

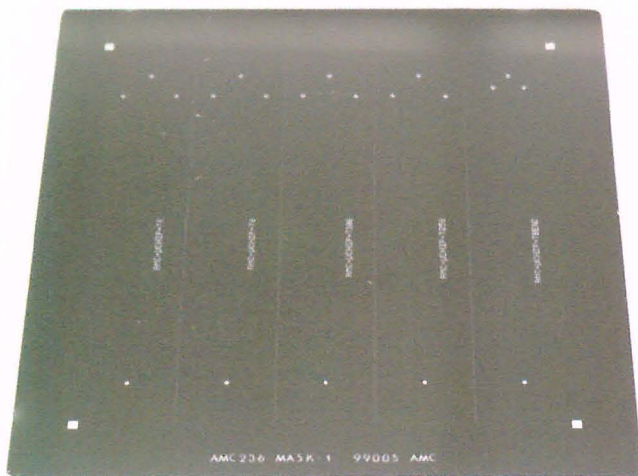


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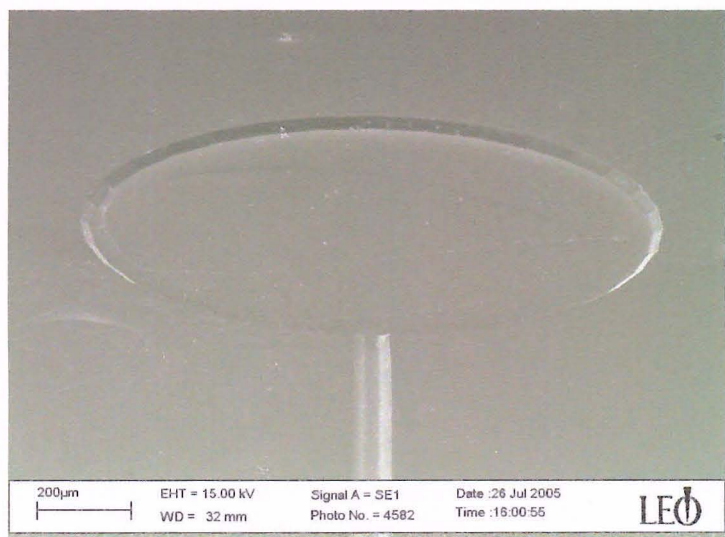
NanoFab Glass Microfluidic Device Fabrication Manual

Complete Process Description and Trouble-Shooting Guide
Version 1.0

Kelly Tai
July 2005



Microfluidic Devices Mask



SEM Image of a Microfluidic Channel (200x)

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Chapter 1

Introduction:

Processesing required to fabricate glass microfluidic channels and devices

Microfluidics – the manipulation of small volumes in an enclosed microchip – has become an increasingly popular technology for biochemical applications such as DNA separation and single cell analysis. Microfabrication techniques used in the realization of microfluidic devices offer several advantages. It is ideally suited to producing the same component with exactly the same specifications in large volumes. Other advantages include a reduction in sample and reagent consumption, the ability to automate liquid handling, and a reduction in analysis time due to the shortened diffusion zones associated with a miniaturized system. Microfabrication techniques also make it possible for microfluidics to be integrated with other components to form total analysis systems (μ TAS).

More and more glass substrates are being used to fabricate microfluidic devices. The high chemical resistance of glass makes it ideal for devices used for biomedical applications. Glass is also suitable for applications that require optical detection. Lastly, glass is economical and relatively easy to process using the standard techniques developed for silicon.

For some applications it may be desirable to incorporate electrodes for applying separation voltage or for electrochemical sensing. The electrodes are usually fabricated from a biocompatible material such as gold.

1.1 Glass: A Basic Primer

Glass is the name for a family of materials primarily composed of silica along with secondary materials (usually metal oxides) to modify its properties (melting point, mechanical properties etc.). You need to ensure that you are using the proper glass for your application.

Sheets of optically flat glass are made by float processing. A layer of molten glass is extruded from a furnace onto the surface of a tub of molten tin. The top surface of the glass cools in air and the bottom surface of the glass cools on the molten tin. In this way, optically smooth surfaces are formed on the top and bottom without additional grinding and polishing.

How the glass is made affects its properties. Firstly, the side that touches the molten tin will contain traces of tin. The dissolved tin causes problems with the fabrication of etched structures in the glass. Therefore, when fabricating microfluidic devices, all of the processing must be done on the side that was exposed to air. Suppliers will often mark which side contains tin. Secondly, the cooling speed of the glass during float processing will affect its surface roughness. If the glass was made quickly, it will have a wavy surface.

Table 1. Glass Compositions

Glass	SiO ₂	B ₂ O ₃	Al ₂ O ₃	NaO	K ₂ O	Other
Soda Lime	72%	N/A	N/A	13%	N/A	11% CaO 4% other
BoroFloat®/Borosilicate	70-80%	7-15%	1-7%	0-5%	0-5%	0-8% other
Pyrex (Corning 7740)	80%	13.1%	2.25%	3.5%	1.1%	0.05% Fe ₂ O ₃
Wheaton Borosilicate	69.15%	10.8%	5.9%	8.6%	1.2%	CaO, MgO, ZnO
Corning 0211	65%	9%	2%	7%	7%	7% ZnO 7% TiO
Corning Vycor	96.4%	3%	0.5%	N/A	N/A	N/A

There are many types of glass and similar glasses from different suppliers may have different properties (e.g. Borosilicate glass). It should also be noted that since the properties that the glasses are optimized for are typically not what we use the glass for, companies can change their recipes without notice.

Common Types of Glass:

- BoroFloat®: a borosilicate glass.
- Corning 0211: microscope cover glass. A high-quality glass that can only be purchased in relatively thin sheets.
- Soda Lime: cheap glass. Used in microscope slides and mask blanks.
- Pyrex: boro-aluminosilicate glass. Higher melting point than soda lime glass. Commonly used for microfluidics.
- There are many other commercial glasses available. Manufacturers cover many of these glasses and their properties.

Corning: http://www.corning.com/lifesciences/technical_information/techdocs/descglasslabware.asp - 7913

Schott: <http://www.schott.com/english>

4" x 4" 0211 and BoroFloat® substrates are available for purchase through the NanoFab. The side containing tin in the BoroFloat® substrates are marked with a **T** in black permanent ink in the top left-hand corner. Users should re-mark their substrates with a diamond scribe prior to processing (piranha will wipe off the ink).

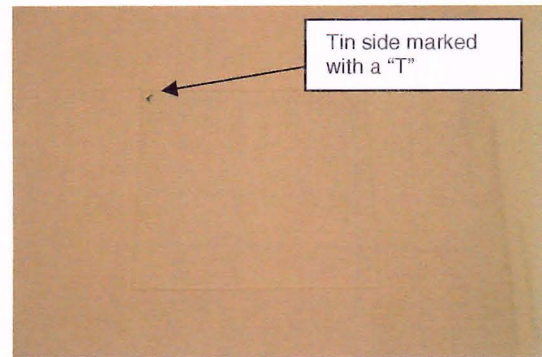


Figure 1. 4" x 4" BoroFloat® Substrate

1.2 Description of Process Steps

DC Sputtering

Sputtering – one type of physical vapour deposition (PVD) – is a method of depositing a thin film onto the surface of a substrate. A basic sputtering system consists of a reaction chamber, a vacuum source, and a power supply. The substrate and sputter target are placed into the chamber. After the vacuum pump evacuates the chamber, a process gas (typically argon) is introduced and converted into a plasma by the large potential generated by the power supply. The energized ions generated from the plasma are accelerated towards the sputter target. The bombardment energy of the ions cause target atoms to be ejected with enough energy to be deposited onto the substrate.

Film properties will vary widely by modifying the sputtering parameters. Process parameters that influence sputtering rate include base pressure, the flow rate and pressure of the process gas, and power.

Photolithography

A photomask design is transferred onto the thin-film masking layer via a series of steps collectively referred to as photolithography. First, a layer of light-sensitive material, photoresist, is spin-coated onto the masking layer. Radiation exposure changes the solubility of the photoresist with respect to unexposed areas. A photomask is then placed on the substrate, and the substrate is exposed to UV light. Finally, the substrate is placed in a developer solution. Exposed photoresist will be removed by the developer, with the result being that the pattern on the mask has been transferred to the photoresist.

Wet Etching

A photoresist image formed on the surface of the wafer is transferred to an underlying thin film layer by etching. Metal layers are typically etched using wet chemistry, more commonly referred to as wet etching. Etchants can be customized to etch just about anything in a controlled manner, making wet etching a simple yet powerful technique. However, one limitation of wet etching is that it is isotropic; wet etches will etch as quickly under a mask as they etch downwards. Wet etching cannot be used reliably to etch features smaller than 3 μm .

Etch reproducibility is also an issue since etch rate is dependent on several parameters. The “rate-determining step” can be:

- By-product accumulation – bath should be changed when etch rate has increased by a factor of 2 to the etch rate of fresh etchant.
- Mass transfer – etch rate increases with agitation of the etch bath
- Temperature – etch rate increases with temperature
- Substrate size – a smaller piece will tend to etch faster simply because it is easier to agitate

Wet etching is commonly performed via a “bucket” etching technique. The substrates to be etched are simply placed in a container filled with the etchant. The etchant may be physically agitated to improve etch rate and uniformity.

Fusion Bonding

Fusion bonding is often employed in forming glass – glass and silicon – silicon bonds. The bonding process requires flat substrates and smooth surfaces. The substrates should also be cleaned of particulates, organics, and metallic contamination immediately prior to bonding; cleanliness is the key to a successful bond. After surface cleaning and preparation, an initial bond is performed at room temperature. Next, a thermal fusion process is performed to increase bond strength. Thermal fusion temperatures are dependent on the properties of the glass, and should be above the annealing point but below the softening point to avoid deformation. The ramping rate of the furnace is also important. If the substrates are heated or cooled too quickly, stress will be induced into the substrates.

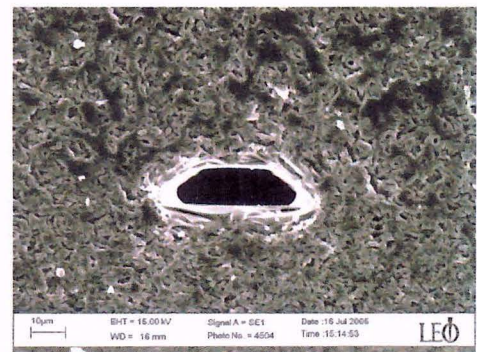


Figure 2. SEM cross section of a glass microfluidic channel (2000x). Note the absence of a gap between the two bonded substrates. Channel is shown upside down, the top plate is on the bottom of this image

1.3 Further Information

Sputtered thin films make suitable etch masks due to their high densities and good adhesion. A commonly used masking layer for glass is a combination of chromium/gold (Cr/Au) for its compatibility with standard microfabrication processes. Cr is used as an adhesive for a gold protective layer. The best Cr/Au masking layers reported to date have a Cr layer of thickness ranging from 20 – 40 nm, and an Au layer of thickness ranging from 150 – 200 nm. Multi-layer films can be conveniently sputtered in the NanoFab using **Doug** (R&D co-sputter deposition system). **Doug** also has RF sputter cleaning capabilities; a 5-minute RF pre-sputter clean should be performed on the substrate in the system immediately prior to DC sputtering. **Doug** is capable of sputtering 1 substrate at a time if sputtering is performed with RF cleaning, and 3 substrates if sputtering is performed without RF cleaning.

The metal etch mask on the glass substrate is then patterned using standard photolithography and chemical wet etching. The NanoFab uses tri-iodide to etch Au, a standard gold etchant consisting of potassium iodate (KI), iodine (I₂), and water. For Cr the NanoFab uses a commercial Chrome Etch containing nitric acid (HNO₃), ceric ammonium nitrate, and water. Over time a layer of chromium oxide forms at the thin-film/glass interface. After Cr etching it is sometimes visible as a translucent gray film. This oxide is removed with a dip etch in Au etchant.

Glass etching in the NanoFab is performed using a HF-based wet chemical etch. The isotropic etch produces hemispherical channel structures. HF reacts with the oxides in the glass to produce insoluble precipitates that act as masking spots during etching. Therefore, HNO₃ is added in the mixture to convert the precipitates to soluble salts, decreasing etch roughness. HF is extremely hazardous. As such, prior to glass processing users should be aware of first aid measures for HF (calcium gluconate).

Electrodes are fabricated using standard techniques for deposition, lithography, and wet etching. For biological applications, a biocompatible material such as Ni should be used as the adhesion layer for Au electrodes. To achieve proper fusion bonding the combined thickness of the electrodes (adhesion layer + electrode) should not exceed 100 nm. The electrodes can be placed on the device substrate or a blank cover substrate, and each method has its advantages and disadvantages. Fabricating the electrodes onto the device substrate requires the use of a photoresist that is at least as thick as the topography (features will have poor coverage if the surface topography is of a similar thickness or greater than the photoresist), limiting this technique to microfluidic designs with shallow channel depths. Fabricating the electrodes onto the cover substrate simplifies photolithography, but complicates alignment during bonding.

Depending on device application, access ports can either be drilled into the etched device substrate or drilled into a blank cover substrate:

- For devices without electrodes, it is recommended that access ports be drilled into a blank cover plate. This eliminates the risk of breaking a device substrate during the drilling process. A set of mask dimensions should be provided when submitting your request. Refer to **Microfluidic Devices Photomask** for a list of dimensions of the standard mask.
- For devices with electrodes, it is recommended that access ports be drilled into the device substrate. Drilling into the device substrate eliminates the problems associated with fabricating electrodes over etched features. The Cr/Au/photoresist masking layer acts as a template during the drilling process.

The ECE department machine shop is capable of performing this service using a diamond drill bit or a waterjet cutter. Crystal bond used to secure the cover substrate during drilling can subsequently be removed using methanol or ethanol.

Masking layers for the device substrate are then stripped off using wet chemical etching. Immediately before bonding the cover and device substrates are removed of contamination. The initial bond is performed at room temperature. Several attempts may be required to obtain satisfactory bonding and alignment between the two plates. Finally, a thermal fusion process is performed in a box furnace to increase the bond strength of the microfluidic devices.

1.4 Microfluidic Devices Test Photomask

The microfluidic devices photomask (ID AMC236-MASK-1) contains four designs. There are a total of five devices on the mask:

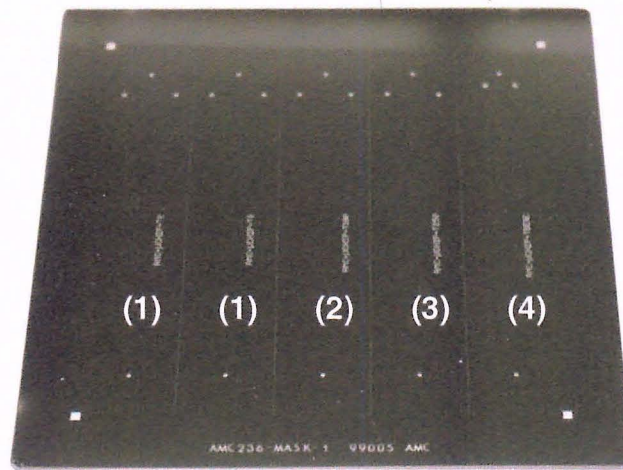


Figure 3. Microfluidic Devices Photomask: (1) AMC- μ CHIP-T0 (2) AMC- μ CHIP-T100 (3) AMC- μ CHIP-T250 (4) AMC- μ CHIP-TBEND

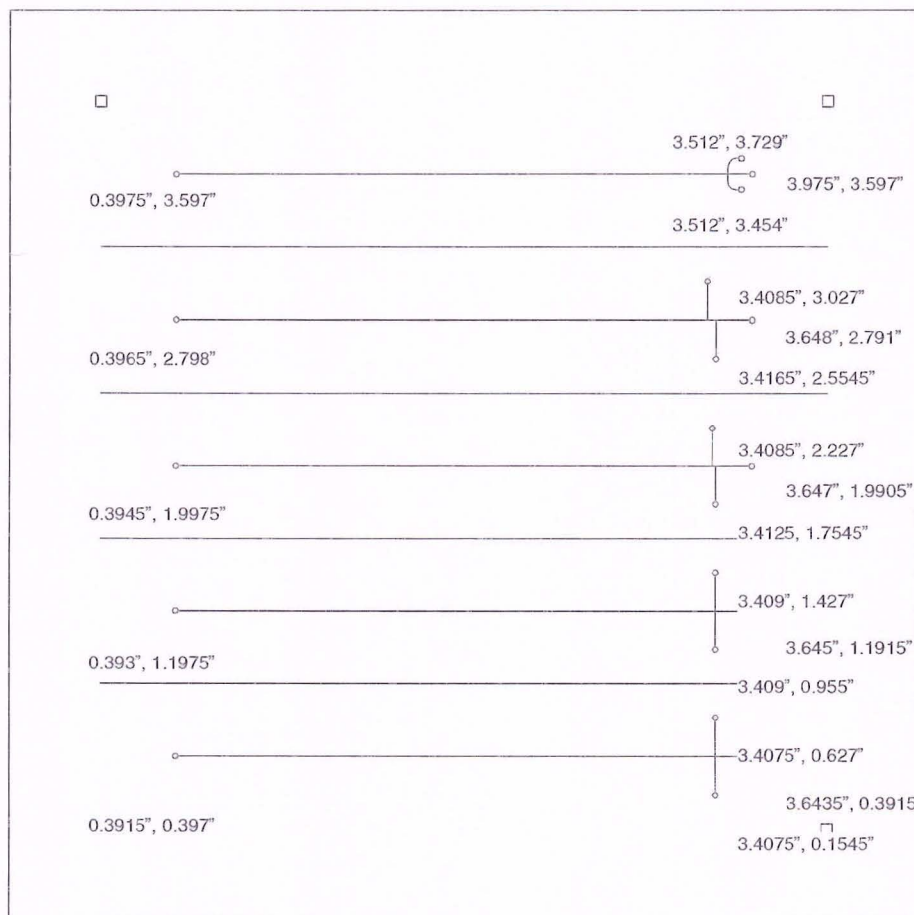
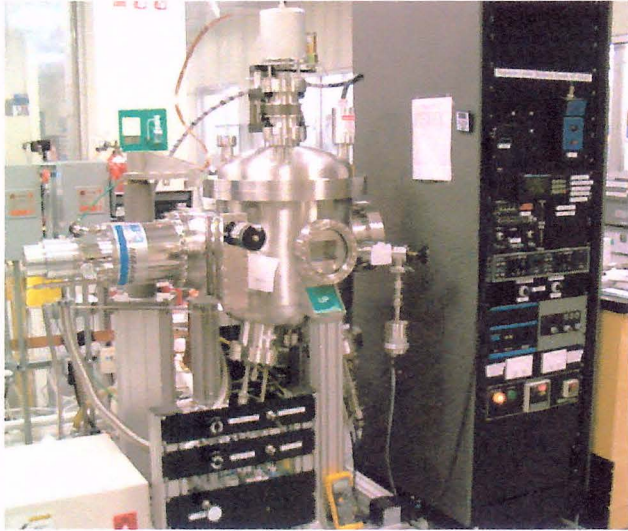


Figure 4. Microfluidic Devices Photomask Dimensions

1.5 Equipment Used to fabricate devices

Deposition Equipment



R & D Co-Sputter Deposition System (Doug)

A planar magnetron sputter system with two sources. The substrate holder has heating and RF biasing capabilities.

Processing Equipment



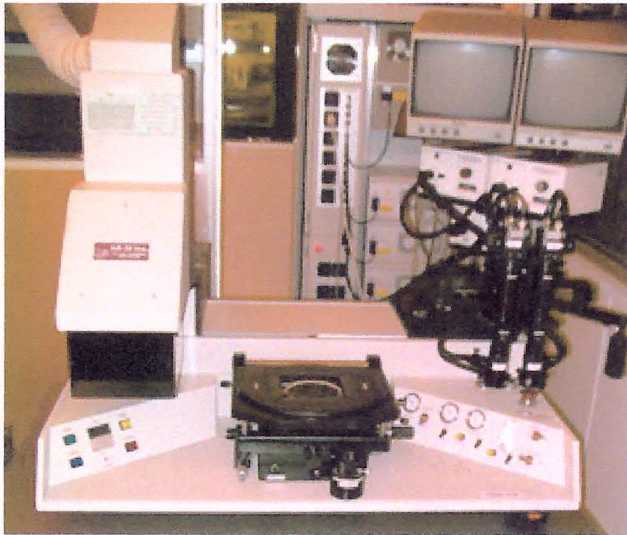
Wet Deck and Spin Rinse Dryer

The wet deck is used for all chemical processing including piranha cleaning and wet etching. The spin rinse dryer is an automated rinser and dryer.



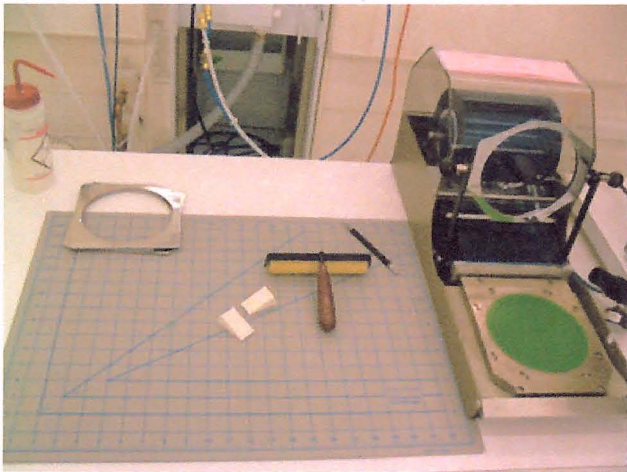
Lithography Process Station

The solitec spinner is used to spin coat photoresist. The convection oven is used to bake the substrates. The wet deck is used to develop and rinse substrates that have been exposed.



AB-M Contact Mask Aligner (Oscar)

The mask aligner is used to expose samples to UV light to transfer the mask pattern to the resist on the sample.



Glass Bonding Area



Box Furnace

The furnace is used to thermally fuse substrates that have been bonded.



Diamond Touch Dicing Saw

This dicing saw is dedicated to non-silicon substrates such as glass, quartz, and ceramic. It is used to dice the bonded substrates into individual devices.

Characterization Equipment



Alphastep Profilometer

The profilometer measures film thickness by running a stylus attached to a capacitor sensor over the surface of a film.



Inspection Microscope

The microscope is used for substrate characterization throughout processing.

References

Cheng J, Kricka LJ, editors. Biochip Technology. Philadelphia, Pa: Harwood Academic, 2001.

Minqiang Bu, Tracy Melvin, Graham J. Ensell, James S. Wilkinson, Alan G. R. Evans, A new masking technology for deep glass etching and its microfluidic application, *Sensors and Actuators A* 115 (2004) 476 – 482.

Mårten Stjernström, John Roeradde, Method for fabrication of microfluidic systems in glass, *J. Micromech. Microeng.* 8 (1998) 33 – 38.

Che-Hsin Lin, Gwo-Bin Lee, Yen-Heng Lin, Guan-Liang Chang, A fast prototyping process for fabrication of microfluidic systems on soda-lime glass, *J. Micromech. Microeng.* 11 (2001) 726-732.

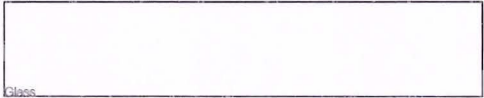
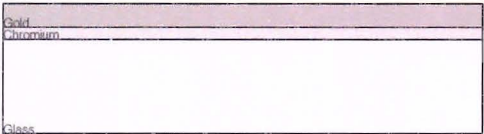
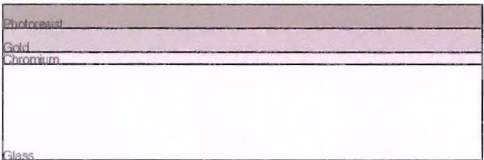
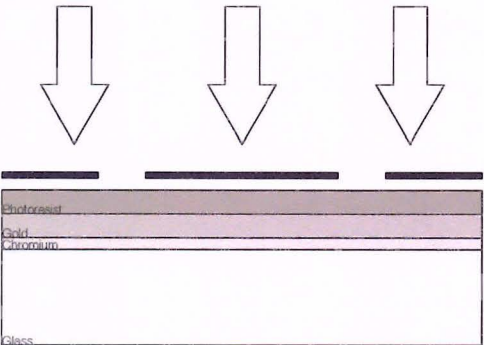
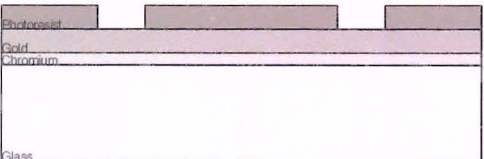
Ciprian Iliescu, Jianmin Mio, Francis E.H. Tay, Stress control in masking layers for deep wet micromachining of Pyrex glass, *Sensors and Actuators A* 117 (2005) 286-292.

Chapter 1 Appendix A: Glass Data Sheets

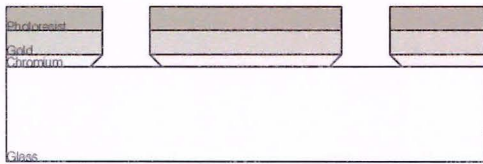
Chapter 2

Process Flow Diagrams

2.1 Fabricating a Glass Microfluidic Device: Process Flow:

- 1)  Substrate and Mask Cleaning:
Remove contaminants with piranha (3:1 H₂SO₄:H₂O₂)
- 2)  Masking Layer Deposition:
20 - 40 nm Cr, 150 - 200 nm Au.
- 3a)  Masking Layer Photolithography, Part A:
Spin on photoresist.
- 3b)  Masking Layer Photolithography, Part B:
Expose photoresist.
- 3c)  Masking Layer Photolithography, Part C:
Develop photoresist.

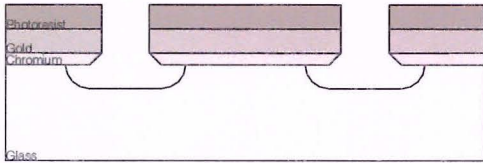
4)



Masking Layer Etching:

Wet-etch Au/Cr masking layer.

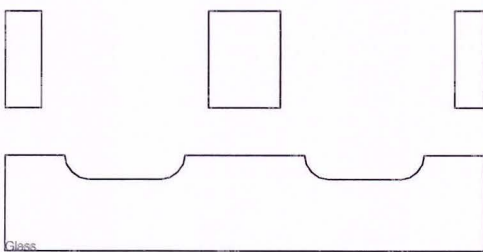
5)



Glass Etching:

Wet-etch substrate.

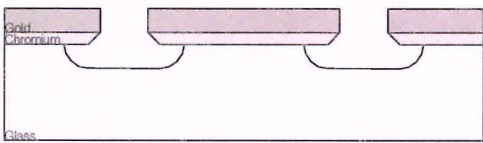
6)



Drilling Access Ports:

Drill ports into cover substrate. Clean cover substrate with methanol/ethanol.

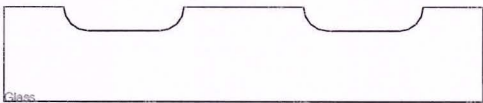
7a)



Device Substrate Stripping, Part A:

Remove photoresist in acetone bath, followed by a 3:1 piranha.

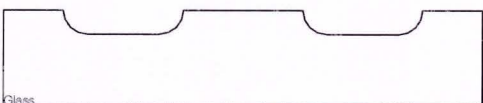
7b)



Device Substrate Stripping, Part B:

Remove Au and Cr masking layers by wet-etch.

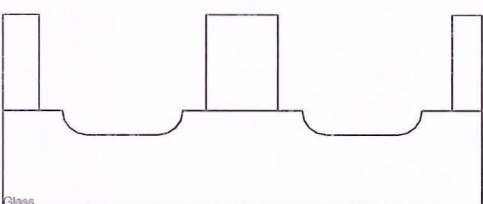
7c)



Device Substrate Stripping, Part C:

Strip remaining traces of metal with a 2:1 piranha.

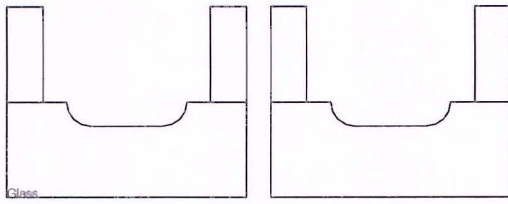
8)



Fusion Bonding:

Remove contaminants from substrates with a 3:1 piranha. Prep bonding surfaces with soap and water. Bond at room temperature and anneal.

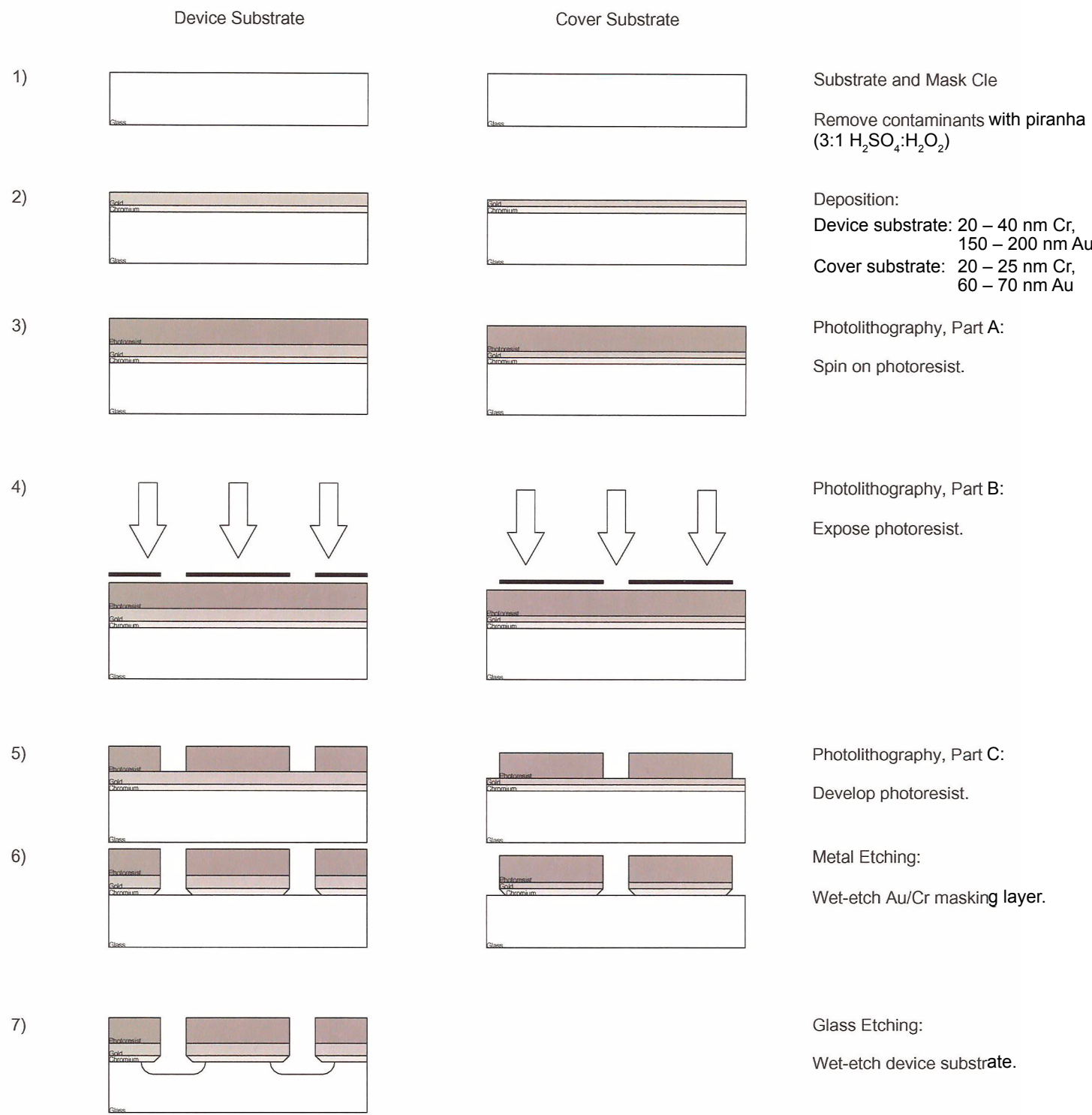
9)



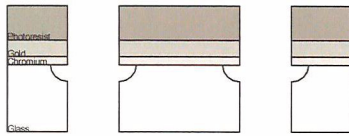
Device Dicing:

Cut bonded substrates into individual devices.

2.2 Process Flow: MicroFluidic Devices with Gold Electrodes



8)



Drilling Access Ports:

Drill ports into device substrate and clean with methanol/ethanol.

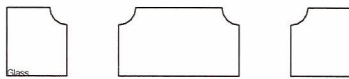
9)



Substrate Stripping, Part A:

Remove photoresist in acetone bath, followed by a 3:1 piranha.

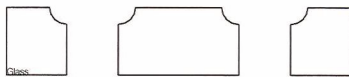
10)



Substrate Stripping, Part B:

Remove Au and Cr masking layers by wet-etch.

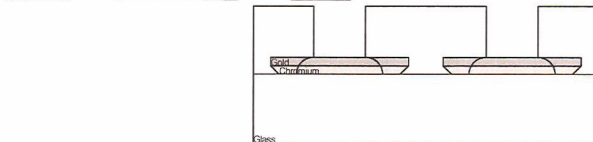
11)



Substrate Stripping, Part C:

Strip remaining traces of metal with a 2:1 piranha.

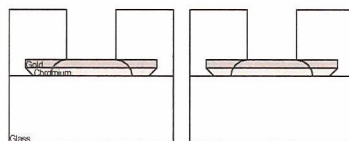
12)



Fusion Bonding:

Remove contaminants from substrates with a 3:1 piranha. Prep bonding surfaces with soap and water. Bond at room temperature and anneal.

13



Device Dicing:

Cut bonded substrates into individual devices.

Chapter 3

Characterization of Gold/Chrome Masking Layers for Glass Etching

3.1 Experiment

In glass etching, adhesion of the masking layer to the substrate is crucial. Poor film adhesion offers a path for the glass etchant to creep into the film/glass interface, resulting in undercutting. Zero undercutting would produce the theoretical best case, a 1:1 ratio of sideways etch to downwards etch. Residual stress in the masking layer is also important. A thin film under high tensile stress will peel back on itself to increase undercutting.

This document reports on the masking layer technologies available in the NanoFab. Using a masking layer of 30 nm Cr / 180 nm Au as a baseline, the performance of thin films obtained from various sputtering systems was examined.

Corning 0211 glass substrates were utilized in this experiment. Immediately prior to film deposition each substrate was cleaned in piranha (3:1 H₂SO₄:H₂O₂) for 15 minutes, rinsed with DI water, and spin-dried.

Substrate	Sputtering System	Base Pressure (Torr)	RF Clean Prior to Deposition
Sample 1	Doug	10 ⁻⁶	N/A
Sample 2	Doug	10 ⁻⁶	5 minutes
Sample 3	Doug	10 ⁻⁵	10 minutes
Sample 4	Doug	10 ⁻⁷	5 minutes
Sample 5	Floyd	10 ⁻⁸	5 minutes

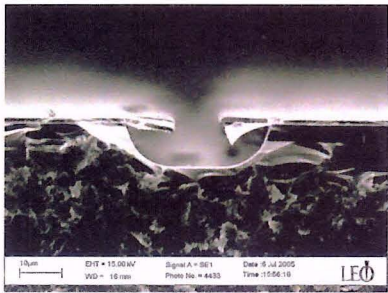
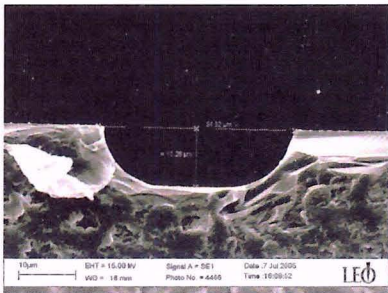
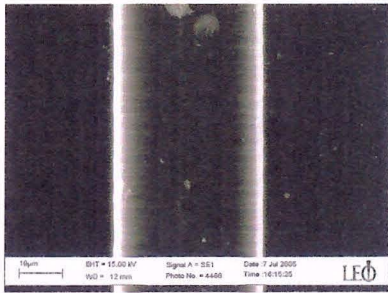
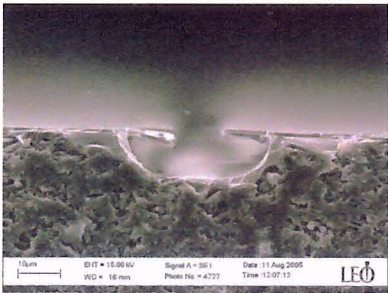
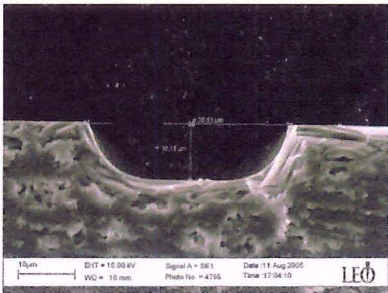
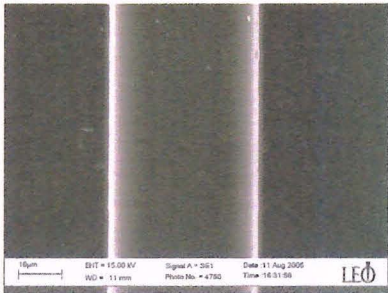
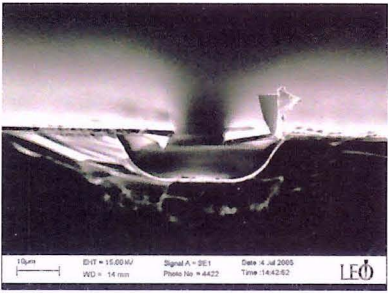
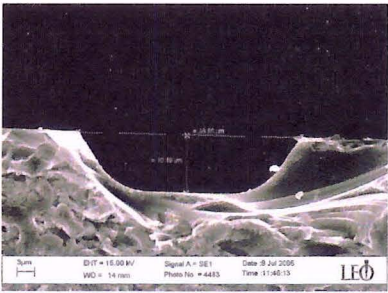
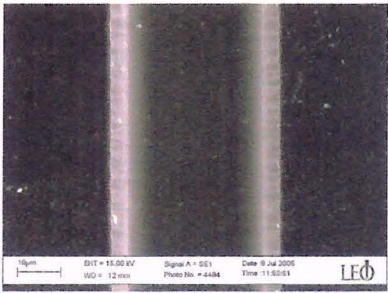
Table 1. Test Parameters

HPR 504 photoresist was used as a mask for Cr/Au etching. Prior to each exposure the photomask was cleaned in a cold piranha bath for 15 minutes. Cr/Au masking layers were etched using the standard chemical wet etches used in the NanoFab (tri-iodide for Au and a commercial Chrome Etch for Cr). Finally, a HF-based wet chemical etch was used to etch a depth of approximately 10 μ m. The etch bath was agitated with a magnetic stir bar to improve etch uniformity.

Table 1 summarizes the test parameters for each sample. Unlisted sputtering parameters such as process gas flow rate/pressure and power were kept constant for each substrate (with the exception of Sample 5, which was sputtered on a different system).

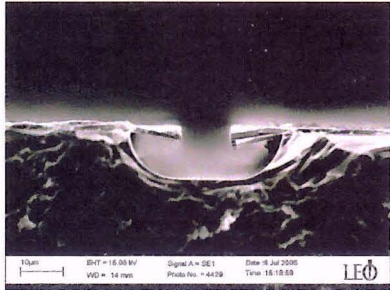
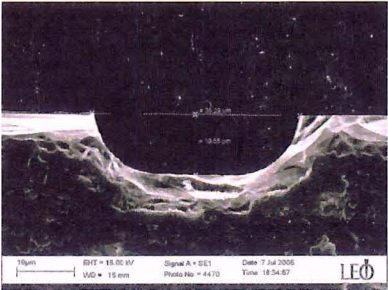
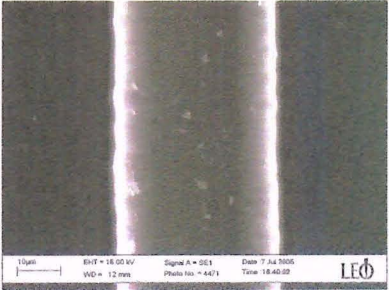
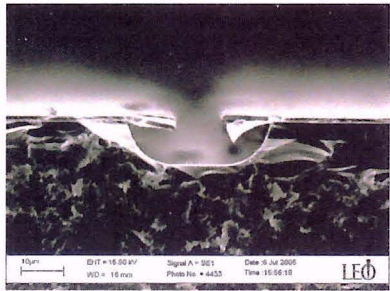
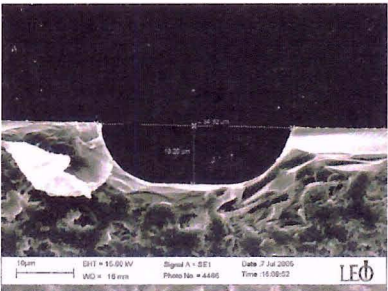
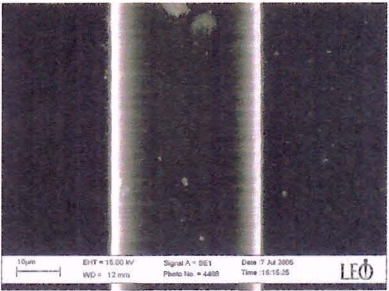
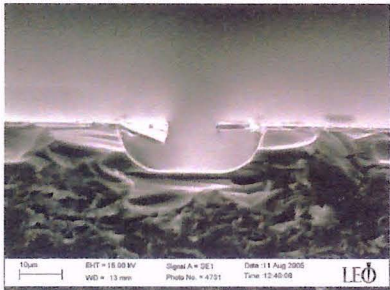
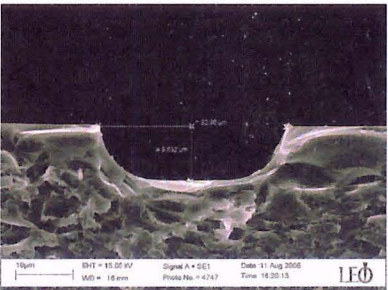
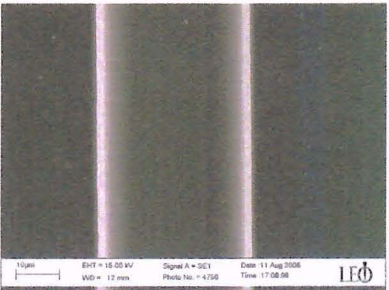
To investigate the effects of undercutting, cross-sections of a 10- μ m etched channel were taken from each substrate and analyzed.

3.2 Effects of Base Pressure

Base Pressure	Cross-section with masking layers after glass etching (3000x)	Cross-section after glass etching (4000x)	Overhead view after glass etching (3000x)
10^{-8} Torr (Sample 2)			
10^{-7} Torr (Sample 4)			
10^{-6} Torr (Sample 5)			

The overhead SEM images verify that masking layer adhesion improves with decreasing base pressure. With the 5-minute RF pre-sputter clean performed on all 3 samples prior to deposition, the etched edges are already fairly smooth at base pressure of 10^{-6} Torr. However, the aspect ratio of the etched structures decreases with base pressure; the calculated aspect ratio is **0.83** at 10^{-6} Torr, drops negligibly to **0.82** at 10^{-7} Torr, and falls to **0.68** at 10^{-8} Torr.

3.3 Effects of RF Pre-Sputter Cleaning

RF clean prior to deposition	Cross-section with masking layers after glass etching (3000x)	Cross-section after glass etching (4000x)	Overhead view after glass etching (3000x)
0 min (Sample 1)			
5 min (Sample 2)			
10 min (Sample 3)			

As expected, overhead SEM images verify that masking layer adhesion improves with RF pre-sputter cleaning. Sample 1 illustrates the ragged edges characteristic of non-uniform adhesion. A 5-minute RF pre-sputter clean prior to deposition yields noticeably smoother edges and an increase in aspect ratio from **0.76** to **0.83**. When the RF pre-sputter time is doubled to 10 minutes, masking layer adhesion appears to have improved with a slight decrease in aspect ratio (**0.80**).

Chapter 4

Protocols and Detailed Process Descriptions

This chapter describes in detail how to process fabricate glass blanks into microfluidic channels and devices. This is a step by step guide in how to make the devices.

The focus of this document is on the fabrication of microfluidic devices without electrodes. However, due to the similarities in the fabrication processes for microfluidic devices with and without electrodes, the document has been organized as follows:

- The procedure is divided into subsections. Each subsection lists the instructions required to complete one step in the process flow.
- Instructions are written for fabricating devices without electrodes.
- When applicable, at the end of each subsection deviations/additions to the instructions are provided for users who would like to fabricate devices with electrodes.

This chapter is broken into process sections or protocols. Detailed descriptions on how to operate the equipment is given in the appendices.

Step 1: Substrate and Mask Cleaning.....	3
Step 2: Masking Layer Deposition.....	4
Step 3: Masking Layer Photolithography.....	5
Step 4: Masking Layer Etching.....	8
Step 5: Glass Etching	10
Step 6: Drilling Access Ports	13
Step 7: Device Substrate Stripping.....	14
Step 8: Fusion Bonding	16
Step 9: Device Dicing	21

Appendix A: Glass etching calculations using the Alphastep

Appendix B: Preparing the cover substrate for bonding

Appendix C: Bonding using the AB-M Mask Aligner

Step 1: Substrate and Mask Cleaning

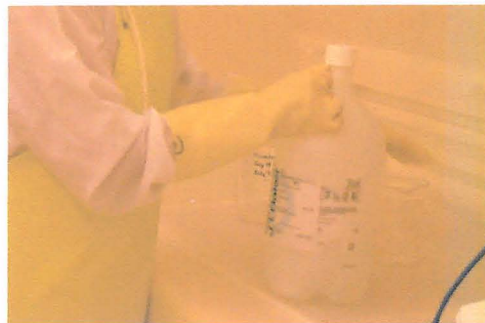
Estimated completion time: 4 hours

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Wetdeck• Spin Rinse Dryer	<ul style="list-style-type: none">• 4" x 4" glass substrate• H_2O_2• H_2SO_4

1. Place the substrates into a Teflon carrier.
2. Label 2 glass beakers for the following: 1) H_2SO_4 ; 2) H_2O_2 . Label 1 glass container for the piranha mixture.
3. Determine the volume of piranha required to immerse the substrate, and calculate the amounts of H_2SO_4 and H_2O_2 required to make a 3:1 piranha. For example, 1000 mL of piranha requires 750 mL of H_2SO_4 and 250 mL H_2O_2 .
4. Put on safety gear.
5. Pour required amount of H_2SO_4 into its corresponding beaker.
6. Pour H_2SO_4 into the glass container.
7. Pour required amount of H_2O_2 into its corresponding beaker.
8. Add H_2O_2 to the glass container.

The temperature of the mixture will exceed 100°C . Piranha cleans best at temperatures greater than 90°C .
9. Immerse the substrates in the piranha mixture for 15 minutes.
10. Remove the substrates from the piranha mixture and perform a 5-cycle DI water rinse in the dump rinser.
11. Dry the substrates using the spin rinse dryer.
12. Allow the piranha mixture to cool to at least 40°C .
13. Immerse the microfluidic devices mask in the piranha mixture for 30 minutes.
14. Remove the mask from the piranha mixture and perform a 5-cycle DI water rinse in the dump rinser.
15. Dry the mask with a N_2 gun.

Devices with electrodes: The cover substrate should also be piranha cleaned prior to sputtering.



Step 7



Step 8



Step 9



Step 11

Step 2: Masking Layer Deposition

Estimated completion time: 2 hours

Equipment	Materials and Supplies
<ul style="list-style-type: none">Doug (R&D co-sputter deposition system)	<ul style="list-style-type: none">4" x 4" glass substrate0.0625" Au and Cr sputter targets

Refer to the operation manual for instructions on how to use Doug.

1. Replace the sputter targets with gold (Au) and chromium (Cr).
2. Uninstall the multi-substrate platter using a Phillips screwdriver.
3. Load substrate into chamber.

Substrate alignment will affect film uniformity, so load the substrate carefully. Non-uniformity of the masking layer results in variations in photoresist thickness and etch rates across the substrate.

4. Pump down the chamber to at least 2.6×10^{-6} Torr (this will take >30 minutes). Record the base pressure.
5. Turn on the Mass Flow Controller (MFC) and introduce the process gas (argon) into the chamber. Establish the following conditions:

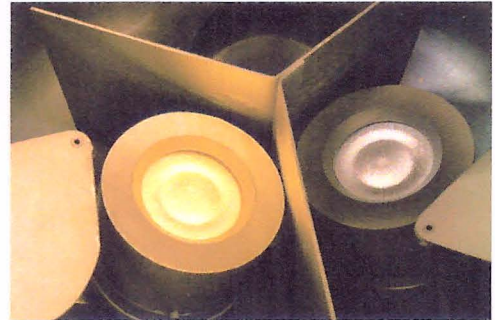
Flow rate 50 sccm
Pressure 7×10^{-3} Torr

Defects in the masking layer will form pinholes in the substrate after glass etching. Take this into consideration if sputtering parameters deviate from the given values.

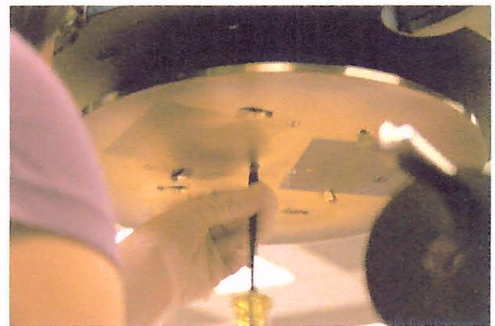
6. Turn on substrate rotation. Set speed control to 2.
7. Turn on DC/RF power supplies. Set the following parameters for the sputter targets:

Cr	300 W
Au	75 W
8. Using one of the sputter guns, light the plasma to establish RF biasing. The baratron pressure may need to be tuned to light the plasma. Time the RF bias for 5 minutes.
9. Burn in the Cr target for 5 minutes.
10. Sputter Cr for 1 minute and 20 seconds.
11. Burn in the Au target for 1 minute.
12. Sputter Au for 6 minutes.
13. Turn off power supplies, substrate rotation, and the MFC. Allow the substrate to cool for 15 minutes.
14. Vent the chamber to 7.6×10^{-2} Torr. Unload substrate.
15. Pump down the chamber.

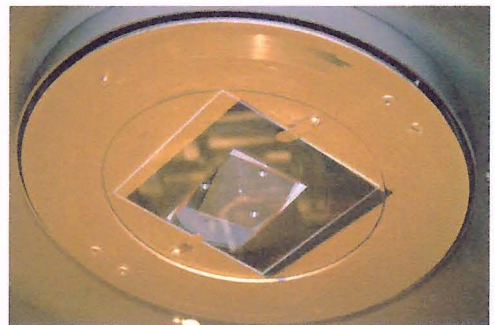
Devices with electrodes: Repeat Steps 3 – 15 on the cover substrate. Total sputtering time should produce a combined electrode thickness no greater than 100 nm.



Step 1



Step 2



Step 3

Step 3: Masking Layer Photolithography

Estimated completion time: 60 – 90 minutes

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Solitec Spinner• Convection Soft Bake Oven• AB-M Mask Aligner• Lithography Wet Deck• Inspection Microscope• Alphastep Contact Profilometer	<ul style="list-style-type: none">• 4" x 4" glass substrate:<ul style="list-style-type: none">20 – 40 nm Cr150 – 200 nm Au• HPR 504 Photoresist• Shipley Microposit 354 Developer• Acetone and Isopropanol

1. Check that the following parameters are set for the Solitec Spinner:

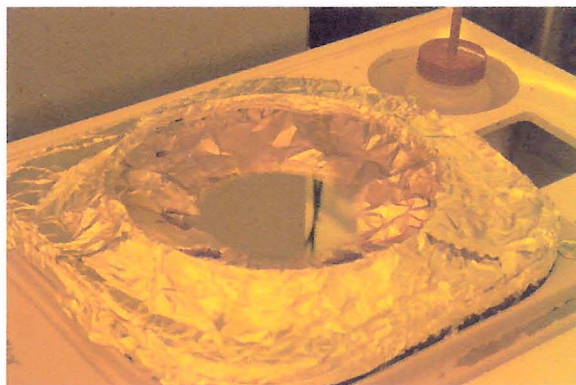
Spread: 500 rpm, 10 seconds
Spin: 4000 rpm, 40 seconds

2. Ensure that the large metal chuck (3.5" diameter) is installed on the spinner.
3. Pour enough HPR 504 photoresist into a labeled glass beaker to coat one glass substrate (5 – 10 mL). Cover this beaker with a second glass beaker.
4. Position the substrate and secure onto the metal chuck by pressing **VACUUM**. Turn on the spinner by pressing **START** to visually verify that the substrate is centered. Reposition if necessary.

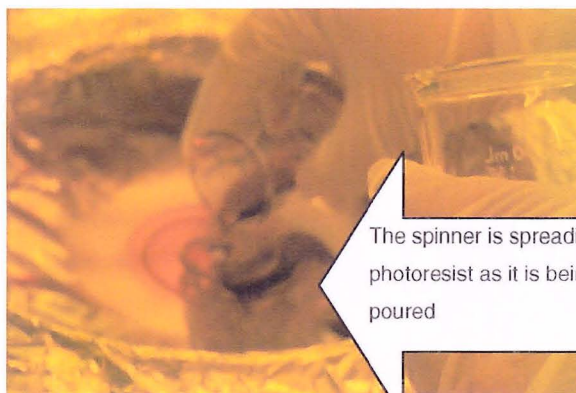
Be careful when working with glass. Glass substrates are fragile and have sharp edges!

5. Blow loose particles off of the substrate with the N₂ gun.
6. Press **START**. As the substrate is spinning, pour on a small amount of photoresist. Place the lid onto the spinner.
7. Once the spinner has stopped remove the lid, turn off the vacuum, and retrieve the substrate.
8. Visually verify that an even layer of photoresist has been spun onto the substrate. If necessary, clean off the substrate with acetone and isopropanol, and repeat Steps 4 - 7.
9. Place the substrate on a rack and bake in the convection soft bake oven for 30 minutes at 115°C.
10. Cool the substrate for 15 minutes.
11. Verify that the exposure time setting on the AB-M contact mask aligner is 4 seconds.
12. Remove mask shield. Load the mask using the pins on the mask holder as guides. The chrome on the mask should be face down. Place the mask shield over the mask after verifying that the O-rings on the holder and shield are secure. Turn on the mask vacuum.

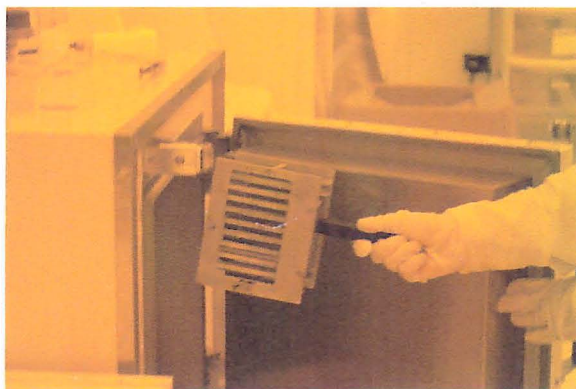
13. Verify that the mask chuck fits the substrate. Replace the



Step 4



Step 6



Step 9

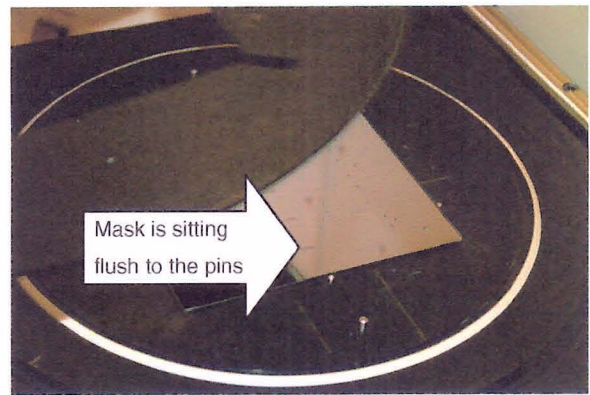
mask chuck if necessary.

14. Lift the mask frame and place the substrate onto the chuck.
15. Turn on the substrate vacuum to secure the substrate onto the chuck.

If the pressure gauge for the substrate vacuum does not change, increase N_2 flow through the mask aligner.

16. Lower the mask frame.
17. Raise the chuck until a small gap remains between the chuck and the mask. Level the chuck by pressing the chuck-leveling button.
18. Adjust the rotation, vertical positioning, and horizontal positioning of the chuck until the substrate is in line with the mask design.
19. Slowly raise the chuck until the substrate comes into contact with the mask. As the two surfaces meet, one will see concentric bands of light form due to thin film interference.
20. Turn on the contact vacuum to secure the substrate to the mask and turn off the substrate vacuum.
21. Expose the substrate for 4 seconds.
22. Turn on the substrate vacuum and turn off the contact vacuum.
23. Lower the chuck, raise the mask frame, and turn off the substrate vacuum.
24. Take the substrate off of the chuck.
25. Pour enough 354 developer into a labeled glass pan large enough to immerse the substrate.
26. Place the substrate into the developer. Start the timer.
27. Develop the substrate for approximately 20 seconds. Agitate the developer using a gentle rocking motion. Exposed photoresist will darken and dissolve off of the substrate in the developer.

Photoresist development is a visual process. Development time is strongly dependent on byproduct, so change the developer bath often. For good results, use the suggested development time as a guideline and pay close attention to the physical process.
28. Using tweezers, remove the substrate from the developer when no more photoresist comes off of the substrate. Stop the timer.



Step 12



Step 13



Step 14

29. Immediately rinse off the substrate with DI water.

Photoresist does not stop developing until the developer is washed off the substrate. Remember that a few seconds of additional development time will overdevelop the photoresist.

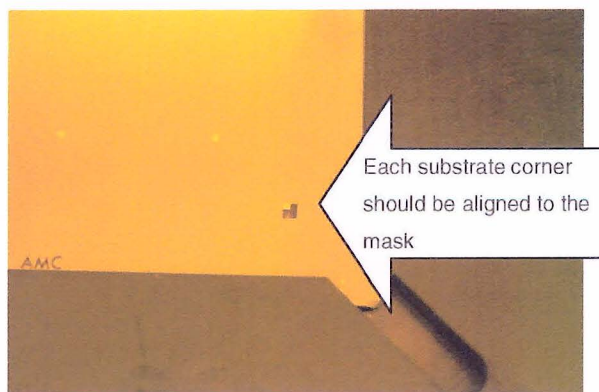
30. Dry the substrate with the N₂ gun.

31. Inspect the substrate on a microscope. Look for underdevelopment, overdevelopment, and miscellaneous defects. Any defects will be transferred to the etch mask.

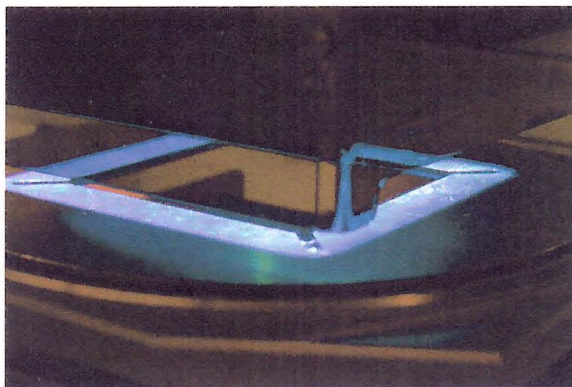
*Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of defects commonly encountered in photolithography.*

32. Using the Alphastep contact profilometer, measure the thickness of the photoresist. Readings should be consistent over the substrate.

Devices with electrodes: Repeat Steps 1 – 32 with the cover substrate.



Step 18



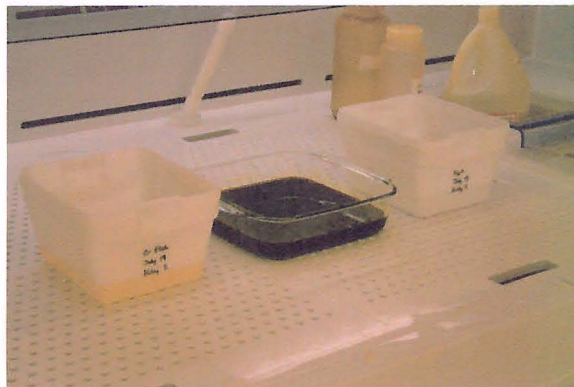
Step 21

Step 4: Masking Layer Etching

Estimated completion time: 30 minutes

Equipment	Materials and Supplies
<ul style="list-style-type: none">Wetdeck	<ul style="list-style-type: none">4" x 4" glass substrate:<ul style="list-style-type: none">20 – 40 nm Cr150 – 200 nm AuGold (Au) EtchantChrome (Cr) Etchant

- Label 1 glass container for Au Etch. Label 2 glass or plastic containers for the following: 1) Cr Etch; 2) Water.
- Fill each container with enough solution to submerge the substrate.
- Place the substrate in a substrate carrier (etch side up).
- Using a scooping motion, dip the substrate in and out of the Au etch bath (this motion causes the etchant to flow over the substrate). 5 - 10 dips are required to etch the gold. The end point is visual; as the gold is etched away, the remaining gold residue will appear brown. The blue-gray chrome film will become visible when the gold is completely etched away. When all of the gold has been removed, immediately dip the substrate into the container of water.

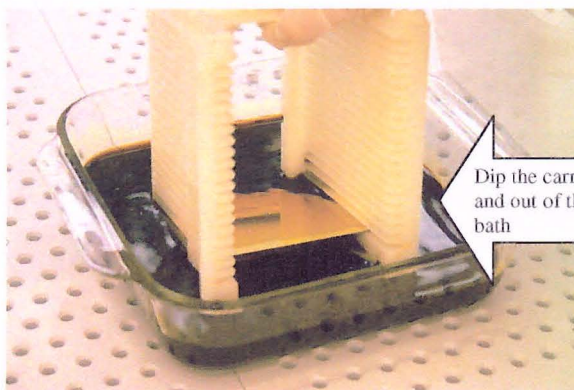


Step 2

Remember that it's better to under-etch than over-etch!

- Follow with a rinse of DI water.
- Dry the substrate with the N₂ gun.

Poor drying technique will result in particles drying onto the substrate. Hold the substrate in the palm of your hand. Aim the N₂ gun away from the body down the substrate surface. Proper drying technique is especially important for gold etching because gold will stick onto the substrate.



Dip the carrier in and out of the etch bath

Step 4

- Refill the container for DI water with fresh water.
- Inspect the substrate on a microscope. Look carefully for under-etching, over-etching and miscellaneous defects.

Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of defects commonly encountered in metal etching.

- Using a scooping motion, dip the substrate in and out of the Cr etch bath (this motion causes the etchant to run off of the substrate). 5 – 10 dips are required to etch the chrome. The chrome film will appear darker (as the oxide layer is reached) immediately before it is etched away. When all of the



Tilt the carrier to allow the etchant to run off before rinsing in water

Step 4

chrome has been removed, immediately rinse off the etchant by dipping the substrate into the container of water.

10. Follow with a rinse of DI water.
11. Dry the substrate with the N₂ gun.
12. Refill the container for DI water with fresh water.
13. Inspect the substrate on a microscope.

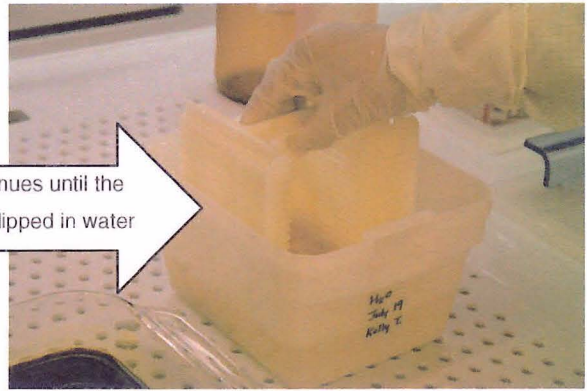
Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of defects commonly encountered in metal etching.

14. Hold the substrate up against light. A gray layer of oxide may be visible on the substrate.
15. Dip the glass piece into the Au etch bath for 1 second. Rinse off the etchant by dipping the substrate inside the container of water.
16. Follow with a rinse of DI water.
17. Dry the substrate with the N₂ gun.
18. Hold the substrate up against light. If there was a visible layer of oxide it should now be removed.
19. Perform a final inspection of the substrate on a microscope.

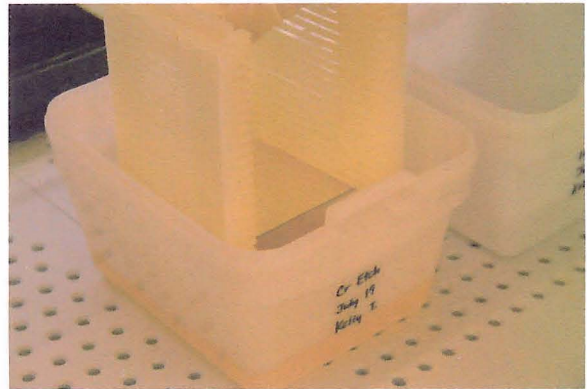
Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of defects commonly encountered in metal etching.

20. If the etchants can be re-used, store them in plastic containers with name, date, and chemical name. If the etchants cannot be re-used, dispose of them in their corresponding waste bottles.

Devices with electrodes: Repeat Steps 1 – 20 with the cover substrate. Remember that it will etch faster since the metal films sputtered on are not as thick.



Step 4



Step 9

Step 5: Glass Etching

Estimated completion time: 60 – 90 minutes

Refer to **Appendix A: Glass Etching Calculations Using the Alphastep** for details on how to perform all calculations in this procedure.

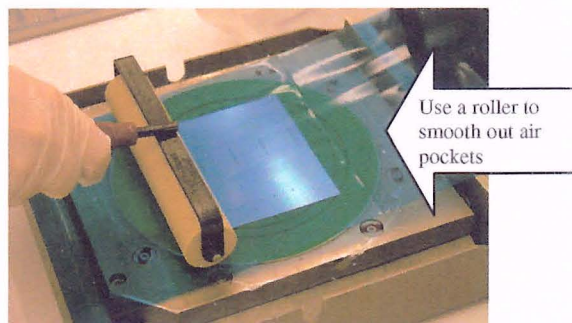
Note: HF is extremely hazardous. Be aware of the first aid measures for HF in the NanoFab.

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Heated Wafer Mounter• Wetdeck with Drop Deck• Magnetic Stir Plate• Alphastep Contact Profilometer	<ul style="list-style-type: none">• 4" x 4" glass substrate:<ul style="list-style-type: none">20 – 40 nm Cr150 – 200 nm Au• Glass Etchant• Calcium Chloride

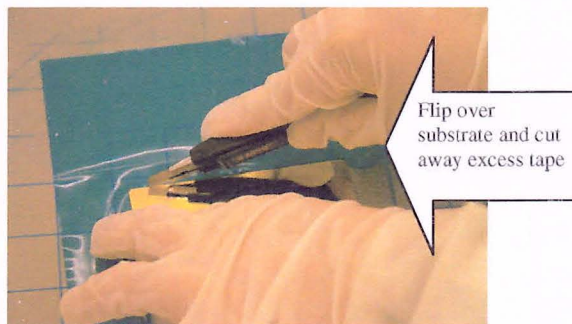
1. Select one feature that appears multiple times throughout the substrate. Using the Alphastep contact profilometer, measure the thickness of the masking layer by taking the average of at least three readings.
2. The HF solution will etch Corning 0211 glass at approximately $1.5 \mu\text{m}/\text{min}$ and Borofloat® at approximately $0.4 \mu\text{m}/\text{min}$. Calculate the approximate time the substrate will need to be in the etchant.

To calculate an accurate etch rate the substrate should be etched for at least 5 minutes. If the required etch is less than $10 \mu\text{m}$, an additional "dummy" substrate will be required.

3. Label 2 plastic containers for the following: 1) Glass Etchant; 2) H_2O .
4. Cover the backside of the substrate with adhesive tape on the wafer mounter. Verify that there are no air pockets meeting the edges of the substrate.
5. Place the first container on a stir plate in the drop deck. Set the stir plate's speed control to 3. Place a 1.5" magnetic stir bar in the center of the container and verify that the stir plate is operational.
6. Place the substrate in a substrate carrier (etch side face-up).



Step 4



Step 4

Insert a plastic divider into the carrier. Using the red iron rod, it should be possible to leverage the bottom of the carrier above the magnetic stir bar when it is placed in the container of glass etchant.

7. Put on safety gear.
8. Pour glass etchant into the first plastic container.
9. Fill the second plastic container with DI water and place it in the drop deck.
10. Etch rate calculation:

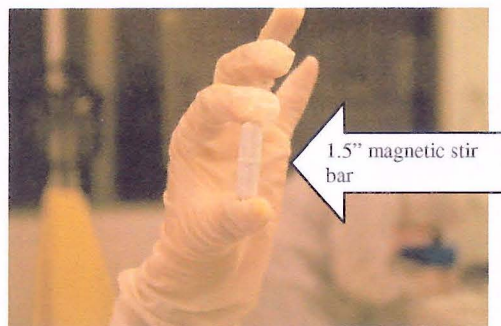
If the required etch is less than 10 μm , Step 10 should be performed on the "dummy" substrate.

- a. Using the iron rod, carefully immerse the substrate in the etch bath. Start the timer.
- b. At about 10 seconds before 5 minutes has elapsed, remove the substrate from the etch bath. Tilt the substrate holder so that the glass etchant runs off of the substrate.
- c. When 5 minutes has elapsed, dip the substrate into the container of DI water. Perform a 5-cycle rinse in the dump rinser.
- d. Dry the substrate with the N_2 gun.
- e. Using the Alphastep, measure the etch depth of the features selected in Step 1. Determine the remaining etch time required.

Note that the etch rate of the glass etchant is temperature-dependent. For deep etches you may find that the rate changes significantly over the course of an etch.

11. Substrate etch:

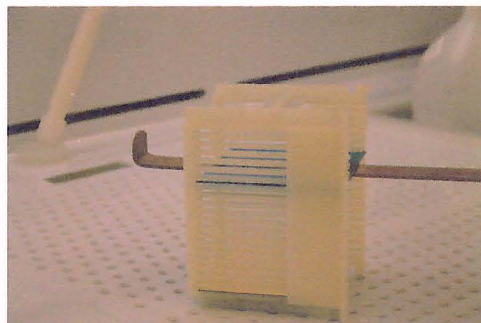
- a. Re-fill the second plastic container with DI water and place in drop deck.
 - b. Using the iron rod, carefully immerse the substrate in the etch bath. Start the timer.
 - c. At about 10 seconds before the required time has elapsed, remove the substrate from the etch bath.
 - d. When the required time has elapsed, dip the substrate into the container of DI water. Perform a 5-cycle rinse in the dump rinser.
 - e. Dry the substrate with the N_2 gun.
12. Using the Alphastep, measure the etch depth of the features selected in Step 1. If the channel was under-etched, calculate the



Step 5



Step 5



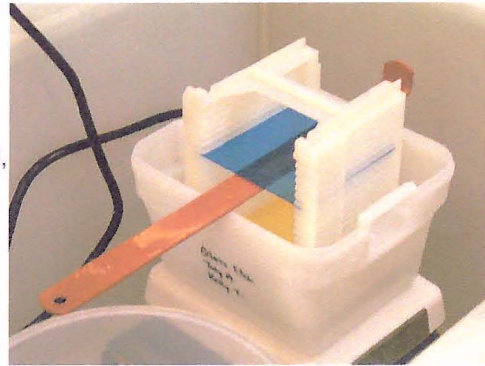
Step 6



Step 9

amount of time required to complete etching and repeat Step 11.

13. Remove the tape from the backside of the substrate.
14. If the etchant can be re-used, store in plastic bottle with name, date, and chemical name. If the etchant cannot be re-used, it must be neutralized with calcium chloride before it can be aspirated.
15. Rinse work surfaces and containers with calcium chloride. Clean up as required.



Step 10

Step 6: Drilling Access Ports

Estimated completion time: 60 minutes

Note: This document outlines the cleaning procedure after access ports have been drilled.

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Wetdeck	<ul style="list-style-type: none">• 4" x 4" glass substrate (access ports drilled)• Large plastic petri dish• Bristle brush• Methanol/ethanol• H₂O₂• H₂SO₄

1. Fill the petri dish lid with enough methanol to immerse the substrate.
2. Immerse the substrate in the methanol bath. Place the petri dish on top to prevent evaporation from taking place. Allow the substrate to soak for 15 minutes.
3. After 15 minutes, the crystal bond should be softened. Use a bristle brush to remove the softened crystal bond from the substrate.
4. Dispose of the methanol in a solvent waste bottle.
5. Repeat Steps 1 – 4.
6. Make a 2:1 piranha.
7. Immerse the substrate in piranha for 15 minutes.
8. Remove the substrate from the piranha mixture and perform a DI water rinse in the dump rinser for a 5-cycle rinse.
9. Dry the substrate using the spin rinse dryer.
10. Aspirate piranha once it has cooled to less than 40°C.

Step 7: Device Substrate Stripping

Estimated completion time: 2 hours

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Wetdeck• Spin Rinse Dryer• Alphastep Contact Profilometer	<ul style="list-style-type: none">• 4" x 4" glass substrate• Acetone• H₂O₂• H₂SO₄• Gold (Au) Etchant• Chrome (Cr) Etchant

Part A: Photoresist stripping

1. Place the device substrate, mask side face-up, into a substrate carrier.
2. Label 1 glass/plastic beaker for acetone.
3. Pour enough acetone in the beaker to immerse the device substrate.
4. Immerse the device substrate in acetone for 10 minutes.
5. Remove the device substrate from the beaker and perform a 5-cycle rinse in the dump rinser.
6. Dry the device substrate using the spin rinse dryer.
7. Dispose of the acetone in a solvent waste bottle.
8. Place the device substrate into a Teflon carrier.
9. Make a 3:1 piranha (see page 1 for instructions). The piranha will remove any remaining photoresist on the device substrate.
10. Immerse the device substrate in piranha for 15 minutes.
11. Remove the device substrate from the piranha mixture and perform a DI water rinse in the dump rinser for a 5-cycle rinse.
12. Dry the device substrate using the spin rinse dryer.
13. Aspirate piranha once it has cooled to less than 40°C.

Devices with electrodes: Repeat Steps 1 – 7 with the cover substrate.

Part B: Metal stripping

14. Place the device substrate, mask side face-up, in a substrate carrier.
15. Label 2 plastic/glass containers for the following 1) Au Etch; 2) Cr Etch.
16. Fill each container with enough etchant to submerge the device substrate.
17. Submerge the device substrate in the Au etch bath. Occasionally agitate the etch bath to remove by-product building up on the substrate. The blue-gray chrome will become visible as the gold is etched away.
18. Rinse thoroughly with DI water and dry with the N₂ gun.
19. Submerge the device substrate into the Cr etch bath. Occasionally agitate the etch bath to remove by-product building up on the substrate. The chrome layer will appear to darken immediately before it is etched away.

20. Rinse thoroughly with DI water and dry with the N₂ gun.
21. Submerge the device substrate into the Au etch bath for 1 minute. Occasionally agitate the etch bath to remove by-product building up on the substrate. Remove the substrate from the etch bath.
22. Rinse thoroughly with DI water and dry with the N₂ gun.
23. If the etchants can be re-used, store them in plastic containers with name, date, and chemical name. If the etchants cannot be re-used, dispose of them in their corresponding waste bottles.

Part C: Substrate Cleaning

24. Make a 2:1 piranha (see page 1 for instructions).
25. Immerse the device substrate in piranha for 15 minutes.
26. Remove the device substrate from the piranha mixture and perform a DI water rinse in the dump rinser for a 5-cycle rinse.
27. Dry the device substrate using the spin rinse dryer.
28. Aspirate piranha once it has cooled to less than 40°C.

Part D: Inspection

29. Inspect the device substrate on a microscope. Carefully examine each device to determine overall yield.
*Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of defects commonly encountered in glass etching.*
30. Using the Alphastep, measure the profiles of selected areas. Verify that the bottoms of the etches are smooth.
The profilometer tip is 10 nm wide, and as such, profiles obtained from the Alphastep are approximate. As the tip moves downwards into a channel, it will make irregular contact with the sides of the etched areas.

Step 8: Fusion Bonding

Estimated completion time: 24 hours

For applications involving smaller substrates where substrate alignment is critical, it is possible to perform the bonding step using the AB-M mask aligner. Refer to **Appendix B: Bonding Using the AB-M Mask Aligner**.

Note: Success with bonding is heavily dependent on the “cleanliness” of the clean room. It is advisable to attempt bonding only when the number of users in the NanoFab is relatively low.

Equipment	Materials and Supplies
<ul style="list-style-type: none">• Wetdeck• Glass Bonding Station• Thermolyne Box Furnace	<ul style="list-style-type: none">• 4" x 4" glass device substrate• 4" x 4" glass cover substrate• H_2O_2• H_2SO_4

Part A: Surface Cleaning

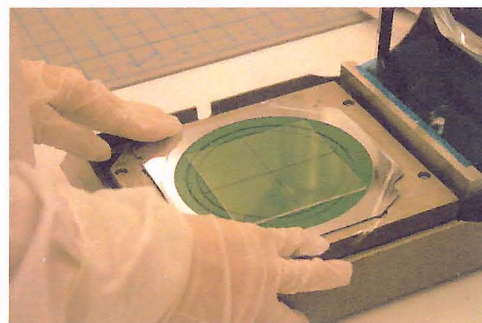
Make a 2:1 piranha (see page 1 for instructions).

Immerse the substrates in the piranha mixture for 15 minutes.

Remove the substrates from the piranha mixture and perform a DI water rinse in the dump rinser for a 5-cycle rinse.

Immerse the substrates in a container of H_2O . This prevents particulates from settling onto the substrates before bonding takes place.

Aspirate piranha once it has cooled to less than 40°C.



Step 9

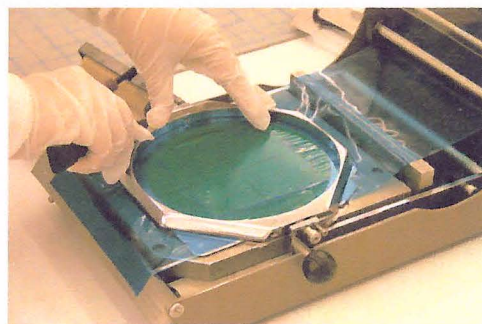
Part B: Bonding

Wipe down work surfaces with isopropanol.

Turn on the wafer mounter. Warm up for approximately 15 minutes or until the chuck is warm to the touch.

Take the device substrate out of the container of H_2O . Dry with a N_2 gun.

Center the device substrate onto the wafer mounter chuck,



Step 12

bond surface down.

Center a wafer frame onto the chuck.

Place a layer of tape over the device substrate and wafer frame. Smooth out the tape using a roller.

Cut off excess tape around the wafer frame. The tape should now be securing the device substrate to the wafer frame.

Check that the following parameters are set for the High Pressure Cleaning Station:

Clean Time = 5

Dry Time = 9

Place the device substrate inside the High Pressure Cleaning Station, ensuring that the wafer frame is secure. Close the lid of the cleaning station and flip the power switch to **ON**. The cleaning station should automatically begin washing.

Open the lid and take out the device substrate after the cleaning station has finished its drying cycle.

Place the device substrate, bond surface up, on the work surface.

Store the device substrate underneath a large glass petri dish.

Repeat Steps 15 – 22 for the cover substrate.

Devices with Electrodes: If you are working with small electrodes, cleaning the cover substrate in the high-pressure washer may lift them off. Refer to Appendix C for instructions on how to prep the cover substrate for bonding.

Remove the petri dish covering the device substrate.

Place two wafer frames on top of the wafer frame holding the device substrate. These two frames will act as a spacer between the device substrate and cover substrate while initial bonding takes place.

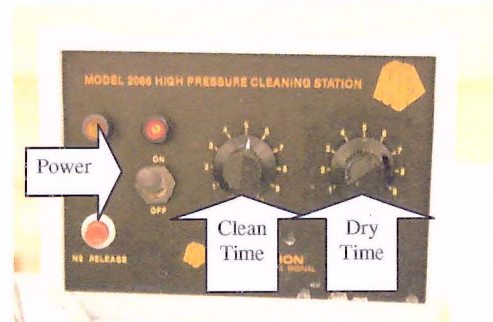
Place the cover substrate, bond surface down, on top of the stack of wafer frames.

Press on the tape to the left and right of the cover substrate, and then the tape to the front and back. Repeat until the two plates begin to form a bond. Once the two substrates make contact, the bonding process will take place fairly quickly.

Cut away the tape holding the bonded substrates to their respective frames.

Inspect the bonded substrate on a microscope.

Refer to the **Microfluidic Devices Fabrication Inspection Guide** for a list of commonly encountered defects encountered in bonding.



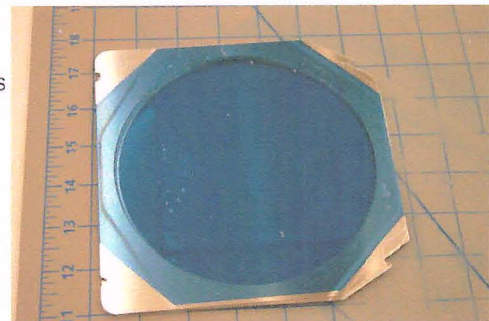
Step 13



Step 14

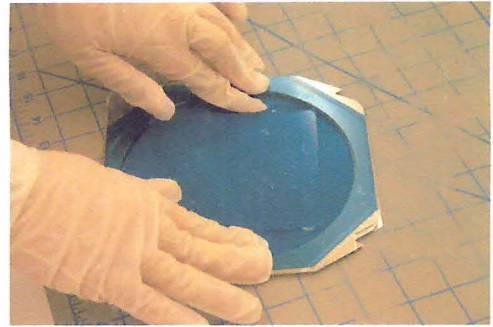


Step 20



Step 21

If the substrates need to be re-bonded, pry the substrates apart using an X-acto knife and repeat Steps 1 – 25.



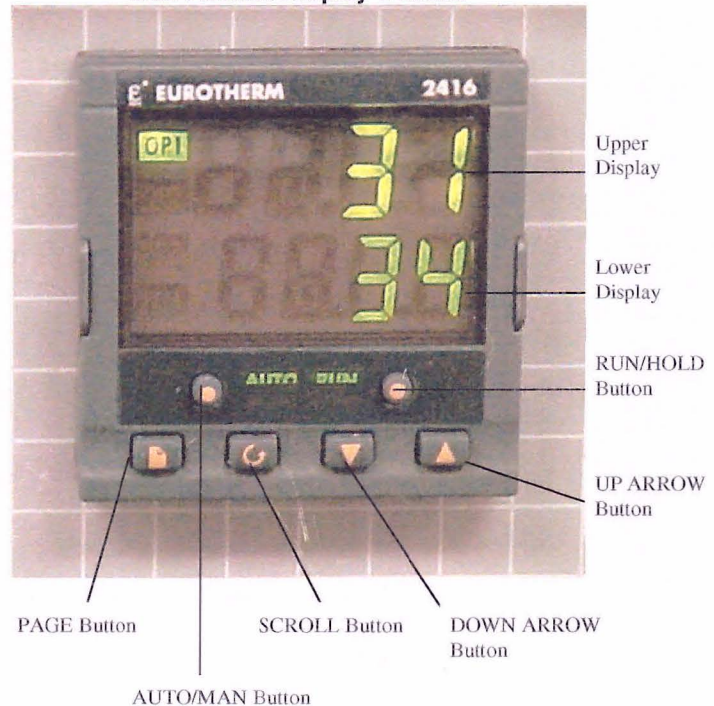
Step 22

Part C: Annealing

Thermolyne Box Furnace



Box Furnace Display Window



26. Turn on the Thermolyne Box Furnace Controller. By default the furnace will adjust its temperature to the previously entered setpoint on the Lower Display. Adjust the setpoint to 23°C using the **UP** and **DOWN** buttons.
27. Program the box furnace for a maximum temperature ramping rate of 10°C/minute and an anneal temperature of 600°C for 120 minutes:
 - a. Press the **PAGE** button until the display reads **ProG LiSt**.
 - b. Activate the deviation band holdback:

Press the **SCROLL** button until the display reads **Hb**. Press the **UP** and **DOWN** buttons to toggle the holdback type to **bAnd**.

Press the **SCROLL** button until the display reads **Hb.U**. Toggle the holdback value to **5**. This will hold the program whenever the temperature deviates above or below the setpoint by more than 5°C.

c. Set up additional parameters:

Press the **SCROLL** button until the display reads **rmP.U**. Toggle ramp units to **min**.

Press the **SCROLL** button until the display reads **dwL.U**. Toggle dwell units to **min**.

Press the **SCROLL** button until the display reads **Cyc.n**. Toggle number of cycles to **1**.

d. Set up temperature ramp-up:

Press the **SCROLL** button until the display reads **SEG.n**. If necessary, toggle to segment number **1**.

Press the **SCROLL** button until the display reads **tYPE.n**. Toggle to **rmP.r** (ramp rate).

Press the **SCROLL** button until the display reads **tGt**. Toggle the target setpoint to **600**.

Press the **SCROLL** button until the display reads **rAtE**. Toggle the ramp rate to **10.0**°C/minute.

e. Set up annealing time:

Press the **SCROLL** button until the display reads **SEG.n**. The segment number should automatically be set to **2** (toggle if necessary).

Press the **SCROLL** button until the display reads **tYPE.n**. Toggle to **dwEll** (dwell units).

Press the **SCROLL** button until the display reads **dur**. Toggle the annealing time to **120.0** minutes.

f. Set up temperature ramp-down:

Press the **SCROLL** button until the display reads **SEG.n**. The segment number should automatically be set to **3** (toggle if necessary).

Press the **SCROLL** button until the display reads **tYPE.n**. Toggle to **rmP.r** (ramp rate).

Press the **SCROLL** button until the display reads **tGt**. Toggle the target setpoint to **23**.

Press the **SCROLL** button until the display reads **rAtE**. Toggle the ramp rate to **10.0**°C/minute.

g. End the program:

Press the **SCROLL** button until the display reads **SEG.n**. The segment number should automatically be set to **4** (toggle if necessary).

Press the **SCROLL** button until the display reads **tYPE.n**. Toggle to **End**.

Press the **SCROLL** button until the display reads **End.t**. Toggle to **dwEll** (an indefinite dwell).

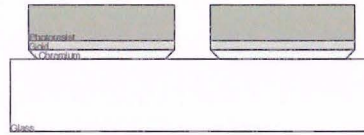
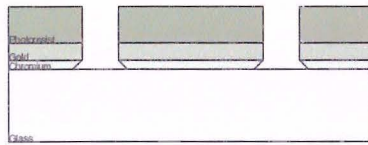
28. Press the **PAGE** button until the display returns to the home display, showing the present chamber temperature in the upper display and the Setpoint in the lower display.

29. Load the bonded substrates inside the furnace chamber.

30. Press the **RUN/HOLD** button once to start the program. The **RUN** light should illuminate. The bonded substrates will require several hours to cool down.

31. Take the substrate out of the box furnace. Inspect the bonded substrate on a microscope. Check if spots that had failed to bond earlier had increased in size, decreased in size, or remain unchanged.

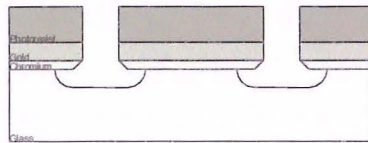
6)



Metal Etching:

Wet-etch Au/Cr masking layer.

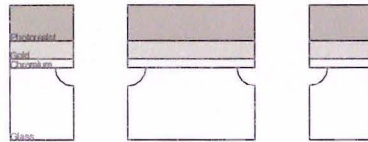
7)



Glass Etching:

Wet-etch device substrate.

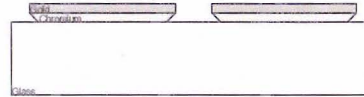
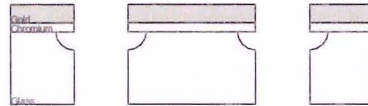
8)



Drilling Access Ports:

Drill ports into device substrate. Clean device substrate with methanol/ethanol.

9)



Substrate Stripping, Part A:

Remove photoresist in acetone bath, followed by a 3:1 piranha.

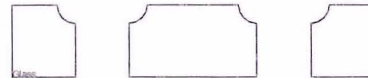
10)



Substrate Stripping, Part B:

Remove Au and Cr masking layers by wet-etch.

11)



Substrate Stripping, Part C:

Strip remaining traces of metal with a 2:1 piranha.

Devices with electrodes: In the box furnace the heat will cause the Cr adhesion layer for the electrodes to oxidize. Annealing should be performed in a vacuum oven.

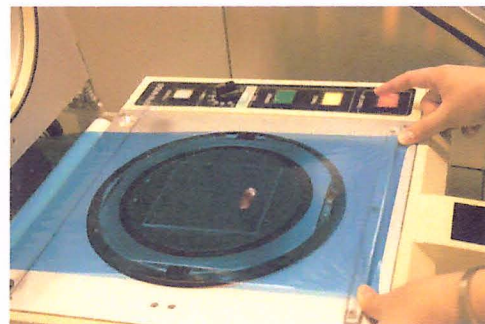
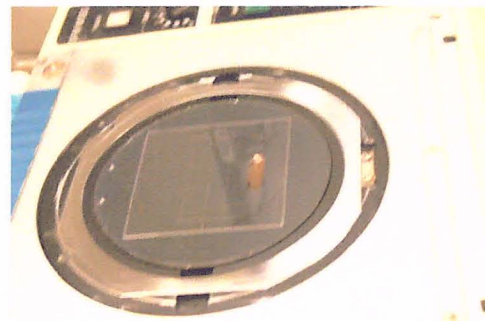
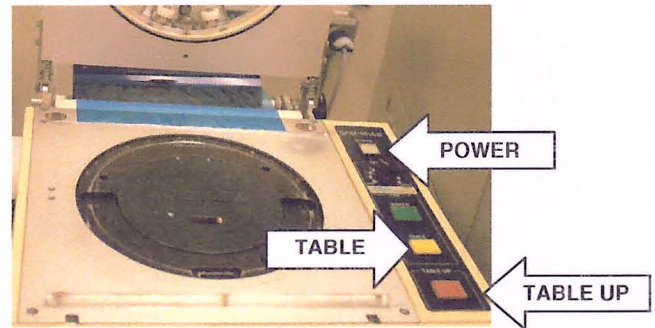
Step 9: Device Dicing

Estimated completion time: 30 minutes – 1 hour

Equipment	Materials and Supplies
<ul style="list-style-type: none">Diamond Touch Dicing Saw	<ul style="list-style-type: none">4" x 4" bonded glass substrate

Refer to the operation manual for full instructions on how to use the Diamond Touch Dicing Saw.

1. Turn on the DFM-M150 Wafer Mounter. Allow it to warm up until the chuck is warm to the touch (5 – 10 minutes).
2. Center a wafer frame onto the chuck.
3. Center the substrate onto the chuck. Press **TABLE** to secure the substrate.
4. Place a layer of tape over the substrate and wafer frame. Press **TABLE UP** to raise the chuck; the tape is now stretched over the substrate. Smooth out the tape using a roller. Air pockets can be deflated with a X-acto knife. Cut the tape off the dispenser.
5. Place a second layer of tape on top of the substrate and wafer frame.
6. Cut off excess tape around the wafer frame. The tape layers should be securing the substrate to the wafer frame.
7. Press **TABLE UP** to lower the chuck and **TABLE** to turn off the vacuum securing the substrate. Carefully peel off the tape securing the substrate. Allow the frame to cool down for several minutes until it is cool to the touch.
8. Turn on the Diamond Touch Dicing Saw.
9. Turn on the spindle. Set the spindle speed:
Corning 0211: 10000 RPM
Borofloat®: 14000 RPM
10. Retrieve spindle height. Since the blade's diameter will change after each cut, its height must be retrieved each time the dicing saw is operated. Turn off the spindle.



11. Place the wafer frame onto the chuck and turn on the vacuum.
12. Align the chuck to a dicing line.
13. Position the dicing saw to the starting point of the cut.

The dicing saw cuts from right to left. To ensure a straight cut the contact point of the blade should be positioned approximately 1" away from the substrate.

14. Adjust the spindle height to cut the top layer of the bonded substrate:

Corning 0211: 0.018 inches
Borofloat®: 0.086 inches

15. Set the feed rate to 0.1 inches/second.
16. Turn on the spindle.
17. Turn on water. Adjust water pressure to 20 GPH.
18. Cut the substrate.
19. Adjust the spindle height to 0.006 inches to cut the bottom layer of the bonded substrate. Make a second cut.
20. If multiple cuts are required, re-position the dicing saw and repeat Steps 14 – 19.

*The dicing saw can be re-positioned manually using the direction pad, or by selecting **MOVE Y** and inputting a defined distance. A positive value will move the dicing saw forward.*

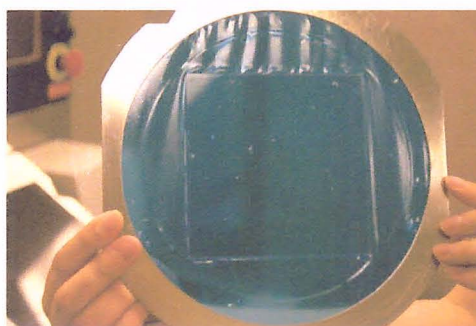
21. Turn off the spindle. Press **BACK**.
22. Dry the substrate using the N₂ gun. Turn off the vacuum and retrieve the wafer frame.
23. Turn off Diamond Touch Dicing Saw.
24. Cut away excess tape and peel back carefully to free the substrate.



Step 5




Step 6



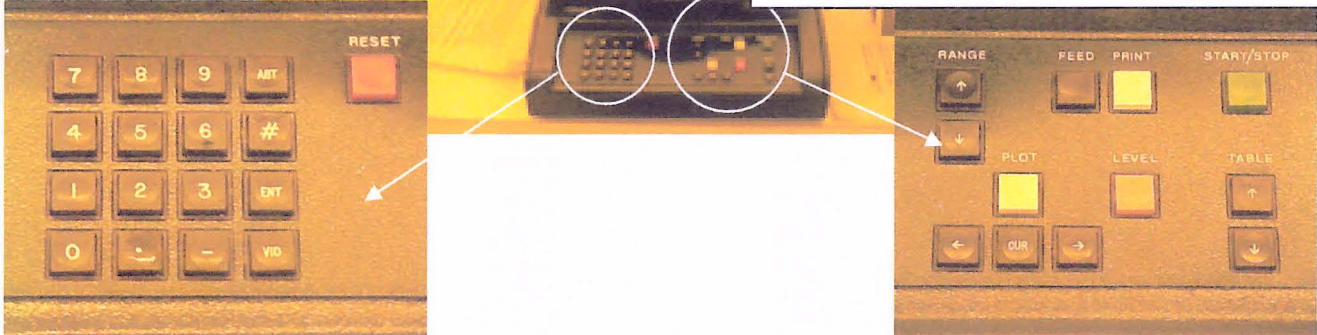
Step 7

Appendix A: Glass etching calculations using the Alphastep



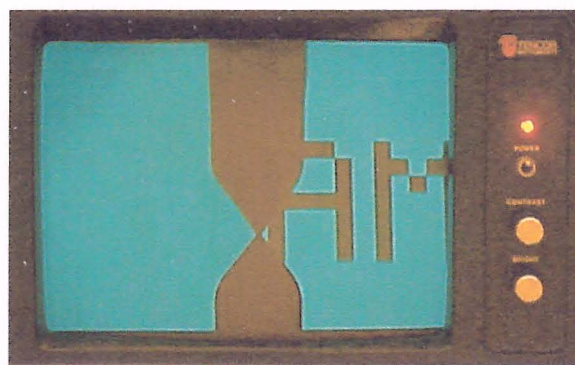
Setting measurement parameters

1. Press the **ENT** key to access the parameters.
2. Set the scan distance/speed to 400 μm at 1 s/ μm by toggling the \uparrow and \downarrow keys under **RANGE**.
3. Set the scan direction by toggling the \leftarrow and \rightarrow keys beside the **CUR** key.



Obtaining a measurement

1. Load the substrate onto the Alphastep stage.
2. Select one mask feature that appears multiple times on different areas of the substrate, such as one of the letters of the mask ID.
3. Use the \uparrow and \downarrow keys under **TABLE** to bring the substrate into focus. Adjust rotation, vertical positioning, and horizontal positioning so that the stylus is placed at the desired starting point of the scan. The stylus should be positioned so that the start and end points of the scan are level (this

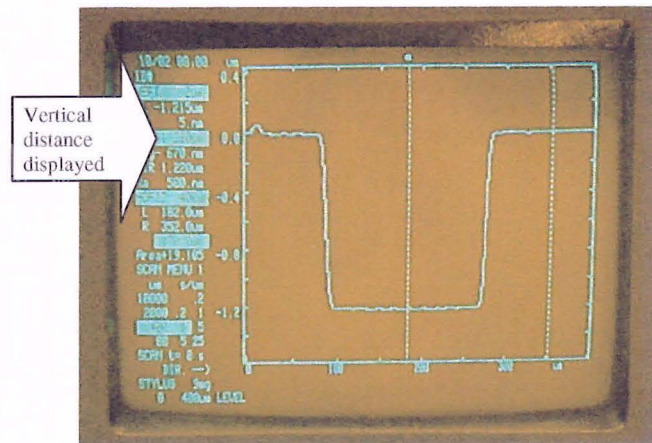


Step 3

eliminates the need to level the plot).

4. Press the **START/STOP** key to start scanning. A plot of the scan profile will be automatically displayed.

5. On the plot, use the **CUR** key to toggle the measurement points and the \leftarrow and \rightarrow keys to move the measurement points. Etch depth should be measured from the center of the etched channel as illustrated on the right.



Step 5

Sample Calculations

Step 1: Before glass etching, measure the depth of the masking layer.

From Figure 1, the etch depth of the masking layer is 1.220 μm .

Step 2: Etch the substrate for 5 minutes to calculate etch rate.

From Figure 2, the etch depth of the masking layer + glass channel is 11.07 μm .

$$\text{Etch Depth} = 11.07 - 1.220 = 9.85 \mu\text{m}$$

$$\text{Etch Rate} = \frac{\text{Etch Depth}}{\text{Etch Time}} = \frac{9.85 \mu\text{m}}{5 \text{ min}} = 1.97 \mu\text{m} / \text{min}$$

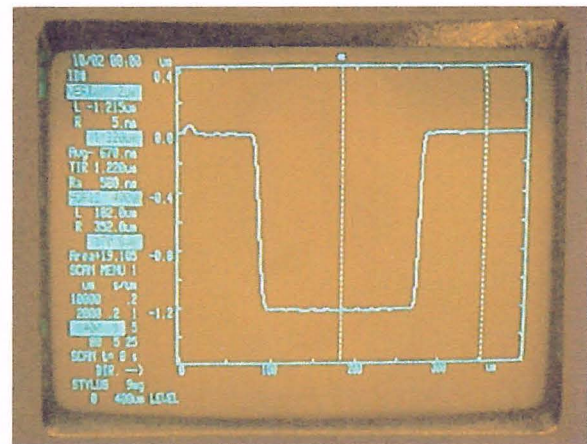


Figure 1. Before Etch

Remaining etch time for a 25 μm channel:

$$\begin{aligned}
 &= \frac{\text{Desired Etch Depth} - \text{Etch Depth}}{\text{Etch Rate}} \\
 &= \frac{(20 - 9.85)\mu\text{m}}{1.97\mu\text{m/min}} \\
 &= 5.15 \text{ min}
 \end{aligned}$$

Step 3: Etch the substrate for another 5.15 minutes (5 min 9 s) to etch 25 μm channels.

From Figure 3, the etch depth of the masking layer + glass channel is 26.10 μm .

$$\text{Etch Depth} = 26.10 - 1.220 = 24.88\mu\text{m}$$

Note that etch rate is temperature-dependent. For deep etches you may find that etch rate changes by ± 0.2 $\mu\text{m}/\text{min}$ over the course of one etch.

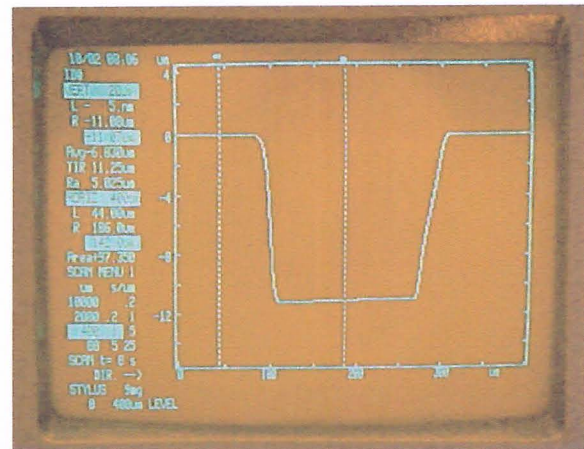


Figure 2. After Test Etch

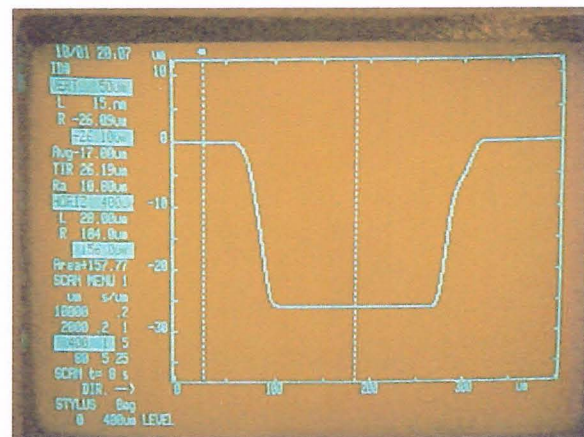
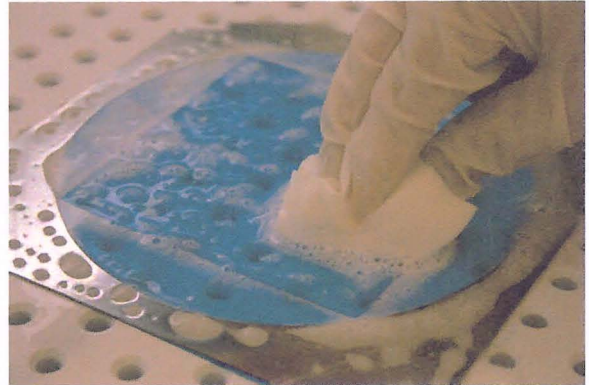


Figure 3. After Substrate Etch

Appendix B: Preparing the cover substrate for bonding

1. Turn on the wafer mounter. Warm up for approximately 15 minutes or until the chuck is warm to the touch.
2. Take the cover substrate out of the container of H_2O . Dry with a N_2 gun.
3. Center the device substrate onto the wafer mounter chuck, electrode side down.
4. Center a wafer frame onto the chuck.
5. Place a layer of tape over the device substrate and wafer frame. Smooth out the tape using a roller.
6. Cut off excess tape around the wafer frame. The tape should now be securing the device substrate to the wafer frame.



Step 7: Soap decrease friction between the two glass substrates during bonding

7. Using a sponge and the stock bottle of soap water for bonding, scrub the substrate for 3 minutes.

Concentrate on scrubbing the substrate edges. Users most often handle substrates by gripping the substrate edge, making these areas more susceptible to contamination.

8. Rinse the substrate thoroughly with DI water.
9. Check that the following parameters are set for the High Pressure Cleaning Station:

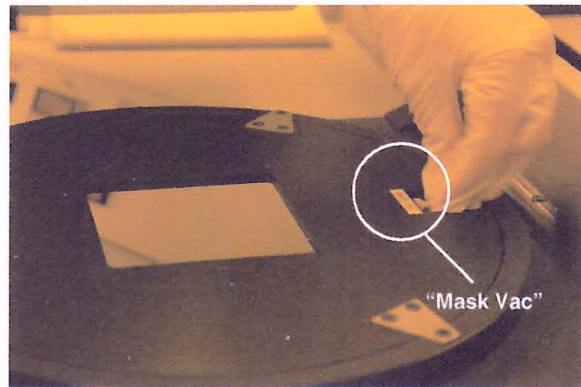
Clean Time = 0
Dry Time = 9

10. Place the substrate inside the High Pressure Cleaning Station, ensuring that the wafer frame is secure. Close the lid of the cleaning station and flip the power switch to **ON**. The cleaning station should automatically begin washing.
11. Open the lid and take out the substrate after the cleaning station has finished its drying cycle.

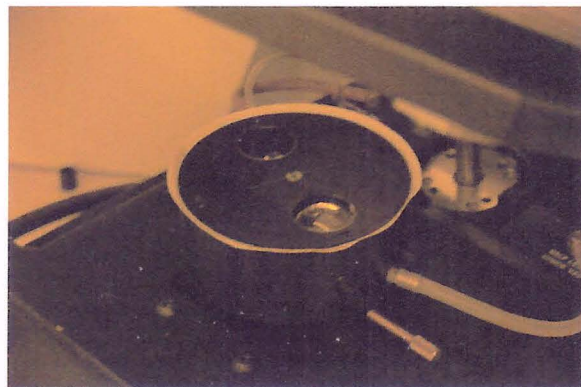
Appendix C: Bonding using the AB-M mask aligner

Equipment	Materials and Supplies
<ul style="list-style-type: none">• AB-M Mask Aligner with CCD Alignment System (Ernie or Oscar)	<ul style="list-style-type: none">• One device substrate (dimensions smaller than 4" x 4")• One cover substrate (4" x 4")

1. Replace the mask holder with the bonding jig for 4" x 4" square substrates:
 - a) Turn off mask vacuum.
 - b) Unscrew the side screws to loosen the mask holder.
 - c) Disconnect the mask vacuum tube.
 - d) The top face of the jig has a label identifying the location for the mask vacuum. Insert the jig into the mask aligner. Thread the mask vacuum tube through the small opening in the jig.
 - e) Reconnect the mask vacuum tube.
2. Replace the chuck with the 4" IR backside alignment chuck.
3. Lift the mask holder. Cut excess tape away from the cover substrate and place on the underside of the bonding jig, bond surface down. For the mask vacuum to hold the substrate it must be aligned with the resting screws. Turn on the mask vacuum, increasing nitrogen flow if necessary.
4. Place the device substrate onto the chuck, bond surface up.
5. Lower the mask holder.

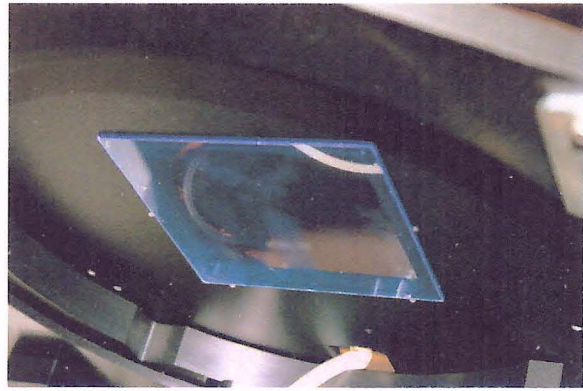


Step 1d

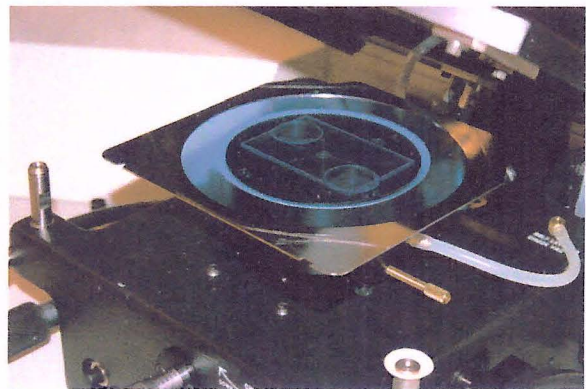


Step 2

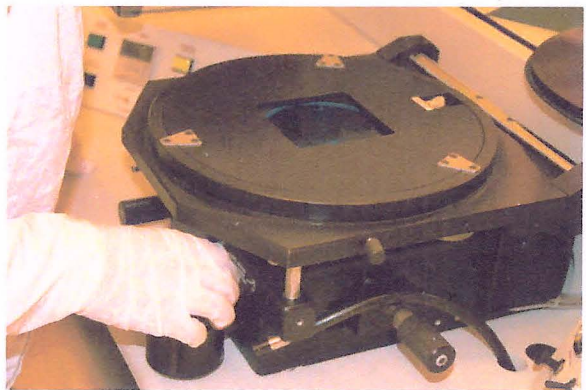
6. Raise the chuck until a small gap remains between the two substrates. Level the chuck by pressing the chuck-leveling button.
7. Using the alignment microscope, adjust the rotation, vertical positioning, and horizontal positioning of the chuck until the two substrates are aligned.
8. Slowly raise the chuck until the two substrates make initial contact. As the two surfaces meet, concentric bands of light form due to thin film interference.
9. Turn off the mask vacuum.
10. Press gently on the sides of the cover substrate. A bond should begin to form on the edges of the device substrate.
11. Raise the mask frame and turn off the substrate vacuum.
12. Take the bonded substrate out of the mask aligner.
13. Inspect the bonded substrate on a microscope. Areas that failed to bond will appear as a series of concentric rings of light centered around the contaminant that inhibited bonding. If the substrates need to be re-bonded, pry the substrates apart using an X-acto knife and return to Step 1 of **Fusion Bonding**.



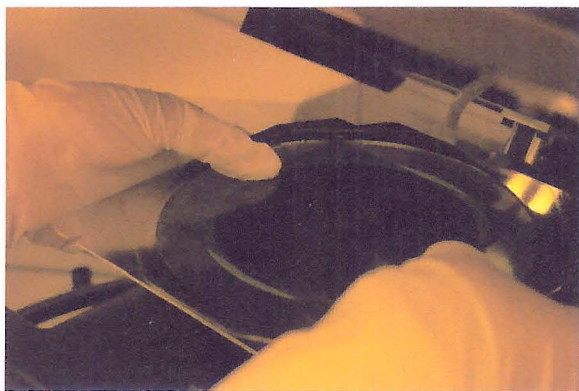
Step 3



Step 4



Step 8



Step 10

Chapter 5

Process Run Cards

Process run cards, or as they are also known, process travelers, are a check list that travels with the substrates during all processing. The run cards describe each step in order and ensure that the wafers are processed correctly, and any difficulties noted. Run cards are critical when fabricating a complex device. It is very easy to forget a step or not note down a problem step. If this occurs and the process fails, you are relying on your memory to determine why the process failed. This is not very effective and leads to large amount of wasted time and frustration. All real world processes (i.e. in industry) use run cards for these reasons.

This chapter contains the run cards required to fabricate a glass microfluidic device. The NanoFab strongly suggests that you follow this example and develop run cards for your process.



Microfluidic Devices Run Card v1.0

Name:

Date:

Step 1: Device Substrate and Mask Cleaning

Temperature:

Humidity:

Step #	Process	Comments/Descriptions
1	Label 2 glass beakers for the following: 1) H_2SO_4 ; 2) H_2O_2 Label 1 glass container for the piranha mixture.	
2	Determine the volume of piranha required to immerse the substrate.	Piranha volume:
3	Calculate the volumes of H_2SO_4 and H_2O_2 required to make a 3:1 piranha.	H_2SO_4 volume: H_2O_2 volume:
4	Pour H_2SO_4 into the glass container for the piranha mixture.	
5	Pour H_2O_2 into the glass container for the piranha mixture.	
6	Immerse the substrate in the piranha mixture for 15 minutes.	
7	Remove the substrate from the piranha mixture.	
8	Perform a 5-cycle DI water rinse in the dump rinser.	
9	Dry the substrate in the spin rinse dryer.	
10	After the piranha mixture has cooled to at least 40°C , immerse the mask in the piranha mixture for 30 minutes.	
11	Remove the mask from the piranha mixture.	
12	Perform a 5-cycle DI water rinse in the dump rinser.	
13	Dry the mask with a N_2 gun.	

Step 2: Masking Layer Deposition

Temperature:

Humidity:

Step #	Process	Comments/Observations
1	Load substrate into chamber.	
2	Pump down the chamber.	Base Pressure:
3	Introduce the process gas (argon) into the chamber. Establish the following conditions: Flow rate - 50 sccm Pressure - 7×10^{-3} Torr	
4	Set the following parameters for the sputter targets: Chromium - 300 W Gold - 75 W	
5	Perform a 5 minute RF bias.	
6	Burn in chromium target for 5 minutes.	
7	Sputter chromium for 1 minute and 20 seconds.	
8	Burn in gold target for 1 minute.	
9	Sputter gold for 6 minutes.	
10	Cool the substrate for 15 minutes.	
11	Vent the chamber to 7.6×10^{-2} Torr.	
12	Unload substrate from chamber.	
13	Pump down the chamber.	

Step 3: Masking Layer Photolithography

Temperature:

Humidity:

Step #	Process	Comments/Observations
1	Pre-heat the convection soft-bake oven to 115°C	
2	Set the following parameters on the Solitec Spinner: Spread: 500 rpm, 10 seconds Spin: 4000 rpm, 40 seconds	
3	Spin coat HPR 504 photoresist onto substrate.	
4	Visually verify an even layer of photoresist has been spun onto the substrate.	
5	Bake the substrate in the convection oven for 30 minutes.	
6	Cool the substrate for 15 minutes.	
7	Set up the mask and align the substrate.	
8	Expose the substrate for 4 seconds.	
9	Develop the substrate in 354 developer for approximately 20 seconds.	
10	Inspect the substrate on a microscope.	
11	Measure photoresist thickness using the Alphastep.	



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Name:

Date:

Step 4: Masking Layer Etching

Temperature:

Humidity:

Step #	Process	Comments/Observations
1	Label 3 plastic or glass containers for: 1) Au Etchant; 2) Cr Etchant; 3) H ₂ O.	
2	Fill each container with enough solution to submerge the substrate.	
3	Etch Au layer.	
4	Rinse the substrate with the DI water gun.	
5	Dry the substrate with the N ₂ gun.	
6	Inspect the substrate on a microscope.	Observations:
7	Etch Cr layer.	
8	Rinse the substrate with the DI water gun.	
9	Dry the substrate with the N ₂ gun.	
10	Inspect the substrate on a microscope.	Observations:
11	Check for a layer of oxide on the substrate.	
12	Dip substrate into Au etch bath for 1 second.	
13	Rinse the substrate with the DI water gun.	
14	Dry the substrate with the N ₂ gun.	
15	Hold the substrate up against light. The oxide layer should now be removed.	
16	Inspect the substrate on a microscope.	Observations:

Step 5: Glass Etching

Temperature:

Humidity:

Step #	Process	Comments/Observations
1	Measure the average thickness of the masking layer.	Thickness:
2	Calculate expected etch time.	Etch time:
3	Label 2 plastic containers for the following: 1) Glass Etchant; 2) DI water.	
4	Cover the back side of the substrate with tape.	
5	Verify the stir plate is operational.	
6	Pour glass etchant into the first container. Turn on magnetic stirrer.	
7	Fill the second container with DI water and place in the drop deck.	
8	Immerse the substrate in the etch bath and start the timer.	
9	At about 10 seconds before 5 minutes has elapsed, remove the substrate from the etch bath. When 5 minutes have elapsed, dip the substrate into the container of DI water.	
10	Perform a 5-cycle rinse in the dump rinser.	
11	Dry the substrate with the N ₂ gun.	
12	Measure the average etch depth.	Etch depth:
13	Calculate the required etch rate.	Etch rate/minute: Remaining etch time:
14	Immerse the substrate in the etch bath and start the timer.	
15	At about 10 seconds before the required time has elapsed, remove the substrate from the etch bath. When the required time has elapsed, dip the substrate into the container of DI water.	
16	Perform a 5-cycle rinse in the dump rinser.	
17	Dry the substrate using the spin-rinse dryer.	
18	Measure the average etch depth.	Etch depth:
19	Remove tape from substrate backside.	



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Name:

Date:

Step 6: Drilling Access Ports

Temperature:

Humidity:

Step #	Process	Comments/Descriptions
1	Fill the petri dish lid with enough methanol to immerse the substrate	
2	Immerse the substrate in the methanol bath for 15 minutes.	
3	Remove the softened crystal bond with a bristle brush.	
4	Dispose of the methanol.	
5	Repeat Steps 1 - 4.	
6	Perform a DI water rinse in the dump rinser for a 5-cycle rinse.	
7	Dry the substrate with the N ₂ gun.	
8	Make a 2:1 piranha	
9	Immerse substrate in piranha bath for 15 minutes.	
10	Remove substrate from piranha bath and perform a 5-cycle rinse.	
11	Dry the substrate in the spin rinse dryer.	

Step 7: Device Substrate Stripping

Temperature:

Humidity:

Part A: Photoresist Stripping		
Step #	Process	Comments/Observations
1	Label 1 glass/plastic container for acetone.	
2	Pour enough acetone in the container to immerse the substrate.	
3	Immerse substrate for 10 minutes.	
4	Remove the substrate and perform a 5-cycle rinse in the dump rinser.	
5	Dry the substrate with the N ₂ gun.	
6	Make a 3:1 piranha	
7	Immerse substrate in piranha bath for 15 minutes.	
8	Remove substrate from piranha bath and perform a 5-cycle rinse.	
9	Dry the substrate in the spin rinse dryer.	
Part B: Metal Stripping		
Step #	Process	Comments/Observations
6	Label 2 glass/plastic containers for the following: 1) Gold Etch; 2) Chrome Etch	
7	Fill each container with enough etchant to immerse the substrate.	
8	Submerge the substrate in Au etch bath.	
9	Rinse thoroughly with DI water and dry with the N ₂ gun.	
10	Submerge the substrate in Cr etch bath.	
11	Rinse thoroughly with DI water and dry with the N ₂ gun.	
12	Submerge the substrate in Au etch bath for 1 minute.	
13	Rinse thoroughly with DI water and dry with the N ₂ gun.	
Part C: Substrate Cleaning		
Step #	Process	Comments/Observations
14	Make a 2:1 piranha	
15	Immerse substrate in piranha bath for 15 minutes.	
16	Remove substrate from piranha bath and perform a 5-cycle rinse.	
17	Dry the substrate in the spin rinse dryer.	
Part D: Inspection		
Step #	Process	Comments/Observations
18	Inspect the substrate on the microscope.	Observations:



Microfluidic Devices Run Card v1.0

Name:

Date:

Step 8: Fusion Bonding

Temperature:

Humidity:

Part A: Substrate Cleaning

Step #	Process	Comments/Observations
1	Make a 3:1 piranha	
2	Immerse the substrate in the piranha mixture for 15 minutes.	
3	Remove the substrate from the piranha mixture.	
4	Perform a 5-cycle DI water rinse in the dump rinser.	
5	Dry the substrate in the spin rinse dryer.	

Part B: Bonding

Step #	Process	Comments/Observations
6	Center the wafer frame and device substrate (bonding side face-down) onto the wafer mounter chuck.	
7	Roll a layer of tape onto the substrate and frame. Cut away excess tape.	
8	Set the following parameters on the High Pressure Cleaning Station: Clean Time: 5 Dry Time: 9	
9	Rinse and dry the substrate.	
10	Store the device substrate bond surface up underneath a large petri dish.	
11	Repeat Steps 10 - 13 for the cover substrate.	
12	Place 2 wafer frames on top of the device substrate to form a spacer.	
13	Place cover substrate bond surface down on top of the wafer frames.	
14	Bond substrates.	
15	Inspect the bonded substrate on the microscope.	Observations:

Part C: Annealing

Step #	Process	Comments/Observations
16	Turn on the Thermolyne Box Furnace. Program the furnace to ramp at 10°C/minute and anneal at 600°C for 120 minutes.	
17	Place bonded substrates inside the furnace. Leave overnight.	
18	Inspect the bonded substrate on the microscope.	Observations:

Step 9: Device Dicing

Temperature:

Humidity:

Step #	Process	Comments/Observations
1	Centre a wafer frame and the bonded substrates onto the centre of the chuck.	
2	Roll a layer of tape onto the substrate and frame.	
3	Roll a second layer of tape onto the substrate and frame. Cut away excess tape.	
4	Turn on the dicing saw.	
5	Turn on the spindle. Set the spindle speed: Corning 0211: 10000 RPM Borofloat®: 14000 RPM	
6	Retrieve spindle height. Turn off the spindle.	
7	Place the wafer frame onto the saw chuck and turn on the substrate vacuum.	
8	Align the chuck to a dicing line.	
9	Position the dicing saw to the starting point of the chuck.	
10	Adjust the spindle height: Corning 0211: 0.018 inches Borofloat®: 0.086 inches	
11	Adjust the feed rate to 0.1 inches/sec. Turn on the spindle	
	Adjust water pressure to 20 GPH.	
12	Make one cut.	
13	Adjust the spindle height to 0.006 inches.	
14	Make a second cut.	
15	Make additional cuts as necessary by repeating steps 8 - 14.	
16	Turn off the spindle. Press BACK .	
17	Dry the substrate using the N ₂ gun. Turn off the substrate vacuum and take the wafer frame off of the chuck.	
18	Cut away surrounding tape and peel back to free the devices.	
19	Perform a final inspection on the devices underneath a microscope.	Observations:

Chapter 6

Process Troubleshooting and Fabrication Inspection Guide

Introduction



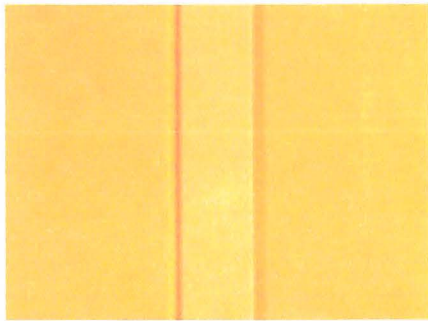
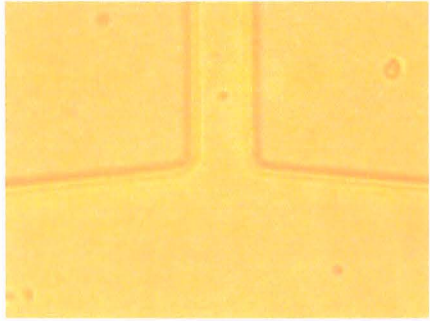
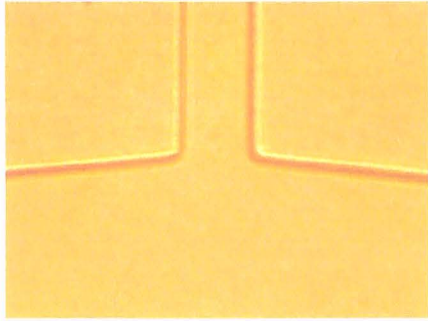
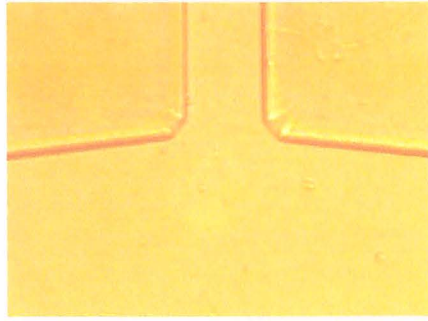
This inspection guide is intended to illustrate common problems encountered in glass processing, and provide recommendations to solve these specific problems when applicable. Take overall yield consideration whenever you feel the need to rework a substrate.

Things to Remember:

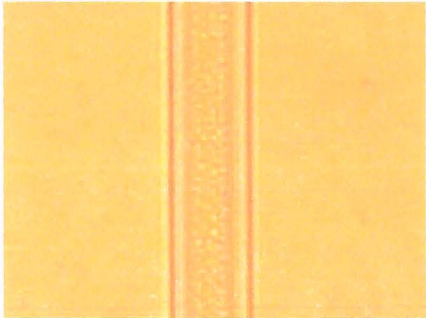
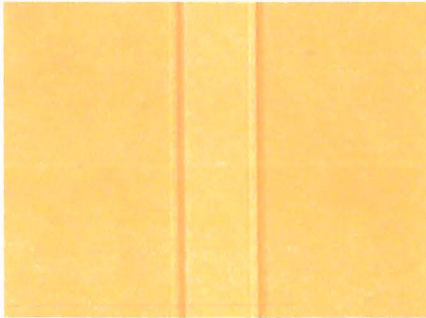
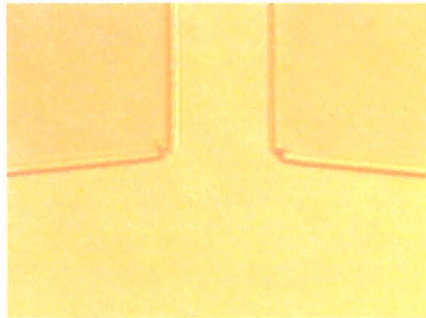
- Defects on isolated areas of the substrate will not impact device operation.
- Don't expect defects to disappear. They will generally propagate throughout processing
- More often than not cleanliness is the cause of defects. A contaminated mask is the likely cause of identical defects repeated on multiple substrates. As a minimum the mask should be cleaned in cold piranha after exposing 4 substrates.

6.1 Photolithography

A comparison of photoresist development times after 4 seconds of exposure:

	Underdeveloped Development time: ~10 seconds	Developed Development time: ~20 seconds	Overdeveloped Development time: ~30 seconds
Observation	Features are smaller than desired or not fully formed.	Features are fully formed and clearly defined.	Features are wider than desired or “smeared” off.
Recommendation	Continue developing until no more photoresist lifts off of the substrate in the developer bath. Re-inspect under microscope.	Proceed to wet etching.	Re-work substrate. Remove photoresist using acetone and IPA. Repeat photolithography.
Optical Image (50x) Channel			
Optical Image (50x) Port			

A comparison of photoresist exposure times prior to 20 seconds of development:

	Underexposed Exposure time: 1 second	Exposed Exposure time: 4 seconds	Overdeveloped Exposure time: 16 seconds
Observation	Areas of exposed photoresist have not dissolved off.	Features are fully formed and clearly defined.	Larger features due to the faster development times associated with longer exposures. Some features have ragged edges.
Recommendation	Re-work substrate. Remove photoresist using acetone and IPA. Repeat photolithography.	Proceed to wet etching.	Re-work substrate. Remove photoresist using acetone and IPA. Repeat photolithography.
Optical Image (50x) Channel			
Optical Image (50x) Port			

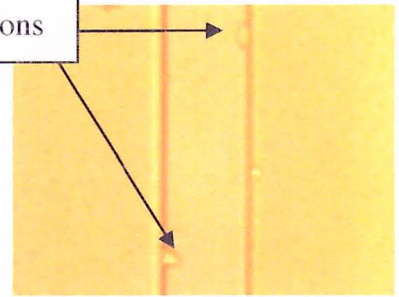
6.2 Miscellaneous Lithography Defects (Page 1 of 2)

Observation: "Nips" along the edges.

Cause/Effect: Substrate contamination, possibly from a dirty mask.

Recommendation: Clean mask with cold piranha. Remove photoresist using acetone and IPA. Repeat photolithography.

Intrusions

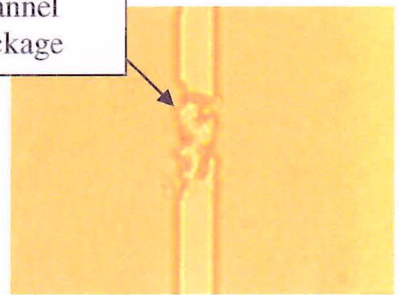


Observation: "Holes" in photoresist.

Cause/Effect: Lift-off from a dirty mask.

Recommendation: Clean mask with cold piranha. Remove photoresist using acetone and IPA. Repeat photolithography.

Channel blockage



Observation: Pieces of photoresist have fallen into areas to be etched.

Cause/Effect: No effect. Because the photoresist is loose, the surface underneath will still be etched.

Recommendation: Proceed with etching.

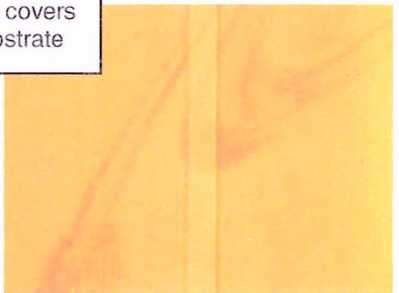


Observation: Fine film of particles covering substrate.

Cause/Effect: Poor rinsing and drying techniques deposit particles back onto the substrate.

Recommendation: Rinse substrate thoroughly with DI water. Hold the substrate in the palm of your hand at an angle, and dry aiming the N₂ gun away from the body down the substrate surface.

A film of gold particles covers this substrate



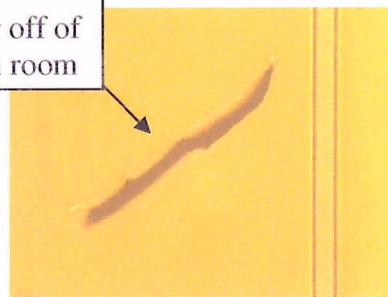
Miscellaneous Defects (Page 2 of 2)

Observation: Large, loose particles lying on top of the photoresist.

Cause/Effect: Particulates, such as fibers from clean room wipes or skin flakes.

Recommendation: Blow off substrate using a N₂ gun. Re-inspect substrate under microscope.

A fiber off of
a clean room

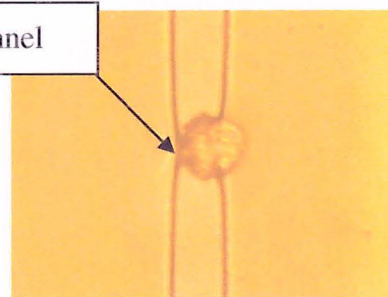


Observation: Dark "spots" embedded in the photoresist layer.

Cause/Effect: Particulates caused by poor handling or contaminated equipment. A dirty mask will lift up photoresist or flattened particles and deposit it onto the next substrate. Photoresist coverage will be uneven over a non-uniform surface, leaving topographical variations unprotected. The defect area may then be etched away, resulting in pinhole formation at the defect point.

Recommendation: Clean mask with cold piranha. Remove photoresist using acetone and IPA. Repeat photolithography.

Channel

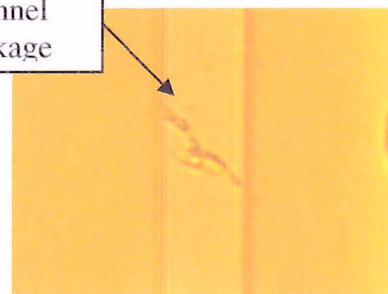


Observation: Ragged edges. The removed photoresist splits up features (e.g. blockages created in channels).

Cause/Effect: Over-baking or over-exposing the photoresist will cause it to crack and peel at the edges. If the same defect is repeated on multiple substrates, a dirty mask is the likely cause.

Recommendation: Clean mask with cold piranha. Remove photoresist using acetone and IPA. Repeat photolithography.

Channel
blockage



Observation: Dark spots underneath the photoresist layer.

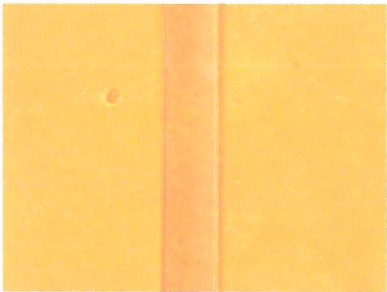
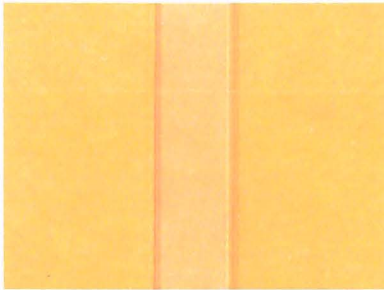
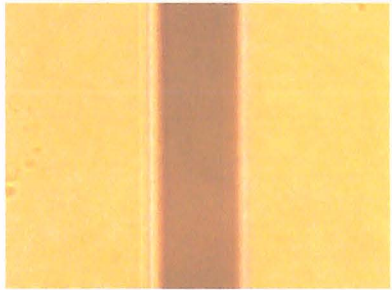
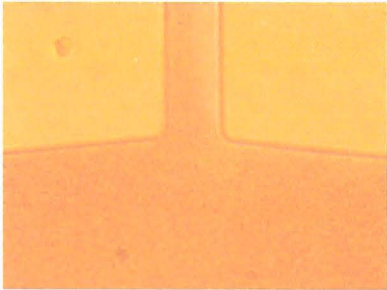
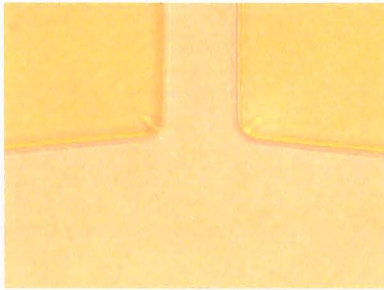
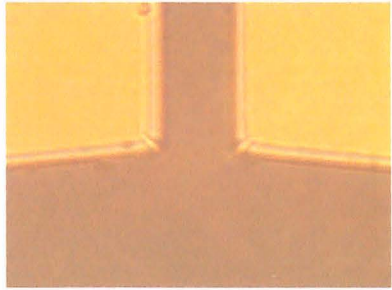
Cause/Effect: Gold has not adhered onto the chromium film. Photoresist coverage will be uneven over a non-uniform surface, leaving topographical variations unprotected. The defect area may be etched away, resulting in a pinhole forming at the defect point.

Recommendation: Remove photoresist using acetone and IPA, strip off metal, and proceed from sputtering.



6.3 Metal Etching

After Mask Etching: A side-by-side comparison

	Underetched	Etched	Overetched
Observations	"Double lines" on edges. Deposits of material in etched areas.	Features are fully formed and clearly defined.	"Double lines" on edges.
Recommendation	Etch the substrate for a few seconds and re-inspect. Note that chrome etches isotropically and may lift off isolated gold deposits.	Proceed to wet etching.	If the metal layer was over-etched to the point where the device dimensions have been altered, the substrate will have to be re-worked.
Optical Image (50x) Channel	After Gold Etch: Deposits are left inside etched regions 	After Gold Etch: Clean edges with no traces of gold 	After Chrome Oxide Etch: Gold was over-etched 
Optical Image (50x) Port			

6.4 Miscellaneous Metal Etch Defects (Page 1 of 1)

Observation: Ends of the channel not etched.

Cause/Effect: Gas bubbles form in the etch bath as a reaction product. Therefore, small features such as the ends of narrow channels may not etch due to surface tension.

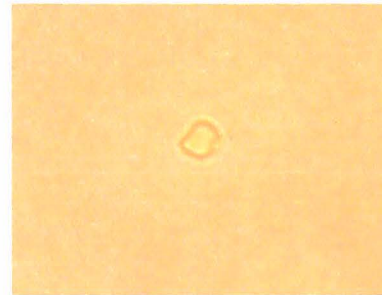
Recommendation: Agitating the etch bath during etching will remove some of the bubbles.

N/A

Observation: Isolated deposits of metal over etched areas.

Cause/Effect: The metal layer may be under-etched. Another possibility is that contamination prevented the area from being exposed during photolithography. Metal deposits will act as a micro-mask; the glass underneath will not be etched.

Recommendation: Replace etch bath with fresh etchant. Etch the substrate for a few seconds and re-inspect. Note that for the case of gold, chrome etches isotropically and may lift off smaller deposits.

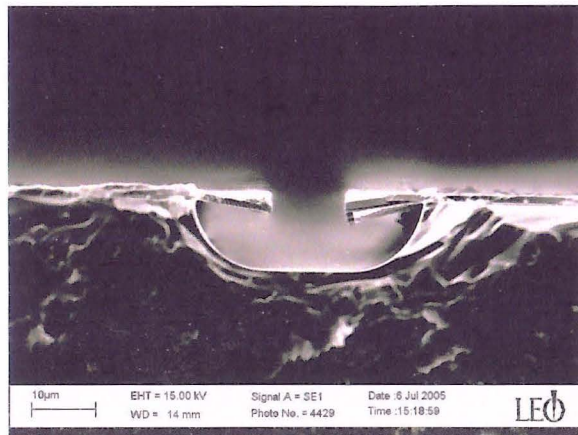


6.5 Glass Etching

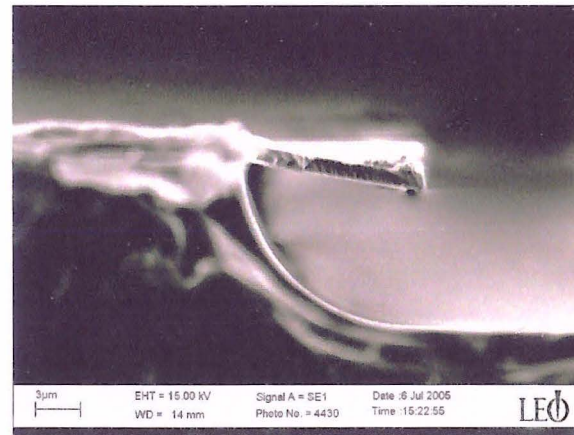
Additional Notes:

- HF etches the oxide layer isotropically at the glass-chromium interface. Therefore, deviation from the mask sputtering parameters (chamber base pressure and RF biasing time) may result in significantly increased undercutting of the masking layer.
- The main thing to look for is obstruction in the fluidic channels. Depending on device application, partial blockage or widening of the channels may be acceptable.

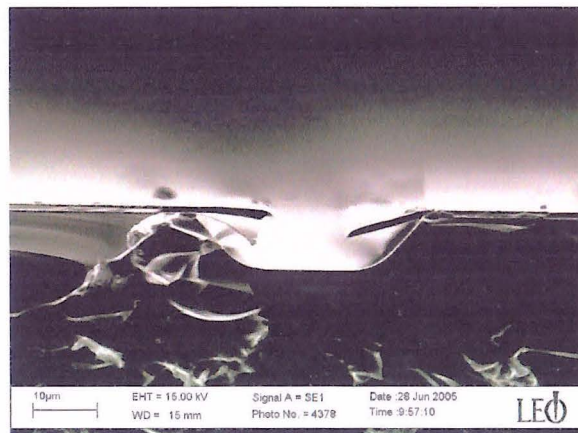
SEM Images of a 10 μm Microfluidic Channel in Corning 0211 Glass:



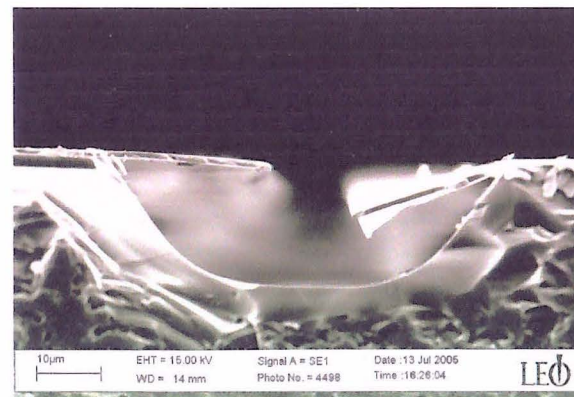
Cross-Section (3000x)



Cross-section (7750x)



Underetched Cross-section (3000x)



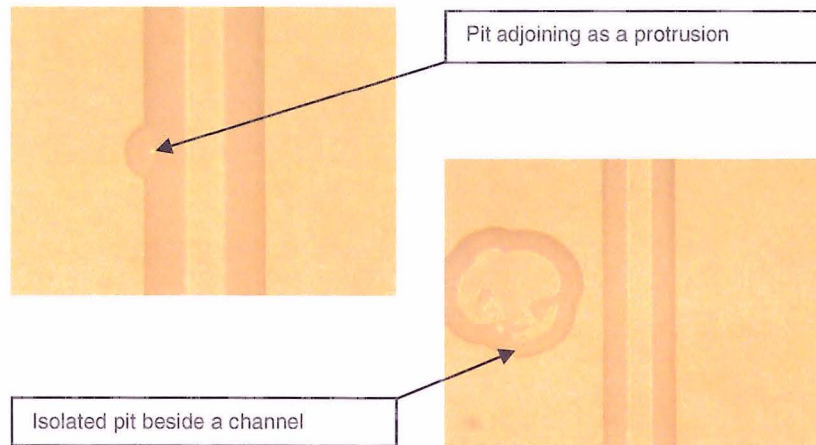
Overetched Cross-Section (3000x)

6.6 Miscellaneous Glass Etching Defects (Page 1 of 2)

Observation: Pits in the glass. May be isolated or adjoining an etched area as a protrusion (i.e. a "bubble" sticking out of a straight edge).

Cause/Effect: Non-uniformity in the etch mask, possibly due to surface contamination or defects in the masking layer.

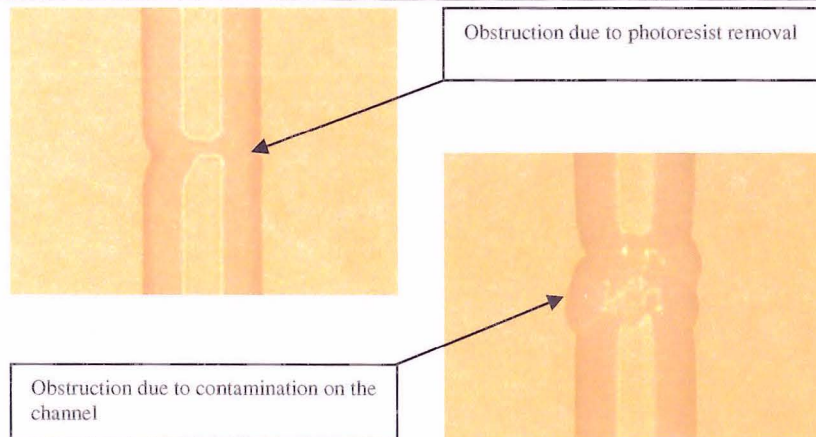
Comments: Isolated pits do not impact device operation. Depending on the device application, small extrusions may be acceptable.



Observation: Obstruction in device channels.

Cause/Effect: Photoresist was removed at the obstruction point. The photoresist may have been scratched off, or during photolithography exposing the substrate with a dirty mask will lift off photoresist. Contaminants may also prevent areas from etching.

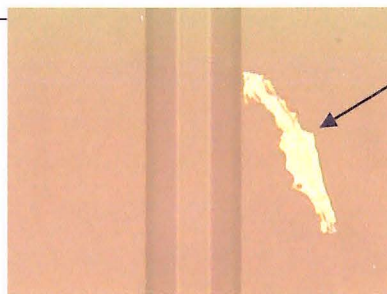
Comments: If the blockage is partial, you may want to obtain a profile using the Alphastep. Depending on the device application, partial blockages may be acceptable.



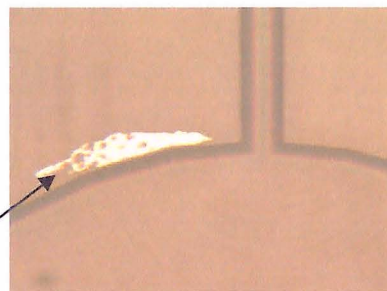
Observation: Deposits of metal on the glass.

Cause/Effect: Mask stripping failed to remove all metal. Deposits of metal are often embedded into substrate defects such as scratches, and can be difficult to remove.

Comments: Repeat Mask Stripping and Substrate Cleaning. Stubborn deposits can be removed using a Q-Tip dipped into etchant.



Metal deposit beside a channel



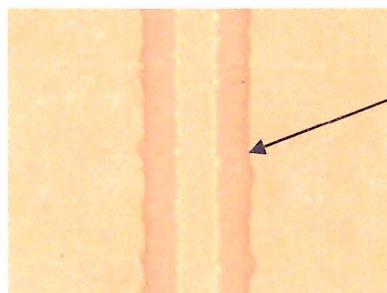
Metal deposit beside a port

Miscellaneous Defects (Page 2 of 2)

Observation: Jagged edges.

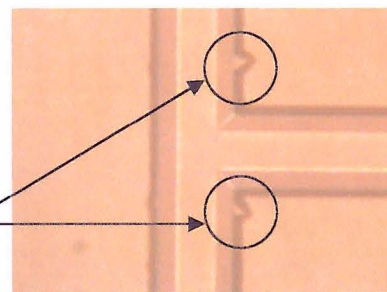
Cause/Effect: Non-uniform adhesion of the masking layer will also produce ragged edges.

Borofloat® only: Float processing techniques result in one side of the substrate containing more tin. The side containing tin was processed.



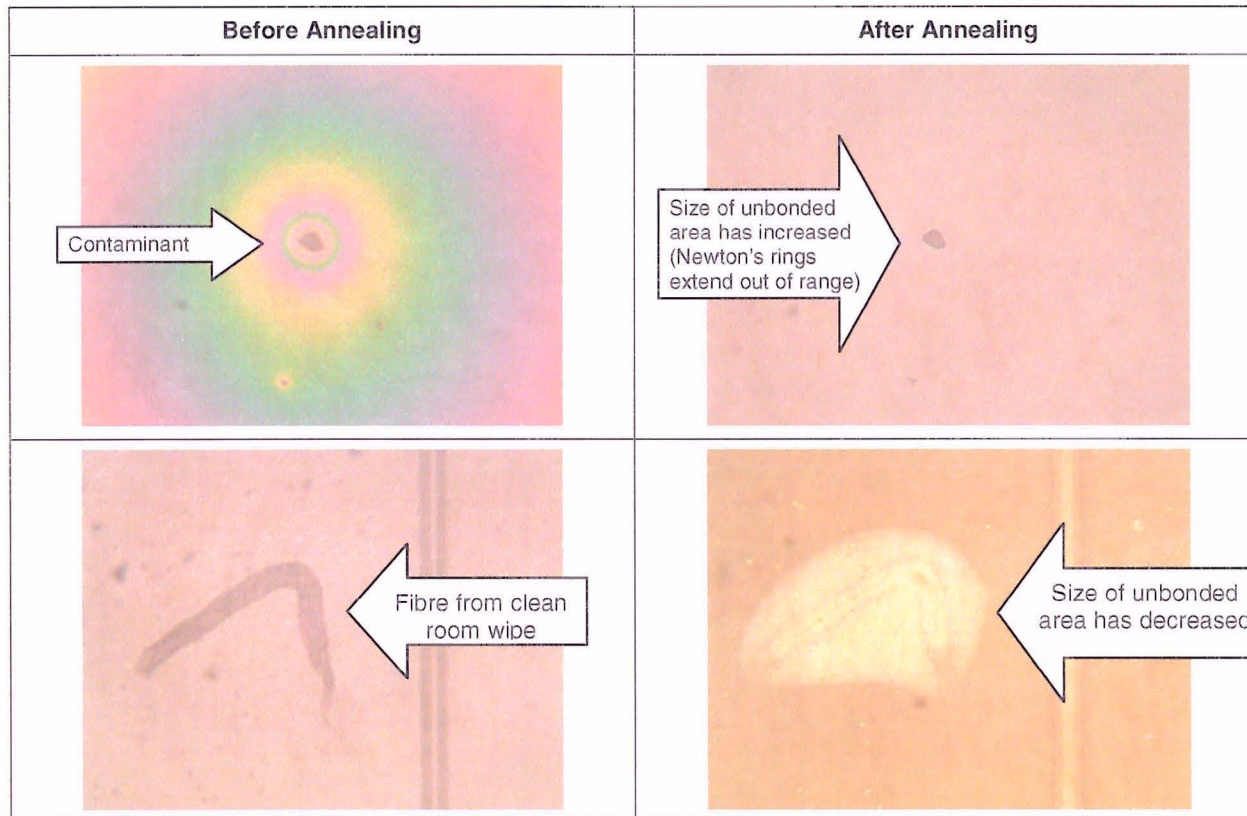
Jagged edges from Borofloat® processing

Jagged edges due to non-uniform adhesion of the masking layer



6.7 Bonding

Areas that failed to bond will appear as a series of concentric rings centered around the contaminant that inhibited bonding. The spots that failed to bond may increase or decrease in size after annealing. Some of the common sources of contamination that prevent bonding are illustrated below (10x magnification):



WaterJet Drilling – Version 1.03
protocol by Paul Dumais
December 2004

This document describes how to get our machine shop to cut coverplates. The process described here is most applicable to glass cutting; however, many other materials can be cut using this protocol without much change (such as PDMS-coated glass).

The WaterJet cutter uses a high-velocity jet of water (2-4 x the speed of sound) to cut through such materials as steel and glass. It uses an abrasive suspended in the water to help cut the material. The WaterJet operator (not you!, in this case Herb) needs to take special precautions when cutting glass because the glass can shatter or crack due to the stress that the water applies to the substrate. Specifically, special care must be taken by the operator to make sure the abrasive and water hits the material at the same time. This requires frequent cleaning of the nozzle since the abrasive slurry tends to clog. Also, other precautions need to be taken due to stringent requirement to maintain a damage-free surface suitable for glass on glass bonding. These include: 1. The WaterJet operator drills holes in a steel chuck before drilling to protect the glass from turbulent backwash. 2. A sacrificial glass layer needs to be bonded to the bottom of the glass plate to be cut. The details are included below.

Preparing the specifications for drilling

Presuming that you already have a file drawn in L-edit, you will need to convert only the needed information from .tdb format to .dxf format. At present, we have software that will do this conversion for holes only.

- ✓ 1. Save a copy of your L-edit design file with a new name. Ideally, you should have the holes and lines to be cut drawn on a separate layer. Hide all layers except the one just mentioned and the one that shows the outline of your wafer/substrate (usually icon/outline). Move all the remaining objects so that the origin of the coordinate system is at the lower left hand corner of your substrate. This zero of the coordinate system needs to be known to the WaterJet operator. Make sure that the mask that was made from this L-Edit file was printed chrome side down. If it was printed chrome side up, then your hole pattern will be a mirror about the vertical center line with respect to the patterned substrates. In this case, you will need to modify your drawing by mirroring it about the same axis.

Note that the WaterJet software does not properly compensate for the actual hole diameter that will be drilled due to factors such as abrasive type, tube type, hole diameter, plate thicknesses and drill speed. Therefore, the hole size specified in the .dxf file needs to be smaller than the desired size. This size reduction depends on the factors listed above and is so far a matter of experience and trial and error. For drilling a single 1.1 mm borofloat glass plate bonded with a 0.5 mm 0211 glass plate, and a desired hole diameter of 2 mm, medium abrasive, medium-low power/pressure (and other factors that the operator finds suitable), the specified diameter needs to be 1.1 or 1.15 mm. These parameters should also hold for drilling similar holes in 1.1 mm borofloat plates bonded together with 2mm thick plate glass. The above is just for your information, always get the proper specification from Herb Dixel prior to drilling, and check the diameter drilled on your first plate to make sure your first guess was suitable, adjust as required.

- ✓ 2. Select all the holes and change the radius to reflect the needed reduction as given to you (see above).
- ✓ 3. Select all objects (substrate outline, holes, cut lines) and select cell...flatten from the L-edit menu. Save the document. Select file...Export Mask Data...Cif and save the .cif file to a directory that you can easily find when you later use the ssh client to transfer it to gaea (one of our Linux servers).

4. Open the ssh Secure Client Shell. Login to gaea. Navigate to:
/home/Archive/Software/Students/paul/WaterJet/Code. There are two important files here: Cift and README. README contains the simple instructions on how to operate the software. Cift is the program that you run on gaea to convert the holes on your .cif drawing to holes on specified in a .dxf drawing. Move your .cif file to this directory (click on the new file transfer window icon to open the secure file transfer client).
5. In the SSH secure shell, type ./Cift . This will start a the menu-driven program. Press 1, <Enter>. Type or paste in the file name of the .cif file you want to convert. Press <Enter>. Press <Enter> again. Press 3, <Enter>. This will write your file to output.dxf. Press <Enter> again. Press q, <Enter> to quit.
6. Using the SSH Secure File Transfer client as before, move output.dxf back to the directory that contains the original .cif file. Open the .dxf file in an appropriate editor (Rhinoceros works). Make sure all the holes are there and make sure that the zero of your coordinate system is at the bottom left of the substrate.
7. In this same editor, draw in any linear cuts that need to be made. Save the file and send a copy to the WaterJet operator via a 3.5" floppy.
8. Using Crystalbond 590 (brown stick), bond one glass substrate to a sacrificial glass plate (usually cheap plate glass as thin as possible ~2 mm). In the case of borofloat where you want to bond only the non-tin sides, make sure that you scribe the word 'Tin' on the side that is marked with a 'T' with permanent marker. Use a clean wipe and acetone to check which side of the glass this 'T' is written on. This 'T' should be removed and with acetone after 'Tin' has been scribed onto the tin side with a diamond scribe. Using a hot plate (preferably a digitally controlled one) set the temperature to 150 degrees C. Place aluminum foil onto the plate (so as not to dirty the hot plate) once this temperature has been reached.
9. To bond one or more plates together with a sacrificial plate, put a glass plate on the hot plate (on top of the foil). Then melt a uniform layer of crystalbond onto the glass. Make sure this layer is thick enough so that any bubbles formed can be squeezed out by pushing down on the glass with a bunched-up cleanroom wipe (so as not to damage the substrate or burn your hands). Some amount of air pockets is usually acceptable as long as you feel that the substrate can be oriented (by turning 90 degrees 1-3 times) in such a way as to make sure none of your holes will hit an air pocket. Having the pattern printed on a transparency can help with this. Note that the plates to be drilled must be oriented such that when the plates are loaded in the WaterJet machine, the sacrificial plate is in the bottom and the plate to be drilled is on top of this with the tin side up. Also, it is advised that another sacrificial cover plate be bonded (with Crystalbond as before) to the top surface of the borofloat glass. This will protect the top surface from excessive damage that would make it difficult to get a good seal with a pipette tip for injecting polymer into the channels.
10. Send the bonded plates with the diskette to the machine shop.
11. After the plates have been cut, they need to be cleaned. First separate each substrate from the sacrificial glass plate. This can be done by heating each plate on a hotplate set to 150 °C (thicker plate touching the hot plate). A piece of tin foil should be placed on the hot plate. When the Crystalbond is suitably hot, you can slowly pry the two plates apart and set each on aside to cool on a piece of tin foil. The sacrificial glass plates can be discarded in a waste bin suitable for sharp objects.

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12. Soak the substrates in methanol for a minimum of 2 hours. The container used for this should be sealed with tin foil to reduce evaporation losses.
13. Use methanol in a spray bottle and/or a brush to remove as much as the Crystalbond as possible.
14. Put your substrates in a hot Piranha (3:1) for 15 minutes. See attached 'Piranha Clean 3:1' protocol.

Attachments: 1. Piranha Clean 3:1 protocol;