the back. This effectively cleaves the wafer. These different samples now allow us to test different annealing conditions and find which works best. This was a good idea but due to our over-etching the aluminum sample we were unable to see anything on it after each test. Our nickel sample also had problems as apparently we did not do a good job getting all the photoresist off. This cause some pretty severe burn marks on our wafers. Obviously these marks became less noticeable as the temperature decreased. We decided to stick with the data the book provided and agreed that the best annealing conditions are 450°C for 5 minutes.

This was a good lab in that we were able to alter our silicon wafers and learn how to properly etch patterns into the metal. It was a little long; however, as we had to wait for the furnace to cool down since the group in front of us had already heated it up. This lab consisted of a lot of sitting around talking about sports, which I guess is not too bad! But once we got our tests done it was neat seeing how the wafers had changed throughout the time we had spent in the lab that day.

## Week 5: Contact Resistance

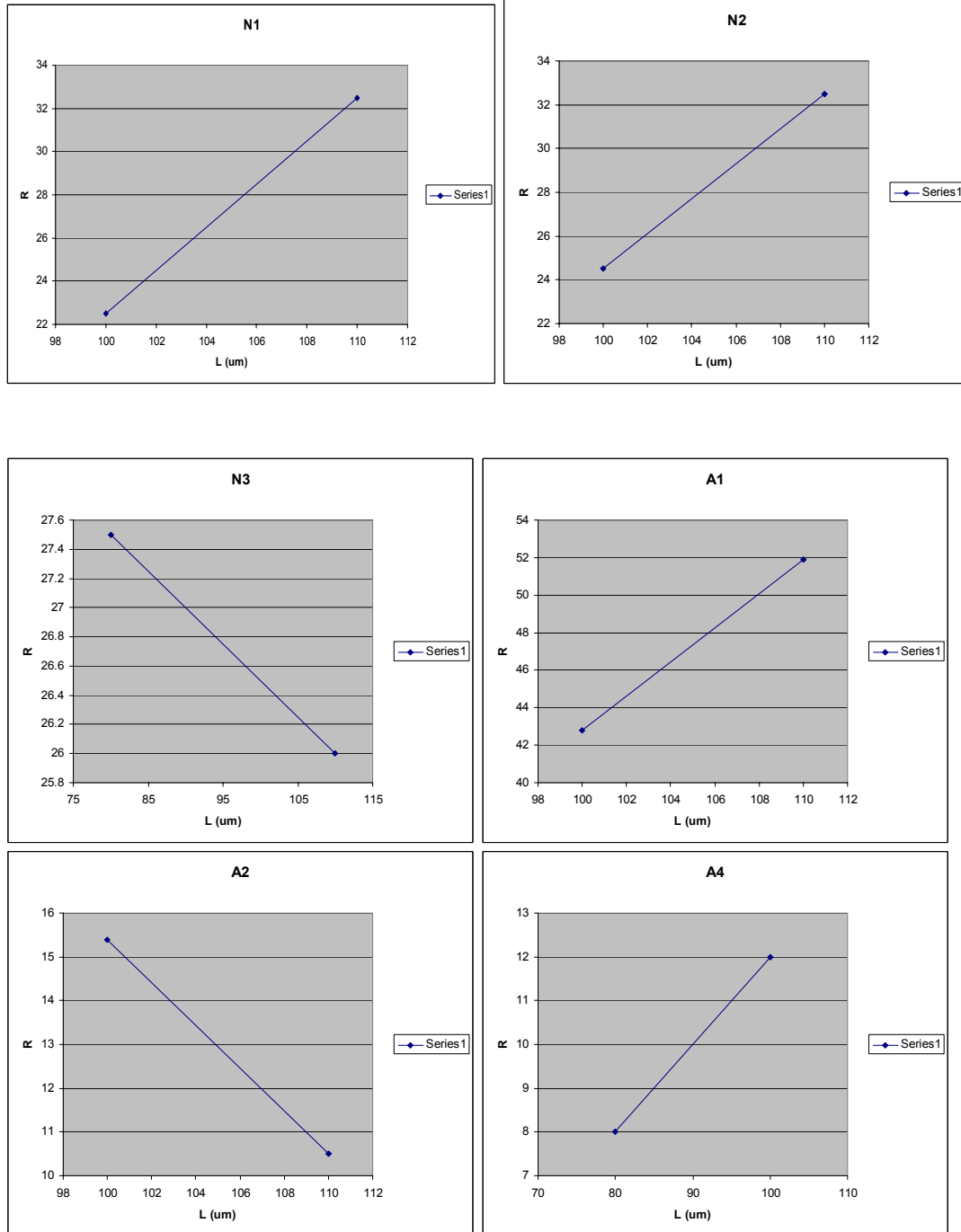
This lab will focus on learning how to use different machines to measure the resistance between two different pads. As the distance between pads increases, the resistance also increases, which makes intuitive sense. To find the resistance, the probe station applies a voltage between two probes and then measures the current flow. The display then shows a specific V-I curve.

From last week's lab we have different sized pads on our wafers. To measure the resistance, it is best to find the largest pads and use those. But unfortunately, this was nearly impossible as our samples were very poor. Most of our data comes from small pads, as seen in the table below. The table includes as much information as we could find. The first aluminum readings we forgot to get the voltage and current.

	Size	Spacing	V	I	R	Spacing	V	R
N1	100x100	110 $\mu$ m	3.25V	100mA	32.5 $\Omega$	100 $\mu$ m	2.25V	22.5 $\Omega$
N2	100x100	110	3.25	100	32.5	100	2.45	24.5
N3	100x100	80	2.75	100	27.5	110	2.6	26
N4	200x100	110	4.6	100	46			
A1	400x400	110			51.9	100		42.8
A2	1200x400	110			10.5	100		15.4
A4	400x200	80	50mV	6.1	8	100	100mV	12
A6	200x200	110	1.5	100	15	100	1.35	13.5

Table 1

To summarize this data, here are a few sample graphs to illustrate the resistance versus pad spacing.



Ideally, the measurements would have been independent of pad size but due to problems with the equipment ours were not. The best annealing conditions were found to agree with our results from the previous week and they are 450°C for 5 minutes. After we took all these measurements we etched the annealed metal and with nickel etch it was very hard to get clean and pitting was evident. However, with aluminum etch, cleaning was incredibly easy and there was no pitting. Most of this lab was done with the group in front of us since our samples from the previous week were pretty pathetic. This resulted in a lot of standing around and not doing anything but it still was good to see how to measure resistance on such a small scale.

## Week 6: Oxide Growth

In this lab we grew an oxide layer to use as the foundation for the dielectric picture in week 7. Proper oxidation growth will also be critical for our MOSFET in future labs. We determined in the pre-lab questions that it would grow about 250 Angstroms in ten minutes of dry oxidation, and it would take about 25 minutes to complete 5200 Angstroms.

1. We began by growing a thin oxide growth (targeted at 1000 Angstroms) on a spare wafer. First we stripped all of our wafers in Buffered HF, and then baked the spare wafer for 45 minutes at 1100 degrees Celsius to make sure we had enough oxidation growth. Using the Nanospec we measured 1012 Angstroms of oxide. The less accurate color chart showed 1250 Angstroms. We learned through this that we needed to overshoot our calculated oxide growth times in order to get a thick enough oxide growth.

2. Next we took our 4 (100) wafers for MOSFET fabrication and 2 (111) wafers for color picture fabrication, and grew a thick oxide using conditions calculated previously. Again we overshot our calculations and baked the wafers for 60 minutes. Our calculated times were probably low because of the inaccuracies of reading the charts from the text book.

3. After pulling them from the oven we saw that we had grown the oxide unevenly because the wafers were only half green. This can be corrected by dipping the over-oxidized part in Buffered HF, but the Phil Brown said he would oxidize new wafers for us for the final week.

4. Finally we measured the oxide thickness using three methods: The color charts, the ellipsometer, and the Nanospec:

- The color charts are a quick but less accurate method used to visually match the wafer's color to a chart located in the lab. We had three colors on our wafers as mentioned about that ranged from 5800-6300 Å. The chart below shows a midrange value.
- The ellipsometer, as explained in the lab notes, uses light from a laser and interference from reflection at a surface, and by measuring the change in polarization of the reflected laser light very precisely measures the thickness of a transparent film. We had trouble using the ellipsometer accurately because even the TA didn't really know how to use it. After 30 minutes of calculations we finally came up with two relative values from the table based on our measurements.
- The third method is the nanospec. The nanospec measures the reflected spectrum from the oxide's surface by illuminating it with a range of wavelengths. We found the nanospec to be the easiest, quickest, and for us, most accurate measuring device. We were able to get a nice range of values and then average the good ones to produce an average oxide thickness for the entire wafer.

Oxide Thickness Measurements (averaged)			
	Color Charts	Ellipsometer	Nanospec
(100)	6000 Å	5774 Å	5921 Å
(110)	6000 Å	5664 Å	5815 Å

## Week 7: Photolithography

This lab was the culmination of all our efforts in the IML this semester. The idea of this lab was to create a color picture using the different colors of silicon dioxide. We used the following image from Finding Nemo, which had a red mask a green mask and a blue mask already made by the lab TA's.

We began by using 2 silicon wafers with a 5200 angstrom thick oxide layer. The basic procedure was:

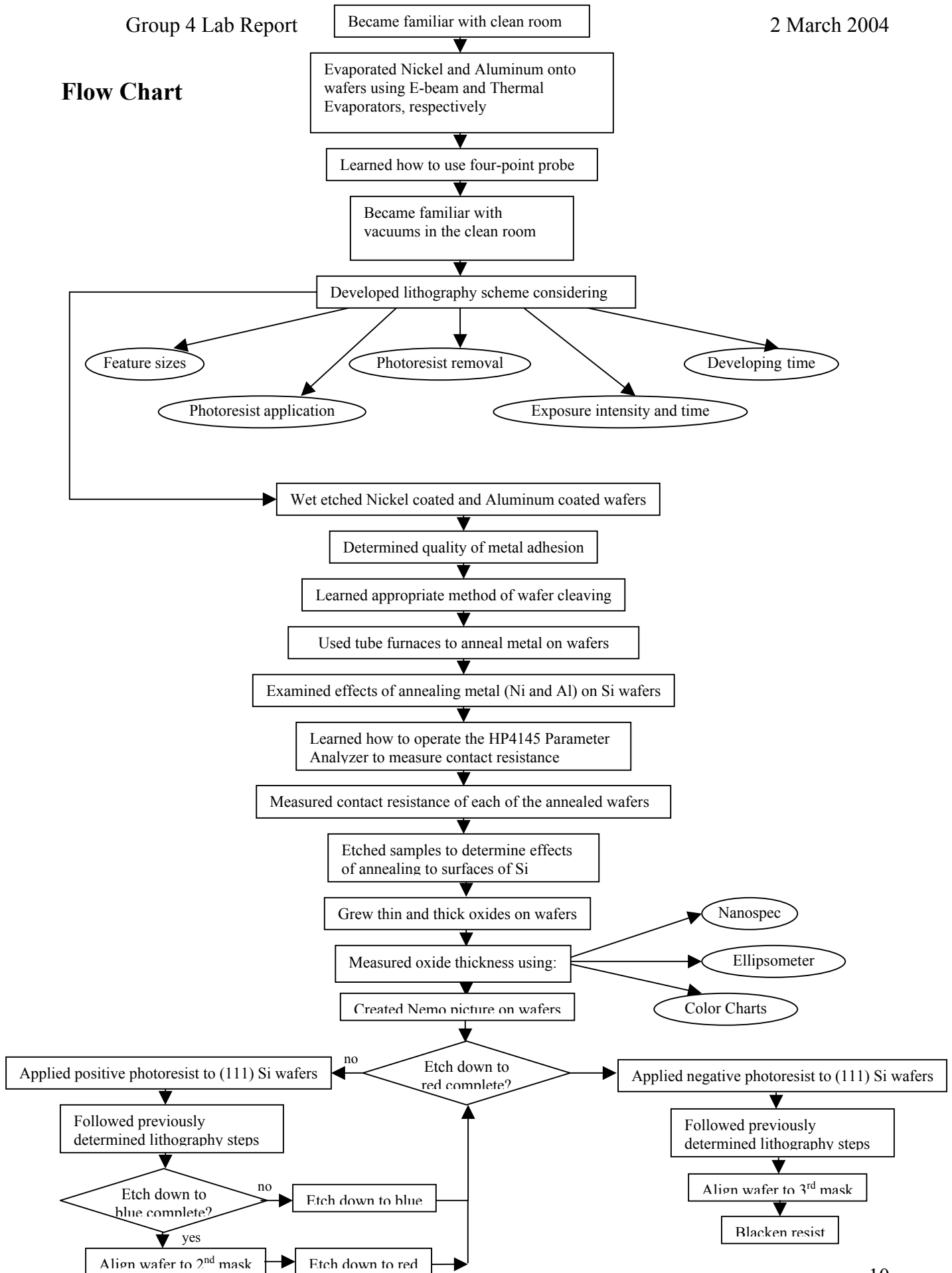
1. Apply positive photoresist and expose to the green mask.
2. Etch the oxide left uncovered by the photoresist until it appeared blue.
3. Apply a new layer of positive photoresist and expose to the blue mask.
4. Etch the oxide left uncovered by the photoresist until it appeared red.
5. Apply a final layer of negative photoresist and expose it to the red mask
6. Bake the remaining resist until it appears black.

Although the procedure appears relatively straightforward, there are several places where things can be messed up. Unfortunately our first wafer was accidentally masked with negative photoresist the first time because the bottle was improperly marked. We also had problems with alignment on the second wafer, and in the end both pictures appeared mostly red.

Despite all this we did manage to improve our procedure down for applying photoresist, using lithography, and etching silicon dioxide, which was the point of the lab. In reality oxide can't produce good RGB colors, i.e. the colors are dull and not perfectly red blue or green. We also learned the importance of properly labeled bottles!



**Flow Chart**



## Conclusion

These first seven weeks in the clean room have been very good for us as we have become accustomed to the clean room and much more comfortable working with some of the equipment. At first it was quite intimidating to go in there as the first week was pretty scary with the video showing all the explosions and the horror stories of people dying from touching hydrofluoric acid. But luckily we are not really scared anymore; we simply know the things we need to respect. This confidence has helped us in getting more out of the labs each week.

Besides from becoming more confident in our abilities, we have been able to actually take pure silicon wafers and do useful things with them in preparation for the second half of the semester, namely building a MOSFET. By learning lithography, etching, annealing, oxide growth, measuring contact resistance, and photolithography we now have many of the skills needed to do productive things in the IML laboratory. These skills will not only help us finish the semester but in our careers as professional engineers.