Silicon Nano-wire (SiNW) Fabrication: Alumina Transfer Unit

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University Of Illinois at Urbana-Champaign, IL

December 17, 2010

Prepared in partial fulfillment of the requirements of the Illinois Scholars Undergraduate Research Program (ISUR), under the direction of Kyle Jackobs, Keng Hsu, and Nicholas Fang in the Nanotechnology group of the Mechanical Engineering department at the University of Illinois at Urbana Champaign.

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Abstract

Silicon Nanowires (SiNWs) are used in several applications such as converting chemical energy and temperature gradient in to electrical energy. SiNWs could be manufactured in several different processes depending on what final product one wants to achieve. In this experiment we are interested in fabricating SiNWs that have separations of about 100nm and a diameter of about 40nm using the process of chemical wet etching. We face a real issue during the last few stages of the manufacturing process when trying to transfer an alumina template floating in buffer solution into an acetone solution then deposit it on Silicon. The purpose of this project was to create a mechanical system that would make this process easier to carry out. To achieve this goal, a simple system was designed and rapid prototyped for testing. The mechanical system behaved as originally predicted; however, there is still some stability issue that needs further investigation.

Introduction

Nano technology is one of the fields that is getting a lot of popularity in Engineering field. There are broad areas of research topics in this field one of which is nano-wire fabrication. Silicon Nano-wire (SiNWs) fabrication can be achieved through several different techniques including vapor-liquid-solid mechanism (VLS), deep reactive ion etching (DRIE), metal-assisted chemical wet etching and more. However, each method separately fails to either restrict the growth of the nanowires in only one direction (axial) or have a diameter less than 100 nm with big separation between each 'hole'. One of the ways to resolve these issues is carrying out a process called chemical wet etching where metallic chromium/gold nanodots were first deposited onto the silicon wafer using anodic aluminum oxide template and eventually chemical wet etching is carried out in a hydrogen peroxide and deionized water. In previous experiments, using this method, it was showed that the silicon nanowires could be precisely controlled to a precision of 10 nm in the range of 40 to 80 nm that is uniform and well aligned.

I spent the majority of the semester learning the process of chemical etching which mainly has two major steps with several subcategories. These steps include:

- 1. Preparing for SiNW manufacturing
- a. Electro-polishing in order to remove any bumps on our aluminium sample \sim 5-10 min in Ethanol and perchloric acid solution in temperature less than 15 $^{\circ}$ C
- b. First Anodization \sim for 8-16 hrs in 0.3M oxalic acid with temperature less than 15 $^{\circ}$ C- this creates small U-shaped holes
- c. Remove Alumina (Aluminium oxide) in Phospheric acid, Chromic Acid, and Water solution this removes the alumina that created our holes converting them to little dots

- d. Second Anodization-same as first Anodization except now only for 2-4 min- creates our U-shaped holes again but this time more closely spaced
- e. Spin sample on PMMA so that our sample would not fall out when we remove the base (Aluminium)
- f. Remove Aluminium in a water, Hydrochloric acid, and cupper chloride solution keeping the sample as long as needed.
- g. Open the pores in a phosphoric acid solution at room temperature for about 20 min this removes the bottom of our U-shaped holes making them || shaped (i.e. just columns).
- h. Remove the PMMA in an Acetone solution and deposit our aluminium oxide (AAO) columns on Silicon
- i. Evaporate Chromium and Gold on our Silicon/AAO sample
- j. Remove AAO and evaporate AU gain
- k. do catalytic etching in hydrofluoric acid and per oxide solution creating the final product
- 2. Do measurements to check the diameter and spacing of the SiNWs (commonly done by another group)

One of the biggest issues we face in this fabrication process is during the transfer of the anodic aluminium oxide template onto the silicon wafer. The aluminium oxide is a very thin film; so we support it with PMMA to prevent it from falling apart, and let it sit in a buffer solution to make less acidic. The last step before depositing the template is transferring it from the buffer solution to an acetone solution for removal of the PMMA. After this step is completed, we deposit the "PMMA free" template onto the silicon wafer. However, we do not have a good way of executing the PMMA removal and alumina transferring steps, which we believe is

causing in poor final result. Therefore, my objective for this semester was to be able to design a system that could make the alumina pattern transfer easier.

Methods

The first step I took was design. Having all the requirements in mind a simple sketch of the system was made. Once a simple concept was achieved, it was translated to a 3D sketch.

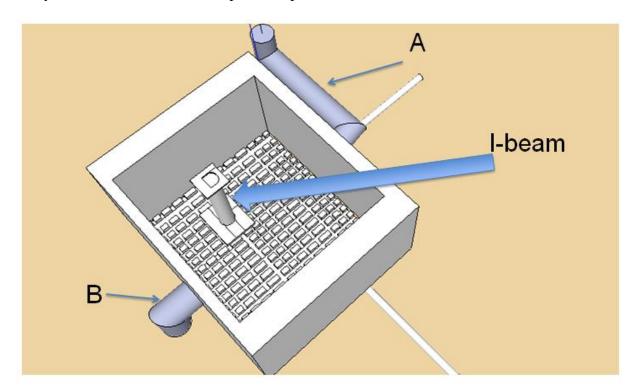
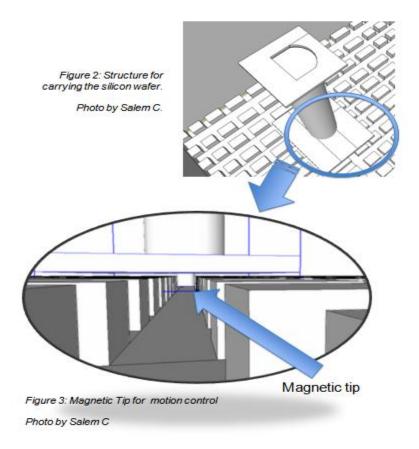


Figure 1: Alumina Transfer System-ATS Top view: Photo by Salem C

The picture above shows a snap shot of the 3D sketch of the alumina (aluminum oxide) transferring system. The two (in later design I added two more tubes symmetrically around the cube) tubes labeled "A" and "B" are used for transferring a solution in and out respectively. The silicon wafer will be hold in the I-beam structure shown below.



The cubes at the base of the rectangle are there to restrict the motion of the I-beam in a predictable manner just for the purpose of simplicity; it would also limit the moment of the I-beam about the base of the cube to some extent. The motion of the I-beam is controlled by a magnetic force attraction between the ''magnetic tip" (shown in Figure 3) and the magnetic bar shown in Figure 4.

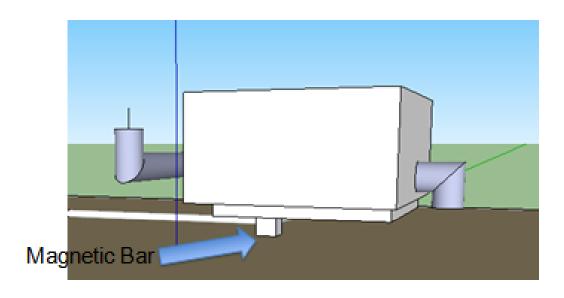


Figure 4: Alumina Transfer System-ATS Side View: Photo by Salem C

At first a simulation of the Alumina Transfer system was made using google sketchup to analyze what kind of motion it could have. In most of the simulations, the I-beam moves in a straight line following the position of the 'magnetic bar.' However, in some situations where the bar magnet position was changed rapidly the I-beam could not keep up and as a result falls sideways as shown in Figure 5 below.

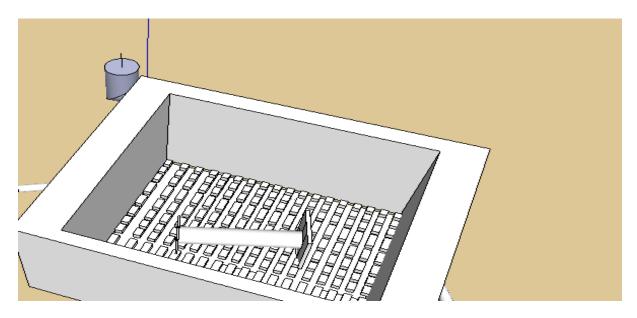


Figure 5: I-beam on its side during simulation: *Photo by Salem C*

The results of the simulations were satisfactory for the most part. However, google sketchup is not really perfect simulation software so to get a more accurate analysis of how the system would work I had to build an actual model and get it rapid prototyped. Therefore, I made the same model depicted in the figures above in ProE for rapid prototyping. Once the rapid prototyped part was done I bought some tubes and valves for the inlet and outlet of flows. Below is the picture of the rapid prototyped system with the tubes and valves attached to it.

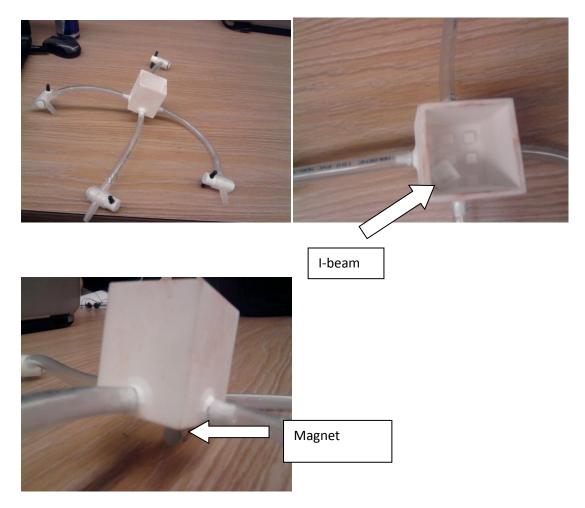


Figure 6: different views of the rapid prototyped part: *Photo by Salem C*

Results and Discussion

According to several of experiments we run, the alumina floats on the buffer solution but it sinks in the acetone solution. This makes sense since acetone is less dense than the buffer

solution. The other result we got was that it takes about a minute to have more than 90% of acetone concentration if we remove the buffer solution our cube while adding acetone at the same time with the same flow rate. This was important during our original theory which was based on the assumption that the alumina pattern would float in both acetone solution and buffer solution. If this assumption was correct, we would first have the alumina template floating in the box filled with the buffer solution. Once the sample is kept in the buffer solution long enough to be neutral, we will open the valves and remove the buffer solution while adding acetone with the same inlet flow rate as the outlet flow rate simultaneously keeping the height constant. However, now we know that the alumina template sinks in acetone. Therefore, we can remove the buffer solution with a very low flow rate at first until the solution's height is leveled with the I-beam. Then we would let the template settle on the silicon which is already placed on I-beam. Once the template is on the I-beam we can just remove all the buffer solution and fill it out with the acetone solution.

At this point we would have the silicon on top of the I-beam and on top of that we would have our alumina template. Once the PMMA removal is completed, which usually takes about 20 minutes in the acetone solution, we would again remove the acetone solution the same way as we removed the buffer solution and we would have the transfer of alumina completed.

During my experiment I was able to get the system working until the buffer removal step. However, I could not find a small enough magnet to fit on the I-beam so the process was not consistent. Since, I am going to be working on this next semester too, I am planning to change up the geometry of the system a little bit so that I could fit magnets at the bottom. Once I get that stable then I would try to do the process and see if we get better Nanowires in this method than the method we are using right now which is just transferring the template by using tong.

Plans for Next Semester

Next semester I am planning to address several problems. The first problem I want to address is the problem of visibility. Right now it is really hard to track where exactly in the solution the film is sitting visually. One way of approaching this problem is to use ultraviolet light. Acetone does not reflect UV light whereas alumina does. Therefore, if we shine a UV light and try to detect the reflection, we could possibly be able to track the film.

The second problem I want to address is, as stated above, make the system I designed more stable and compare the final results.

The third problem I want to address is the problem of multiple transfer and deposition. Right now the system is designed to transfer and deposit one pattern at a time. If this system works as expected, I want to add more features so that it would be able to do several depositions/transfers at a time.

Finally, I want the system to be able to do all the processes, which include tracking the position of the alumina template, moving the I-beam so that it is always below the template, do multiple transfers, and control the inlet and outlet flow automatically. For this I am planning to do some more reading on control system applications.

Reference

Jinquan Huang; Sing Yang Chiam; Hui Huang Tan; Shijie Wang; Wai Kin Chim. Fabrication of Silicon Nanowires with Precise Diameter Control Using Metal Nanodot Arrays as a Hard Mask Blocking Material in Chemical Etching. Department of Electrical and Computer Engineering, National University of Singapore, 4 Engineering Drive 3, 117576, Singapore, and ‡Institute of Materials Research and Engineering, A*STAR (Agency for Science, Technology and Research), 3 Research Link, 117602, Singapore, *Nano Res.* 2010