

# ELECTROHYDRODYNAMIC MICROPUMP

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**Abstract**—This report details the process for fabrication of a electrohydrodynamic micropump which was developed through 6 weeks.

## I. INTRODUCTION

An Electrohydrodynamic Micropump is microsystem device capable of displacing a liquid through an electromagnetic field without any moving parts, taking advantage and enhancing the capillarity properties. [1]

An Electrohydrodynamic Micropump is the implementation of robust micropump technology with no moving parts. The fabrication of a EHD micropump is a relatively simple process which is ideal to illustrate microfabrication process, since only one mask is needed. The process used in the fabrication of the EHD micropump utilizes the fundamental steps that are widely used in the machining industry applications such as semiconductors (Diode, MOS, BJT, etc.) and MEMS (accelerometers pressure sensors, etc.) which techniques are fundamental to the fabrication of integrated circuits such as microprocessors and RAM memory.

This report goal is to reflect the work done in the Fabrication Lab of Northeastern University for the Micro and Nano Fabrication Class during the Spring 2014 term. This report is based in the Laboratory Experiments and Procedures Manual and the work done throughout Friday morning sessions.

The EHD micropump was build on a p-type, boron doped silicon wafer  $< 100 > \pm 5^\circ$ , single face polished with a  $76.2 \pm 0.5\text{mm}$  diameter (Part N°. 46, Lot N° 11/1024)

## II. OXIDATION

The oxidation process consist of growing a silicon dioxide layer above the silicon wafer. This layer of oxide has many desired properties that make silicon the preferred material for the fabrication of IC. Some of the silicon dioxide properties are: being an excellent electrical insulator (with resistivity higher than  $1 \times 10^{20}\Omega\text{cm}$ ); high breakdown electric field; The interface between the silicon and the silicon dioxide is highly stable and easy to reproduce; The silicon dioxide is a great diffusion mask

for dopants and there exists a good etching selectivity between silicon and silicon dioxide.

When we started the our first lab session, we did not mark our wafers as directed by the manual, since by scratching the silicon we would released some silicon particles from the wafers into the lab, hence being a risk of contaminating the clean room.

### A. Pre-diffusion clean

Before we start the oxidation process it is critical to be absolutely certain that there are no undesired particles on the surface of the wafer. Since we are not sure about the conditions in which the wafer has been packed, moved and store previous to the moment of the oxidation. We have to assume that there is a thin layer of contaminants on the surface of the wafer which will diffuse when the wafers are brought to the temperature needed for oxidation, and causing undesired behavior in the final product.

Throughout the pre-diffusion clean process we followed closely the recipe from the manual with a few exceptions:

1) *Piranha etch*: removes organics and some materials. During our process we did not heat the solution, because we fabricated the piranha solution at that moment. The solution reaction release enough energy to heat our process if the peroxide is added to the acid.

2) *Cascade Rinse*: Until resistivity  $> 1\Omega$

3) *50:1 HF solution*: removes the native thin film of oxide

4) *Cascade Rinse*: Until resistivity  $> 1\Omega$

5) *H<sub>2</sub>O:HCl:H<sub>2</sub>O<sub>2</sub>*: removes remaining metal impurities.

6) *Cascade Rinse*: Until resistivity  $> 1\Omega$

7) *Rinse dryer*: Make sure that the wafer was clean before putting it into the dryer. 7.5 min dryer follow by a 150 sec rinse follow by a 300 sec dryer. When taking the wafer out of the machine we most check that the wafer completely dried.

After the pre-diffusion cleaning process. It is important to make sure that the wafers are completely dry and should be placed into the diffusion boat. It is important to keep in mind that this is going to be our first process. Any contaminate on top of the wafer will be diffused

into the silicon during the oxidation process. Water that is left to dry on the wafer and touching the wafer with our hands(even with gloves) are the principal source of contaminates at this stage.

### B. Oxidation

The silicon layer that is grown in this step will define the active areas where our micropump will be formed. The diffusion boat that was loaded with the wafers in the previous step was place in the Bruce diffusion furnace (Horizontal furnace) [Fig 1]. This furnace is kept on all the time, because the energy needed to heat up the furnace is very high. It is more efficient to keep the furnace in an idle temperature when is not in use than to turn it on and off as it is needed.

Throughout the oxidation we follow the following steps for wet oxidation:

0) *Idle state*: The temperature of the furnace is set to  $1000^{\circ}\text{C}$

1) *Boat Loader Pull*: Pulls out the boat loader from the furnace, Takes about 22 min.

2) *Boat Loader Push*: Push in the boat loader to the furnace, Takes about 12 min. At this point the parameters of the furnace should be set to: 0.9slpm  $\text{O}_2$  and 18 slpm  $\text{N}_2$  and kept like that through the rest of the oxidation process.

3) *Temperature ramp up*: 10 min temperature ramp to  $1100^{\circ}\text{C}$ . The  $\text{O}_2$  flow has to be kept at 0.9 slpm.

4) *oxidation*: Let the wafers oxidize for 22 min at  $1100^{\circ}\text{C}$ . (wet oxidation)

5) *Temperature ramp down*: 10 min temperature ramp down to  $1000^{\circ}\text{C}$ . The  $\text{N}_2$

6) *Boat Loader Pull*:  $\text{N}_2$  flow is reduced to 6 slpm and the boat loader is pull out. This takes 20min.

### C. Inspection

Once the oxidation process is complete. We have to make sure that the process was successful.

1) *Visual Inspection*: We do a visual inspection looking for large defects and dust on the wafer. It is possible to determine the thickness of the oxidation layer with this method. By analyzing the color of the wafer surface under white light. Visual inspection is not common to determine the thickness of the oxide because it is difficult to differentiate some colors form others. Also since the colors correspond to the light wave length, the colors will be the same at different oxide length. Making this process unreliable.

2) *Nanospec*: To get an accurate oxide thickness reading, we used an Nanospec [Fig 2]. Since the wafer is not fully flat; and also, due to the growth of oxide through wet oxidation, the oxide has low quality. We took nanospec measurements in different point of the



Fig. 1. Brunce Furnace: Used for oxidation.

wafer, obtaining the following results:  $5114\text{\AA}$ ,  $5114\text{\AA}$ ,  $5116\text{\AA}$ ,  $5116\text{\AA}$ ,  $5114\text{\AA}$ .

Since the minimum measure error is of the Nanospec is  $100\text{\AA}$  we can conclude that the oxidation is even through out the wafer.



Fig. 2. Nanospec thickness measurment.

## III. PHOTOLITHOGRAPHY AND WET ETCHING OF ALUMINUM

The photolithography process consists of transferring a geometric patterns from a mask to the wafer surface

through a series of steps.

Since the photolithography requires a step in which UV light is used as a tool. The photolithography process is performed in a class 10 room with yellow light. Yellow light wavelength is significantly larger than the UV wavelength for the photolithography process, making the use of yellow light ideal to light the room, since it would not affect the photolithography process.

Before we started the photolithography a 130nm layer of aluminum was sputter [Fig. 3] on the surface of the wafer. This layer of aluminum will be useful in the liftoff process to form the electrodes in our EHD micropump.



Fig. 3. Perkin Elmer 2400 SPUTTER: used to sputter Al.

#### A. Photoresist coat and soft bake

Before each process the wafer should be clean with nitrogen. If by eye inspection the wafer does not look clean, solvents must be used.

*1) Photoresist coat:* The wafer is placed in the spinner [Fig 4] and properly aligned to the center of the spinners vacuum chuck. Using a pipette, we added enough (Shipley S1813) positive photoresist to cover at about  $\frac{1}{3}$  of the surface of our wafer and spinned for 45 sec at 4,000 RPM. This will form a  $1.3\mu m$  photoresist thin layer.

*2) Soft bake:* Once the layer of photoresist was added to to surface of the wafer. The wafer was placed on a hot bake plate for 1 min at  $115.5^{\circ}C$ . The soft Bake removes solvents form the photoresist and improving the the adhesion. Once this step is complete the wafer most be inspected, if the surface of the wafer is not even the lithography process most be repeated.

#### B. Expose and Develop the device wafers

*1) Wafer Alignment:* The mask that is being used should be cleaned (with acetone, isopropane, DI water and blow dry with nitrogen) before placing it into the Quintel aligner. [Fig 5]



Fig. 4. Brewer Spinner: used to spin PR.

Since we only required one mask to build the EHD micropump, there is no need for alignment marks. The mask is hold in place by activating the vacuum, and the wafer is introduced to the developer and aligned to the mask with the joystick.

*2) Exposure:* The wafer is exposed to UV light for 6.5 sec

*3) Development:* The wafer is removed from the Quintel aligner and secured into a wafer holder. The wafer holder was dipped and agitated in the Developer (Shipley Microposit MF-319) for 50 seconds. The wafers are remove from the developer, Dip into a DI water container, squirt with DI water and dry with nitrogen.

*4) After development inspection (AID):* The wafer was place under the microscope[Fig 6] for analysis. When we looked through the microscope, we observed a silver area, where the photoresist was removed; that area corresponds to the area that will form the electrodes. The segments of the wafer where photoresist was not removed were red colored.

In this experiment there was no need to redo the lithography process since all the areas in the wafer looked as was expected. Some areas looked as if the developing was not ideal, but those areas were very small and isolated, or where part of a pump that we would not test.

#### C. Etching Aluminum layer

*1) Etch the exposed Aluminum layer:* On this step, we dipped the wafers into Phosphoric-Acetic-Nitric Aluminum etchant at  $60^{\circ}C$  for 30 seconds (at this point the shaker went into the cassette). A moment after the wafer were dipped in the etchant, we could observed the Aluminum pealing (etching) away from the wafer at a rate of about  $100 \frac{\mu m}{sec}$ . This reaction started sooner in the area closer to the bottom of the container. Once the 30 seconds were over we introduced the cassette with



Fig. 5. Quintel 4000 Mask Aligner.

wafers into a container with DI water for 1 min, then we rinse the cassette with DI water and dried with nitrogen.



Fig. 6. Nikon Optiphot 200D Microscope

**2) After etching inspect (AEI):** After the aluminum etch, we place the wafer again under the microscope. Figure 7 shows a segment under the microscope of the EHD micropump. During the inspection is hard to see the amount of undercut, since we are looking from a vertical angle, but we can clearly see a shadow that forms under the aluminum.

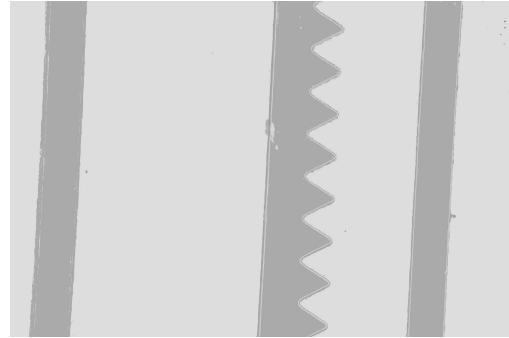


Fig. 7. Microscope picture of wafer in AEI: Lighter region is oxide, darker region is Aluminum

#### IV. LIFTOFF PROCESS FOR FABRICATION OF THE METAL ELECTRODES

The aluminum liftoff process was used to create the microelectrodes for the micropump. Before the session, 900Å of Au were sputter on the wafers. Throughout this process we placed the wafers in acetone followed by etching the the sacrificial aluminum layer that was added on the previous process. If we keep repeating these two steps, results in enhancing the yield on the micropatterns.

##### A. Acetone dip in a beaker

When we got to this session, we found the wafers already in the beaker with acetone. They had been there for about a day. The fact that this differs from the 30 min that the manual suggested is not important. Since from the materials that are on our wafers, the acetone can only dissolve photoresist.

##### B. Etch Aluminum Layer

**1) PAN etch:** The wafer was placed in a beaker with PAN aluminum etchant. Almost instantly the reaction started bubbling out the aluminum, with the gold on top of it. All the aluminum was out of the wafer after one minute.

**2) Sonic etch:** This step was not included in the manual. In order to get some of the remaining undesired material. The wafer was placed in a petri dish with acetone pass it though sonic etching for about 30 seconds. After this step the wafer was rinse with acetone.

##### C. Inspection

The wafer was inspected to see if there was remaining material to be etched. If we found any undesired material, we repeated the sonic etch step until we got the desired pattern.

#### D. Microscope Inspection and Zyglo imaging

1) *Microscope Inspection:* The wafer was placed on the digital microscope, where we captured images with different magnification factors. And measured the EHD micropump features.

The following images were obtained from the digital microscope.



Fig. 8. Zygo NuView 6000 optical profilometer

Figure 9 shows the first microscope capture with a 10X magnification. The black marks are particles that should have been removed in the etching step. The blue-gray region is the oxide layer, while the yellow region are the gold electrodes.



Fig. 9. 10X magnification

Figure 10 shows a 100x magnification of the EHD micropump. In this picture, we can see smaller undesirable particles than in Figure 9. We can also see in Figure 10 a shadow in the borders of the electrodes that correspond to the undercut beneath the electrodes.

Figure 11 shows the measurements achieved from the Image-Pro Plus software tool

From Figure 11 we know that the minimum length in the sawed electrode is  $18.5\mu m$ , the maximum length of



Fig. 10. 100X magnification

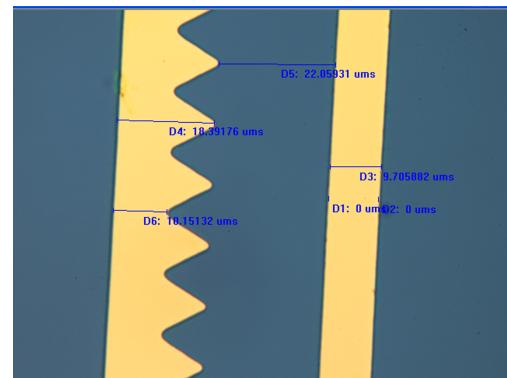


Fig. 11. 100X magnification measurements

the sawed electrode is  $18.39\mu m$ , the distance between the flat and sawed electrodes is  $22.06\mu m$  and the length of the flat electrode is  $9.71\mu m$

Figure 12 attempts to measure the undercut under the electrode. The undercut measured is  $0.15\mu m$

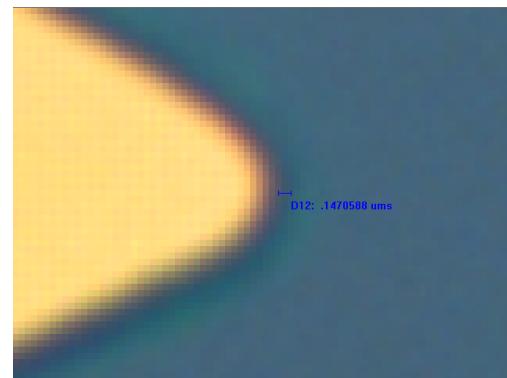


Fig. 12. 100X undercut measurements

2) *Zyglo Imaging:* After we measured graphically our EHD micropump we used the Metro Pro Zyglo Microscope Application to get a deeper analysis of the dimensions of our device profile.

Figure 13 shows the height surface map of the electrodes. where blue is lower and red is higher. This mapping is a 2D image where the undercut cannot be measured.

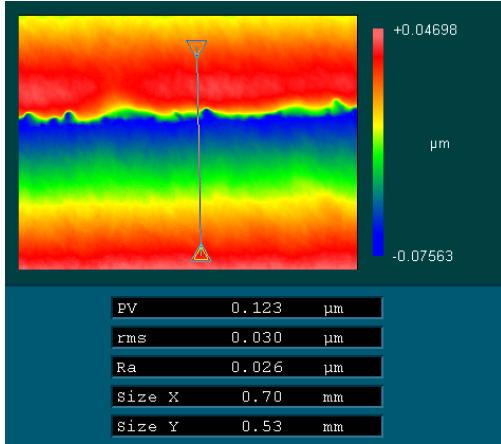


Fig. 13. Electrode surface map

Figure 14 shows a 3D model of our wafer. This representation is not accurate, since the measurements are gathered from a 2D image. But it is a good approximation of the profile of our device.

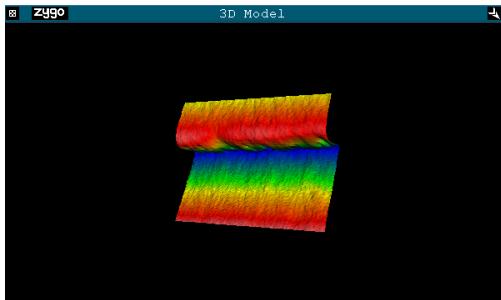


Fig. 14. Electrode 3D model

Figure 15 shows the the surface profile of our wafer.

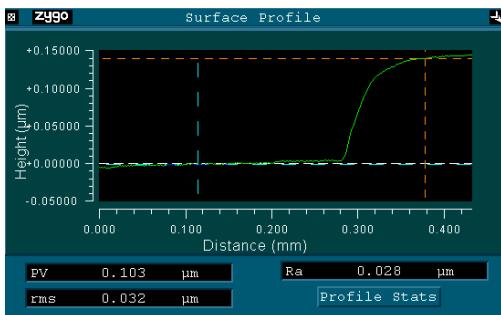


Fig. 15. Surface Profile Plot

## V. PDMS CHANNEL FABRICATION

Soft lithography or PDMS (Polydimethylsiloxane) fabrication is an effective and low cost method to build micro or nanostructures. In this process, an elastomeric wafer can be formed with patterns or structures in its surface with a feature size ranging from 30 nm to 100 $\mu$ m [2]. This method is routinely used to create inexpensive test devices including microfluidics chips and cell culturing templates.

For our device, the PDMS will be used to channel the particles on our device.

### A. Mix PDMS prepolymer

On this step we were required to mixed PDMS elastomer with a curing agent (**Sylgard® 184**). First we added 1g of the curing agent to a petri dish and then we applied 10g of the polymer base. We mixed the components with a mixing fork, until we got an uniform milky color. At this point the solution is liquid with a high viscosity.

### B. Degas the pre-cured PDMS

During the mixing step, the PDMS was cured a little because of the air in the room. Also some unwanted air bubbles where formed in the solution while we mixed it. the following steps where used to degas the solution:

1) *Apply vacuum*: The petri dish with pre-cured PDMS was placed inside of the vacuum desiccator, where we observed how the air bubbles slowly raised to the surface.

2) *Vent vacuum desiccator*: When we observed that most bubbles accumulated in the surface, we vent the chamber. The difference in pressure popped to bubbles.

Step 1) and 2) where repeated a few times, until popping the bubbles became more difficult. For our application the quality of the PDMS is not an important factor, since it is only going to be used to channel the particles to the EHD micropump.

### C. Cure the PDMS

The PDMS will be completely cured if we leave it a room temperature for a couple of days.

Once we got rid of most of the bubbles we placed the petri dish inside an oven [Fig 16] at 90°C and left the lab. One hour after the TA took the finished PDMS out of the oven.

## VI. ATTACHING CHANNELS TO MICROPUMP

PDMS surface can be treated using O<sub>2</sub> plasma. The surface of PDMS treated this way, can be bond to other layers such as SiO<sub>2</sub> PDMS. In this process. We cut the



Fig. 16. Fisher Scientific Vacuum Oven

PDMS that was fabricated in the previous process, then treat the PDMS surface in the ICP tool [Fig 17] and then bond it to the EHD micropump wafer creating the channels.



Fig. 17. Unaxis ICP etch (PlasmaTherm 790)

#### A. Slice the PDMS mold into proper dimensions

Aided with a scalpel knife, we took the PDMS wafer out of the petri dish and placed it over a firm surface. We cut eight strips from the PDMS wafer with length of about 1/4”.

#### B. Surface treatment on both Si wafer and PDMS

In this step we used the Inductively Coupled Plasma (ICP) tool. To change the properties on the surface of

the PDMS and Si wafer. It is important to remember that the PDMS properties will only change for a short period, therefore the PDMS will have to be attached to the Si wafer surface before the desired properties of the PDMS are lost.

When doing the plasma treatment it is important to closely monitor the temperature and oxygen pressure. If there is photoresist on the wafer it might burn out contaminating the tool.

1) *Si Wafer:* First we treated the silicon wafer for 1 minute inside of the ICP Tool

2) *Si Wafer and PDMS:* We placed the PDMS strip on top of the wafer, without covering the final position of the PDMS channel. Operated the ICP tool for 30 seconds.

#### C. Attach the PDMS layer to the Si wafer

When the time was done we turned the PDMS upside down, placed the PDMS on the top and bottom of the EHD micropumps and pressed the PDMS so it would stick to the Si wafer surface. After the PDMS sealed to the Si wafer. The Si Wafer was place in a hot plate for 15 min at 90°C with DI water.

#### D. PDMS attachment test

We performed an attachment test by pulling the PDMS from the Si wafer. We observed that the PDMS has a different color when it is sealed to the Si wafer. When it is not well attached it is possible to differentiate it by air under the PDMS.

## VII. MICROPUMP TESTING

The final process done to build our device consist on testing. By testing we can determine if all the process used to build the EHD micropump were successful. The testing measurements were made under the Signatone probe station [Fig 18].

#### A. Micropump functionality

The wafer with the EHD micropumps was placed in a petri dish. We place the petri dish in the probe station. We added a few drops of IPA with beads on top of one of the micropumps. The power supply was connected to the probes and turned on.

When the power turned on, the particles suspended on the IPA started vibrating and floating away from the surface of the wafer. once the particles raised far enough from the wafer they slowly start moving to the left, increasing its speed as the voltage increased.

When the power was inverted, the particles fell suddenly back to the surface of the wafer while the wires were disconnected, and when the polarity was inverses the

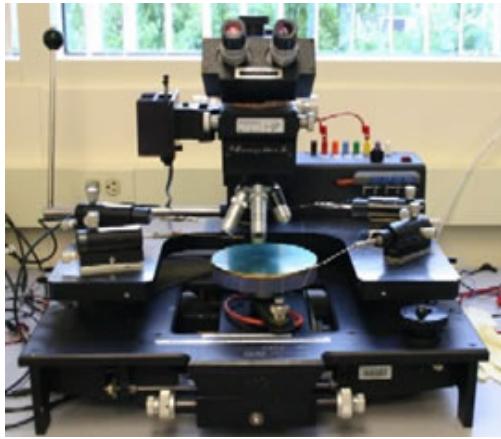


Fig. 18. Signatone probe station

particles in the IPA started floating again, and started moving the right accelerating until they reach what seemed to be the same speed as before or slightly lower.

#### B. Effects of PDMS on the micropump operation

1) *Test with PDMS:* : When we tested the micropump with the PDMS channels, we noticed that the flow of the IPA was not in a straight line. It seemed as if some vortices formed in the center of the area that we were looking at. Probably this is due to hydrodynamic properties caused, since the channels affect the IPA flow.

2) *Test without PDMS:* : When we tested the micropump without the PDMS channels, we noticed that we needed to place more IPA on the surface, since there was nothing that would hold the IPA on top of the micropump. When observed under the microscope, we saw that the flow was more linear than when we did the same test with the PDMS. But the time we had to observe the behavior of the micropump was reduced, because the IPA would tend to spread along the whole wafer.

The PDMS channel testing is better, since it will yield the same result every time with the same amount of IPA and beads, even though there is a formation of vortices. While the testing without the PDMS channel might look better it is hard to measure and replicate.

#### VIII. CONCLUSION

Through out six session we had a practical approach to the fabrication process. In which we developed a functional *Electrohydrodynamic Micropump*. During the first three weeks we started from a polished silicon wafer, following the fabrications steps of: oxidation, photolithography, and lift-off. Then we spent the last three weeks preparing for testing by fabrication the

PDMS, Attaching PDMS channels to the micropump and finally testing the micropump. These lab sessions were a great aid to our understanding of the class lecture concepts and show us how each fabrication step are linked and how they affect the final product.

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**IX. ANALYSIS QUESTIONS****A. Oxidation**

**2.a.** What is the oxide thickness predicted from oxidation theory, assuming an  $H_2O$  ambient? (Use the equation for oxide thickness, and A, B/A from the text, or the table from the text!) In fact, the ambient during the oxidation was a mixture of oxygen and  $H_2O$ . As a lower limit on the expected oxide thickness, determine the oxide thickness predicted by oxidation theory for a dry oxidation at the same temperature. Comment qualitatively on the thickness expected from the actual composition of the oxidizing ambient, remembering that the partial pressure of  $H_2O$  is less than one atmosphere, and comment on your results with respect to your predictions. Note that wet oxidation rates are often specified for 640 Torr of  $H_2O$ , corresponding to a mixture of water and oxygen in the oxidizing ambient.

$$\text{since } (t + \tau) \gg \frac{A^2}{4B}$$

$$X_{dry} = \sqrt{(1200\text{sec})(0.0178)} = 0.59\mu\text{m}$$

$$X_{wet} = 2.8\mu\text{m}$$

**2.b.** What is the color given by the color chart for the oxide thickness predicted for your process? Pink

**2.c.** What is the observed color? What thickness correspond to the observed color?  
something between 0.48 and 0.52  $\mu\text{m}$

**3.a.** How much variation was there among your Nanospec measurement? (The apparent non-uniformity may either reflect real non-uniformity in the oxide thickness or random measurement errors.)

The variation was of 1 Å, this is meaning less, since the variation is smaller than the measure error.

**3.b.** How do the two methods of thickness determination compare with each other and with theory? What are some possible reasons for the differences?

Both, the nanospec and the eye inspection are based on measuring the wavelength of the light that bounces on the wafer. The difference is that the nanospec has a better resolution for measuring those the size that can be found on a wafer.

**4.a.** Estimate the percentage error in determining the thickness using the color chart.

the thickness error can be 8% if we know the expected thickness but don't quite notice the difference between some colors, can be up to 78% if we don't have previous knowledge of what might be the oxide thickness. And can even be higher if the person who is measuring the wafer by sight has any type of daltonism.

**4.b.** Propose an experiment (a set of measurements) to determine the random error in the Nanospec measurements, and to distinguish the random error from thickness variations across the wafer.

In order to determine the random error from the spectrometer we could move the wafer with the nanospec to different coordinates. This way we would measure the same thickness at different coordinates.

The difference in measurement can be translated into the nanospec error.

**5.** Ideally, through the processing sequence so far, the face of the wafer should be entirely featureless except for the very light crite marks. Is it? is there any dust visible under lamp inspection? comment. We did not notice any features on the wafer, dust or any other unwanted characteristic.

**B. Photolithography**

**1.** How do you know that all of the photoresist is removed from the exposed areas during the after development inspection (ADI)?

By looking into the microscope it is easy to tell if there are any PR visible particles. The color of the PR is red while the AL is silver. So if there area red particles on the wafer that means that there is still some PR on the wafer.

**3.** What are the various colors you notice the wafer during ADI? What materials does each of these color correspond to?

Red  $\Rightarrow$  Photoresist

Silver  $\Rightarrow$  Aluminum.

**4.** Are the structures accurately reproduced in the patterned metal electrode areas on the wafer?

Because of proximity masking was used, there is a small discrepancy between the mask and the wafer pattern due to optical diffraction. But it was not noticed in through the microscope.

**5.** Interpret the results of your AEI. What are the various colors you notice on the wafer during AEI? What material does each of the colors correspond to? In particular, was the etch unifrom, and were any other deviations from the expected result visible?

The result was very good with no observable deformities or deviations from expected results

Silver  $\rightarrow$  Aluminum Blueish  $\rightarrow$  Silicon dioxide.

**C. Lift-off**

**1.** Did you see some flakes coming from of your wafer (during DI water rising) after the first acetone

dip process. What are these flakes made of, Cr/Au, aluminum, photoresist or a combination?

The wafer was already in acetone when we got to the lab.

**2.** Does it matter if you etch for 2 mins versus 5 during the Al etch process?

It does not matter how long you leave the wafer in the etchant, It will only etch aluminum.

**3.** What was your yield (roughly) after the first round of acetone dip/Al etch process? Is it necessary to do the second round? Did the yield improve after repeating the acetone dip and Al etch process?

After the first round most of the Al was etch, some teams did a second round of the acetone dip/Al etch and soindic etch to improve results.

**5.** What magnification was used for the Zygo imaging tool? How about the optical microscope? What is the resolution for the Zygo tool in the vertical axis, in the lateral axis (x and y)?

The magnification of the Zygo tool was 10x and 100x. the Nikon Optiphot 200D Microscope magnification used was 100x

The Zygo resolution for the vertical axis used was 130nm, but can be improve by using a different magnifying lens.

We did not use lateral axis, but the Zygo tool has a resolution of  $0.37\mu m$  resolution on the x and y axis.

**6.** What is unique about the Zygo imaging tool compared to the optical microscope? In other words, what feature does it provide compared with the optical microscope? What is the resolution limit in the horizontal direction for the Zygo and what is the resolution limit in the z-direction.

The Zygo imaging toll can measure depth crating an aproximate profile image of the surface of the wafer.

The Zygo tool is capable of measuring the undercut, but for our results the profile is not fully accurate, since we only measured the vertical axis. The Zygo resolution in the z-axis is 130nm, but can be improved by changing the lenses.

#### D. PDMS Channel Fabrication

**1.** What would happen to the process if the PDMS prepolymer didn't mix well?

It would break when we take it out of the petri dish.

**2.** What would be the impact to the final PDMS layer if air bubbles weren't fully removed?

If air bubbles weren't remove, the PDMS would not work as an insulator.

Since our PDMS will be used to channel a liquid, and we are not requiring any insulator properties for our device, this would not matter in our application.

**3.** Can you think of an application for this PDMS process?

Sensors that can be embedded in the body.

Aesthetic Implants.

Adhesive

Sealants for aquariums or pipes.

#### E. Attaching Channels

**1.** How does the oxygen plasma treatment change the surface properties of the PDMS layer?

It cleans the wafer and the PDMS so they can stick better to each other.

**2.** What's the meaning of hydrophobic and hydrophilic properties? How do you know if a material is hydrophobic or hydrophilic? Suggest a simple means for testing.

Hydrophobic is a material that repels water (cannot be solved in water).

Hydrophilic is a material that accepts or absorb water (gets wet with water or can form a solution in water).

A simple experiment to test if a material is hydrophobic or hydrophilic is to dip a spoon with a small sample of that material into a water container, and take the spoon out of the water right after dipping. If the material looks dry, that material is hydrophobic, if the material looks wet that material is hydrophilic.

**3.** How do you confirm or convince yourself that the plasma treatment really works or that the surface property of the PDMS material really changed after this oxygen plasma treatment?

The PDMS will stick strongly to the wafer

You can see a difference in color form where it worked and where it did not.

You can pull the PDMS if it comes out easily it the plasma treatment did not work. If it was hard to rip the PDMS from the wafer then it worked (Not recommended to rip the PDMS off the wafer).

**4.** What is the pupose of fabricating the PDMS channel for your micropump?

To prevent the testing liquid from going outside the pump area.

**5.** What if any, are the limitations of the PDMS channels? Suggest one or more ways of overcoming the limitations you have mentions:

If there PDMS has a large area, it will not fully stick to the wafer. This can be improved by cutting the channels as thin as possible.

There is just a short time to place the PDMS on the wafer after the plasma treatment. This can be overcome if the location where the PDMS is going to be is well planned before the plasma treatment and the PDMS is treated as close as possible to its final location on the wafer.

#### *F. Micropump Testing*

**1.** At what voltage did you see the beads moving from the micropump? Does increasing the voltage constantly increase the beads flow velocity? What is the polarity of the voltage that is applied to the emitter end, and in which direction do the beads move?

In this part of the experiment we used 40V dual output. The positive terminal was connected to the micropump anode electrode and the negative terminal was connected to the micropump cathode electrode. When the power source was turned on the beads started accelerating to the left (towards the cathode).

When we increased the polarity of the voltage source the flow velocity was increased.

When we change the polarity (positive terminal to cathode and negative terminal to anode) the flow changed direction but the velocity magnitude seemed lower.

**2.** Do you observed strange behavior, i.e. vortices forming? Do you see them while testing the micropump one with channels or one without channels? The vortices, do they form laterally or vertically, can you tell for sure? Try to suggest a reason why the vortices are forming and a means to reduce them.

When using the micropumps with PDMS channels we observed that vortices were formed. These vortices bent the horizontal trajectories in a vertical direction.

I believe that the vortices form because of fluid dynamic interaction between the test fluid and the PDMS walls. We could reduce this vortices by placing the PDMS further from the pump, or by lowering the power applied to the micropump.

# FABRICATION OF CARBON-NANOTUBE DEVICES

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**Abstract—**This report details the process for fabrication of a carbon-nanotube structure that is assembled between two electrodes.

## I. INTRODUCTION

A carbon nanotube is a cylindrical single molecule made out of single carbon atoms with a diameter of as little as 1 nm and a length up to micrometers.

Carbon nanotube can be thought as a single layer of carbon that has been wrapped into a cylinder. Nanotubes have a wide range of mechanical and electrical properties; Nanotubes are the strongest fibers than can be made, and nanotubes can be processed to behave as semiconductors or metals. All the properties of carbon nanotubes are very promising since its applications would be very vastly once the knowledge about carbon nanotubes will be enough to extend the maximum length of the nanotubes.[1]

## II. CARBON NANOTUBES ASSEMBLY

### A. Processing

Before we arrived to the fabrication laboratory a silicon wafer was processed with e-beam lithography to pattern electrodes where our nanotube was going to be grown and the nodes where the probe is going to be connected for testing.

*1) Dielectrophoretic Assembly:* Is one of the methods to attach nanomaterials to micro devices. Dielectrophoresis causes no damage and less contamination in the assembly process.

The process followed in the fabrication lab was the following:

The test die was placed under the **Wentworth Labs Signatone Probstation** [Fig 1], the probes were carefully connected to the electrodes. We applied a solution of DI water and with  $150 \frac{ng}{ml}$  multi-wall concentration from **Nanolab®**. The **Agilent 33220A signal generator**[Fig 2] was powered with  $5V_{pp}$  at  $5MHz$  for 30 seconds. The electric energy creates a magnetic field between the electrodes that attracts the carbon atoms to the end points of the electrodes. When the time was over we kept the power on and dry the die by blowing nitrogen to the die.

Once the die was dry the nanomaterial solidified and we turned off the power.

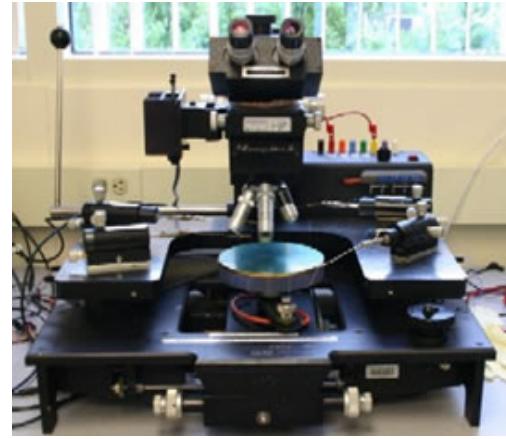


Fig. 1. Signatone probe station



Fig. 2. Signal generator: Agilent 33220A

The force that forms the carbon nanotube, can be modeled as a homogeneous sphere surrounded by a conducting dielectric medium:

$$F_{Dep} = \frac{\pi r^2 l}{3} \epsilon_m R_c \left\{ \frac{\epsilon_p^* - \epsilon_m^*}{\epsilon_m^*} \right\} \nabla |\bar{E}|^2$$

where  $\frac{\pi r^2 l}{3}$  is the volume of the electric field,  $\epsilon_m$  is the medium (DI water) dielectric constant,  $\epsilon_p$  is the nanocarbon dielectric constant and  $\nabla |\bar{E}|^2$  is the electric field.

### III. SCANNING ELECTRON MICROSCOPY (SEM) IMAGING

The SEM [Fig 3] is a tool used for higher magnification images than the microscope. While a microscope can magnify up to 100x or 200x, the SEM can magnify images up to 50,000x. This is useful in fabrication process for debugging and finding fabrication problems.



Fig. 3. SEM- Zeiss Surpa 25

Fig. 4 shows a picture of our sample inside of the SEM tool. Using the camera inside the SEM tool to aligned the sample to the SEM imaging sensor.



Fig. 4. Alignment camera view

The SEM is a very delicate tool that utilizes high voltages and is very sensitive to manipulation. Because of this the tool was operated by the TA at all time and the students were observers.

This session was particular hard to follow due to the great amount of students that were on the lab at this

session [Fig. 5]. And all the data gathered from the SEM tool was on a computer screen.



Fig. 5. Picture of students looking at the results

#### A. Metal Coating

In order to get images with the SEM tool, a thin metal layer is deposited in the surface of the wafer. The SEM tool has a metal deposition chamber, where the coating took place. The metal coating was done in a previous session and we did not get to see the operation of the coating chamber.

#### B. SEM Imaging

After the metal coating, the nanotubes samples were loaded to the SEM holder (this was also performed in a previous session).

The following pictures were gathered from the SEM-Zeiss Surpa 25 tool.

Fig. 6 shows a picture of a die with a multi-wall nanotube formed when applied 3V during the assembly process Magnified 23,230x.

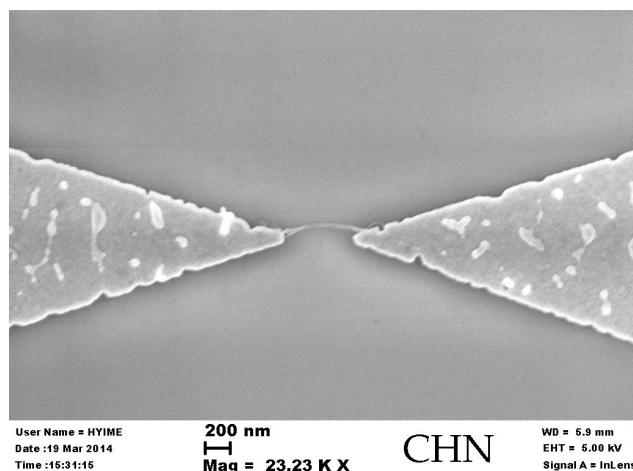


Fig. 6. Nanotube assembled with 3V, magnified 23,230x

Fig. 7 shows a picture of a die with a multi-wall nanotube formed when applied 5V during the assembly process Magnified 23,230x.

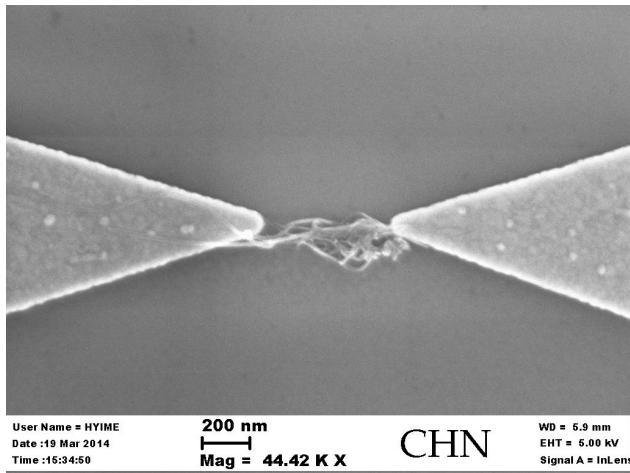


Fig. 7. Nanotube assambled with 5V, magnified 44,420x

Fig. 8 shows a picture of a die with a multi-wall nanotube formed when applied 5V during the assembly process Magnified 659x. The marks on the contacts where made with the probe station touched the die in order to close the circuit to create the nanotubes.

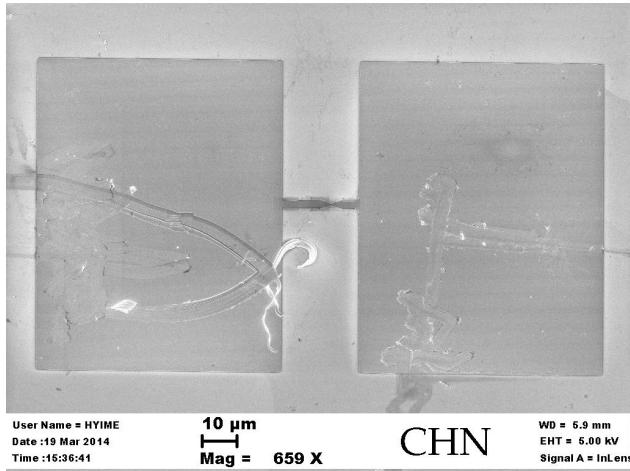


Fig. 8. Nanotube assambled with 5V, magnified 659x

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## IV. CONCLUSION

In this report we detailed the process for fabricating carbon-nanotubes. We notices that if we applied 3V signal we formed a single nanotube that forms following the electromagnetic field formed between the gold electrodes. We also notice that if we applied a higher voltage, we will form a bigger nanotube, with may ramifications which affects its quality.

**V. ANALYSIS QUESTIONS****A. Carbon nanotubes assembly**

**1.** What type of nanostructure (multi or single walled carbon nanotubes) did you use during the assembly experiments? What is the difference between them?

We used multi-walled nanotubes. These structures have a bigger diameter, can grow longer lengths, and have more carbon layers than single walled structures.

**2.** What is the voltage and frequency used during the assembly experiments? How long was our assembly time?

We used  $5V_{pp}$  and  $3V_{pp}$  to form the nanotube structures with a frequency of 5MHz for 30 seconds.

**5.** As usual, be certain to comment on any deviations from the process described here and on any observation that you think might be significant.

We used a syringe to add the nanotube carbon solution. Once the drop was on the die, the process cannot be seen and can only be monitored until the exceeding solution is cleaned (wiped out).

**B. SEM imaging**

**1.** How long was the duration for the metal coating (in hummer coater), can you see a difference in color on your sample? If so, what is the new color?

This part of the process was already done in a previous session and we did not get to see it.

**2.** Did you see a plasma during coating? What type of gas is used inside the hummer tool? What material is used to coat the microplatform? Also, where is the metal source located inside the chamber?

This part of the process was already done in a previous session and we did not get to see it.

**3.** Why did we use the coating? What is the associated problem in the SEM imaging tool called and which type of materials (metal, semiconductors, insulators) does one expect to see this type of problem?

We used the coating to improve the image quality gathered from the SEM tool. Insulating materials will not form a good thin layer, due to the nature of the deposition method used in the chamber (DC instead of RF deposition).

**4.** How did you attach your device to the sample holder? It was attached with carbon tape.

**5.** At what vacuum level do you turn on the e-beam gun?

The system pressure was set to  $2.06 \times 10^{-6} mBar$  and the gunge chamber was set to  $1.45 \times 10^{-6} mBar$

**6.** Some SEMs come with an EDX option, does this SEM have one? What is an EDX used for, ask the TA or find online.

This SEM tool has the EDX option but we did not use it during the lab session. EDX stands for Energy Dispersive X-ray spectroscopy. Which is used for elemental analysis or chemical characterization.

**7.** The hardest part of taking quality SEM images is setting up the tool. Pay attention to the way the Ta does this operation. Notice the equipment settings. What is your acceleration voltage?

The acceleration voltage was set to 5kV

**8.** Try to capture a few (1-2) defective devices or artifacts (Yes, no matter how good you are or how clean your lab is, there are still defects). Explain this defects, lithography, assembly, etching.

There are some scratches on the electrodes which were made when the probe was connected to form the nanotube structure.

There are some sub-micron spots on the electrodes which were made in previous steps such as lift off.

There are some sub-micron defect in the shape of the electrodes, due to lithography defects.

There is a round mark around the die, that was caused when we poured the drop of carbon nanotube solution.