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ABSTRACT

DESIGN AND FABRICATION OF A MICRONEEDLE FOR THE IMPLANTATION OF FLOATING MICROSTIMULATORS

by
Faysal Ahmed

Neural prosthetics are used to stimulate any remaining functional nervous tissue in order to restore function in visual, auditory, or other physiological components associated with the nervous system. Microelectrodes have been used in stimulating and detecting electrical activity in neurons. When stimulating or recording neural tissue activity using microelectrodes, there are usually many problems and difficulties in obtaining the correct functionality and results. There is great difficulty in placing these microelectrodes in a specific location in the central nervous system due to various problems with the methodology used. Many different techniques such as the traditional interconnected microelectrodes have been an arduous process and often times ineffective. A promising technique using the technology of micro electro mechanical systems (MEMS) eliminates the difficulty of placing and delivering microelectrodes in desired areas. MEMS technology enables the use of micro/nanometer sized features by micromachining and microfabrication. The reduced size of the devices produced allows the development of many products used for several different applications.

Placing electrodes in the central nervous system have been a very difficult process. This thesis focuses on the design and fabrication of a microneedle that will have the capability of penetrating the neural tissue and delivering floating microstimulators pumped out by fluid through a microfluidic channel.

DESIGN AND FABRICATION OF A MICRONEEDLE FOR THE IMPLANTATION OF FLOATING MICROSTIMULATORS

by
Faysal Ahmed

A Thesis
Submitted to the Faculty of
New Jersey Institute of Technology
in Partial Fulfillment of the Requirements for the Degree of
Master of Science in Biomedical Engineering

Department of Biomedical Engineering

August 2008

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APPROVAL PAGE

DESIGN AND FABRICATION OF A MICRONEEDLE FOR THE IMPLANTATION OF FLOATING MICROSTIMULATORS

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To my parents, my brother Azfar, and my sister Javairia for their support and stuff.

"I wasn't like every other kid, you know, who dreams about being an astronaut, I was always more interested in what bark was made out of on a tree. Richard Gere's a real hero of mine. Sting. Sting would be another person who's a hero. The music he's created over the years, I don't really listen to it, but the fact that he's making it, I respect that. I care desperately about what I do. Do I know what product I'm selling? No. Do I know what I'm doing today? No. But I'm here, and I'm gonna give it my best shot."

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TABLE OF CONTENTS

Chapter **Page**

1 INTRODUCTION 1

1.1 Objectives 1

1.1.1 Microneedle Design and Implantation 1

1.1.2 Microfabrication Development 3

1.1.3 Device Testing in Agarose Gel 5

2 BACKGROUND 6

2.1 MEMS Technology 6

2.2 Microfluidics 7

2.3 Neural Prosthetics 7

2.4 FLAMES 9

2.5 Microneedle Background 10

2.6 Dektak Profilometer 11

2.6.1 Dektak Operation 12

3 MODELING AND DESIGN FEATURES 14

3.1 Pro Engineer 14

3.2 AutoCAD 16

3.2.1 Microneedle Periphery 17

3.2.2 Microfluidic Features 18

3.2.3 Vias 19

4 FABRICATION 22

TABLE OF CONTENTS
(Continued)

Chapter **Page**

4.1 Silicon Wafers........ 22

4.1.1 Wafer Properties........ 22

4.1.2 Thin Wafer Handling........ 24

4.2 Cleaning........ 24

4.2.1 NMP-Clean........ 25

4.2.2 Piranha-Clean........ 26

4.2.3 M-Pyrol Clean........ 26

4.3 LPCVD 27

4.3.1 Silicon Nitride........ 28

4.4 Photolithography 28

4.4.1 Photoresist Application........ 28

4.4.2 Photomasks........ 31

4.4.3 Mask Allignment........ 34

4.4.4 UV Exposure and Development........ 35

4.5 Etching........ 37

4.5.1 Reactive Ion Etching........ 37

4.5.2 Deep Reactive Ion Etching........ 39

4.6 Wafer Bonding........ 45

4.6.1 Photoresist Bonding........ 46

4.6.2 Wet Thermal Oxidation........ 46

TABLE OF CONTENTS
(Continued)

Chapter **Page**

4.6.3 Fusion Bonding and Hydrophilization.. 48

4.6.4 Post Bond Annealing.. 51

4.7 Microneedle Removal... 53

4.8 Microfluidic Connector.. 54

4.8.1 Microfluidic Connector Bonding... 55

5 FINITE ELEMENT ANALYSIS SIMULATIONS.. 56

5.1 ANSYS... 56

5.2 Buckling of Microneedle Shank.. 57

6 CONCLUSION...... 60

APPENDIX A PHOTOMASKS 63

APPENDIX B TRAVELER FOR PHOTORESIST BOND... 67

APPENDIX C TRAVELER FOR FUSION BOND.. 70

APPENDIX D 2D PROCESS FLOW-PHOTORESIST BONDING..................................... 73

APPENDIX E 2D PROCESS FLOW-FUSION BONDING....... 82

REFERENCES 92

LIST OF TABLES

Table	Page

3.1 FLAMES and Well Sizes. 21

4.1 Recipe for Reactive Ion Etching of Silicon Nitride................................. 38

4.2 Deep Reactive Ion Etching Program. 42

4.3 Deep Reactive Ion Etching Recipe. 42

4.4 Wet Thermal Oxidation Program.. 47

4.5 Wet Thermal Oxidation Gas Amounts.. 48

4.6 Annealer Program. 52

LIST OF FIGURES

Figure		Page
1.1	Flames implantation through microneedle	2
2.1	EmKay Sisonic Microphone [6]	6
2.2	Cochlear Implant [7]	8
2.3	FLAMES device	9
2.4	In-Plane Needles [8].	11
2.5	Out-of-Plane Needles [9]	11
2.6	Dektak Profilometer	11
2.7	Close up of Dektak Profilometer showing stylus and camera	12
3.1	Microneedle shown before bonding	14
3.2	Bonded microneedle	15
3.3	Microneedle side and top view	16
3.4	Microneedle dimensions in millimeters	17
3.5	Microfluidic channel (bottom wafer)	18
3.6	Microfluidic channel dimensions in millimeters	19
3.7	Vias (top wafer)	19
3.8	Vias dimensions in millimeters	20
4.1	Common wafer thinning procedure [15]	23
4.2	Wafer with silicon nitride layer	23
4.3	Common LPCVD system [10]	27
4.4	Feature patterned in the mask to edge the microneedle periphery	31
4.5	Photomask of needle periphery	32

LIST OF FIGURES
(Continued)

Figure		Page
4.6	Microfluidic channel feature centered in microneedle...........................	32
4.7	Photomask of microfluidic channel..	33
4.6	Vias shown in microneedle...	33
4.7	Photomask of vias..	34
4.8	Alignment marks..	35
4.9	UV exposure methods [2]...	36
4.10	Typical reactive ion etching chamber [13]......................................	38
4.11	DRIE anisotropic etch profile [15]..	39
4.12	DRIE anisotropic etch of the microfluidic channels...........................	41
4.13	DRIE anisotropic etch of the vias..	41
4.14	Snapshot of etched microfluidic channels taken under the microscope.........	43
4.15	Snapshot of the vias taken under the microscope..............................	43
4.16	Snapshot of the microneedle periphery taken under the microscope............	44
4.17	Microfluidic channel view on Dektak..	44
4.18	Microneedle periphery view on Dektak...	45
4.19	Thermal oxidation furnace..	47
4.20	Silicon to silicon fusion bonding [1]..	49
4.21	Fusion bonding setup [16]..	50
4.22	Microneedles placed in petri dish..	53
4.25	Bottom half of microneedle with microchannel seen under microscope........	53
4.26	Nanoport features and dimensions [18]...	54

LIST OF FIGURES
(Continued)

Figure **Page**

5.1 Effective length of column with restraint [21].. 57

5.2 Distributed load applied to the top face.. 58

5.3 X-Component of displacement plot.. 59

5.4 Force vs. maximum displacement plot.. 59

6.1 Metal Contacts shown in red on microneedle...................................... 61

CHAPTER 1

INTRODUCTION

1.1 Objectives

The objectives of this thesis are to present the design, fabrication, and testing methods conducted into creating a prototype for a BioMEMS (Bio Micro Electro Mechanical System) micrconeedle. The BioMEMS microneedle has microfluidic features for electrode delivery applications. The electrodes used for delivery in the microneedle will be FLAMES (Floating Light Activated Micro Electrical Stimulators). MEMS and microfluidic technology have allowed this prototype to be created, and these technologies will be further explained in order to understand the processes described in this thesis. The microfabrication development for the creation of the microneedle was made possible through the equipment at the Microelectronics Fabrication Center (MFC) at the New Jersey Institute of Technology (NJIT). Performance of the microneedle will be measured by an initial stage of testing using agarose gel. This chapter introduces and describes the technologies along with applications and background information on the design, functionality, and the fabrication methods of the device.

1.1.1 Microneedle Design and Implantation

This microneedle was designed to deliver the FLAMES devices into living tissue. The FLAMES would be pumped out by a fluid pumping mechanism through a microchannel. The microneedle consists of many different components in order for it to function properly. The microneedle was designed to penetrate tissue. In order for efficient penetration, the microneedle was designed to be very thin almost resembling a two

dimensional form to eliminate lateral stresses. The internal features of the microneedle contain the microfluidic reservoir, well, and channel. These design features would then yield options for certain fabrication techniques such as etching and bonding. The idea behind the fabrication of the microneedle was to have processing done on two different wafers then have them bonded them together. The top wafer would have etched features going all the way through the wafer exposing features of the bottom wafer. The bottom wafer consists of an etched microfluidic channel. The FLAMES fabricated from Boston University, would then be inserted into a via located near the tip of the microneedle. The via was designed based on the dimensions of the FLAMES devices. The outer areas contain the, microfluidic reservoir, port, and capillary tube shown in Figure 1.1.

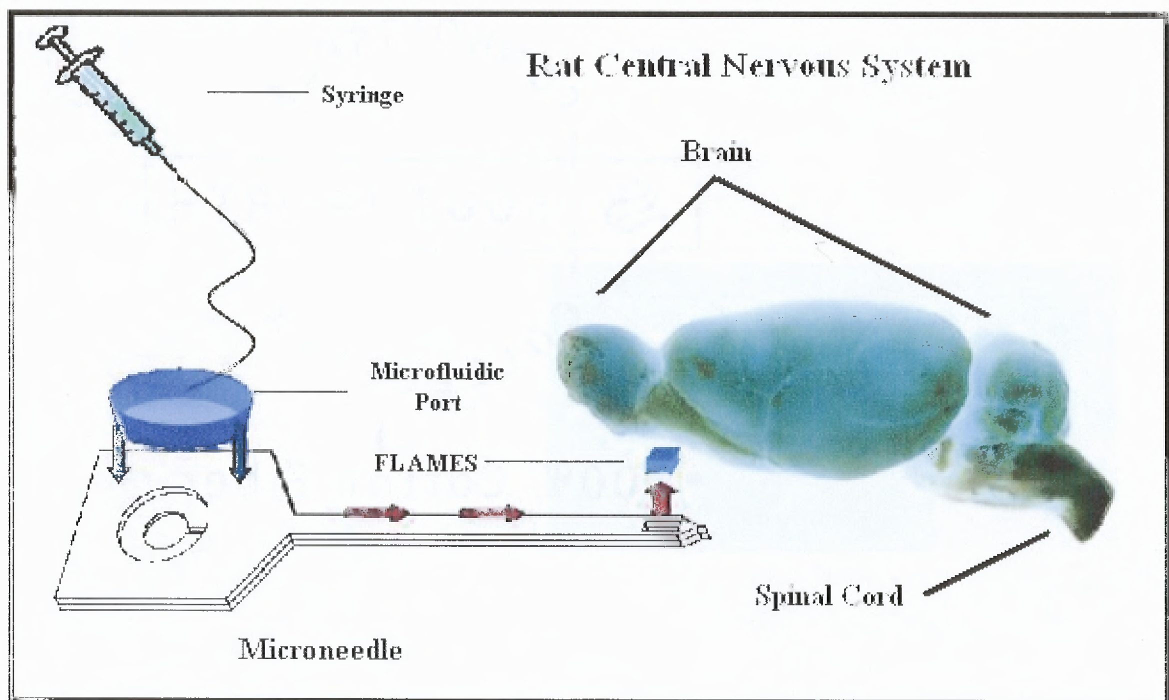


Figure 1.1 Flames implantation through microneedle.

The electrode delivery mechanism is powered by the fluid pressure at the inlet of the channel. When the microneedle is positioned in the desired area, a FLAMES device placed at the outlet of the channel will be pushed out. The flow through the microneedle

will be pressure driven through a syringe. The syringe is used to pump saline solution into the microfluidic reservoir then through the channel and into the well pushing out the stationary electrode. Figure 1.1 shows the penetration of the microneedle into the tissue, and delivering the FLAMES device. The FLAMES will then be used to stimulate neural tissue.

1.1.2 Microfabrication Development

The microfabrication methods used in creating a prototype for the device were made possible through the equipment at the MFC. There were certain characteristics of the design of the microneedle that had to be developed. The first of these characteristics was to have an area for the FLAMES to be inserted and firmly locked in place. An area would also need to be dedicated for the insertion of fluid to enter the microneedle in order for it to pump fluid through the channel. This led to the first fabrication need for the microneedle to have wholes or "vias" that would be designed to specifically fit the FLAMES devices and a microfluidic port. A second characteristic that the microneedle needed was a microchannel running inside the microneedle similar to a hypodermic needle. In terms of the fabrication, this characteristic meant that a fluidic channel needed to be etched and then be sealed in some way. The fabrication decision involved in the seal would be provided by a bond between wafers. A last characteristic the microneedle needed to possess would be to have the ability to penetrate living tissue in a way where there would be no threat in breaking the needle. This characteristic required the needle to be thin so it would eliminate lateral stresses applied from the tissue to the microneedle. Taking these characteristics into account, a general fabrication method was formed through testing and experimenting with different fabrication equipment at the MFC.

equipment at the MFC. Creating these special characteristics on the needle posed many different options for the fabrication.

There were many initial tests done before a well detailed set of microfabrication steps and procedures were implemented. There were several different fabrication options in creating the needed characteristics for the microneedle. The vias need to be etched, and the options of dry and wet etching had to be tested. Also, the bonding of the wafers could either be done by fusion or photoresist bonding. Considering the options, the bonding would be the more important factor as it would set the fabrication standard for the rest of the fabrication steps. Fusion bonding was to be experimented as the first option due to its reliability and tight seal. This option however, failed as the wafers separated in the dry etching chamber due to the low vacuum pressures. Fusion bonding would also need a more sensitive fabrication procedure consisting of more effective cleaning methods mainly because of the sensitivity of the surfaces being bonded. This would lead to the only other option in using photoresist as a bonding adhesive. An initial experiment of testing how well wet etching with potassium hydroxide (KOH) was also conducted and deemed too destructive as the chemical penetrated in between the wafers and also possibly finding a way into the channels. Dry etching was then the first option as it would also be effective and efficient when etching small features. These tests would lead to the first set fabrication methods of using dry etching and bonding the wafers with photoresist which would be much more effective in reliability and reproducibility. The other fabrication steps consisted of standard microfabrication methods used in many other common MEMS devices processed throughout the industry such as lithography, thin film deposition, and chemical cleaning.

1.1.3 Device Testing in Agarose Gel

After fabrication of the microneedle, the device was tested using agarose gel, imitating some conditions of living tissue. Agarose gel is an ideal and permeable medium in which a fluid can be pumped through the microchannel. This gel is widely used as a medium for microbiological experiments. Once the microneedle is submerged into the agarose gel, a syringe was then be connected through a capillary tube to the microfluidic port. Once the connection was made, a fluid such as a saline solution was then pumped through the port into the microchannel. Once the fluid reached the tip of the microneedle, the pressure should cause the FLAMES device to be released. This testing method will help simulate how the microneedle will function in living tissue so it can then be used in animal testing.

CHAPTER 2

BACKGROUND

2.1 MEMS Technology

Since the invention of the transistor at Bell Labs, technology has been leaning towards becoming smaller and simpler. The manufacturing process of IC's led to the creation of microelectronics. MEMS function on the micron level and contain electrical and mechanical components required for the functionality of their devices. Since, they are able to function on the micron level, MEMS provide many advantages. To understand the scale of a MEMS device, Figure 2.1 shows a MEMS microphone in comparison to a match stick. This technology then began to influence many other areas such as the medical, automotive, and telecommunications industries. BioMEMS are devices which use MEMS technology and are able to function in biological environments due to their functionality at the cellular and molecular levels.

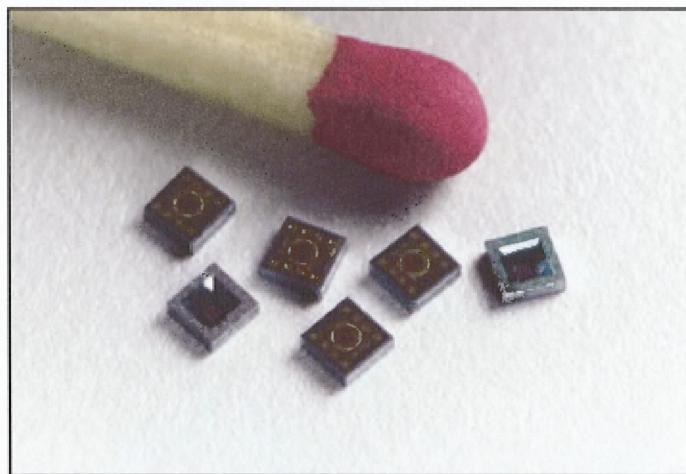


Figure 2.1 EmKay Sisonic Microphone [1].

2.2 Microfluidics

Microfluidics were first introduced in printers where they aided in the control and deposition of ink. The microfluidic industry has since expanded and has found itself being used in a variety of many different applications and more importantly biotechnology. In the biotechnology industry, the major contribution it has been successful in creating are the lab on a chip devices. These devices enable a single microchip to perform many different lab tasks at once. Microfluidic devices have at least one or more microchannels with at least one dimension less than one millimeter. Biological fluids which can be used in these devices include blood samples, bacterial cell suspensions, protein or antibody solutions, and various buffers [2]. In biotechnology, microfluidic devices have the functionality of measuring molecular diffusion coefficients, fluid viscosity, pH, chemical binding coefficients, and enzyme reaction kinetics [2]. They are also used in electrophoresis, cell separation, immunoassays, and DNA analysis [2]. The microfluidic channel feature fabricated in this microneedle however, is not intended for any of the previously mentioned measuring applications or common functions found in most microfluidic devices.

2.3 Neural Prosthetics

There is a wide spectrum of neural prostheses research being conducted in various areas of the sensory and motor components of the central nervous system. These neural systems contain visual and auditory prostheses, deep brain stimulation, bladder and bowel control and respiration. To understand the natural function of the nervous system, stimulation and recordings of neural signals must be fully observed to obtain consistent

results. By detecting and analyzing these results of the natural function of the nervous system, more advancements can be made in neural prostheses. Neural prosthetic devices help the nervous system by restoring the function lost due to injury or disease. Some common neural disorders that can benefit from neural prosthetics include Parkinson's disease, spinal cord injury, Lou Gehrig's disease, epilepsy, Tourette Syndrome, blindness, and hearing loss. One of the most popular and major neural prosthetics used today in auditory prostheses are cochlear implants, shown in Figure 2.2, which function by stimulating the auditory nerve via an implanted electrode array.

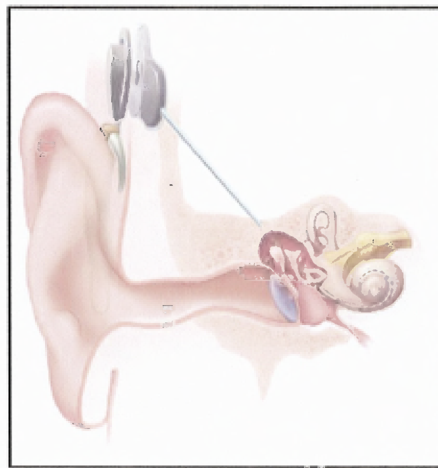


Figure 2.2 Cochlear Implant [3].

Stimulation of nerves is usually done through surface electrodes or electrodes implanted near the nerves. Implantation of electrodes with interconnects however, can pose many problems with the setup of the interconnections on the surface or in the tissue causing damage in some cases. A new concept in implantable micro stimulation is being investigated in our laboratory at NJIT, which eliminate interconnections, and can be activated for stimulation through light energy.

2.4 FLAMES

Implantable micro stimulation is a new approach to stimulating neural tissue and will allow for more efficient testing. Electrical stimulation of damaged neural tissue is being used in treating a number of neural disorders. Implantable microstimulators also eliminate the interconnects which are used in the much more traditional stimulation method with microelectrode arrays. A prototype of an implantable micro stimulator was designed through collaboration with Boston University and fabricated at Ecole Polytechnique Fédérale de Lausanne (EPFL) in Switzerland. This device is also known as a floating light activated micro electrical stimulator or FLAMES which are passive devices with multiple photodiodes connected in series and are able to provide neural stimulation by being activated with a beam of near infrared (NIR) light. Light penetrates into the neural tissue a few millimeters before reaching the FLAMES. Before in vivo testing of the FLAMES devices, the charge injection capacity of the contacts and light transmittance in neural tissue is currently being tested in our lab. The fabrication of the FLAMES was conducted by standard microfabrication techniques.

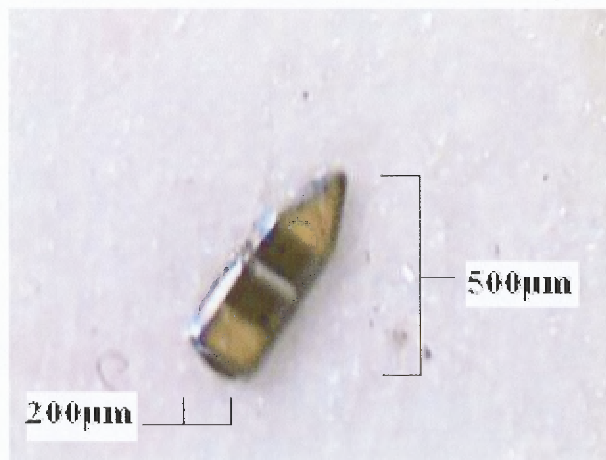


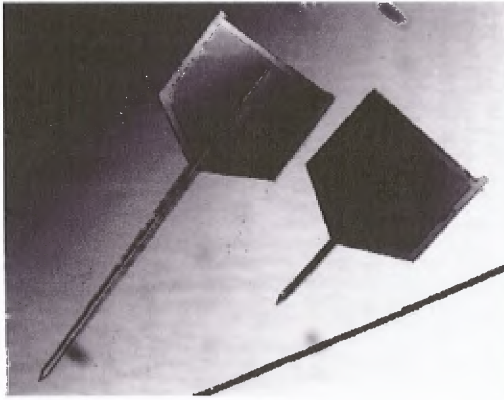
Figure 2.3 FLAMES device.

The FLAMES devices consisted of Aluminum metal contacts, a silicon dioxide

(SiO_2) layer, and vias that were etched for TiN sputtering. Ion implantation was used to form highly doped positive (p+) and highly doped negative (n+) contact regions. The total device size of the FLAMES measures 200x500μm while each contact was 60x200μm.

2.5 Microneedle Background

To create microneedles through MEMS fabrication, there are usually two types of needles that can be fabricated in different ways. These different styles are called "in plane" and "out of plane" microneedles. Both these types of needles give certain advantages and disadvantages, but are effective in different applications. In-plane needles are fabricated lying flat on the wafer, and are fabricated using dry etching. Out of plane needles protrude out of the wafer and are usually fabricated through chemical etching. These types of etching allow for high aspect ratio structures. There have been a few groups who have designed and fabricated microneedles with microfluidic ports. One such group at the University of Michigan, were able to fabricate a functional microneedle with microheaters and a fluidic port. The microfluidic channels in these microneedles were fabricated without wafer bonding and instead used sacrificial layers that were etched. The microneedles however, shown in Figure 2.4 were fabricated for the purpose of chemical analysis and fluid sampling.



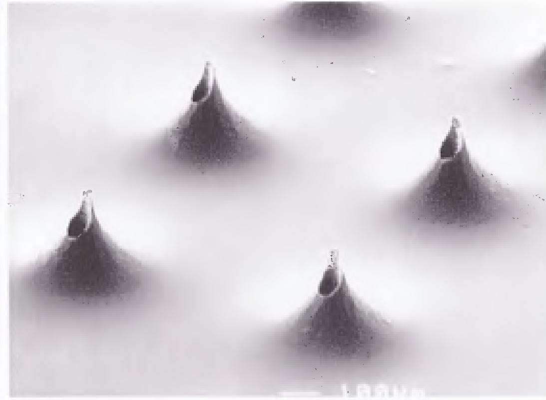


Figure 2.4 In-Plane Needles [4]. **Figure 2.5** Out-of-Plane Needles [5].

2.6 Dektak Profilometer

To get an accurate measurement of the etched features, a measuring instrument needed to be able to provide a surface profile of the wafers. The Dektak 2A profilometer was used to measure the etched features. The Dektak profilometer measures any vertical feature including photoresists, thin film depositions, and etched features in the range of 65μm to 200A°. The material or wafer to be measured is placed on a tray or stage underneath a stylus. A light and camera are also focused on the stage.

Figure 2.6 Dektak Profilometer.

The profilometer works by placing the diamond tipped stylus onto the surface of the material, and then moving it along the surface from the bottom to top or vice versa. The stylus is sensed by an linear variable differential transformer (LVDT) and then stored in the systems memory. The information is then shown as a line graph of the surface topology on a video screen.

2.6.1 Dektak Operation

To turn the Dektak on, a main power switch located on the back of the system was switched on. The "video" button was then pushed to display the different specifications. The wafer can be moved on the stage by two knobs underneath. The feature can then be seen on the video screen. The magnification of the camera can also be adjusted by twisting the lens if needed. Once the feature is clearly seen on the display it is ready to be scanned.

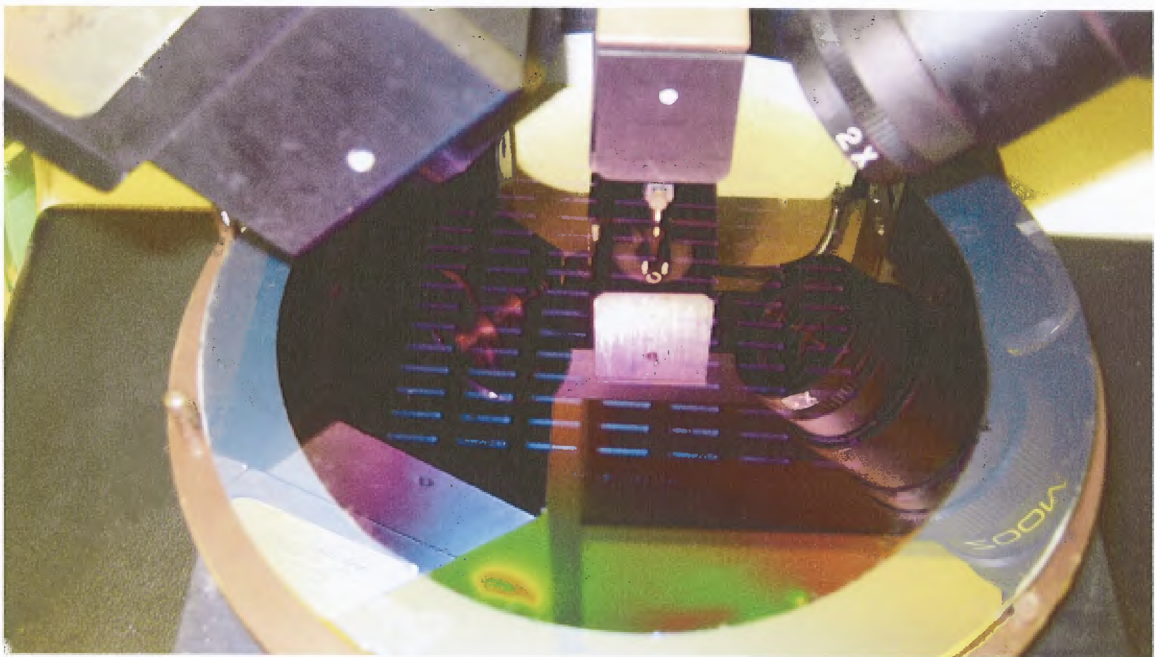


Figure 2.7 Close up of Dektak Profilometer showing stylus and camera.

The "stylus" button is then pushed to move the stylus down onto the etched feature. To

enter a program the "program" button is pushed and different parameters can be set. A scan length of 1mm was entered to move the stylus along the profile of the feature. After the chosen parameters are set, the "enter" button is pressed and the scan is ready to be performed. By pressing the "scan" button, the stylus moves along the surface profile. The "R" button activates the reference cursor and the "M" button activates the measuring cursor. Once both cursors are moved to the place on the graph where the surface is level, the "level" button is ready to be pressed, and makes the two points level. The "top" button is then pressed to enter a value for the top of the vertical axis. Once the plot is level and the vertical axis has the correct range, the thickness can then be measured by moving the M cursor to the highest peak of the graph.

CHAPTER 3

MODELING AND DESIGN FEATURES

3.1 Pro Engineer

To visualize and better understand the concept of the design of the device, a 3D model was constructed in Pro Engineer. There were two parts created then assembled together resembling the top and bottom wafers. The model was drawn to scale except for the thickness which was made a little thicker than actual for viewing purposes. Two files were constructed in Pro Engineer. The first part was the bottom wafer with the etched microfluidic features. The second part was the top wafer with the "vias". Vias are etched holes going through the entire wafer. Figure 3.1 shows both the wafers aligned but not yet bonded together.

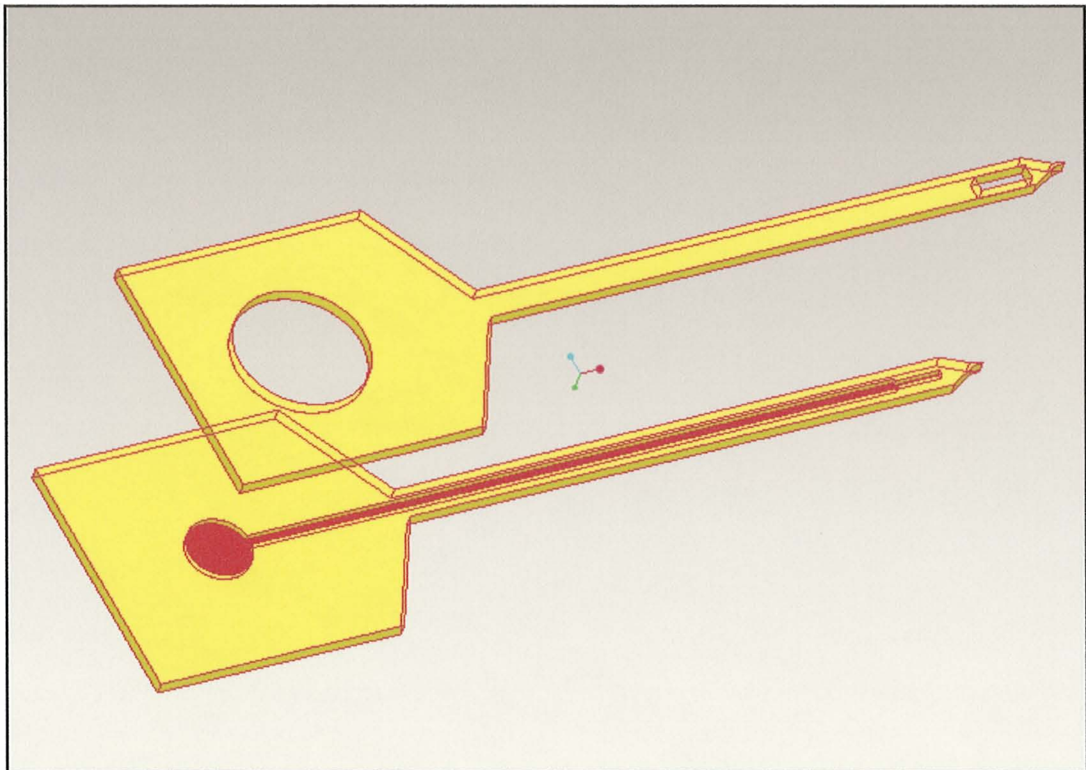


Figure 3.1 Microneedle shown before bonding.

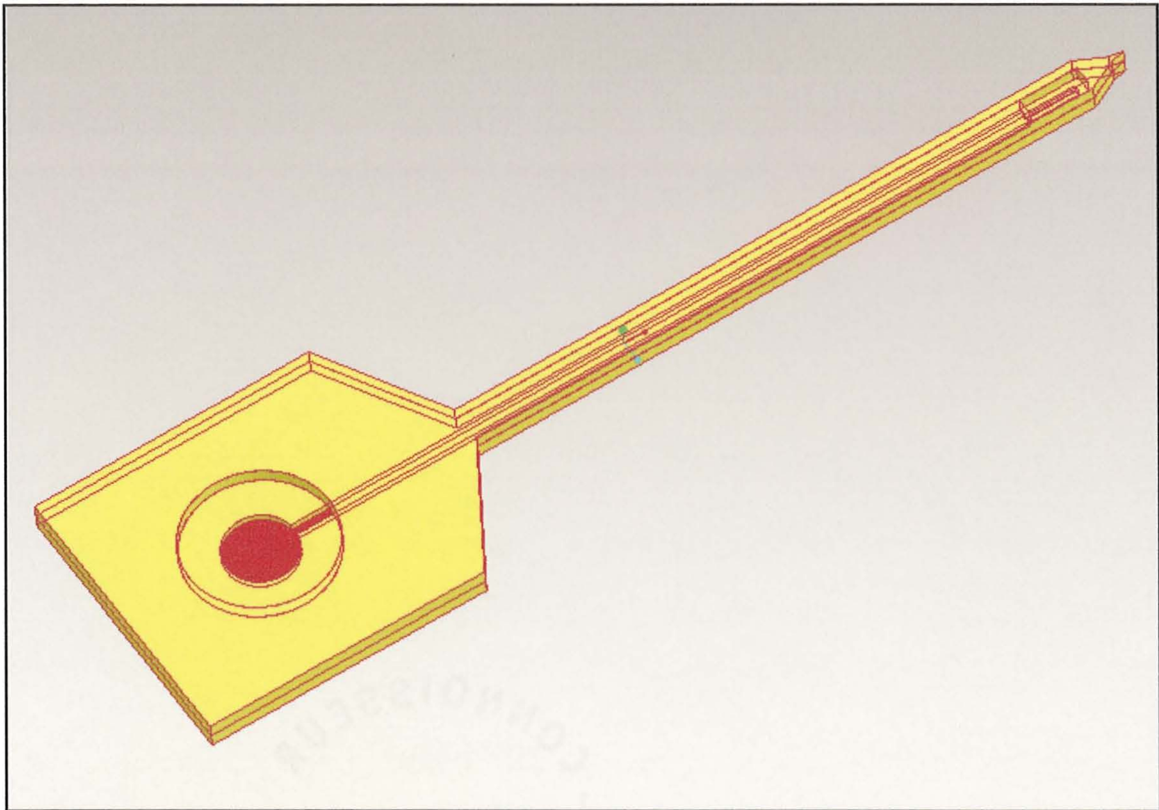


Figure 3.2 Bonded microneedle.

The first step into modeling the microneedle was done by the protrusion feature in Pro Engineer. An outline of the microneedle was sketched and then protruded to a certain thickness. The microfluidic features were created through the protruding feature. The second part was created to resemble the top wafer. This part was also a protrusion of the microneedle with the same dimensions of the first wafer. The top wafer needed etched features going directly through the wafer exposing features of the bottom wafer. To etch the features on this wafer the protrude cut was again used. An assembly file was then created to align and mate the two wafers together which is shown in Figure 3.2. A 3D model of the microneedle and thickness of 200μm total is shown in Figure 3.3.

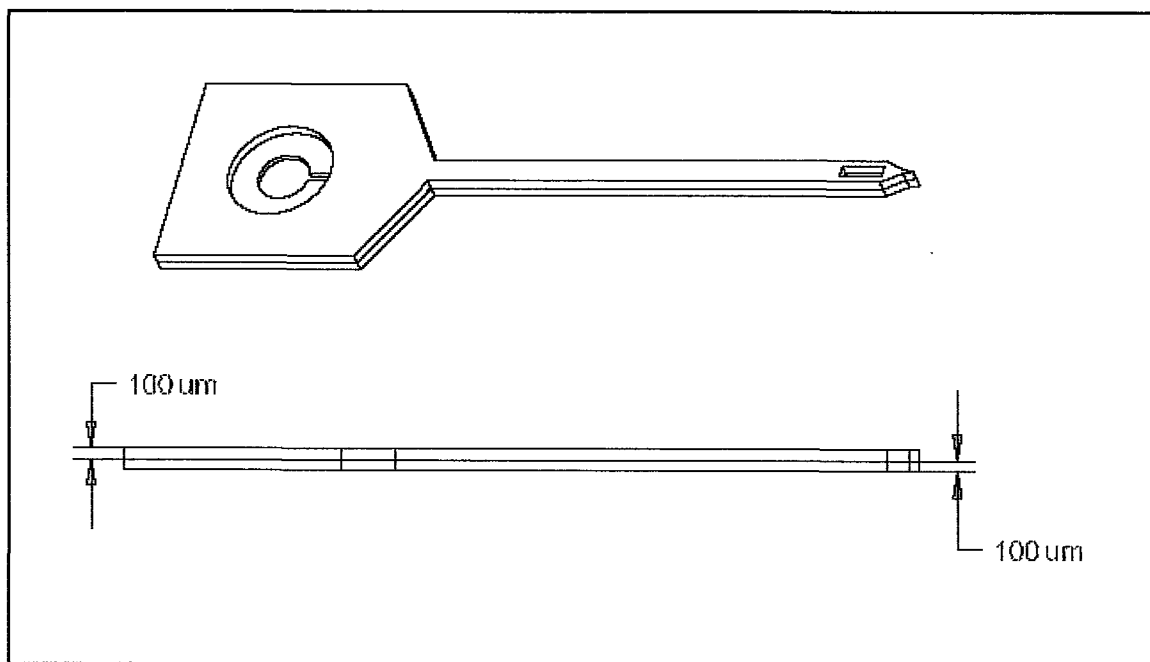


Figure 3.3 Microneedle side and top view.

3.2 AutoCAD

Computer aided design (CAD) is a technique that allows the design and drafting of mechanical parts. AutoCAD is a software program for 2D and 3D drafting. CAD is used to design almost everything from small simple machines to buildings, houses, and other structures. AutoCAD is used in many different disciplines such as architecture, civil, and mechanical engineering. AutoCAD's natural file format is in DWG, which is used for 2D and 3D design drafting.

The microneedles were first sketched using dimensions from a design drawn on paper. A device in the micron range was desired. There were seven different microneedle well sizes. The well on the top wafer had seven different sizes based on the FLAMES made at Boston University; therefore there were seven microneedle device designs. The different well sizes caused the dimensions of the channel width and end to be different in

all seven devices.

3.2.1 Microneedle Periphery

The periphery of the microneedle was first constructed on paper. The entire length of the microneedle was 7.3mm long. To penetrate the tissue a long thin needle-like protrusion was required. The back part or base of the microneedle was needed to serve as a holder, and also needed to be large enough to connect to the microfluidic connector. To penetrate the tissue efficiently the needle part of the device was designed to be 4.5mm long and 245μm thick. The base of the needle was designed to be 2mm in length and width, to serve as a stable holder for the 1x1um microfluidic connector.

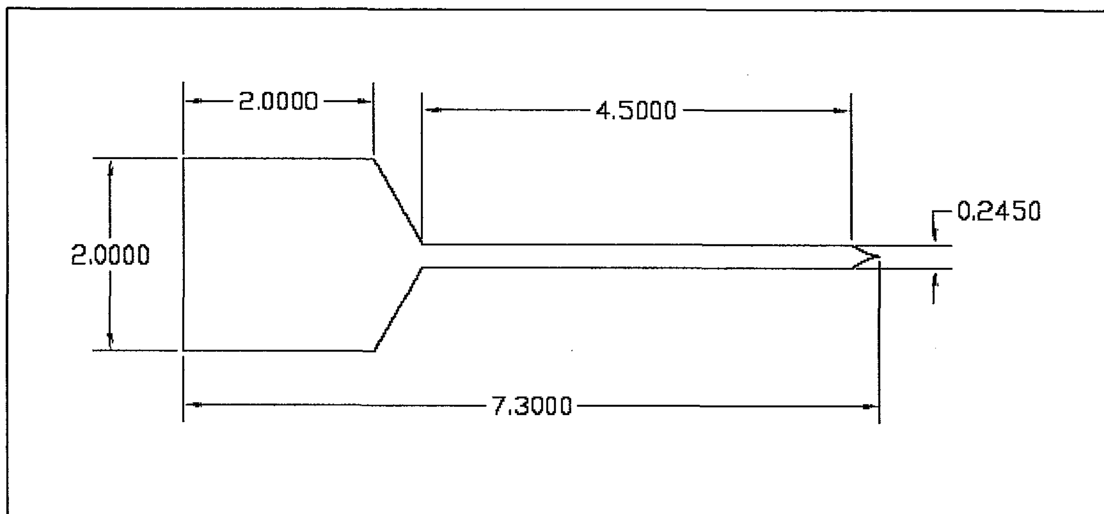


Figure 3.4 Microneedle dimensions in millimeters.

3.2.2 Microfluidic Features

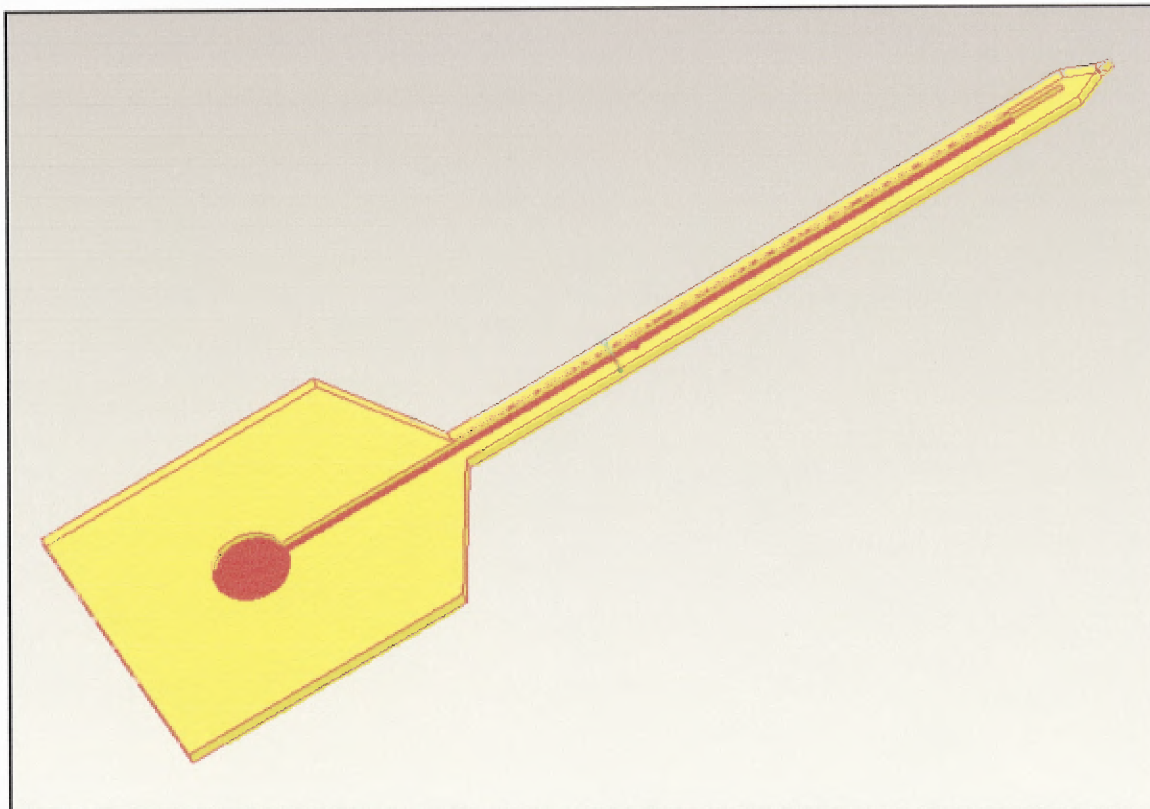


Figure 3.5 Microfluidic channel (bottom wafer).

Figure 3.5 shows the top view of the bottom wafer. The features shown are of the microfluidic reservoir, channel, and well. The microfluidic channel design was constructed to be centered through the microneedle. This microchannel extends out from the reservoir and ends near the tip. The dimensions are shown in Figure 3.5. The length of the channel is around 5.3 mm and 80 μm in width. There was no set length in the microchannel, as long as it connected the reservoir and extended out to the via near the tip. The width however was centered through the center of the needle and set to 30% of the width of the needle. The reservoir was set to around 500μm in diameter to accommodate the incoming fluid pumped in by the syringe. The microchannel becomes smaller in size near the tip. This was done to be centered underneath the well.

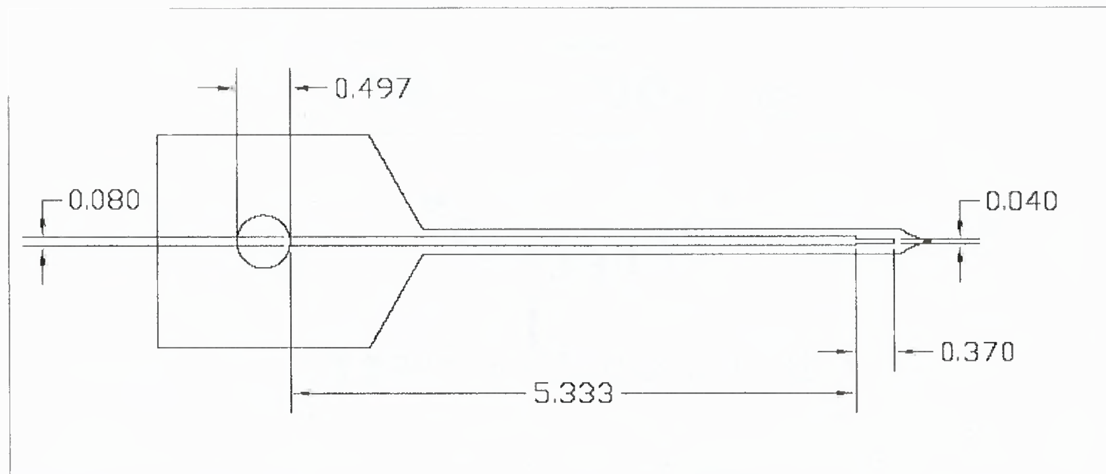


Figure 3.6 Microfluidic channel dimensions in millimeters.

3.2.3 Vias

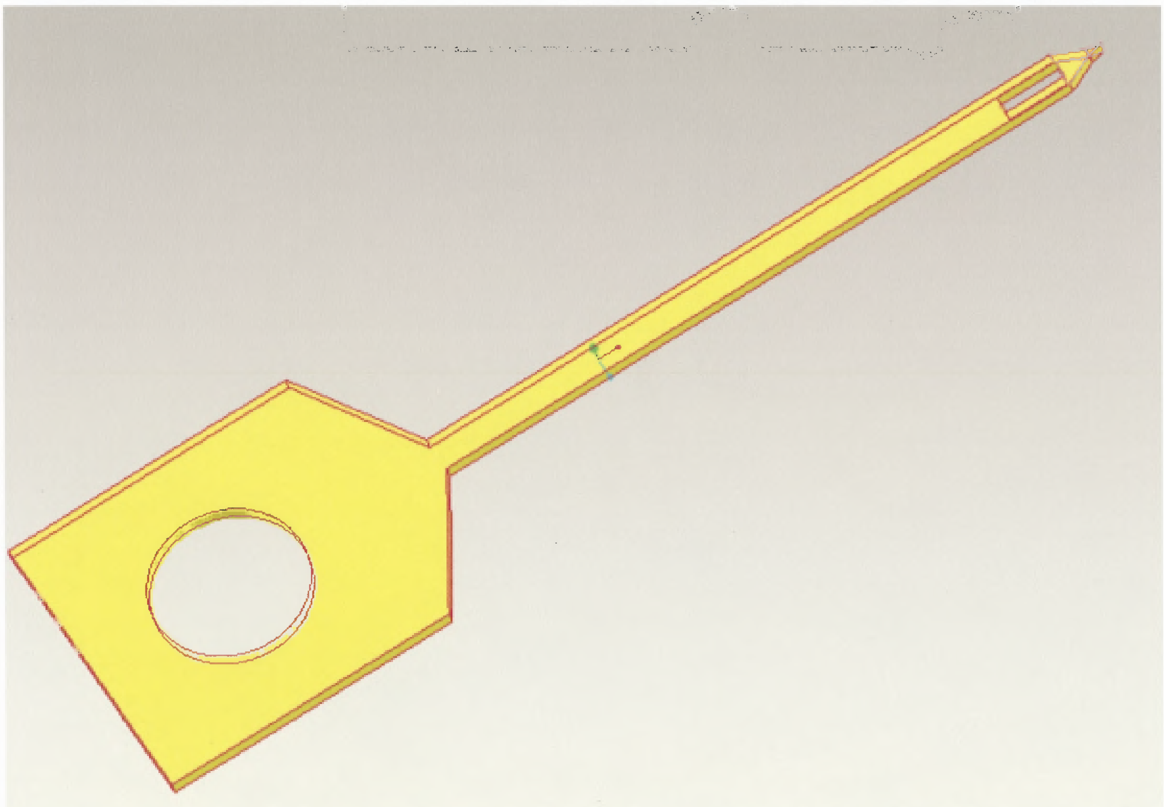


Figure 3.7 Vias (top wafer).

Vias were designed on the top wafer to expose some of the etched features in the wafer bonded below it. The via exposing the microfluidic reservoir was designed to be

1.02mm. This was designed to accommodate the microfluidic port which was 1mm in diameter. The via was designed to be a little bigger than the port so there would be a tight fit. The port had a diameter of 1mm and an inner diameter of 360μm.

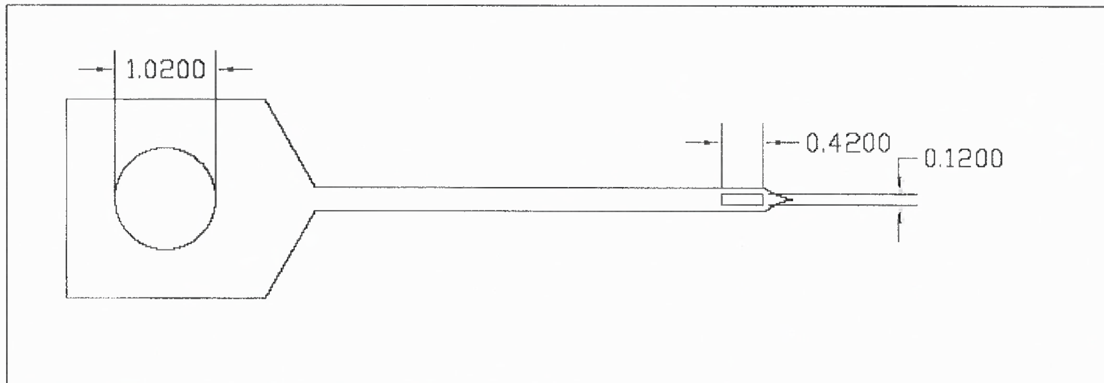


Figure 3.8 Vias dimensions in millimeters.

The via near the tip of the needle was designed to accommodate seven different FLAMES sizes. These sizes were calculated by allowing extra room for the electrodes to fit. The seven FLAMES sizes are shown in table 3.1. The well sizes were designed to be 10 to 20 microns bigger than the FLAMES sizes to create a tight fit.

Table 3.1 FLAMES and Well Sizes

# of Well Sizes	FLAMES Size (width (um) x length (um))	Well Size (width (um) x length (um))
1	419 x 90	420 x 120
2	470 x 90	470 x 120
3	626 x 220	630 x 250
4	838 x 220	840 x 250
5	929 x 220	930 x 250
6	565 x 90	565 x 120
7	1050 x 220	1050 x 250

CHAPTER 4

FABRICATION

4.1 Silicon Wafers

Silicon wafers were used as the substrate to fabricate the devices. Silicon is used in most integrated circuits and has essential properties that make it an ideal material to work with. Silicon is one of the best semiconductors. It also easily permits deposition of an oxide layer for dielectric surfaces. Biocompatibility of silicon is being tested in numerous studies currently with recent findings indicating that silicone causes minimal chronic response in neural tissue particularly if the surface is treated to have microfeatures [6].

4.1.1 Wafer Properties

The wafers used for the fabrication needed to meet certain criteria in order for the microneedles to function properly. The wafer diameter was to be of 4 inches, with a thickness of 100μm. The wafer thinning process is conducted by using a 2000 grit back grinding/polishing saw which thins the wafers and then polishes the backside. A common thinning procedure is shown below. During thinning, a wafer is placed on an abrasive material and a protective film and weight are placed on top. The abrasive then is spun and acts as a saw to grind down the wafer.

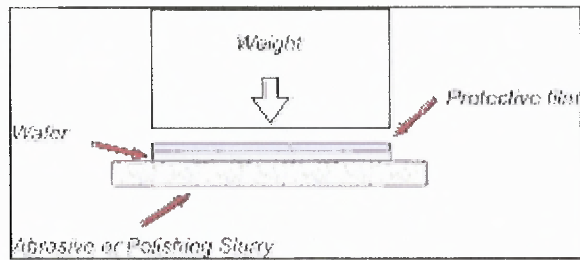


Figure 4.1 Common wafer thinning procedure [7].

The wafers also needed to have a high resistivity so we would be able to see through the wafer better using the infrared lamp. Seeing through the wafer allows for better alignment during procedures such as photolithography and bonding. A resistivity greater than 1ohm-cm^2 was needed so the wafers could be visible under infrared light. Low dopants give the wafer a higher resistivity. A standard orientation of <100> was preferred due to etching techniques used. The wafers were also needed to be double side polished (dsp) so there would be no rough surfaces to deal with. Rough surfaces will not allow wafers to bond or be patterned on. A layer of silicon nitride of 2000A° was also needed to be used as a mask for the etching.

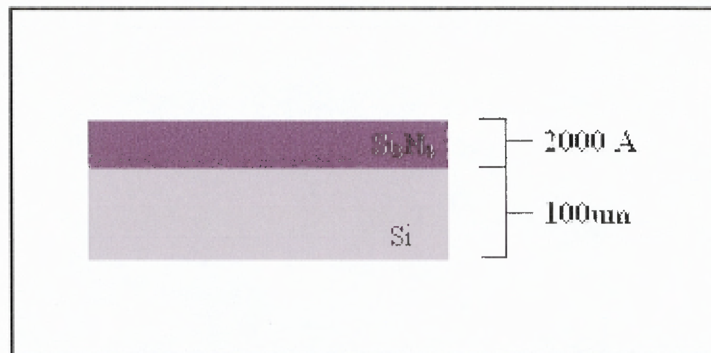


Figure 4.2 Wafer with silicon nitride layer.

4.1.2 Thin Wafer Handling

The wafers used in this project were very thin and thus fragile. The wafers had to be handled very carefully in order to reduce the risk of cracking, chipping, or breaking. Any surfaces the wafers were placed on was also a key issue, because of the risk of the wafers sticking to the surface due to the surface properties and small wafer thickness. The wafers also had to be handled with only Teflon tweezers because any other material such as steel would easily crack or break the wafers.

4.2 Cleaning

Cleaning is an essential part of microfabrication because each process requires clean surfaces in order for the reactions and other phenomena occurring on the surface to be successful. Cleaning of the wafers was conducted with standard cleaning procedures. Although the clean room minimizes airborne particles, there are always contaminants in gasses used in many of the fabrication tools. Contaminants can be in the form of particles, metals, organics, inorganics, native oxides, and micro roughness [8]. Drying methods are also a very important step in cleaning. Some common methods used are lamp drying, vacuum drying, nitrogen blowing, and spinning. All these methods are effective but also have special requirements.

Cleaning was mandatory before each photolithography, and bonding step because these processes are very sensitive to particle contamination. The cleaning methods used were either Piranha, NMP, or M Pyrol clean. These cleaning methods are used in order to remove substances from the wafer. The only drying method used to dry the wafers of this project was to gently waive a nitrogen gun around the wafer since the wafers were very

thin. Any type of rotational movement or spin drying would cause the wafers to break. The wafers had to be dried off carefully and slowly with a nitrogen gun.

4.2.1 NMP-Clean

N-methyl-2-pyrrolidone (NMP) is a chemical compound with low toxicity which can be mixed with water and solvents such as ethyl acetate, chloroform, and benzene [9]. NMP is also used in cleaning to remove organic material from the surfaces of substrates. In many cleaning procedures, a type of NMP clean is usually the first cleaning step used on unprocessed silicon wafers.

Before beginning the NMP-Clean procedure, wearing a face mask and rubber gloves were required as a precautionary. A wet chemical bench produced by JST Custom Fabrication Inc. housed various baths. For the NMP clean, the secondary bath, and a deionized water bath were used. The primary bath serves as a photoresist stripper while the secondary bath is used for new wafers. The wafers were placed into a Teflon wafer tray with handle. The wafers were then submerged into the secondary bath containing the NMP solution for 10 minutes. Next, the wafers were taken out and dumped into a deionized water bath for another 10 minutes. The de-ionized overflow switch was turned on so the water could circulate in the bath. The wafers were then dried very carefully using a nitrogen gun at low speeds. To ensure complete dryness the wafers were then taken to the oven for and placed in a quartz tray for 5 minutes at a temperature of 85°C.

4.2.2 Piranha-Clean

A Piranha-clean (P-clean) was performed to remove any remaining particles off the wafer. This is a more thorough clean and is used before bonding. The P-Clean station was a similar setup to the NMP clean station. The station consisted of a wet chemical bench provided by JST Custom Fabrication Inc. The wafers were placed in a Teflon tray and then submerged in the Sulfuric Acid (H_2SO_4) bath at 110 °C for 10 minutes. The wafers were then dumped in a 65°C circulating hot de-ionized water bath for 10 minutes. The final step was to submerge the wafers in cold de-ionized water at room temperature for 10 minutes.

4.2.3 M-Pyrol Clean

1-Methyl-2-Pyrrolidone (M-Pyrol) is another chemical compound used in cleaning silicon wafers. This type of clean is performed commonly when removing photoresist. It also has applications in polymerization mediums, latex paints, industrial cleaning, and solvents [10].

The M-Pyrol clean was used to strip away photoresist. The M-Pyrol clean was performed at the same wet bench as the NMP-Clean. The first step in the M-Pyrol clean was to place the wafers into the Teflon tray and dip them in the primary bath at 95°C for 10 minutes. The next step was to submerge the wafers in a secondary bath at 95°C for 10 minutes. The final step was to rinse the wafers in a circulating cold de-ionized water bath for 10 minutes. The wafers were then dried with a nitrogen gun slowly and carefully.

4.3 LPCVD

Chemical Vapor Deposition (CVD) is a process in which a film of solid material is deposited onto the substrate made possible by a chemical reaction which takes place during the deposition. Low Pressure Chemical Vapor Deposition (LPCVD) is a type of Chemical Vapor Deposition usually done inside a chamber or reactor kept at vacuum that uses gasses at low pressures to form a thin film on the substrate. In a basic LPCVD setup a quartz tube is heated by a furnace while gasses enter the chamber and exit through the opposite side, shown in Figure 4.3 [11].

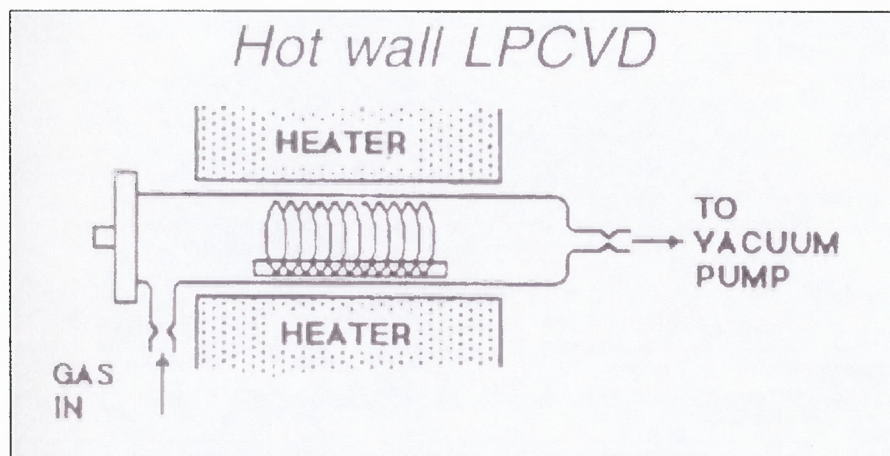


Figure 4.3 Common LPCVD system [11].

Adsorption occurs when the gasses flow to the surface of the wafers causing a reaction. The temperature causes the deposition rate to vary. The temperatures involved in LPCVD can be anywhere from 550 to 900°C and set at pressures of 1 to 8 torr [11]. Some of the advantages LPCVD has over other thin film deposition methods is that it deposits the film at a high uniformity and can also house many wafers at a time. The weaknesses of LPCVD are the high temperature conditions and deposition rates which can cause problems to the features already processed.

4.3.1 Silicon Nitride

Silicon Nitride (Si_3N_4) is used in many MEMS fabrication processes especially for its masking abilities for wet and deep etchants. It can also provide a barrier to diffusion of water and has effective resistance to oxidation [12]. When using LPCVD, silicon nitride is usually deposited at temperatures between 700 and 800°C. Since the fabrication of the microneedles was going to need deep reactive ion etching, silicon nitride served as a suitable mask. To make the fabrication process less time consuming wafers with a 2000A° layer of silicon nitride were ordered. University Wafer used LPCVD and deposited a stoichiometric silicon nitride. Stoichiometric silicon nitride is made from the two gasses, dichlorosilane and ammonia, which is the reaction shown in the equation below. The dichlorosilane helps the thin film to be more uniform. In LPCVD, the gas flows are in a 10:1 ammonia to dichlorosilane ratio [11].

$$3SiCl_2H_2 + 4NH_3 \longrightarrow Si_3N_4 + 6HCl + 6H_2$$

4.4 Photolithography

Photolithography is a patterning process in which a desired pattern can be imprinted onto silicon substrates or polymers. In most photolithographic systems, the substrate is placed on a plate locked into position. A mask is placed above the substrate and then ultra violet light is exposed.

4.4.1 Photoresist Application

The patterning is formed by a polymer coating called a photoresist, which is deposited on top of the surface of the substrate. A photoresist is an organic polymer and ultra violet sensitive material that is applied to the surface of the substrate. Positive and negative

photoresists are the two different types of photoresists that react differently to ultra-violet exposure. Positive photoresist which is exposed to ultra violet light becomes soluble to photoresist developer, while negative photoresist which is exposed to ultraviolet light is insoluble to photoresist developer [13]. The photoresist is usually spin coated onto the substrate by a spinner. A varied amount of photoresist is deposited in the center of the substrate. The spinner is then started and usually set to speeds of 1500 to 8000 rotations per minute (rpm) depending on the film thickness desired [11]. The centrifugal forces cause the photoresist to spread evenly onto the surface. The substrate is then usually heated or soft baked at about 75°C to 100°C on a hot plate, to harden the viscous form of the polymer.

There were two different photoresists tested during the fabrication, the Shipley S1813 which is a 2μm thin photoresist and the 5μm AZ P 4620 thick photoresist made by Clarient Co. The thin photoresist was first used to see how it would hold up in deep reactive ion etching. The photoresist would wear away after two to three hours of etching. Due to this complication the AZ P 4620 thick photoresist was used instead.

The Shipley S1813 positive thin photoresist was tested first. Once the wafers were dried in the oven, they would be ready for photoresist application and placed on the spinner. Wafers are put on the spinner then locked into position by a vacuum from underneath so the wafer will not spin off the spinner. During the first trial, the center of the wafer was sucked into the spinner resembling an indentation. Due to the thin size of the wafer, it was also feared the wafer could break from the high vacuum force of the spinner. This was an unexpected problem, and to have good patterning done on the wafer, the photoresist needs to be spread evenly across the wafer. To overcome this problem an

idea to reduce the force of the vacuum was implemented by placing some porous material in between the wafer and spinner. To reduce the vacuum force on the spinner, cotton chemical wipes were cut in the shape of the wafer and placed on top of the spinner, creating a filter effect and reducing the vacuum force. A foot pedal was used to switch on the spinner and stay on for one minute. The speed of the spinner was controlled by a rotating dial. Once the wafer lay flat on the spinner, five drops of HMDS (Hexamethyldisilazane) primer were applied onto the center of the wafer for 1 minute at 800rpm's. HMDS priming aids in giving the photoresist better adhesion. Next, a dropper with a large bulb was used to extract the photoresist from its container and then squeezed to release the S1813 photoresist on the center of the wafer. The amount of photoresist applied was required to cover a three inch diameter on the wafer. The spinner was spun for a minute at 2000rpm's. Once the spinner stopped, a uniform 2μm layer of photoresist was formed on the surface of the wafer. A soft bake was then performed at 115°C for five minutes, to further improve the adhesion of the photoresist. The wafer was placed into a quartz boat and loaded into the oven for five minutes.

During the second trial of the fabrication, the AZ P 4620 thick positive photoresist was tested. Similar to the application of the thin photoresist, five drops of HMDS primer were applied onto the center of the wafer for one minute at 800rpm's. Next, the thick photoresist was applied from a plastic dropper and squeezed to release the AZ P 4620 photoresist on the center of the wafer. The amount of photoresist applied covered around three inches of the wafer. The spinner was spun for a minute at 3000rpm, a much faster rate than the thin photoresist. Once the spinner stopped, a uniform 5μm layer of photoresist was formed on the surface of the wafer. A soft bake was then performed at

110°C for 25 minutes.

4.4.2 Photomasks

A photomask is designed to pattern the desired imprint onto the substrate. Ultraviolet light penetrates the clear areas of the mask and is blocked out by the dark areas. This is how a dark field mask functions, while a light field mask has the opposite effect. A photomask can be made out of glass, quartz, or even a transparency. Photomasks are usually designed using AutoCAD or other design software. Many companies are able to produce these photomasks using high resolution laser printing. The photomasks were constructed using AutoCAD 2005 software. Creating these photomasks are the first steps in designing many MEMS devices. In the initial design phases, sketches made on paper were copied into AutoCAD. Every feature of the device was copied and divided into layers. The layers were also made to divide the device into different masks. There were three masks needed for the microfluidic channel, the vias, and the periphery of the microneedle.

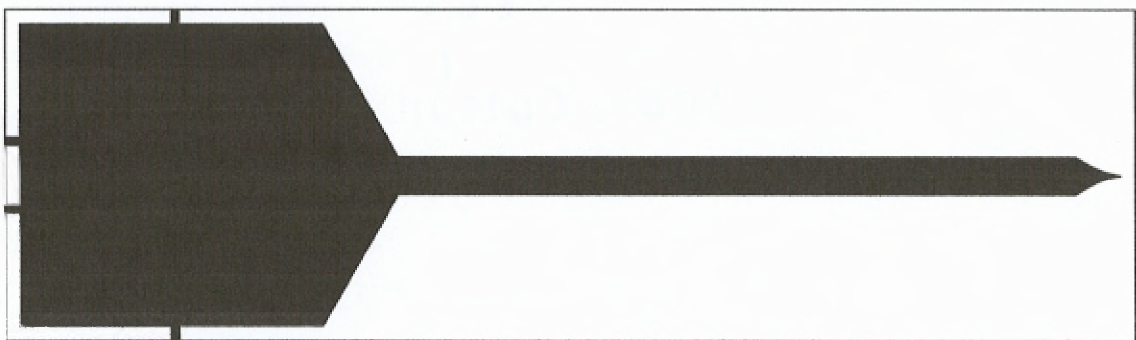


Figure 4.4 Feature patterned in the mask to edge the microneedle periphery.

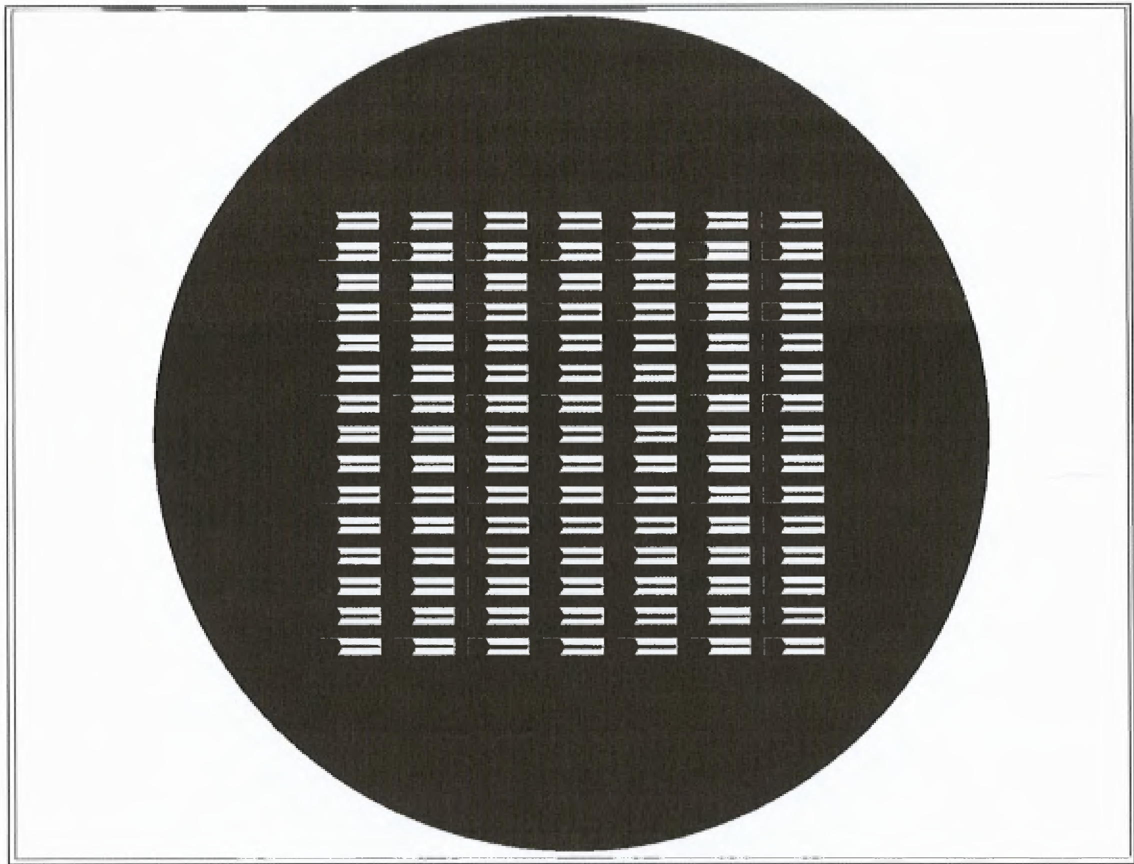


Figure 4.5 Photomask of needle periphery.

Figure 4.6 Microfluidic channel feature centered in microneedle.

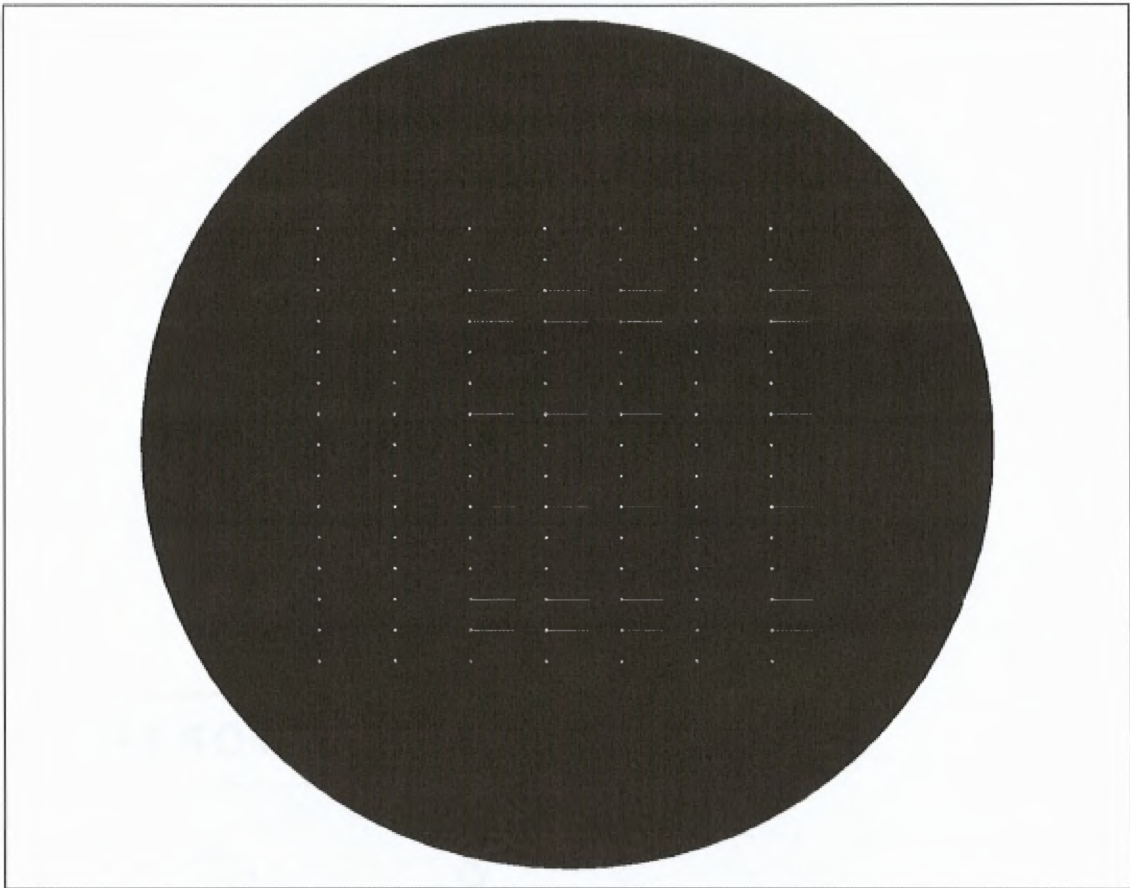


Figure 4.7 Photomask of microfluidic channel.

Figure 4.8 Vias shown in microneedle.

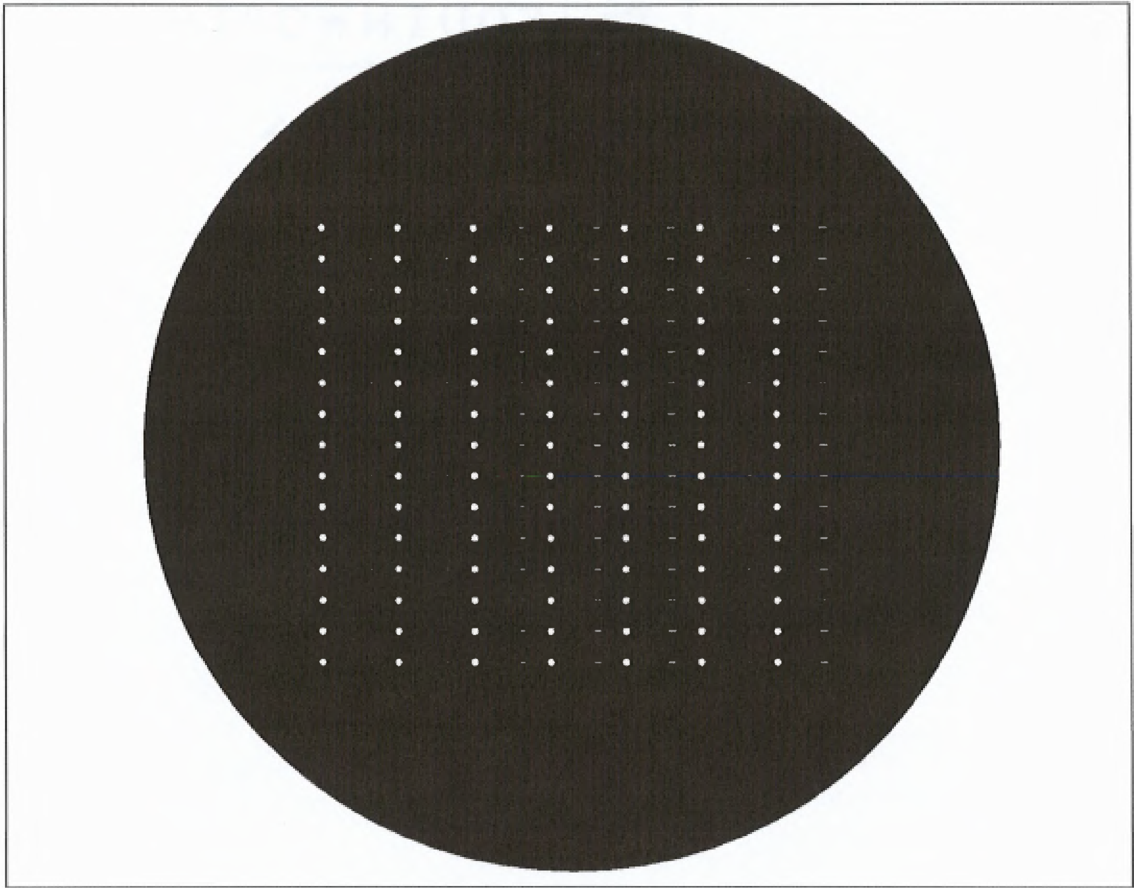


Figure 4.9 Photomask of vias.

After the designs of the devices were created in AutoCAD, the DWG files were vendored out to FineLine Imaging. FineLine Imaging printed out the DWG files onto transparencies. The resolution of each mask was 16,256 dots per inch (dpi) or around 1.56μm.

4.4.3 Mask Alignment

Mask alignment is a crucial step in photolithography. The photomask must be aligned with the wafer in the correct area so the pattern is imprinted onto the wafer. The photomasks involved in the later steps must be aligned to the pattern of the photomask that precedes it. Ultimately, all the photomasks should be aligned with each other to

obtain the desired features. The mask aligner used in the clean room was a Karl Suss MA6 mask aligner. This type of mask aligner uses ignition voltages of 30kV and operating voltages of 180V, with currents of 5 to 30amps [14].

Figure 4.10 Alignment Marks.

Due to some complications with mask alignment, a brute force method was conducted. This method comprised of aligning the mask with the wafer using the infrared lamp which is mostly used to see through wafers. First, the mask was placed face down on the tray. Next, the wafer was carefully placed on top of the mask, and then visually aligned by viewing the monitor. The wafer was then taped at two ends to prevent it from moving, with the photoresist side lying down on the mask. The mask and taped wafer were then loaded into the photolithography system for patterning.

4.4.3 UV Exposure and Development

The photolithography system in our clean room contains a Mercury Xenon lamp. This lamp produces ultraviolet light which is exposed to the mask and onto the photoresist. In most photolithographic systems there are three primary ways of exposure which are contact, primary, and projection. Each of these methods differs in how far away the masks are to the wafer or how the light is projected from the mask to the wafer.

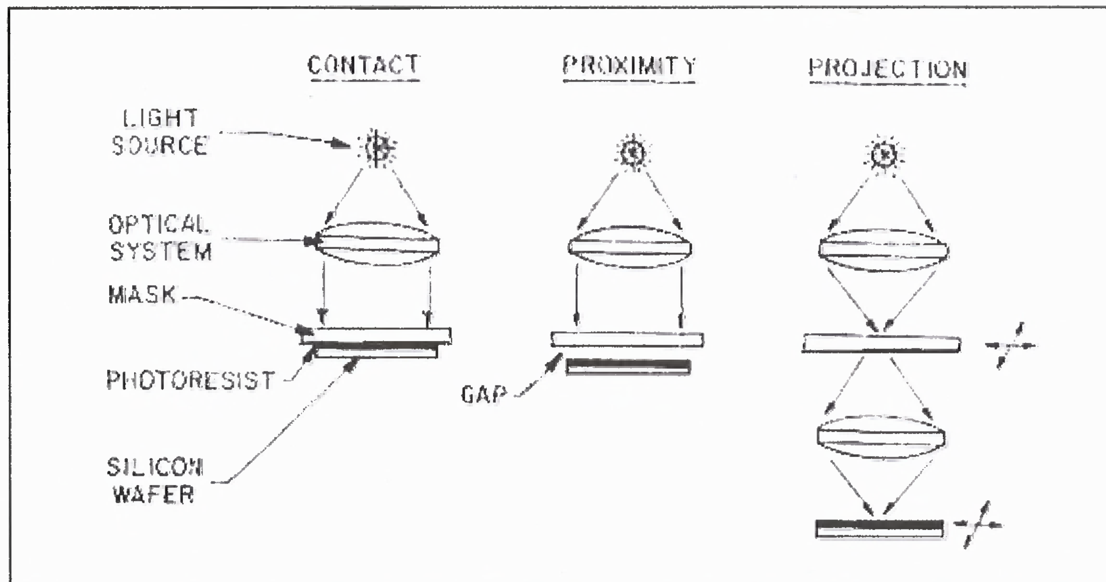


Figure 4.11 UV exposure methods [13].

The ultraviolet exposed areas of the photoresist become soluble to photoresist developer such as acetone. Solubility of the photoresist depends on a threshold or exposure energy. For positive photoresists higher exposure energy will cause the photoresist to become completely soluble [11]. At the end of most photolithographic procedures, a hard bake is performed at about 105°C.

The clean room houses a contact exposure photolithography system. After UV exposure, the wafer was then placed in a Petri dish to be prepared for development. Shipley Microposit MF319 developer was used because of its unique formulation designed to work with S1813 photoresist. The MF319 was poured onto the wafer to remove the UV exposed photoresist. Development time was around two minutes, and then the solution was emptied into a waste container. Deionized water was then poured into the Petri dish to rinse the wafer. The contents in the Petri dish were dumped into a special waste container for the MF319 developer. The wafer was then dried by a nitrogen gun. For the thick AZ P 4620 photoresist, the developer used was the AZ 400. This

developer however was needed to be diluted with water before pouring onto the wafer. Development time was around two minutes.

4.5 Etching

Etching is usually performed on substrates which have had photolithography and other additive processes completed on them. Etching is a subtractive process that removes material from the substrate by wet or dry techniques. Etching can be isotropic or anisotropic. Isotropic etching removes material in all directions while anisotropic etching etches material at certain angles. Wet etching involves the substrate to be immersed in certain types of chemical solutions at varied temperatures. Some common wet etchants used in microfabrication are Potassium Hydroxide (KOH), Hydrofluoric Acid (HF), and Phosphoric Acid (H_3PO_4). Reactive Ion Etching (RIE) and Deep Reactive Ion Etching (DRIE) were the two types of dry etching used in the design of the microneedles.

4.5.1 Reactive Ion Etching

RIE is a type of dry etching that uses plasma to remove material from a substrate. The plasma is created under a low pressure in a vacuum chamber. Varied amounts of gasses depending on the etch process enter thru the top of the chamber and exit thru the bottom. The etch setup is shown in Figure 4.12. A radio frequency (RF) electromagnetic field is then formed by two electrodes which direct ions toward the surface of the substrate shown below.

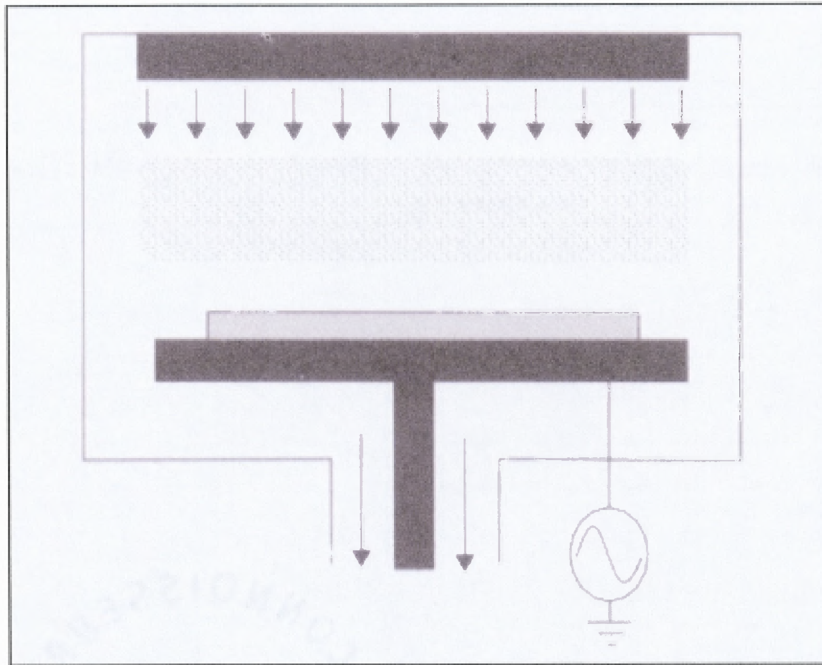


Figure 4.12 Typical Reactive Ion Etching Chamber [8].

At a few hundred watts, an electric field is set at a standard of 13.56 megahertz and creates a plasma by ionizing the gas molecules that are stripped of electrons which accelerate up and down in the chamber [8]. These electrodes are placed on the top and bottom of the vacuum chamber with the substrate being placed in between shown above. The system used in the clean room was a Phantom Minilock Reactive Ion Etcher manufactured by Trion Technology. The parameters set for etching were the amount of gas, pressure, and RF power. The recipe used is shown below in table 4.1.

Table 4.1 Recipe for Reactive Ion Etching of Silicon Nitride

Surface Etched	Total Time(min)	Gas Amount (sccm)	Pressure (mTorr)	RF Power (W)	Etch Rate (Å/min)
Si_3N_4	2	$SF_6 = 50$	250	100	1000
Si_3N_4	1	$CF_4 = 50$	300	200	1000

4.5.2 Deep Reactive Ion Etching

Another type of reactive ion etching, which is also becoming more popular in the IC industry is DRIE. This type of etching is able to etch thru hundreds of microns and form high aspect ratio structures. The main difference between DRIE and RIE is that in DRIE two different gasses are used in the chamber. The etch process used in DRIE produces an anisotropic etch shown in Figure 4.13. This process stems from a technology known as the "Bosch Process" [15]. In DRIE, a polymer is created on the surface of the substrate while the second gas etches the substrate. The polymer etches away slowly while also protecting the side walls from being etched [15]. This process creates high aspect ratio structures and also higher etch rates.

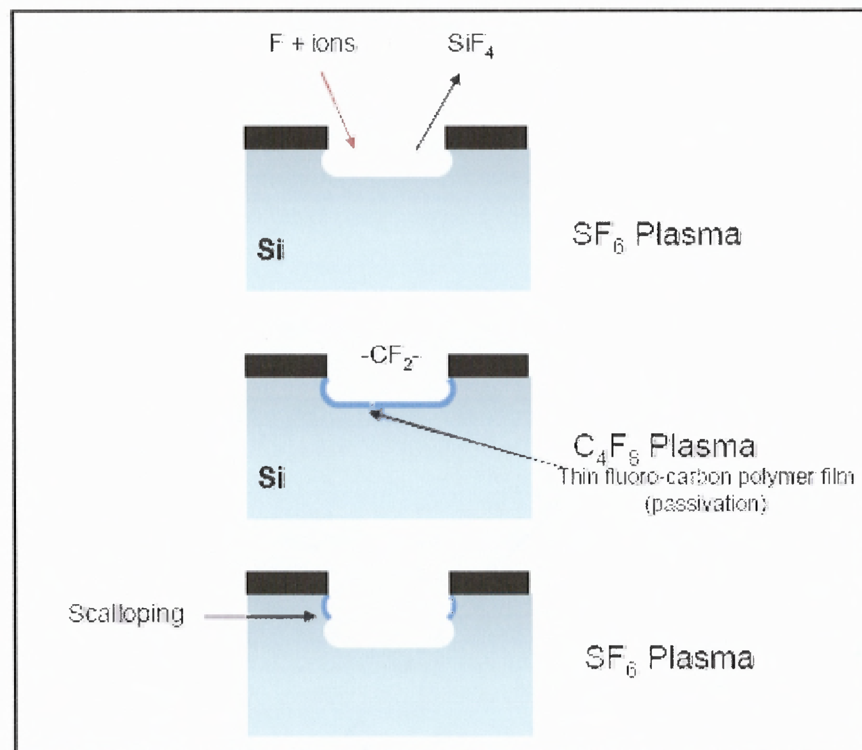


Figure 4.13 DRIE anisotropic etch profile [16].

In the clean room, deep reactive ion etching was used to deep etch silicon. It was used to create the microfluidic channel, the vias, and to etch out the periphery of the

wafer. The deep reactive ion etcher model that was used was the SLR series and was manufactured by PlasmaTherm. It is a two chamber deep reactive ion etcher in which one chamber at vacuum is used for loading and transferring to the other chamber for an even lower pressure at vacuum. The first chamber has a regular pump which levels the pressure to 1mtorr, while the second chamber has a turbo pump that reduces the pressure to even lower values (10e-4mtorr). Octafluorocyclobutane (C_4F_8), Sulfur Hexafluoride (SF_6), Argon (Ar), Nitrogen (N_2), Helium (He), and Oxygen (O_2) gasses were used inside the high vacuum chamber. Each gas played a role in the etching process. Argon was used to provide continuous cleaning of the surface during etching. Helium and liquid nitrogen were used to cool the wafer, chamber, and walls. SF_6 initially etches the silicon and then C_4F_8 forms a polymer layer on the etched surface. O_2 removes the polymer layer and then SF_6 etches the silicon, while the N_2 gas purges and keeps the gasses flowing repeating in loops forming an anisotropic etch profile shown above. After the gasses and pumps were turned on, the patterned wafer was loaded into the first chamber and the recipe for the DRIE process was entered. The chamber was then closed, and a vacuum was applied. Finally, the wafer was transferred to the second chamber via a robotic lever where etching started. In a previous trial, the recipe was set to 150 loops, where it took 16 seconds for each loop. This trial took around 40 minutes, and etched around 20μm of silicon giving an etch rate of 0.5μm/min. An etch depth of around 50μm was desired so the program was set to 300 loops that gave an etch depth of around 40μm.

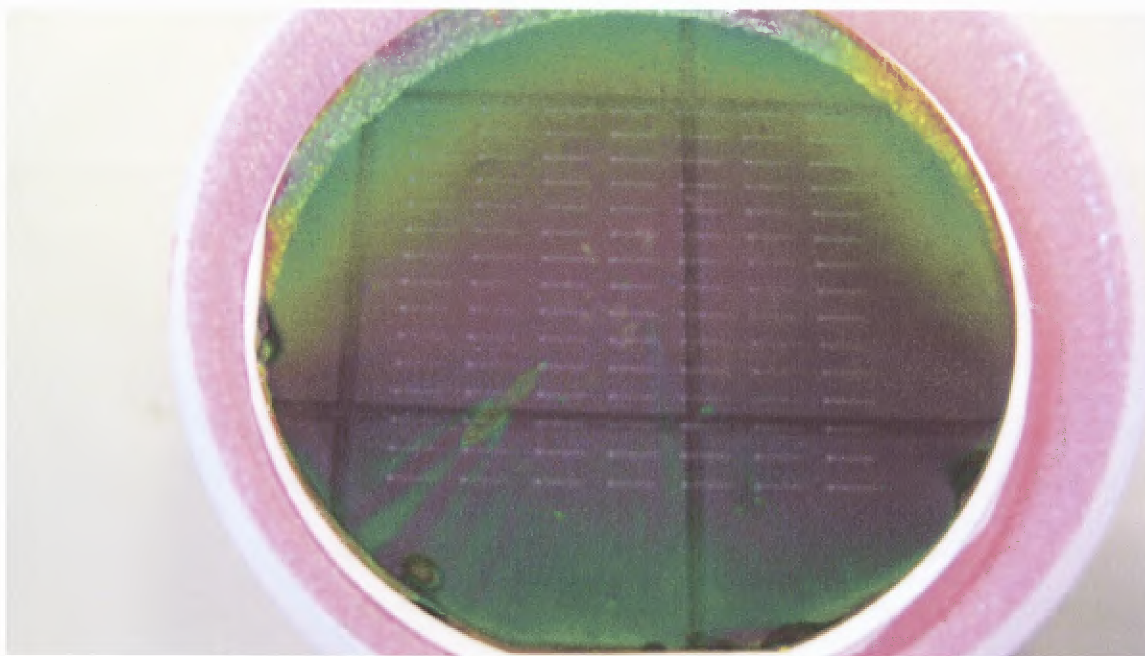


Figure 4.14 DRIE anisotropic etch of the microfluidic channels.

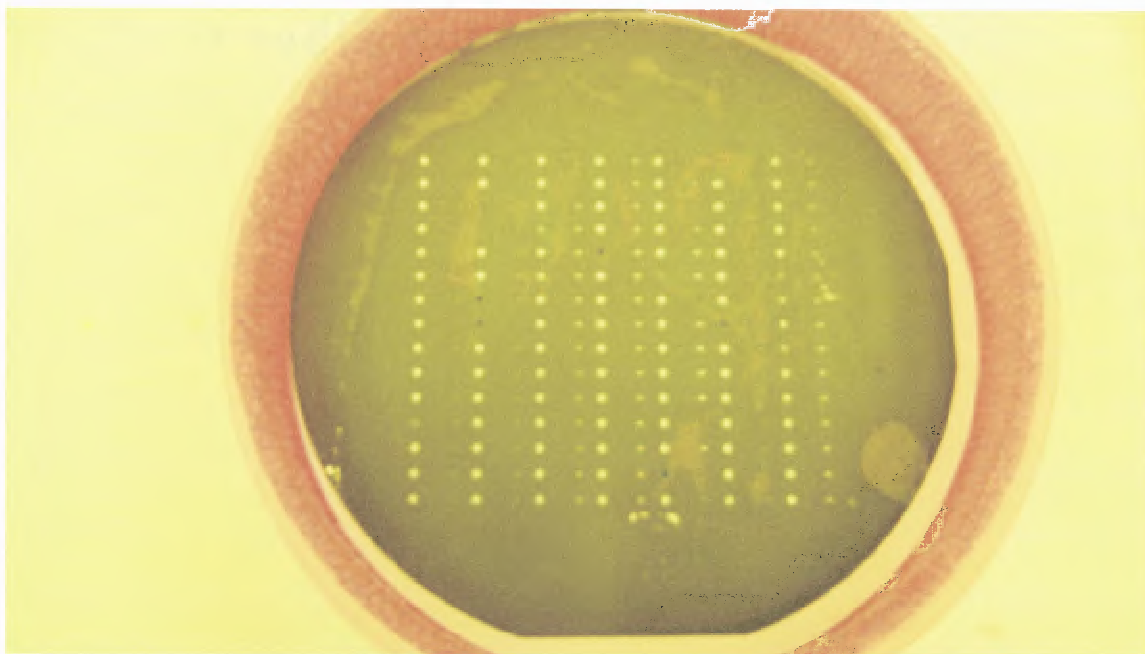


Figure 4.15 DRIE anisotropic etch of the vias.

Table 4.2 Deep Reactive Ion Etching Program

Steps	Process	Time(sec)
1	Initial	10
2	Process Stabilization	60
3	Process Ignition	5
4	Process Deposition	2
5	Process Etch A	4
6	Process Etch B	10
7	Loop	1
8	End	

Table 4.3 Deep Reactive Ion Etching Recipe

Step	Gas Amount (sccm)	Pressure (mTorr)	RF Power (W)	Temperature(C)
1		Bs. Press. = 6000	RF1 = 8 RF2 = 500	LN_2 = 25°C
2	SF_6 = 20 C_4F_8 = .5 Ar = 30	Press. = 13 He Flow = 3.6 He Press. = 2.5	NA	LN_2 = 25°C
3	SF_6 = 20 C_4F_8 = 0 Ar = 40	Press. = 13 He Flow = 3.6 He Press. = 2.5	RF1 = 8 RF2 = 500	LN_2 = 25°C
4	SF_6 = 70 C_4F_8 = .5 Ar = 40	Press. = 22	RF1 = 1 RF2 = 700	LN_2 = 25°C
5	SF_6 = .5 C_4F_8 = 50 Ar = 40	Press. = 23	RF1 = 9 RF2 = 700	LN_2 = 25°C
6	SF_6 = .5 C_4F_8 = 100 Ar = 40	Press. = 23	RF1 = 9 RF2 = 700	LN_2 = 25°C

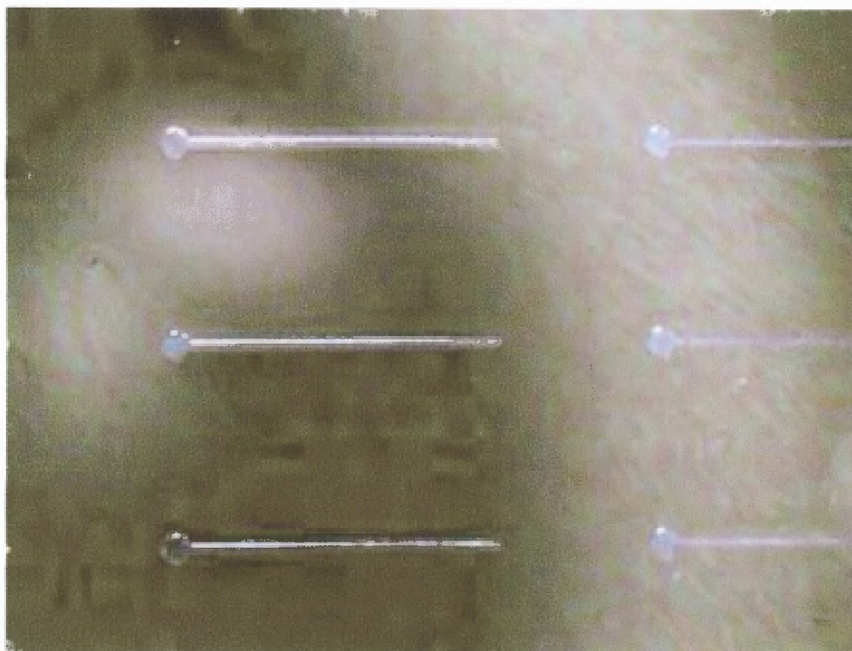


Figure 4.16 Snapshot of etched microfluidic channels taken under the microscope.

Figure 4.17 Snapshot of the vias taken under the microscope.

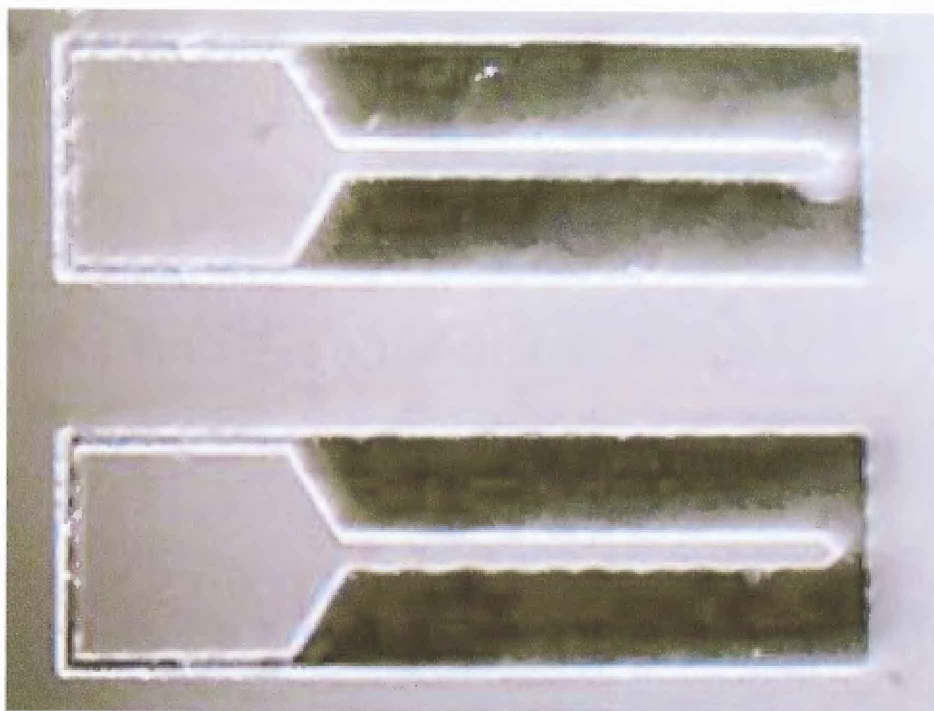


Figure 4.18 Snapshot of the microneedle periphery taken under the microscope.

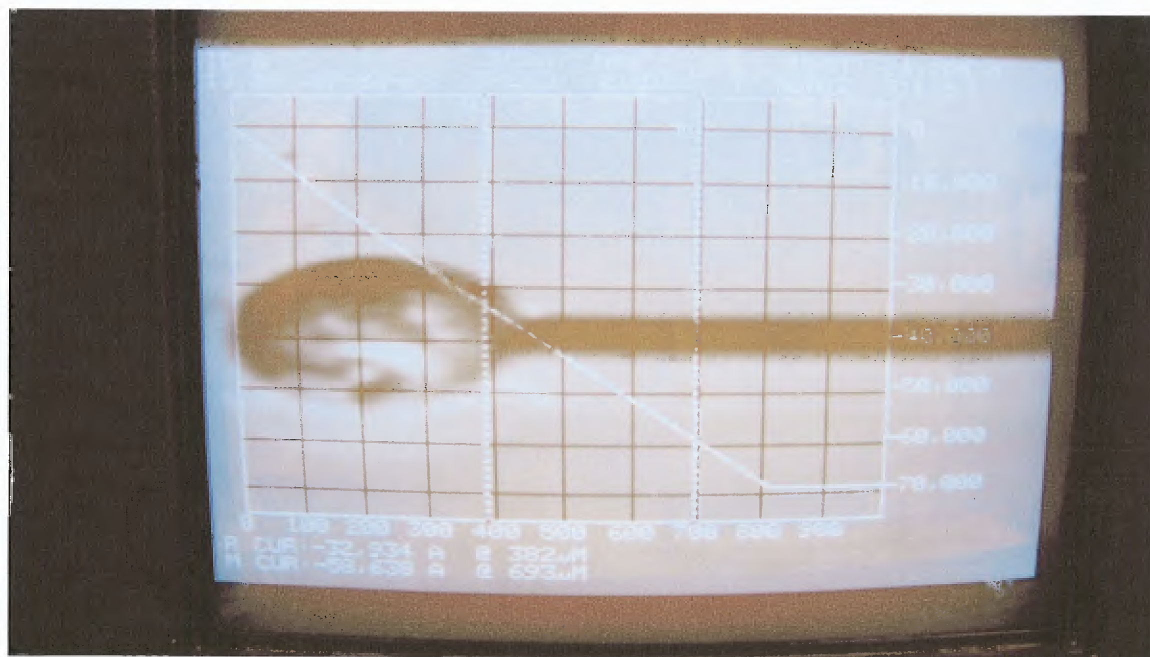


Figure 4.19 Microfluidic channel view on dektak.

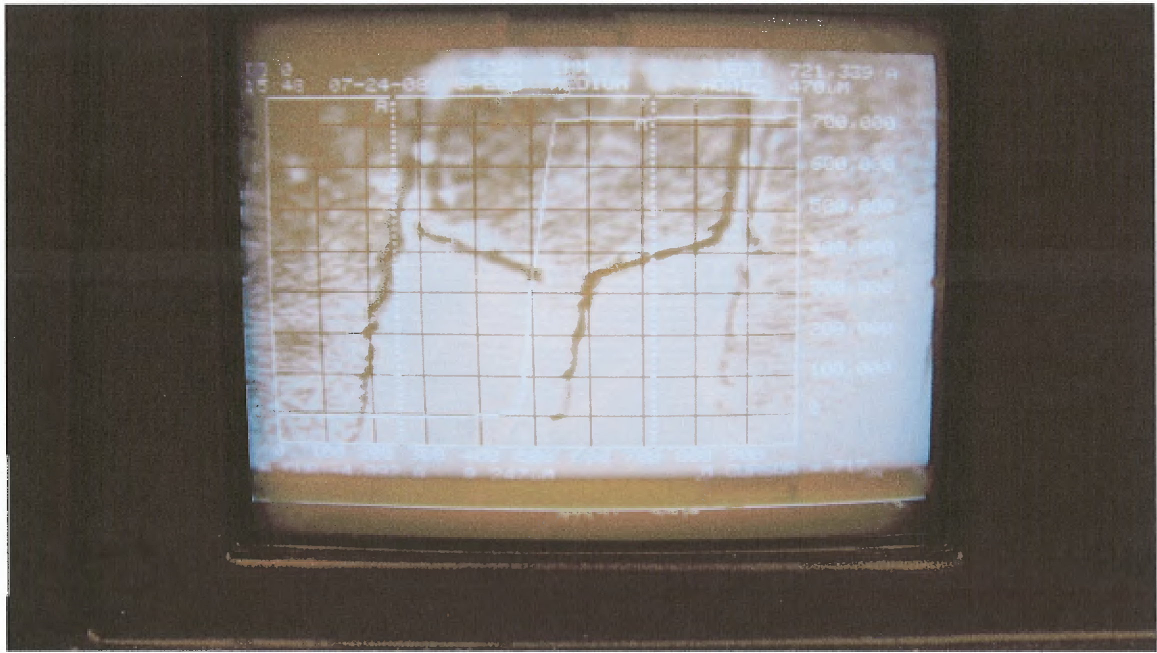


Figure 4.20 Microneedle periphery view on Dektak.

4.6 Wafer Bonding

There are many bonding techniques and methods used in microfabrication. Standard bonding methods have been set for certain substrates such as silicon and glass. Fusion bonding is very widely used to bond silicon to silicon. Anodic bonding is another common performed technique used to bond silicon to glass. Other bonding procedures used in microfabrication involve adhesive materials such as photoresist and glue to form a seal between substrates, however these procedures may not often times form the tightest and most durable bonds. Bonding was required in creating a seal for the microfluidic channel. A second silicon wafer was bonded on top of the wafer forming a tight seal to prevent leakage.

4.6.1 Photoresist Bonding

Due to some complications in dry etching fusion bonded wafers, photoresist was used as an alternate method for bonding. A thin layer of photoresist would be applied to a bare wafer and the etched wafer would be placed on top of the photoresist layer. Once the microfluidic channels were etched, the wafers were ready to bond. Since the channels were etched around 40um deep, a thin photoresist layer of 2μm would be a suitable layer for the wafers to bond. A bare wafer was taken and placed on the spinner. Five drops of HMDS primer were applied onto the center of the wafer for 1 minute at 800rpm's. Next, the S1813 photoresist was applied on the center of the wafer and covered the entire wafer. The spinner was spun for a minute at 2000rpm's and a 2μm layer of photoresist was formed on the surface of the wafer. Next, the wafer with the etched microchannels was then carefully placed on top of the photoresist layer with the flats aligned. The wafers were then placed in the bonder at a pressure of 1000N for ten minutes to tighten the bond. Finally, the wafer was placed into a quartz boat and loaded into the oven for a hard bake at 115°C for 15 minutes.

4.6.2 Wet Thermal Oxidation

Fusion bonding can only be performed if layers of oxide are deposited onto the surfaces of the substrates being bonded. A thin layer of oxide around 2000A° is usually deposited before fusion bonding. Thermal oxidation is a high temperature technique used to grow oxide onto the surface by diffusing into the substrate then reacting with it. Most of these procedures are performed using furnaces at temperatures around 800°C and 1200°C [11]. There is usually a tray or wafer boat made from glass or quartz that is used to move and hold the wafers inside the furnace. The governing equation for wet oxidation is: Si

(solid) + $2H_2O$ (vapor) --> SiO_2 (solid) + $2H_2$ (vapor) [12].

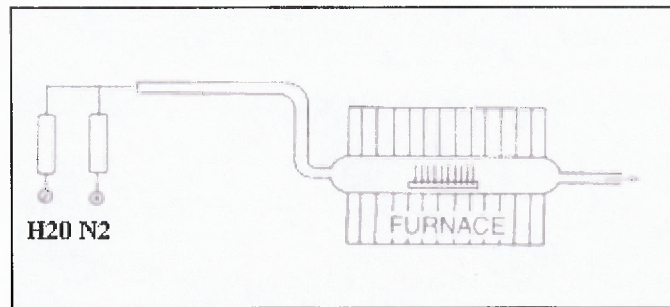


Figure 4.21 Thermal Oxidation Furnace.

The thermal oxidation setup is shown in Figure 4.21. Wafers are loaded into the furnace and then the oxidizing agent which is steam diffuses into the silicon where it reacts and forms an oxide layer.

Table 4.4 Wet Thermal Oxidation Program

	Process	Time(min)
0	Idle	0
1	Push In	20
2	Temp Ramp-Up	45
3	Temp Stab	20
4	Pre-Ox	1
5	Steam-Ox	60
6	Post-Ox	1
7	Anneal	10
8	Temp Ramp-Down	75
9	Pull Out	5

Table 4.5 Wet Thermal Oxidation Gas Amounts

	Gasses	SLPM
1	Nitrogen	30
2	Oxygen	10
3	Bubbler (Water Vapor)	5

After DRIE, an NMP clean was performed to clean the wafers before oxidation. The wafers were then transferred to a quartz boat and loaded into the oxidation furnace. The recipe shown above was then run. Standard oxidation time of the wafers was set to one hour. The oxidation recipe used was a standard for a deposition of a 2500A° layer. The oxidation was conducted at a temperature of 1050°C.

4.6.3 Fusion Bonding and Hydrophilization

Silicon fusion bonding is the process which binds silicon to silicon. This binding of silicon is the product of the hydroxide groups reacting with each other at the surface of the wafers. Fusion bonding is one of the more robust bonding processes compared to other bonding techniques. The surfaces of the wafers have to be very clean when fusion bonding. Any particles or organic materials will cause the wafers to not be bonded at those places.

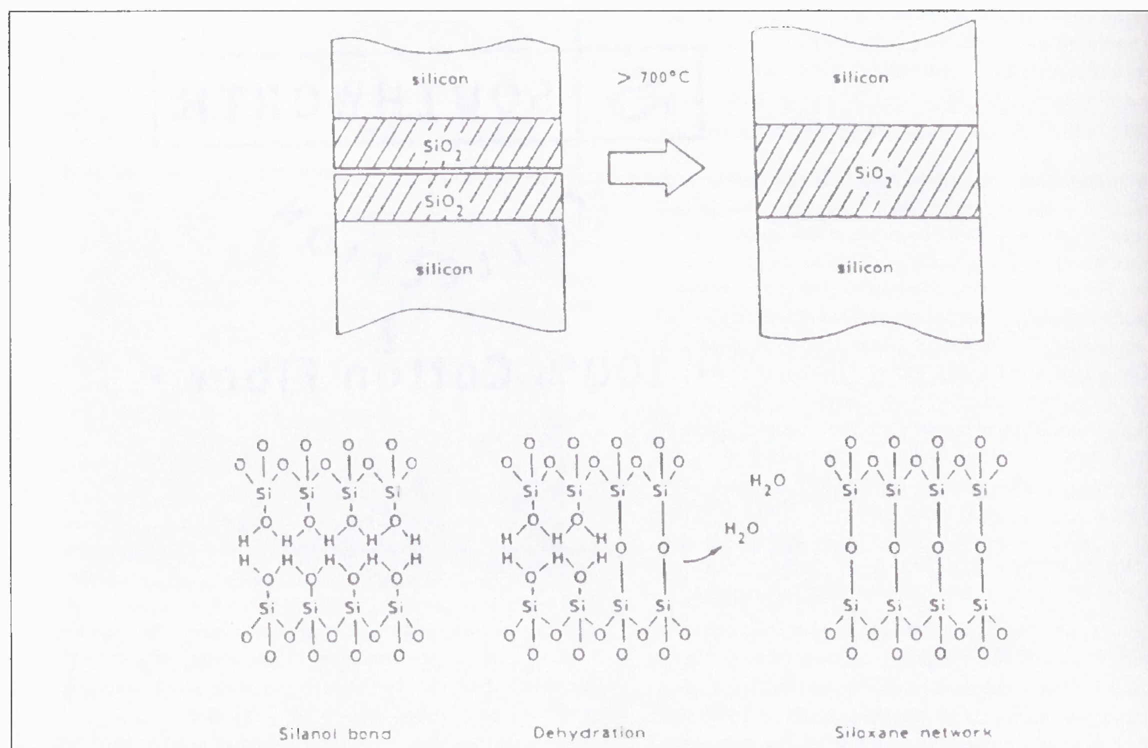


Figure 4.22 Silicon to silicon fusion bonding [11].

Before beginning fusion bonding, hydration and hydrophilization need to be administered on the wafers. Hydrophilization is achieved by submerging the wafers in a H_2O- H_2SO_4 solution diluted with H_2SO_4 [11]. This process forms a layer of O-H bonds on the oxide surface. This is followed by a rinse of deionized water. The wafers are able to bond by themselves under certain pressures. Obtaining a good bond depends on how smooth and clean of organic and foreign materials are present on the surfaces of the wafers. The pressure applied on the wafers in the bonder also determines how well a bond is achieved.

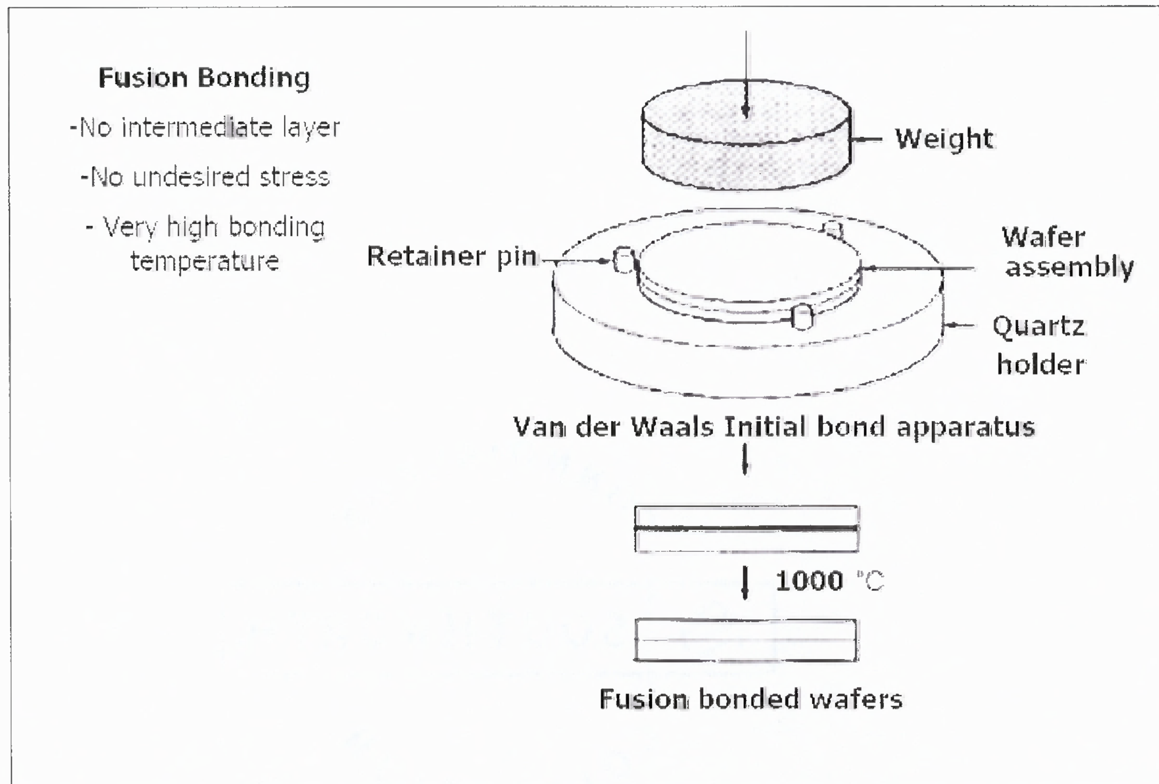


Figure 4.23 Fusion bonding setup [17].

The bonder used was the EV501S and manufactured by the EV Group, which specializes in wafer bonding technology. To start the bonder, nitrogen gas was turned on to circulate inside the chamber. The wafer bonder apparatus includes a chamber which is opened by unscrewing four large knob screws. On the top of the bonder a dial is used to control the depth of the wafer stack. The depth used is usually the total thickness of the wafers combined but is also a parameter that can be changed if the set depth does not bond the wafers. Even though the total wafer thickness was 1200μm, the dial was set to 750μm after larger depths were not successful in previous bonding experiences. Time is the second parameter which can be changed, but most wafers will either bond or not in five minutes. The wafers are then placed on a chuck that aligns the wafers by their primary flats. Since thin wafers are harder to bond, two 500μm thick wafers were then

placed on a metal chuck with the flats aligned by two metal pins. These two thick wafers were used to help the thin wafers bond more efficiently, creating a larger depth in the total wafer thickness. A thin wafer was then placed on the top of the two 500μm wafers. Three metallic bonding levers were then placed on the top of the thin wafer. The second thin wafer was then placed and aligned on top of the metallic levers and bottom thin wafer. The lid was then closed and locked into position by screwing back the knob screws. The next steps were conducted by using the bonder program. After the chamber is closed, a state update is done to secure that the chamber is properly closed. A maximum force of 1000N is a standard set parameter which is uniformly pressed on the wafer stack. Next, the wafer depth is checked by clicking the wafer pin icon that drops a small pin on the top of the wafer stack to locate where the stack begins. The metallic lever icon is then clicked to release the levers from in between the two thin wafers. Finally, the electrode icon is then clicked to start the uniform force of 1000N being applied by a quartz plate on the wafer stack. The wafers can be inspected after a period of five minutes. By clicking the electrode icon, the force was stopped and the chamber was opened to inspect the wafers. Bonding was detected by carefully moving tweezers around the edges and observing the sides of the wafers for gaps. The force applied creates van der Waal forces between the wafers, which are then annealed to create a more secure and strong bond.

4.6.4 Post Bond Annealing

Annealing is a type of thermal treatment needed to complete the fusion bonding process. The temperatures at which the annealing process is performed is anywhere between 700°C to 1100°C [18]. During this heating process, the wafers adhere and form Si-O-Si

bonds. Wafers annealed at temperatures above 1000°C for two hours usually result in higher strength bonds [18].

Table 4.6 Annealer Program.

	Process	Time(min)	Temperature(°C)
0	Idle	0	500
1	Push In	20	500
2	Temp Ramp-Up	45	1100
3	Anneal	30	1100
4	Pre-Ox	1	1100
5	Temp Ramp-Down	60	500
6	Pull Out	1	500

After the wafers were taken out of the bonder and inspected, they were ready to be annealed. They were placed into a quartz boat and immediately loaded into tube #8 for annealing. The recipe shown above was then run. A total annealing time of 30 minutes was set.

4.7 Microneedle Removal

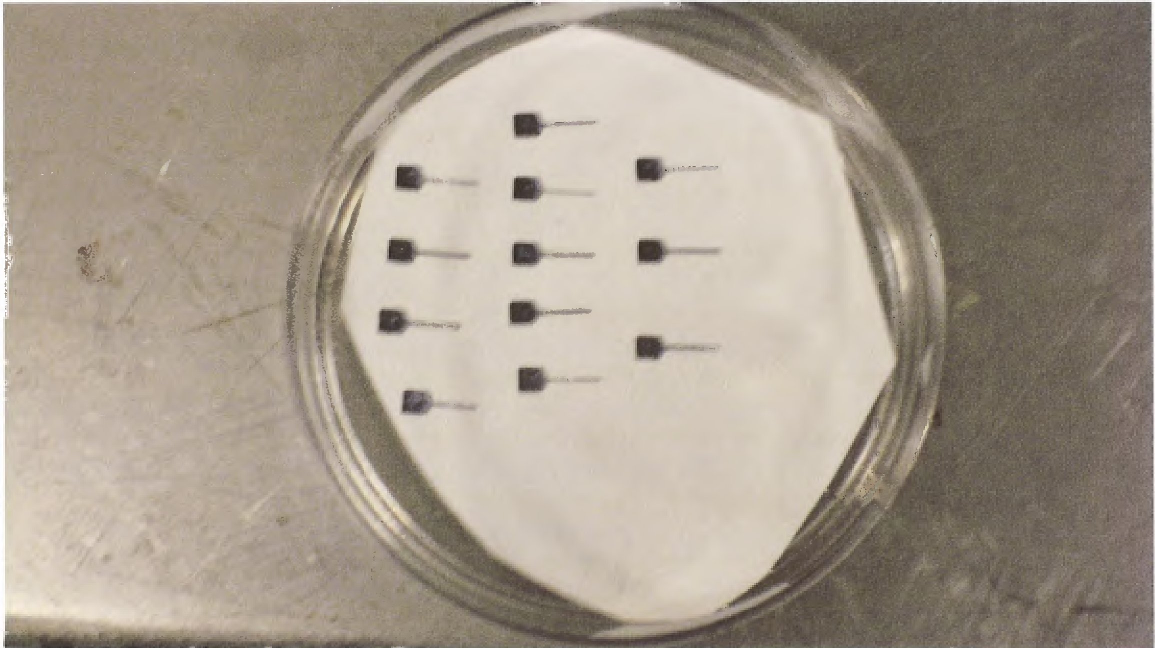


Figure 4.24 Microneedles placed in petri dish.

Figure 4.24 shows the microneedles poken out of the bottom wafer. Once the periphery of the microneedle was etched all the way through the wafer, the microneedles were ready to be poken out. The microneedle removal process from the wafer was very simple. Any pressure added to the microneedle would quickly separate it from the wafer. In some cases the microneedles popped out by themselves due to the shear movement of the wafer. Figure 4.25 shows a snapshot of a single microneedle under the microscope.

Figure 4.25 Bottom half of microneedle with microchannel seen under microscope.

4.8 Microfluidic Connector

A microfluidic connector was needed to enclose and supply a port to connect a tube to pump fluid into the channel. Fluidic ports are commercially available, but there are few companies which manufacture these ports on the micro and nano meter levels. One such company that does manufacture microfluidic ports used for lab-on-a-chip applications and many other microfluidic devices is Upchurch Scientific. A microfluidic port was ordered under the "Nanoport Assemblies" product line.

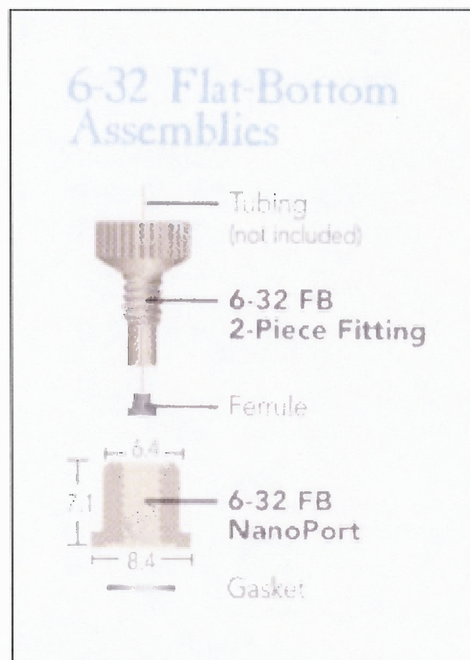


Figure 4.26 Nanoport features and dimensions [19].

These ports are made of inert, biocompatible PEEK polymer and Perlast perfluoroelastomer [19]. The port also prevents contamination of fluid being passed through. The port ordered contained a nanoport, fitting, and ferrule. Since the via to the microfluidic reservoir was around 1mm in diameter, only the ferrule was small enough to consider to use as the connector. The ferrule had an outer diameter of 1mm and depth of

1mm. The inner diameter of the ferrule was 360μm.

4.8.1 Microfluidic Connector Bonding

There were a couple of options to bond the microfluidic connector to the microneedle. The Upchurch Scientific catalog recommended either to use an adhesive ring that was provided or to use an alternative epoxy adhesive. The catalog also mentioned that all the Nanoports will adhere to silicon, quartz, glass, and polymers [19]. The connector would also serve as a holder of the microneedle, therefore the bonding of the connector to the microneedle needed to be high in strength.

CHAPTER 5

FINITE ELEMENT ANALYSIS SIMULATIONS

5.1 ANSYS

ANSYS is a software which produces finite element analysis simulations. The software allows users to solve solid and fluid mechanics problems. When conducting finite element analysis, a set of steps are followed to obtaining the solution. The first of these steps is specifying the geometry of the structure, which depend on what dimension the users model is in. The next step requires the user to specify an element type and the material properties of the solid or fluid being investigated. The model is then constructed or was imported from another modeling software in the beginning. The next step is a crucial step in obtaining an accurate solution. This step is called meshing, which breaks the structure into smaller elements. By breaking the structure down into smaller elements, information on how the structure will react to loading conditions can be acquired. ANSYS uses both the p and h-method, each of which play different roles in producing a solution. Boundary conditions and loads can then be applied after the proper meshing. Various parts of the structure can be selected as fixed supports and other parts can be selected to have external loads. A solution can then be generated with the paramters chosen in the previous steps. In postprocessing, data can be viewed by many different graphs. Many simulations also recquire refining the mesh to achieving a more accurate solution.

5.2 Buckling of Microneedle Shank

Understanding the loads that would require the microneedle to break during the penetration into tissue, required a finite element analysis simulation. When a structure experiences a sudden failure due to high compressive stresses, it is characterized as having elastic instability or buckling. Critical loads are the loads that may be maintained in a deflected shape [20]. The lowest critical load is called the Euler buckling load, Eulers formula is:

$$Pcr = \frac{\pi^2 EI}{L^2} \qquad \text{(Equ. 5.1)}$$

Pcr: Critical load (Pa)
E: Young's Modulus (Pa)
I: Moment of Inertia (m^4)
L: Length (m)

Figure 5.1 shows the buckling of a column with a critical load applied at one end and fixed at the other end. Notice the length experiencing displacement is 70% of the total length. Le denotes the effective column length.

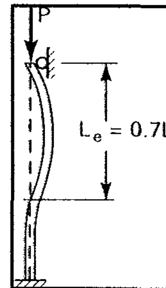


Figure 5.1 Effective length of column with restraint [20].

With the Young's Modulus of silicon being 150GPa, a moment of inertia of 1.63e-16m^4, the critical load was calculated to be 17.029N. A finite element analysis simulation was conducted applying this critical load to observe the displacements. A structural analysis of the shank of the microneedle was conducted using ANSYS. A solid,

brick 8-node 185 element type was chosen to model the shank. Material properties of silicon were entered including a Young's Modulus of 150GPa and a Poissons Ratio of .17 [11]. A model of the microneedle shank was then created by setting the dimensions. A mesh was then established by setting divisions in the solid. The long lines were given 20 elements per division while the smaller faces on either side were given 5 elements per division. Loads and boundary conditions were then applied seen in Figure 5.2. The shank was fixed at the bottom and the critical load calculated was applied to the top face. The solution was then solved and the x-component of displacement was displayed.

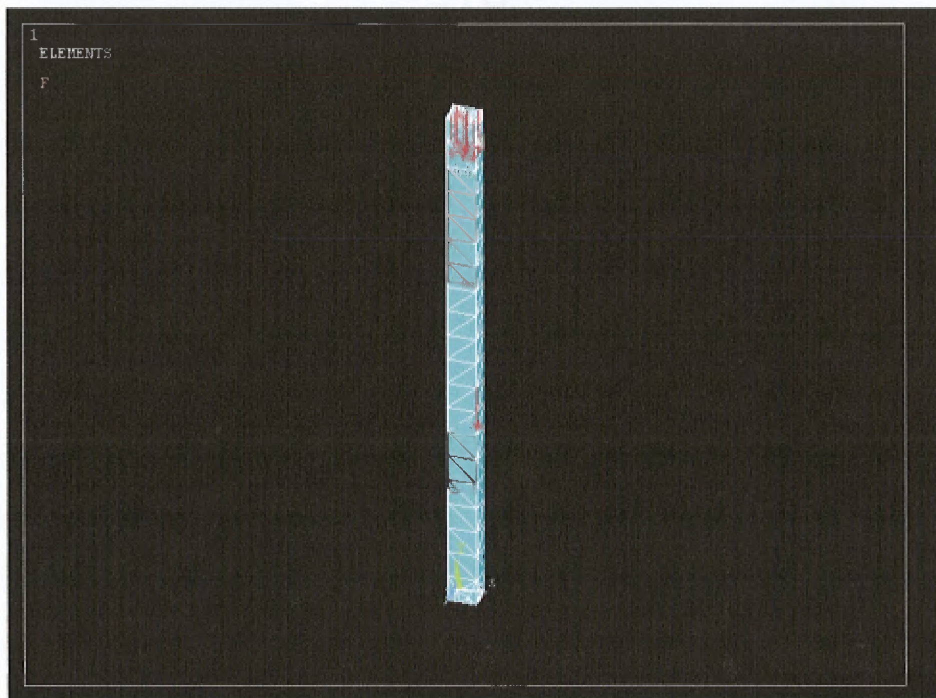


Figure 5.2 Distributed load applied to the top face.

Figure 5.3 shows the x-component of displacement plot. A maximum displacement value of 38.8μm was solved in the ANSYS simulation. In conclusion, the shank of the microneedle would initiate buckling if a pressure of 17.03 N was applied.

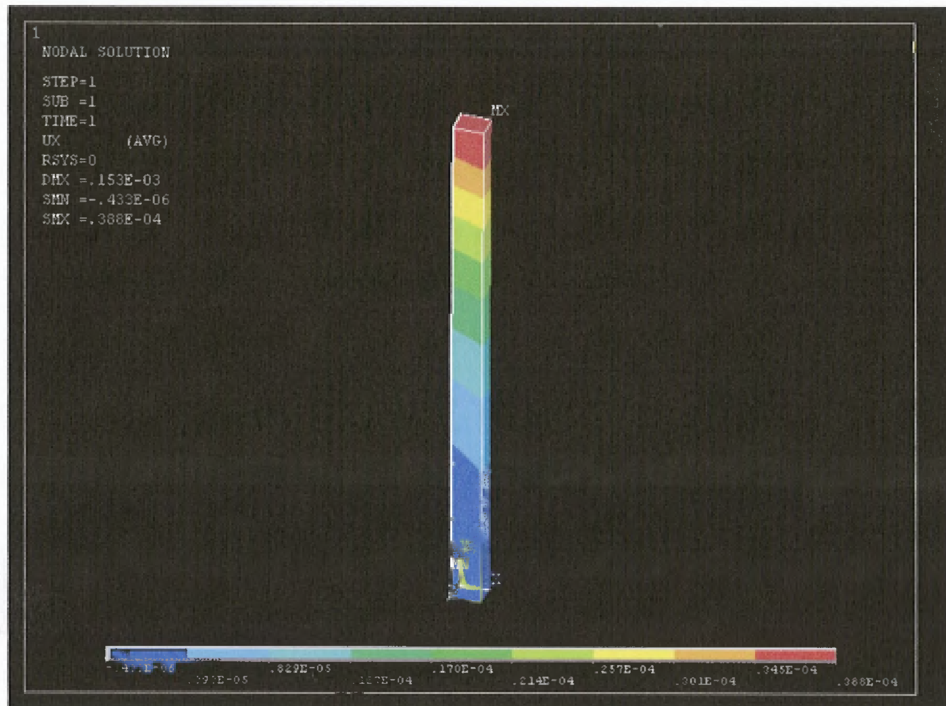


Figure 5.3 X-component of displacement plot.

Figure 5.4 shows the force vs. maximum displacement plot for applied loads of 5N, 10N, 17N, 35N, 50N, and 75N. The increase of applied loads in turn cause an increase in the displacements.

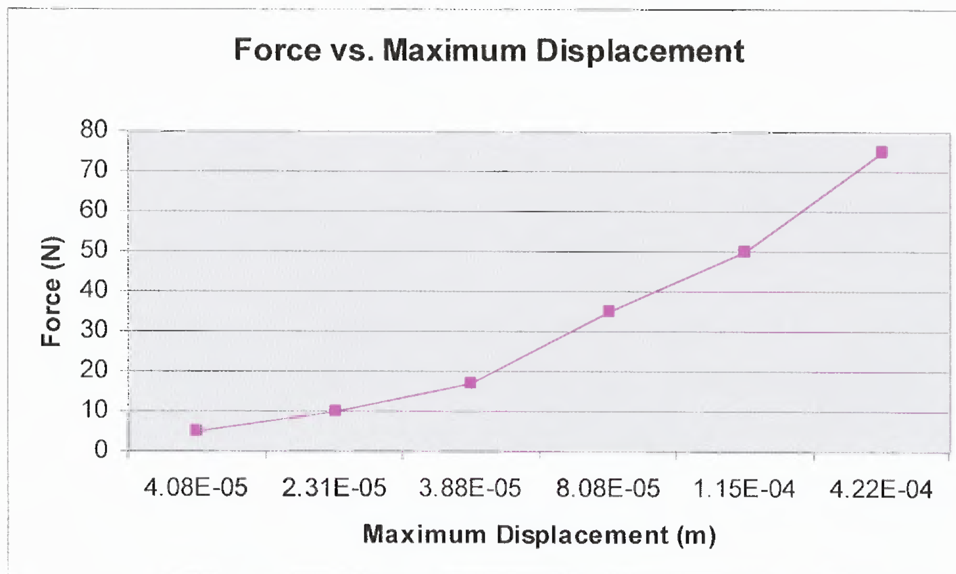


Figure 5.4 Force vs. maximum displacement plot.

CHAPTER 6

CONCLUSION

In conclusion, there were some weak points in the design, but valuable lessons were learned and some alternative ideas came to light to approaching these flaws more efficiently in the future. The weakest point in the design was sealing the microfluidic channel by fusion and photoresist bonding. Fusion bonding the wafers before dry etching (RIE, DRIE), caused the wafers to separate due to the low pressure conditions of dry etching. This problem resulted in processing the wafers separately and bonding them with super glue. Using photoresist as an adhesive to bond the wafers was also unsuccessful due to the design of the periphery of the needle having too much exposed area also resulting in separation of the wafers. Handling of 50-100μm thin wafers was also a difficult task in processing the wafers. More attention was given to the placement and transporting of the wafers while conducting the different fabrication processing on the wafers. Some alternative ways for the implementation of these microneedles were discovered.

Many approaches in sealing microchannels in research are fabricated by depositing sacrificial layers. These sacrificial layers are then etched leaving a film deposited above the sacrificial layer to serve as a seal for the microchannel. Sacrificial layers are wet etched which could result in problems with the other features on the microneedle. To avoid wet etching, an adhesive bond would be suitable for wafer bonding. Adhesive bonds compose of organic or inorganic layers. The adhesive can be deposited on the wafer through spin coating, spraying, or laminating. Once the adhesive

layer is applied to a wafer the other wafer can be placed on top and then be prepared to cure, either by temperature or pressure. Many of the organic layers consist of polymers, while the inorganic layers consist of ceramics. There are four categories of polymer adhesives including, thermoplastic, thermosetting, elastomeric, and alloys. Each of these polymers have varying material properties and parameters associated with there adhesion capabilities. Some polymer adhesives that should be investigated further for use in adhesive bonding will include, UV epoxies, Benzocyclobutene, Polymethylmethacrylate (PMMA), Polydimethylsiloxane (PDMS), Fluoropolymers, Polyetheretherketone (PEEK), Thermosetting Copolyesters, waxes [21].

In Figure 6.1, a microneedle with the addition of metal contacts shown in red, could be a useful design feature for neural stimulation. In the next generation of microneedle development, the function of stimulation near the tip could be a great design feature to detect if the location of the FLAMES are implanted at the targeted neural structures accurately.

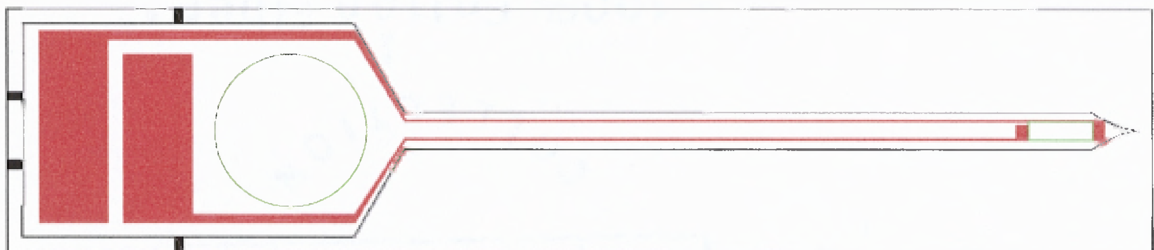


Figure 6.1 Metal contacts shown in red on microneedle.

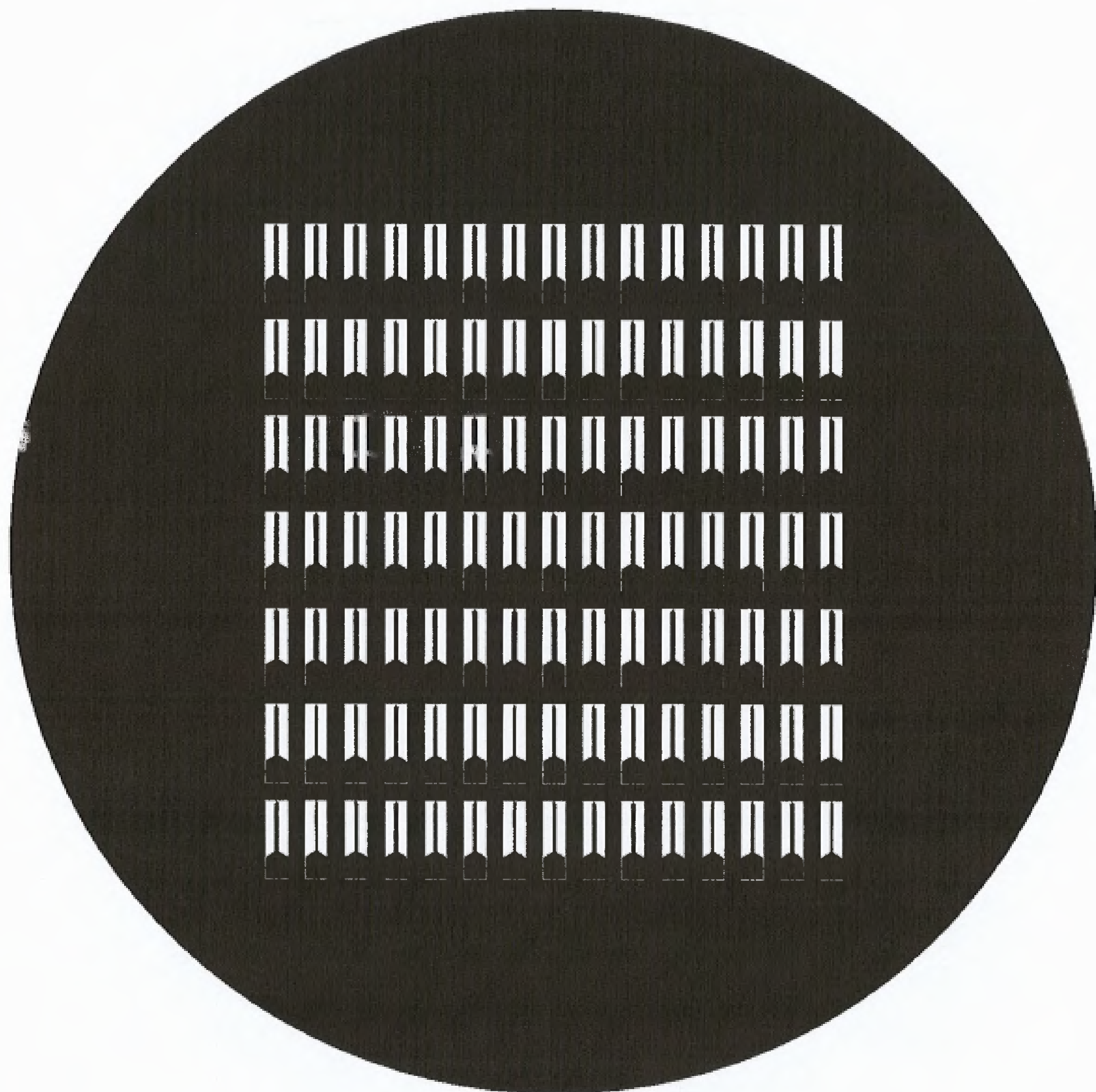
The microneedle could carry out a function of delivering current as well as delivering the FLAMES in certain areas of the central nervous system. Metallic contact pads would be interconnected to a larger set of bonding pads. These bonding pads would then be bonded with external wiring to pass the current. The current would stimulate the tissue through metal contacts. The contacts would be made of thin film metal sputtered or

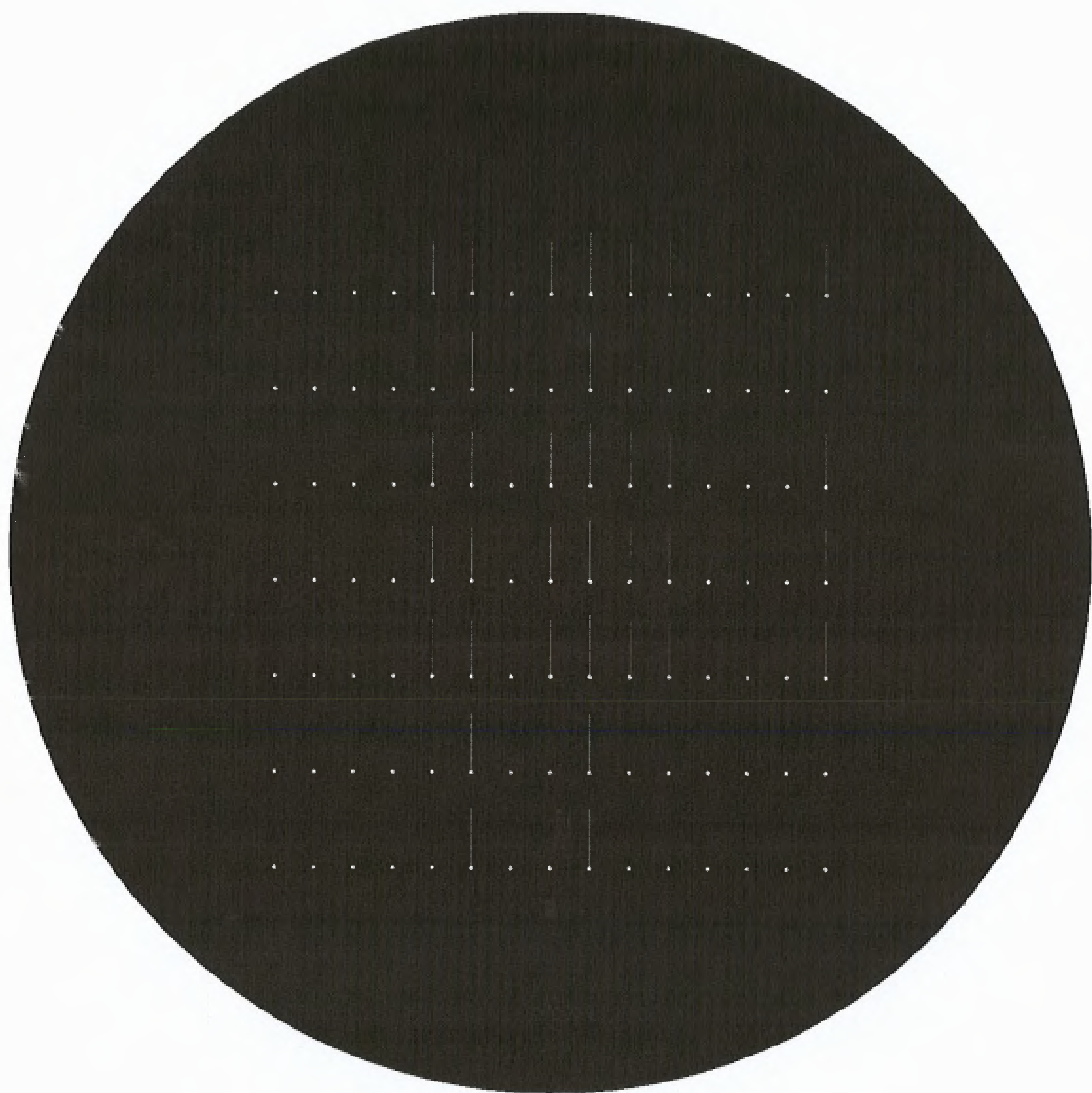
deposited using e-beam. The addition of the metal contacts would be a very conducive feature and should be investigated in future versions of the device.

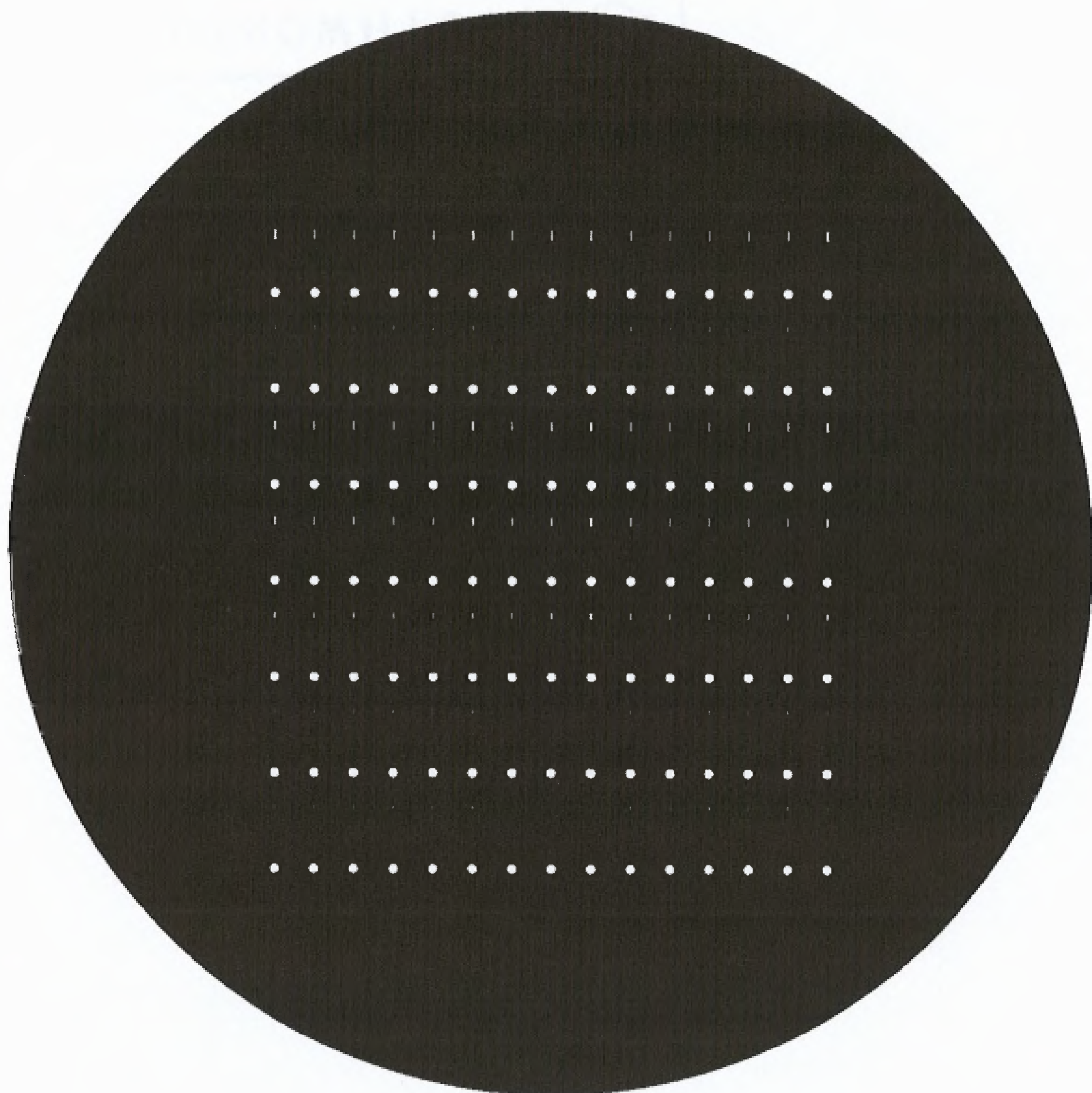
APPENDIX A

PHOTOMASKS

The dark field photomasks of the microneedle periphery, vias, and microfluidic channel were ordered from FineLine Imaging with a resolution of 16,256dpi or 1.56μm.







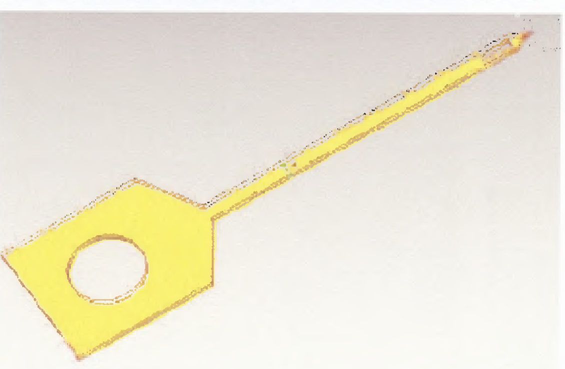
APPENDIX B

TRAVELER FOR PHOTORESIST BOND

A traveler for the photoresist bonding procedure was constructed in Microsoft Excel.

Traveler
Professor - Dr. Mesut Sahin
Faysal Ahmed
Project - Microneedle

Stage	Step	Process	Description	Date/Time	Operator	Comments
			Top Wafer (Mask)			
0. Start	0.1	Inspect	Substrate 4" Diameter, silicon, orientation <100>, Thickness 500 ± 5um			
1. Clean	1.1	NMP-Clean	Secondary Bath, 10 min., 10 min. DI Water			
	1.2	P-Clean	10 Minute H2SO4+H2O2 (5:1) Bath @ 110 C			
			10 Minute Rinse Hot DI water			
			10 Minute Rinse Cold DI water			
			Nitrogen Gun Dry Slowly			
2. Nitride Removal	2.1	RIE Nitride Etch	Room Temperature			
			Thickness=2000 Angstroms			
			SF6=50sccm			
			p=300mT, RF=200W, t=3min			
3. Thermal Oxidation	3.1	Oxidation Deposition	Target thickness for oxide is 3000 Angstroms			
			T=1050 C			
			N2=[illegible]LPM, O2=[illegible]LPM			
			Time=1hr30 min			
4. Photolithography	4.1	Dehydration	Quartz boat in Oven~5min.			
	4.2	Prime	HMDS Primer Spin Coating @800rpm for 1minute			
	4.3	PR Coating	AZ 4620 Spin Coating @4000rpm for 1minute			
	4.4	Soft Bake	Quartz boat in Oven~20min.@ 110			
	4.5	Exposure	Expose for 90 seconds			
	4.6	Develop	Immerse in AZ 400 Developer for 1-1:30 minute			
	4.7	Inspection	Inspect the wafers for residual defects			
	4.8	Hard Bake	Quartz boat in Oven~30min.@ 120			
5. Oxide Etch	5.1	RIE Oxide Etch	T=Room Temperature			
			Thickness=3000 Angstroms			
			CF4=50sccm			
			p=300mT, RF=200W, t=3min			
6. Si Etch	6.1	DRIE Si Etch	Thickness=100um			
			SF6=[illegible]sccm, C4F8=80sccm, Ar=40sccm			
7. PR Removal	7.1	M-Pyrol Clean	10 Minute Primary Bath 95 °C			
			10 Minute Secondary Bath 95 °C			
			10 Minute Rinse Cold DI water			
			Nitrogen Gun Dry Slowly			
	7.2	P-Clean	10 Minute H2SO4+H2O2 (5:1) Bath @ 110 C			
			10 Minute Rinse Hot DI water			
			10 Minute Rinse Cold DI water			
			Nitrogen Gun Dry Slowly			
			(Bottom Wafer/Fluidic Channel)			
8. Nitride Removal	8.1	RIE Nitride Etch	T=Room Temperature			
			Thickness=2000 Angstroms			
			SF6=50sccm			
			p=300mT, RF=200W, t=3min			
9. Thermal Oxidation	9.1	Oxidation Deposition	Target thickness for oxide is 3000 Angstroms			
			T=1050 C			
			N2=[illegible]LPM, O2=[illegible]LPM			
			Time=1hr30 min			
10. Photolithography	10.1	Dehydration	Quartz boat in Oven~5min.			
	10.2	Prime	HMDS Primer Spin Coating @800rpm for 1minute			



Step	Sub-step	Process	Parameters
	10.3	PR Coating	AZ 4620 Spin Coating @4000rpm for 1minute
	10.4	Soft Bake	Quartz boat in Oven~20min @ 110
	10.5	Exposure	Expose for 90 seconds
	10.6	Develop	Immerse in AZ 400 Developer for 1-1:30 minute
	10.7	Inspection	Inspect the wafers for residual defects
	10.8	Hard Bake	Quartz boat in Oven~30min @ 120
11. Oxide Etch	11.1	RIE Oxide Etch	T=Room Temperature Thickness=2000 Angstroms CF4=50sccm p=300mT, RF=200W, t=8min
12. Si Etch	12.1	DRIE Si Etch	Thickness=50um SF6=50sccm, C4F8=80sccm, Ar=40sccm
13. PR Removal	13.1	M-Pyrol Clean	10 Minute Primary Bath 85 °C 10 Minute Secondary Bath 85 °C 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly
	13.2	Pi-Clean	10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly
14. Oxide Removal	14.1	RIE Oxide Etch	T=Room Temperature Thickness=2000 Angstroms CF4=50sccm p=300mT, RF=200W, t=8min
15. Photolithography	15.1	Dehydration	Quartz boat in Oven~5min.
	15.2	Prime	HMDS Primer Spin Coating @ 500rpm for 1minute
	15.3	PR Coating	AZ 4620 Spin Coating @4000rpm for 1minute
	15.4	Soft Bake	Quartz boat in Oven~20min @ 110
	15.5	Exposure	Expose for 90 seconds
	15.6	Develop	Immerse in AZ 400 Developer for 1-1:30 minute
	15.7	Inspection	Inspect the wafers for residual defects
	15.8	Hard Bake	Quartz boat in Oven~30min @ 120
16. Si Etch	16.1	DRIE Si Etch	Thickness=100um SF6=50sccm, C4F8=80sccm, Ar=40sccm
			BOTH WAFERS (Bonding)
17. Photoresist Application	17.1	PR Coating	Shipley S1813 Spin Coating @2000rpm for 1minute
18. Wafer Bonding	18.1	Bonding	Thickness set at 75um Force 1000 Newtons, t=10min.
			Back Side of Bonded Wafers
19. Si Etch	19.1	DRIE Si Etch	Thickness=100um SF6=50sccm, C4F8=80sccm, Ar=40sccm
20. PR Removal	20.1	M-Pyrol Clean	10 Minute Primary Bath 85 °C 10 Minute Secondary Bath 85 °C 10 Minute Rinse Cold DI water Spin Dry/Nitrogen Gun Dry
	20.2	Pi-Clean	10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Spin Dry/Nitrogen Gun Dry

APPENDIX C

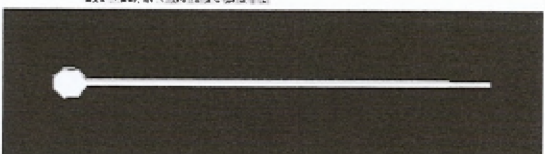
TRAVELER FOR FUSION BOND

A traveler for the fusion bonding procedure was constructed in Microsoft Excel.

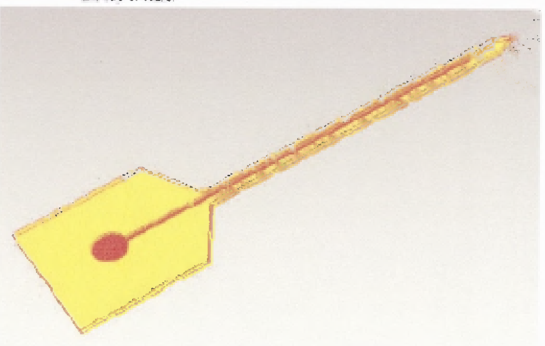
Traveler
Professor - Dr. Mesut Sahin
Faysal Ahmed
Project - Microneedle

Stage	Step	Process	Description	Date/Time	Operator	Comments
			1st Wafer - (Fluidic Channel)			
0. Start	0.1	Inspect	Substrate 4" Diameter, silicon, orientation=<100>, Thickness 400 ± 5um			
1. Clean	1.1 1.2	NMP-Clean P-Clean	Secondary Bath, 10 min., 10 min. DI Water 10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly			
2. Photolithography	2.1 2.2 2.3 2.4 2.5 2.6 2.7 2.8	Dehydration Prime PR Coating Soft Bake Exposure Develop Inspection Hard Bake	Quartz boat in Oven-5min. HMDS Primer Spin Coating @600rpm for 1minute Shipley S1813 Spin Coating @3000rpm for 1minute Quartz boat in Oven-5min. Expose for 20 seconds Immerse in MF 319 Developer for 1-1:30 minute Inspect the wafers for residual defects Quartz boat in Oven-5min.			
3. Nitride Etch	3.1	RIE Nitride Etch	T=Room Temperature Thickness=2000 Angstroms SF6=50sccm P=300mT, RF=200W, t=2min			
4. Si Etch	4.1	DRIE Si Etch	Thickness=50um SF6=130sccm, C4F8=80sccm, Ar=40sccm			
5. PR Removal	5.1 5.2	M-Pyrol Clean P-Clean	10 Minute Primary Bath 88 °C 10 Minute Secondary Bath 88 °C 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly 10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly			
6. Nitride Removal	6.1	RIE Nitride Etch	T=Room Temperature Thickness=2000 Angstroms SF6=50sccm P=300mT, RF=200W, t=2min			
			BOTH WAFERS (Bonding)			
6. Clean	6.1	P-Clean	10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly			
7. Thermal Oxidation	7.1	Oxidation Deposition	Target thickness for oxide is 2000 Angstroms T=1050 C N2=10SLPM, O2=10SLPM Time=1hr30 min.			
8. Clean	8.1	P-Clean	10 Minute H2SO4+H2O2 5:1 Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry			

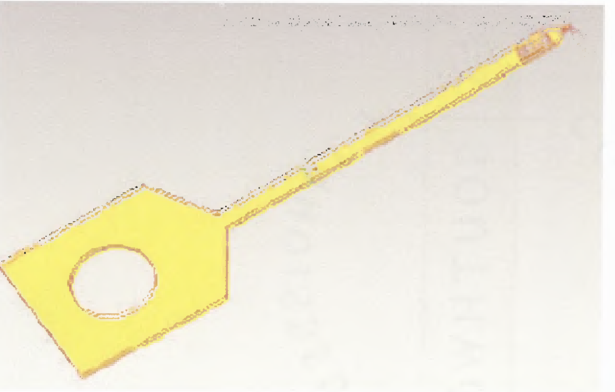
Top Mask of Microfluidic Channel

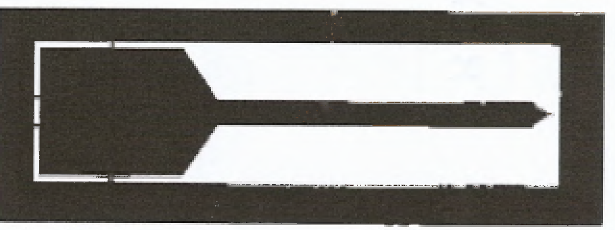


On Top of Wafer



Step	No.	Process	Details
9. Fusion Bond	9.1	Fusion Bonding	Thickness each: 75um Force 1000 Newtons, t=5min.
10. Post Bond - Annealing	10.1	Post Bond Anneal	T=1100 C, N2=7.5SLPM t=30min.
			2nd Wafer - [illegible]
11. Photolithography	11.1	Dehydration	Quartz boat in Oven~5min.
	11.2	Prime	[illegible] Primer Spin Coating @800rpm for 1minute
	11.3	PR Coating	Shipley S1813 Spin Coating @3000rpm for 1minute
	11.4	Soft Bake	Quartz boat in Oven~5min.
	11.5	Exposure	Expose for 20 seconds
	11.6	Develop	Immerse in MF 319 Developer for 1-1:30 minute
	11.7	Inspection	Inspect the wafers for residual defects
	11.8	Hard Bake	Quartz boat in Oven~5min.
12. Nitride Etch	12.1	RIE Nitride Etch	T=Room Temperature Thickness=2000 Angstroms CF4=50sccm P=300mT, RF=200W, t=6min
13. Si Etch	13.1	DRIE Si Etch	Thickness=100um SF6=150sccm, C4F8=80sccm, Ar=40sccm
14. PR Removal	14.1	N-Pyrol Clean	10 Minute Primary Bath 95 °C 10 Minute Secondary Bath 95 °C 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly
	14.2	P-Clean	10 Minute H2SO4:H2O2 (3:1) Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Nitrogen Gun Dry Slowly
			Bonded Wafer
15. Photolithography	15.1	Dehydration	Quartz boat in Oven~5min.
	15.2	Prime	[illegible] Primer Spin Coating @800rpm for 1minute
	15.3	PR Coating	Shipley S1813 Spin Coating @3000rpm for 1minute
	15.4	Soft Bake	Quartz boat in Oven~5min.
	15.5	Exposure	Expose for 20 seconds
	15.6	Develop	Immerse in MF 319 Developer for 1-1:30 minute
	15.7	Inspection	Inspect the wafers for residual defects
	15.8	Hard Bake	Quartz boat in Oven~5min.
16. Si Etch	16.1	DRIE Si Etch	Thickness=300um SF6=150sccm, C4F8=80sccm, Ar=40sccm
17. PR Removal	17.1	N-Pyrol Clean	10 Minute Primary Bath 95 °C 10 Minute Secondary Bath 95 °C 10 Minute Rinse Cold DI water Spin Dry/Nitrogen Gun Dry
	17.2	P-Clean	10 Minute H2SO4:H2O2 (3:1) Bath @ 110 C 10 Minute Rinse Hot DI water 10 Minute Rinse Cold DI water Spin Dry/Nitrogen Gun Dry



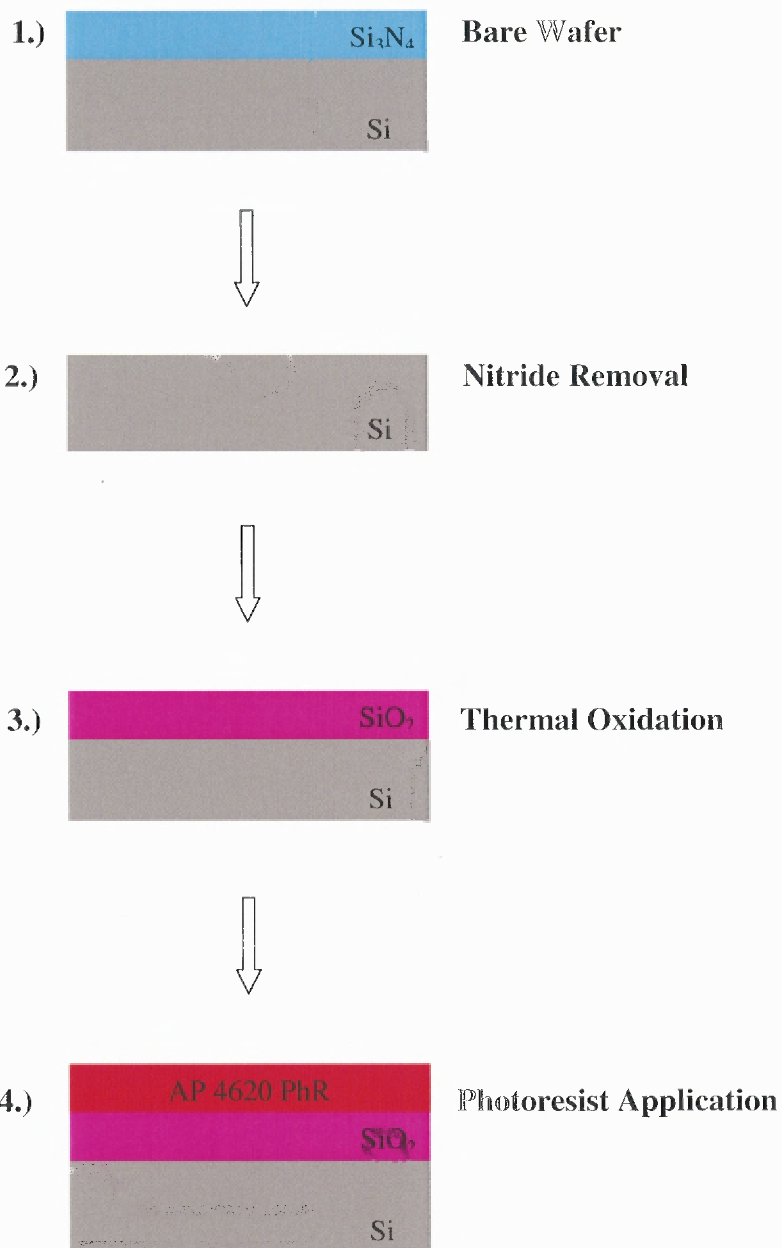


APPENDIX D

2D PROCESS FLOW-PHOTORESIST BONDING

A 2D process flow for the photoresist bonding of the wafers was constructed using Microsoft word.

Vias - Top Wafer of Sandwich



PhotoMask Used for Patterning

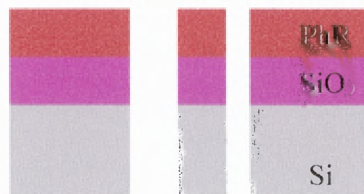
5.)

Patterning, UV Exposure, and Photoresist Development

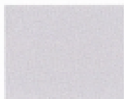
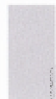
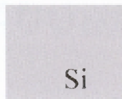
6.)

RIE Oxide Etch

7.)

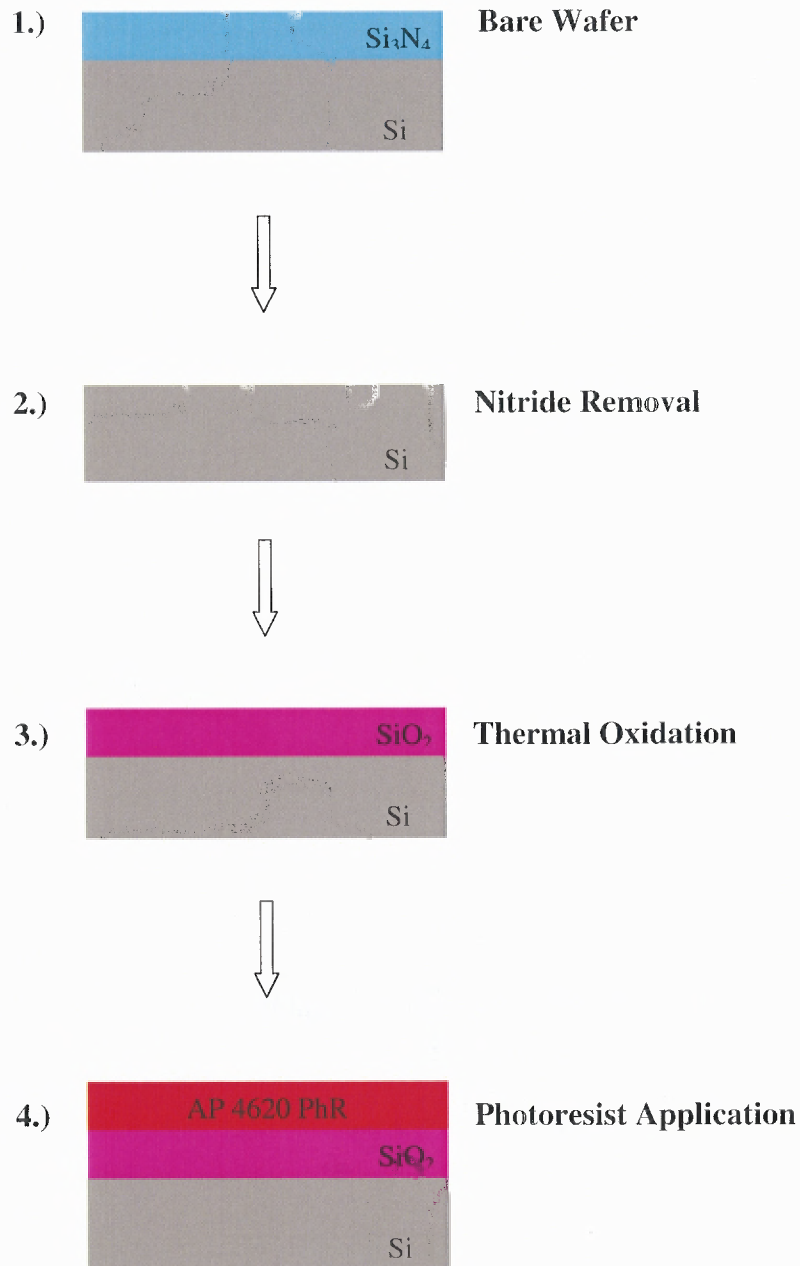


DRIE Silicon Etch

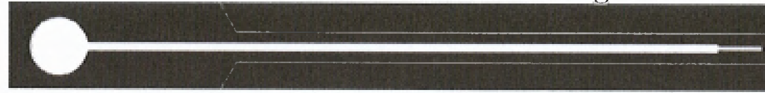
8.)   

Photoresist Removal and RIE Oxide Etch

Fluidic Channel Patterning and Etch – (Bottom Wafer of Sandwich)



PhotoMask Used for Patterning

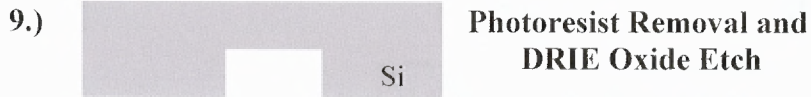


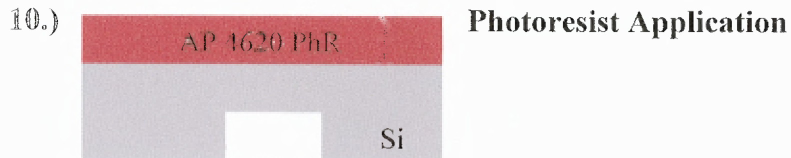
5.) Patterning, UV Exposure, and Photoresist Development

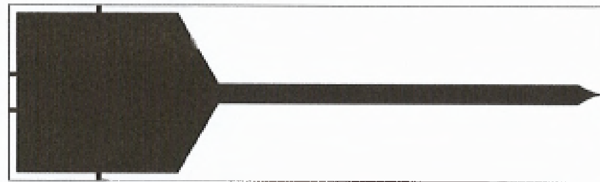
6.) RIE Oxide Etch

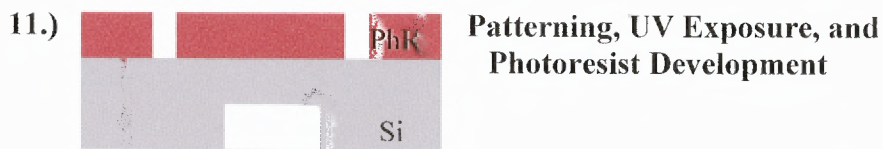
7.) DRIE Silicon Etch

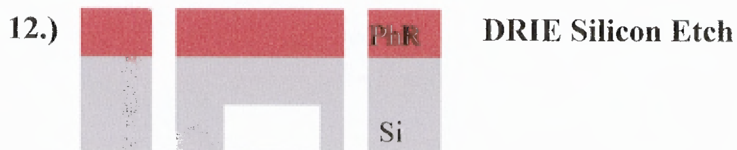
8.) Flip Wafer


9.)
Si
Photoresist Removal and
DRIE Oxide Etch


10.)
AP 1620 PhR
Si
Photoresist Application


PhotoMask Used for Patterning


11.)
PhR
Si
Patterning, UV Exposure, and
Photoresist Development

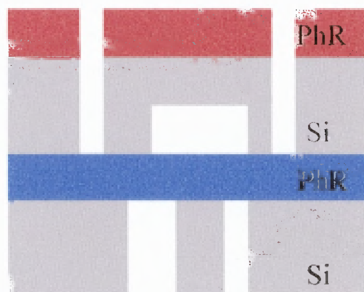

12.)
PhR
Si
DRIE Silicon Etch

Photoresist Bonding – Both Wafers

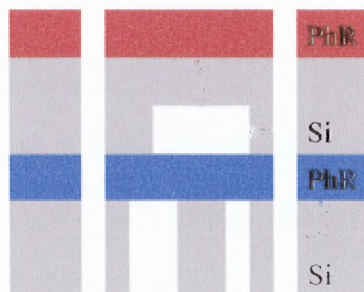
Top Wafer (Vias) **Bottom Wafer**

1.)

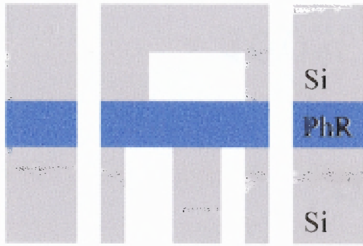
Spin Coat Top Wafer w/thin Photoresist

2.) 

Bond both Wafers In Bonder

3.) 

DRIE Silicon Etch

4.) 

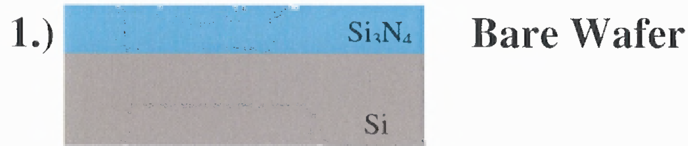
Photoresist Removal

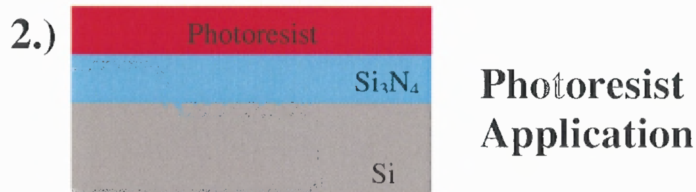
APPENDIX E

2D PROCESS FLOW-FUSION BONDING

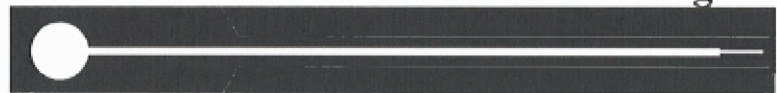
A 2D process flow for the fusion bonding was created using Microsoft word.

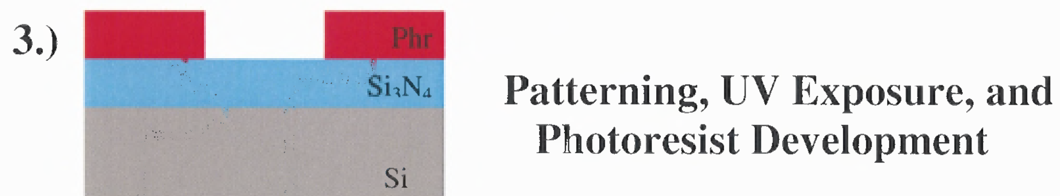
Fluidic Channel Patterning and Etch – (Bottom Wafer of Sandwich)



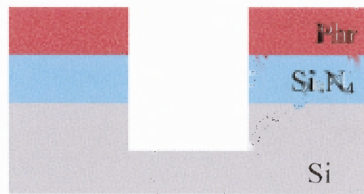


PhotoMask Used for Patterning





5.)

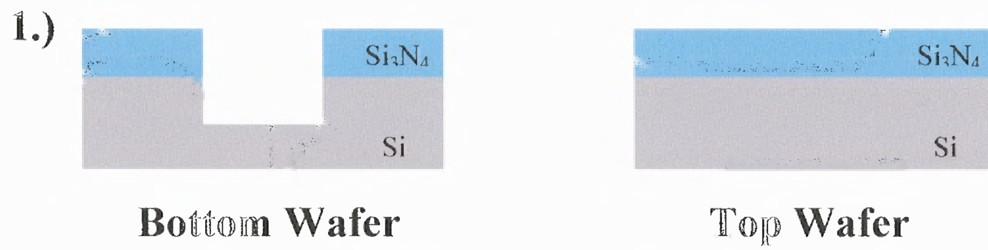


DRIE Silicon Etch

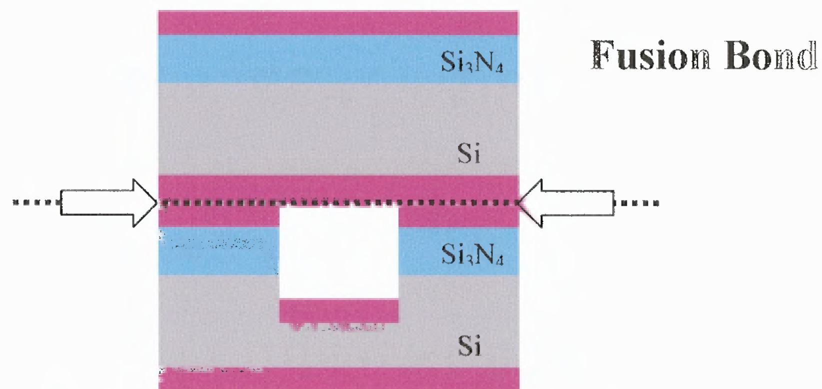
6.)

Photoresist Removal

Fusion Bonding – Both Wafers

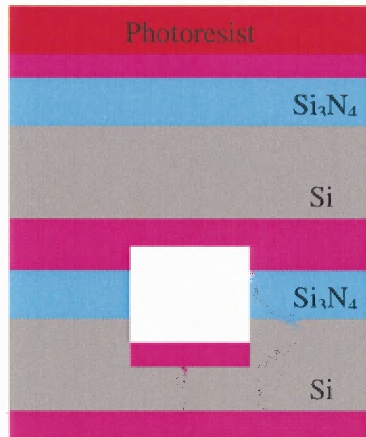


3.)



Vias - Top Wafer of Sandwich

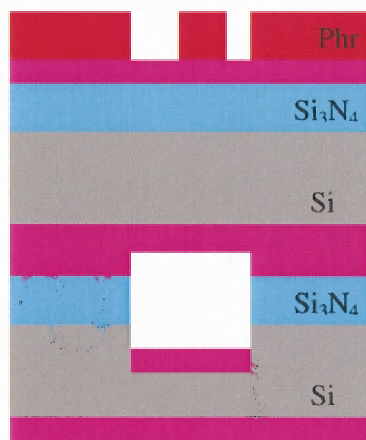
1.)



Photoresist Application

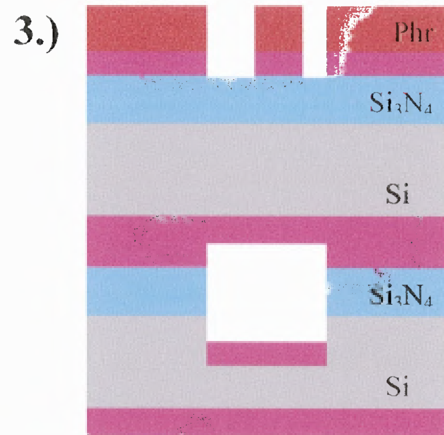
PhotoMask Used for Patterning

2.)



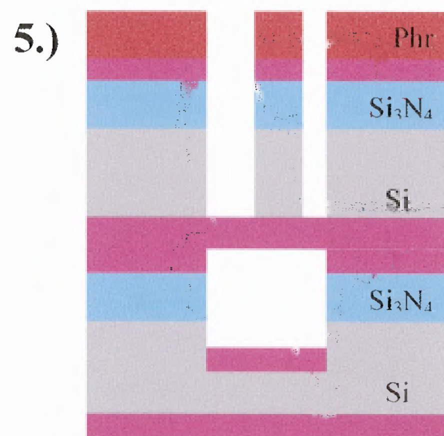
Patterning, UV Exposure, and Photoresist Development

3.)



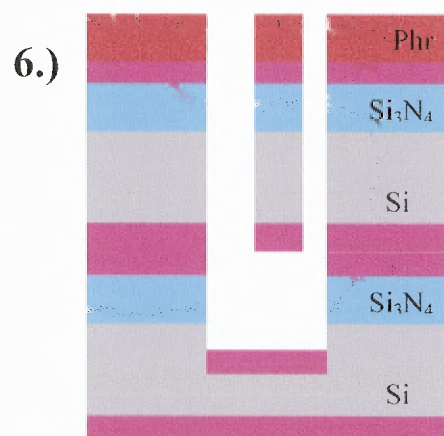
RIE Oxide Etch

5.)



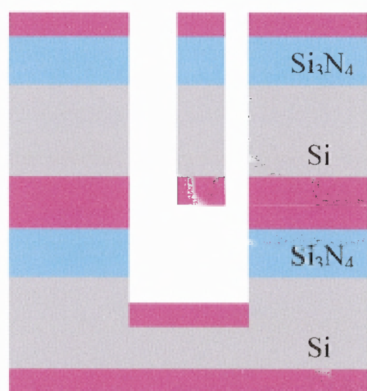
DRIE Silicon and Nitride Etch

6.)



RIE Oxide Etch

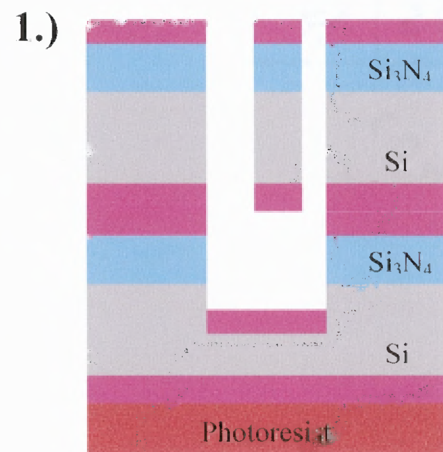
7.)



Photoresist Removal

Needle Periphery Patterning and Etch to Bottom of Sandwhich

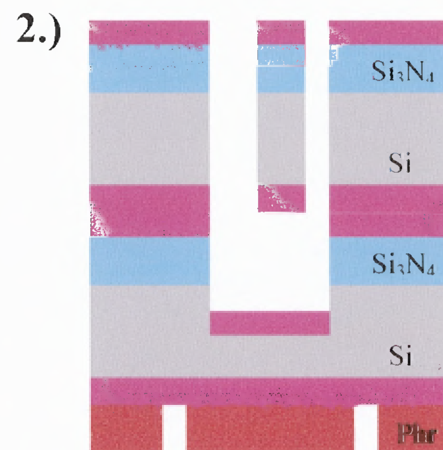
1.)



Photoresist Application

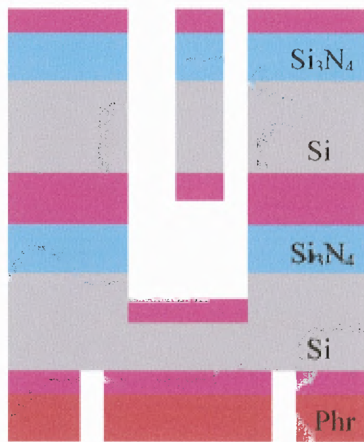
PhotoMask Used for Patterning

2.)



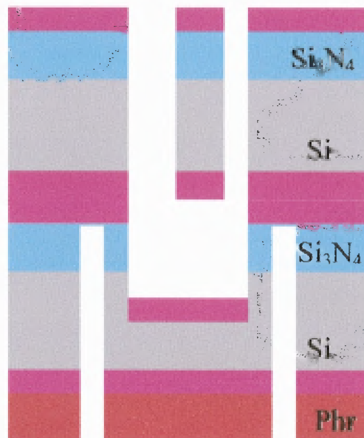
Patterning, UV Exposure, and Photoresist Development

3.)



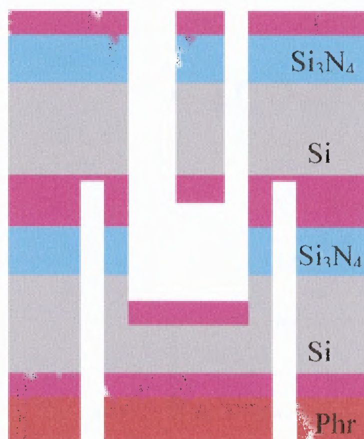
RIE Oxide Etch

4.)



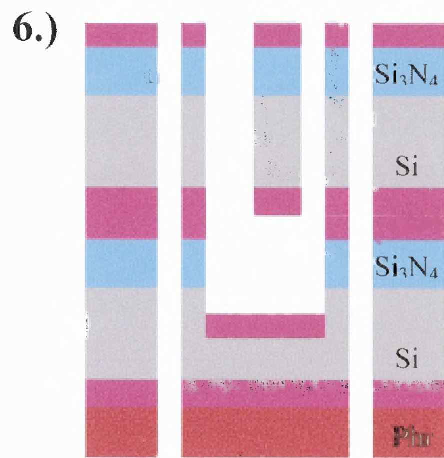
RIE Silicon and Nitride Etch

5.)

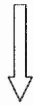


RIE Oxide Etch

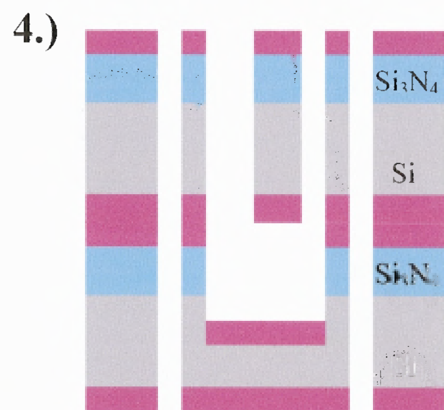
6.)



DRIE Silicon and Nitride Etch



4.)



Photoresist
Removal

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