Dear Dr. Boris Stoeber,

We are enclosing the report “Development of micropump using thermally activated hydrogels”. The report was written by Honza Vohradsky, Jun Wei Fu and Philip Edgcumbe as a requirement for the UBC Engineering Physics APSC 479 report.

The goal of this project is to design and fabricate a thermally activated peristaltic micropump in a monolithic multi-layer polydimethylsiloxane (PDMS) device. Designing a new type of micropump in the field of microfluidics is important because microfluidics is a fast growing field with applications in everything from micro-PCR to digital microfluidics for early cancer detection.

Project objective #1, eliminate leakage, and objective #3, production of portable microfluidic device were completed. Project objective #2, microvalves with response time of <3 seconds incorporated into a micropump was not completed. In early January, 2011 we discovered that replacing 15% Pluronic with 17% Pluronic and spinning on PDMS at 8000rpm allowed for good gel formation and elimination of leakage on the device. In the next few weeks we will test the spin-on technique with 17% Pluronic and hope to complete objective #2 shortly.

Sincerely,

Honza Vohradsky, Jun Wei Fu and Philip Edgcumbe

Enclosure
Executive Summary

The aim of this project is to design and fabricate a thermally activated peristaltic micropump in a monolithic multi-layer polydimethylsiloxane (PDMS) device. The advantage of our micropump over the conventional pressure activated micropumps is that our design only requires one pressure-control valve whereas the conventional approach requires one pressure-control valve for each valve. The goal of the project is to incorporate the newly designed micropump into a fully portable microfluidic device that can be used to pump fluid and cells through the chip. The goal is to design thermally activated valves with a response time of <3 seconds and micropumps that can pump fluid at 0.1 nL/sec. Designing a new type of micropump in the field of microfluidics is important because microfluidics is a fast growing field with applications in everything from micro-PCR to digital microfluidics for early cancer detection.

The equipment and resources of this project fall into three distinct sub-categories. They are: Design, fabrication and testing. For device design we used a computer with 2D AutoCAD drawing capabilities and a printer for transparencies with 10um resolution is required. For fabrication, we used a cleanroom with wet bench, spinner, hot plate, UV light, gold for evaporation, gold evaporator, PDMS mixing facilities, oxygen plasma, plasma bonding and lab space is required. For testing, we used an inverted microscope, pressure source and fluorescent particles.

The project is sponsored by Dr. Boris Stoeber and he has agreed to provide the resources and equipment that we need to make this project a success. This is an exciting project which has the potential to offer a significant new tool to the field of microfluidics.

Project objective #1, eliminate leakage, and objective #3, production of portable microfluidic device were completed. Project objective #2, microvalves with response time of <3 seconds incorporated into a micropump was not completed. Leakage was eliminated by spinning on uncured PDMS onto a glass slide, curing the PDMS and then bonding the glass slide to a PDMS multi-layer device. A portable carriage with pressure sources, microcontroller and power supply was designed and built for carrying our microfluidic device and a program developed for the microcontroller to operate the microfluidic pump. The development of the micropump was not completed because of inconsistent Pluronic gel behavior and it took us until January, 2011 to eliminate leakage in our device. All team members are committed to continuing the project at a collective work rate of 10 hours per week until the micropump works on the portable device.

Key recommendations are to make and test more microfluidic devices with spin-on PDMS and characterize device response time, pumping rate and pressure. Further, we will test the adhesion promoter GE SS412 for improving the spin-on PDMS bonding to glass.
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1 Introduction

1.1 Background and Motivation
We propose to develop a thermally activated micropump and implement the technology in a portable microfluidic device. The thermally activated micropump will be a new kind of tool in the field of microfluidics. Microfluidics is a fast growing field with many interesting applications in biology and beyond.

1.1.1 Technical Background

1.1.1.1 An introduction to microfluidics
The field of microfluidics involves the manipulation of small ($10^{-9}$ to $10^{-18}$ liters) amounts of fluids with channels that are tens to hundreds of micrometers across. The early microfluidic devices were developed to use very small quantities of samples and reagents and to do low cost analysis of chemical solutions. Microfluidics owes its ongoing popularity not only to its size but also due to the behavior of fluids at the microscale. Namely, microfluidics operates in a regime of low Reynolds number (i.e.: the ratio of momentum of a fluid to its viscosity is low) which allows for laminar flow. Further, factors like fluidic resistance, surface tension and energy dissipation starts to dominate the system.

1.1.1.2 Paradigm shift: Application of polydimethysiloxane (PDMS) for multilayer microfluidic chips and valves
Microfluidics, and its associated ability to take advantage of the behavior of solutions at the microscale, was first explored in detail in the 1990s. Manz et al.'s paper written in 1992, is an example of one of the first articles in the field\(^1\). However, the era of rapid and affordable microfluidic device prototyping only came of its own in 2000. In 2000, Unger et al published an article titled: "Monolithic Microfabricated Valves and Pumps by Multilayer Soft Lithography"\(^2\). In this article, Unger et al showed the world how to use Polydimethysiloxane (PDMS) to make simple multilayer valves. Unger's valves are simple to make and simple to operate. The multilayer devices consist of a fluidic channel and control channel. The control channel and the fluidic channel are on top of each other and where they cross they are separated by a thin member (approx. 10μm). The crossing point between the control and fluidic channel is where a valve is. To activate the valve, pressure is applied in the control channel the membrane deflects into the fluidic channel. The membrane is easily deflected because PDMS, the elastomer used to make


the device is a soft material with Young’s modulus of 750 kPa. The pressure of the various valves is controlled outside of the chip by an array of solenoid valves.

1.1.2 State of the art technology - A comparison

1.1.2.1 Current microfluidic valving strategies

Since flow control is an integral part of microfluidic devices, many research groups have proposed alternative designs for valves and pumps. For example, materials with large thermal expansion coefficients have been used to open and close hydrogels have been used to develop pH-sensitive microvalves or other thermally sensitive hydrogels have been heated up by a laser. Each of the proposed valve and pump designs has drawbacks. The large thermal coefficient valves have poor response time, the pH sensitive hydrogels have very limited applications and the laser activation of thermally sensitive valves introduces added complexity and cost to the system.

1.1.2.2 Our proposal - a novel microfluidic valving strategy

Our design uses Pluronic F127, a triblock copolymer, which when heated, has a phase transition from low viscosity to a soft, high viscosity cubic crystalline gel phase. We heat the Pluronic with an on-chip heater that is made of gold and selectively deposited by evaporation. Gel formation controls the multilayer valve by determining whether or not the external pressure reaches and deflects the valve. The advantage of our design is that it promises a fast response time and it is simple to fabricate and operate. We will use the thermally activated micropump to build a low-power portable microfluidic device capable of moving cells in a loop in the microfluidic chip.

1.1.2.3 Currently available portable microfluidics technologies

Microfluidics is a rapidly developing field where applications are being found for the technology at a rapid pace. Currently, there are many commercial implementations of benchtop microfluidic systems capable of carrying out complex experiments at significant time and cost savings. One area of particular promise is being able to deliver medical diagnostic tests in the field. This has two key advantages: firstly, results of the test can be reported immediately, and

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secondly, the cost of doing such a test is dramatically reduced without the need to equip and staff a wet lab. To our knowledge, only electrokinetics have been demonstrated using a portable (i.e. one that can be unplugged from the wall) device.\(^8\)

The company closest to bringing a product to market may be Micronics Microfluidics in the form of immunoassay and immunohematology systems. However, there is no mention of either system or their technologies in the academic literature.

**1.1.3 Alternative strategies**

**1.1.3.1 Choosing polydimethysiloxane (PDMS) over glass for microfluidic chip**

We had to choose between using PDMS or glass for our device. The five main differences between PDMS and glass are: Fabrication process, bonding, softness of material, heat transfer coefficient and heat capacity. For PDMS the fabrication process involves making a photo-resist via UV exposure that once fabricated can be used repeatedly for multiple PDMS chips. PDMS can be bonded to glass very easily via oxygen plasma treatment. PDMS is very soft, it has a Young’s modulus of 750 kPa. Finally, it has a heat transfer coefficient of 0.21 w m\(^{-1}\)K\(^{-1}\) and heat capacity of 1500 w m\(^{-2}\) K\(^{-1}\) (Niu, ZQ; Chen, WY; Shao, SY, et al). For glass, the fabrication process is more involved. Fabrication includes etching glass using hydrofluoric acid (HF) and anodic bond of the silicon to glass. This process uses HF, a dangerous chemical, is more expensive, and no mold is created. Glass is very hard so any valve would have had to be built by putting a PDMS membrane between two glass layers. Glass has a heat transfer coefficient of 0.75 w m\(^{-1}\)K\(^{-1}\) and heat capacity of 834.61 w m\(^{-2}\) K\(^{-1}\) (Niu, ZQ; Chen, WY; Shao, SY, et al). The lower heat capacity of the glass is attractive because our heaters will inevitably heat the surrounding channel walls. A low heat capacity assures that heating the surrounding walls does not interfere too much with the heating or cooling of the solution in the microfluidic chip. Further, unlike PDMS channels, glass channels do not expand under pressure. Expanding channels extend response time because more fluid needs to be cycled in and out of the valve for each cycle. Even though glass has a lower heat capacity and non-expanding fluidic channels, we chose to use PDMS. The advantage of cheaper, simpler and faster fabrication process for PDMS more than outweighs the advantages that glass has to offer.

**1.1.3.2 Chip fabrication considerations**

Another key consideration was the method of fabrication for the fluidic chip. To date, the fabrication procedure we have used is multilayer soft lithography. This process begins with the design of a mask using Solidworks. This is then printed on transparency sheets using laser printers to form a mask. This mask is then used to selectively etch a silicon wafer with UV

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exposure. Using a typical office printer, feature sizes of 250μm are achievable; by using high resolution 20 000 dpi printers, feature sizes of up to 10μm are possible.\(^9\)

The key advantage of this method is that has well characterized, repeatable results in terms of channel dimensions. Drawbacks of it are the need for ~20min of cleanroom time and the turnover time for shipment of high-resolution transparency prints from a US supplier (CAD Art Services).

Recently, novel methods of prototyping soft fluidic chips have been examined, the most notable of which being the use of Shrinky Dink prestressed thermoplastic sheets. This material is used for children’s toys whereby designs can be drawn onto the sheets and then heated to induce shrinking of the material. In fabricating microfluidic devices, Shrinky Dins can be used either to make a mould for the PDMS or be used to make the fluidic chip itself.\(^{10,11}\) Yet another mould making method is to transfer the layout from a laser printed piece of glossy paper to brass plate to selectively shield desired areas from an etching agent.\(^{12}\)

All of these methods are capable of giving feature sizes acceptable to the needs of this project with the benefit of significantly reduced cost, iteration time, and eliminating cleanroom time. However, since we have no firsthand experience with these methods and are not expecting the need for a large number of design iterations, we have opted to continue with the soft lithography process.

### 1.1.3.3 Connecting macro to micro

The gold heater traces are embedded between the PDMS and glass, with only small pads exposed to which leads are attached to provide power. Currently, the leads are attached by soldering, which frequently causes problems with jumpers since the pad spacing is quite tight. Our solution was to re-design our heater pads to fit the standard protoboard spacing and use pogo-pins attached to a protoboard which are perfectly aligned with our heater pads. This design modification streamlined our testing procedures.

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1.1.3.4 Pressure source considerations
To build a portable device, a pressure source to drive the control fluid independent of central lab compressed air is required. For this application, several configurations of camp stove fuel reservoirs were considered, along with a custom solution. Camp stove fuel reservoirs offer fluid capacity ranging from 325 - 975mL and an integrated pump. The fluidic chip is expected to require $1.54 \times 10^{-9}$ m$^3$/s, giving an expected run time of 2.44 days, which is extremely oversized. Instead, the Stoeber lab has a small, custom-designed reservoir which was modified to retain pressure and allow for a pressure relief valve and gauge.

1.1.4 Results from previous experimental work
This is an important project for our sponsor, Dr. Boris Stoeber, because he has worked extensively with Pluronic and is keen to see its applications in microfluidics. In 2005, Dr. Stoeber published an article showing that Pluronic could be used to stop flow in glass channel. In 2010, Dr. Stoeber published an article with graduate student Mr. Vahid Bazargan which is a proof-of-concept article for thermally activated microvalves. This project is a natural extension to that publication because we propose to make a micropump using a similar concept and show how the technology can be applied on a portable device. Critically, we propose to improve the microvalves response time from 20 seconds to 3 seconds and to implement a micropump. We have made significant changes to Mr. Bazargan's design to improve our response time. Philip worked with Dr. Stoeber during the summer to design and develop the micropump. However, the design and fabrication steps proved to be more difficult than anticipated and as of the end of the summer, Philip had sent in two design iterations and fabricated several devices. He was able to form a Pluronic gel in the microfluidic chips and could see valve deflection when a large pressure was applied to the system. However, leakage of PDMS between the gold, glass and PDMS remains an ongoing problem by the end of the summer Philip had not successfully deflected the membrane of the valve via gel formation. At the end of the summer the project was far from done. There is still a lot of work to do to solve the leakage problem, to optimize and characterize the design and to build a portable microfluidic device.

1.1.5 Project Sponsor
The sponsor for this project is Dr. Boris Stoeber, a Professor at UBC that is cross-appointed in electrical and mechanical engineering.

1.1.6 Power consumption calculation
Power Drain:
Arduino = 25mA
Pressure Transducer = 20mW, $I_p = (20mW)/(5V) = 4mA$

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Heaters = \((15\text{mW}) \times 6\)heaters = 90mW, \(I_h = \frac{(90\text{mW})}{(5\text{V})} = 18\text{mA}\)
Total Current = 25mA + 4mA + 18mA = 47mA

Battery Charge = 565mAh = 2034C

Time to drain the battery = \(\frac{(2034\text{C})}{(47\times10^{-3}\text{A})} = 43300\text{s} = 12\text{h}\)

It will take 12 hours of continuous operation to drain the battery.
2 Discussion

2.1 Design

2.1.1 Macroscopic Portable Device

One of our key objectives was the design of a portable device that allowed us to actuate the micropump without any fixed air or electric utilities. To achieve this, we designed a pressure/fluid storage reservoir with power provided from 2 9V batteries controlled by an Arduino Mini.

![Block Diagram of macroscopic portable device](image1)

**Figure 1 - Block Diagram of macroscopic portable device**

![Macroscopic portable device](image2)

**Figure 2 - Macroscopic portable device**

2.1.2 Fluid Supply Systems

The pressure charger and reservoirs for the fluidic and control channels are identical systems designed to supply fluid at constant pressure. The reservoirs are pressurized at the beginning of the experiment; since its capacity is much greater than the volume of fluid delivered, the pressure at the beginning and end can be assumed to be constant.
The reservoir was furnished with a Schrader valve to allow for flexibility in the initial source of pressure. In the lab, this can be provided from compressed air utilities while in the field, a hand bike pump can be used.

2.1.3 Heater Power and Controller
In the past, the heaters have been connected to the fluidic device by soldering wires to heater pads on the glass slide. To improve this, we have made 2 main improvements:

- Pogo pins
- Arduino-controlled heating

2.1.3.1 Pogo Pins
Previously, to power the heaters, wires needed to be soldered by hand to heater pads. This meant that the pads had to be relatively large and there was a high chance of shorting pads together during soldering. Because of how the gold is deposited onto the glass slides, removing and resoldering wires will often damage the pad, rendering the heater unusable.

A pogo pin is a spring-loaded connector commonly used for attaching electronics equipment temporarily to produced systems for testing and initial programming. The spring-loaded action allows the connector to make solid, repeatable contact with the pad and allows for differences in height due to manufacturing variation. Using pogo pins as opposed to allows for a higher density of electrical interconnects to the device, potentially allowing a greater number of heaters and sensors to be implemented in the same envelope; whereas previously, the pad density was governed by the pad area needed for hand soldering, we now are able to use standard protoboard spacing of 0.1” for ease of fabrication. In the future, it will be possible to reduce the pad pitch to slightly larger than the pogo pin tip diameter.

![Round-tipped pogo pin](http://www.sparkfun.com/products/9173)

2.1.3.2 Arduino-Controlled Heating
The Arduino Pro Mini microcontroller has 14 GPIO pins, of which 6 have PWM implemented. The use of a microcontroller to control heating levels allows for greater precision of heating, as well as the ability to experiment with different heating curves. To boost the available current, a basic npn transistor amplifier circuit was used.
The code used to control one cycle of valve operation is shown in Appendix E. The code is quite simple, as all that it needs to do is turn heaters on and off in the proper sequence. It uses two PWM outputs, one for each heater. The voltage to be sent to the heater through the PWM output needs to be high enough to cause Pluronic gel formation but not as high as to cause electrolysis. Therefore the values sent to the PWM outputs will change depending on the heaters and the powers necessary to form gel formation. Theoretically, the power necessary to cause gel formation should be constant and as such the output of the PWM should be determined based only on the resistance of the heater.

However, in reality the power delivered to the Pluronic depends on the heat transfer properties of the heaters and other factors. Therefore the power necessary to turn the heater on needs to be determined experimentally using a DC power supply. This experimental power is equal to the average power output of the PWM, which in turn depends on the duty cycle set in the code. The average power of the PWM output is:

$$ P_{PWM} = \frac{(n_{PWM} + 1)V_{PWM}^2}{256 R} $$

Where $n_{PWM}$ is the value (between 0 and 255) sent to the PWM output in the code, $V$ is the voltage output of the Arduino after amplification and $R$ is the resistance of the heater. This power needs to be equal to the power $P_{DC}$ necessary to cause gel formation which is experimentally determined from voltage as:

$$ P_{DC} = \frac{V_{DC}^2}{R} $$

Where $V_{DC}$ is the power supply voltage necessary to cause gel formation and $R$ is the resistance of the heater. Setting these equal and solving for the PWM value gives:
\[ n_{PWM} = 256 \frac{V_{DC}^2}{V_{PWM}^2} - 1 \]

\( n_{PWM} \) is equal to the variable \( int PWMValveOn \) in the code of Appendix E. Finally, it should be noted that the value \( V_{PWM} \) is not the 5V voltage output of the Arduino board but rather the 18V signal after amplification, i.e. \( V_{PWM} = 18V \).

In addition, the time delays between the different parts of the valve cycle need to be determined and set in the code. These time delays need to be determined experimentally when the response time of the valve is determined and they need to be as low as possible to minimize the valve’s response time. The time delays are likely the same for each valve.

### 2.1.4 Series 4 Design

#### 2.1.4.1 Goals

The Series 4 design is a general revision of the heater and control channel layers. Compared to the Series 3 design, the main goals were:

- Protoboard compatibility
- Integrated temperature sensing
- Greater fluid path length
- Larger valve actuation area
- Removal of other channels and diffusion barriers

#### 2.1.4.2 Protoboard Compatibility

To eliminate the problems associated with soldering wires to the heater pads, we opted to connect the heaters to power sources and other devices using pogo pins soldered to standard pitch (0.1") protoboards. Since no soldering of wires would be needed, the pad size can be much smaller, allowing for greater density of electrical interconnects; whereas the Series 3 heater pads required 61mm\(^2\) for 4 interconnects, we were able to put 6 pads in a 74mm\(^2\) envelope.
2.1.4.3 Integrated Temperature Sensing
Since Pluronic has a small temperature range in which it forms a gel. Both above and below this range, the fluid has identical flow properties, making it difficult to tell if the fluid is above or below the gelation temperature. To measure the temperature, a RTD was implemented by interdigitating two heater systems. In this way, one can be used as a heater and the other as an RTD. An added advantage of this design is redundancy; if using the RTD is not necessary, two independent heaters are available in each heating area.

2.1.4.4 Greater Fluid Path Length
From our experiments on the Series 3 devices, we believe there is a strong relationship between total wall area heated and the ability for the formed gel to prevent flow. In particular, design 3B, which uses dense columns in the heating area instead of zig zag channels, did not appear to hold a gel at all. For this reason, we wanted to increase the total fluid path length under the heating area.

2.1.4.5 Larger Valve Actuation Area
The volumetric flow rate from a peristaltic pump is dependent on the volume of fluid it is able to displace. To increase our pumping capacity, the fluidic channel was tripled through the pumping zone, requiring the valve area to be stretched.
Figure 7 – Control channel heating area details; a) Series 3C; b) Series 3A; c) Series 4B. The orange box indicates the nominal area of flow.
Figure 8 - Time lapse of gel formation and dissolution. Heater voltage 13V was held constant with pressure varied. a) 1psi, no flow; b) 3psi, no flow; c) 5psi, no flow; d) 13 psi with flow.

2.1.4.6 Removal of Other Channels and Diffusion Barriers
Series 3 includes a coolant flow channel and a diffusion prevention channel beside the primary flow path. In characterizing these devices, we saw that the fluid would leak along heater traces into these side channels. Removing these would simplify the overall design and may increase reliability.

Figure 9 - Control channel; a) Series 3B; b) Series 4B. The orange and green boxes indicate the diffusion barrier and the valve actuation areas, respectively.

2.1.4.7 Greater Fluid Path Length
From our experiments on the Series 3 devices, we believe there is a strong relationship between total wall area heated and the ability for the formed gel to prevent flow. In particular, design 3B, which uses dense columns in the heating area instead of zig zag channels, did not appear to hold a gel at all. For this reason, we wanted to increase the total fluid path length under the heating area.
Figure 10 – Control channel heating area details; a) Series 3C; b) Series 3A; c) Series 4B. The orange box indicates the nominal area of flow.

2.2 Testing

2.2.1 Overview

In the context of the deliverables we hoped to achieve, several experiments were designed to address individual issues. Presented chronologically, these tests were:

- **Plasma Bonding:**

- **Parylene Bonding:**

- **Pluronic Rheology:** Since we were unable to form Pluronic gel as expected in the fluidic devices, the quality of the Pluronic itself became suspect. To verify the viscometric properties, rheology was carried out on several samples.

- **RTD:** A key feature of the Series 4 heater design is the addition of an integral RTD. To verify that it functions as predicted and produces repeatable data, the resistance of several samples of this device was tested under known temperature conditions.

- **Electrolysis:** Although the formation of bubbles had been seen within the control channel in the past, this had been attributed to heating the fluid past its boiling temperature. To investigate if electrolysis was possible and how to detect it, an experiment was designed to rule out other sources of bubbles and check for this effect.

2.2.2 Pluronic Rheology

2.2.2.1 Introduction
When we had difficulty getting consistent results for Pluronic gelation in the device, we first attempted to check the Pluronic response by putting it in an oven and on a hotplate. For these experiments, we varied the temperature from ambient to 65 degrees and did not observe gelation at any temperature in this range. As this is far higher than the published gel temperature, this result was very unexpected. These experiments were consistent between over 6 batches of Pluronic made with the following parameters varied:

- Water type: distilled and deionized
- Fluorescent tracer particles: in the mix and not

To undertake a more careful analysis the thermal properties of Pluronic, we used a rheometer to check if the viscosity profile is as expected.

2.2.2.2 Methods

We tested 3 samples using an Anton Paar MRC series rheometer using the cone and plate geometry. Their preparation parameters are as shown in Table 1.

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>Target Pluronic % by wt</th>
<th>Fluorescent Particles [g]\textsuperscript{1}</th>
<th>Water [g]</th>
<th>Pluronic [g]</th>
<th>Pluronic % by wt.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>15</td>
<td>0.114</td>
<td>8.3793\textsuperscript{2}</td>
<td>1.5037</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>15</td>
<td>0</td>
<td>4.2603</td>
<td>0.7497</td>
<td>14.96</td>
</tr>
<tr>
<td>12</td>
<td>15</td>
<td>0.0345</td>
<td>4.2187</td>
<td>0.7848</td>
<td>15.57</td>
</tr>
</tbody>
</table>

\textsuperscript{1}The fluorescent particles are provided to us in a concentrated, 0.02% solution with water.

\textsuperscript{2}This sample was prepared with DI water; subsequent samples were all prepared with distilled water.

Table 1 - Properties of Pluronic batches tested by Rheology

2.2.2.3 Results

The results are presented in Figure 11.
2.2.2.4 Analysis
The erratic viscosity measurements above the gel temperature can be attributed to the fluid being a two-phase system whereby there are areas of gel and liquid, depending on localized temperature differences. This effect also explains why we do not see the fluid return back to a liquid at high temperatures as expected.

2.2.2.5 Conclusion
The lower bound on the gelation temperature varies between 32-34°C. We do not see a temperature at which the gel re-liquefies; this is believed to be since the phase there may be a two-phase (gel and liquid) system present between the cone and plate whereby there are areas of liquid and gel. This means that due to the experimental apparatus, all the data above the lower gelation temperature should be treated as suspect.

2.2.3 Plasma Bonding

2.2.3.1 Introduction
Oxygen plasma bonding is a very popular technique in the field of microfluidics. It is used to create a bond between PDMS to PDMS or PDMS to glass. Oxygen plasma improves adhesiveness by cleaning the surface of contaminants and introducing reactive chemical groups. In PDMS, the -O-Si(CH$_3$)$_2$- group is converted to a silanol group (-OH) which changes the PDMS surface chemistry from hydrophobic to hydrophilic and allows for Si-O-Si bonds between PDMS to PDMS surfaces or PDMS to glass surfaces.

We use the direct bonding via oxygen plasma both for bonding our multi-layer devices to glass and for bonding our multi-layer devices to the 10um of PDMS that is spun onto the glass surface.

2.2.3.2 Methods
The following parameters were varied:

- Power: 15, 30 watts
- Time: 10, 15, 20 seconds
- Weight: 0, 4, 8 pounds

The following parameters were not varied:

- Gas composition: 100% oxygen
- Chamber pressure: 500mtorr

2.2.3.3 Conclusions
There was no apparent link between the variation of the plasma parameters and the quality of the bond. The best 30 watts, 15 seconds and 4 pounds, although even this would yield approximately 30% success.
2.2.4 Parylene Bonding

2.2.4.1 Introduction / Motivation

Parylene bonding is an alternate method of bonding PDMS to PDMS or PDMS to glass. As mentioned above, one of the issues we were having with the device was leakage of Pluronic along the gold heater traces at the glass-PDMS interface. Sometimes the bond between the PDMS and the glass failed completely and the PDMS was partially coming off the glass, rendering the chip unusable. The method that we have been using to bond the PDMS to glass was plasma bonding, a standard bonding method widely used in microfluidics. This method works very well for bonding PDMS to plain glass but the gold heater traces have a significant negative effect on the bond strength (though they are only 100nm in height).

To resolve this issue, we have explored an alternate bonding method, parylene bonding. This method is not as widely used as diffusion or plasma bonding so there is less available literature on the subject. Parylene is the name given to a family of poly(paraxylylene) polymers that are typically used as moisture and dielectric barriers. As such, it is often coated on printed circuit boards and, due to its biocompatibility, also on medical devices. This biocompatibility makes parylene a potentially useful material in the fabrication BioMEMS devices. Parylene is typically deposited on the desired surface by chemical vapour deposition, which also results in the parylene polymerization. There are 3 different types of parylene and the one that we were using for the bonding test is Parylene-C. Should the parylene bonding method prove to be successful, it would make a useful new bonding method for the UBC MEMS group.

2.2.4.2 Method

First of all, the two surfaces to be bonded are coated with a thin layer of parylene. The coating is done in a parylene coating chamber where parylene is evaporated, directed towards the chamber containing the glass slides and PDMS to be coated, and then the vapour deposits on everything inside the chamber with a uniform thickness.

We investigated the bonding process based on available literature and used parameters that other researchers have reported as having produced positive results. The resulting bonding method is as follows. After the coating is finished, the two layers are pressed together in a high temperature environment for 30 minutes. The idea is that during this time the parylene is heated above its glass transition temperature and as a result the chains on the two surfaces interlink, resulting in a bond. The bonding process needs to be done in a vacuum oven as parylene has a tendency to oxidize at high temperatures. Since the Stoeber lab does not possess a vacuum oven, we performed the experiment together with Kevin Heyries, a postdoc in Dr. Hansen’s lab and used the Hansen lab’s vacuum oven and cleanroom.

In addition, the plates need to be pressed together quite strongly. The published papers recommend pressures of 0.5MPa - 16MPa. To minimize damage to the chip and the microfluidic channels, we used a pressure at the lower end of the range, 1MPa. We designed and built a
thermal press held together by 5 bolts which could be adjusted by a torque wrench to achieve the desired pressure.

The temperature of the oven and the baking time were also parameters that needed to be set. According to Noh, Moon et al.\textsuperscript{14} the ideal temperature is between 160°C and 200°C. In fact, the results were the same within experimental uncertainty for this range of temperatures. Therefore we used a temperature of 160°C. The baking time does not have an effect on the bond strength, as long as it is greater than about 10 minutes. Therefore we baked the chips for 30 minutes, then turned off the oven and waited for it to cool down to below 90°C (glass transition temperature of parylene).

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{press.png}
\caption{Press used to apply 1MPa pressure during parylene thermal bonding.}
\end{figure}

\textbf{2.2.4.3 Results}

After the parylene was pulled from the oven, it was allowed to cool to room temperature. Then we placed it under the inverted microscope and attempted to run water through it. However, we have observed complete channel collapse as though the channels did not even exist and the water did not flow through the parylene bonded channels. At a pressure of 30psi the bonding failed. Thus parylene bonding has proved to be a method unsuitable for our purposes, as it resulted in a weak bond and complete channel collapse.

The channels collapsed due to the high pressure that was applied to the PDMS during the thermal bonding. After all, applying 1MPa of pressure to a small channel made of an elastic material is very likely to squeeze it and bond the top of the channel to the bottom of the channel. Therefore to eliminate channel collapse, we need to lower the applied pressure.

After the channel failed, we tried peeling the PDMS off the glass. It was quite easy to peel it off near the edges but the PDMS was strongly bonded to the glass in the center. The reason is that when the elastic PDMS was pressed during the thermal bonding, the pressure was the highest in the center of the chip and decreased towards the outside due to elastic strain in the material. This suggests that pressure has a significant effect on the bonding properties, with high pressure being necessary to bond properly. Therefore to increase the bonding strength we need to increase the applied pressure, contradictory to the channel collapse requirement.

2.2.4.4 Conclusion
Parylene bonding is not a method that can be used in our project and thus was abandoned.

2.2.5 RTD Tests

2.2.5.1 Introduction
The series 4 glass slide heating area comprised of two independent heater systems. These were interdigitated, introducing redundancy as well as the provision to use one as a resistance temperature detector (RTD). The relationship between resistance and temperature is governed by the equation

\[ T = T_0 + \left( \frac{R}{R_0} - 1 \right) \cdot \frac{1}{\alpha} \]

Where: 
- \( T \) = temperature of the device
- \( T_0 \) = ambient temperature
- \( R \) = measured resistance
- \( R_0 \) = resistance at ambient
- \( \alpha \) = temperature coefficient of resistance, a material property

In general, \( \alpha \) is typically found in a table. In our case, since the heater is a combination of chromium and gold, \( \alpha \) is not well-defined. Thus, to find \( \alpha \), we needed to find a relationship between \( R, T_0 \) and \( R_0 \). To achieve this, we used several tests.

2.2.5.2 Methods, Results

2.2.5.2.1 Test 1: Heating one set, detecting with the other
In this experiment, we attached one of the heaters to a power supply and HP 34401A DMM. We then varied the power to the heater and measured the change in resistance seen by the RTD. This was done on two slides, one bare (i.e. no PDMS) and one covered with 5psi 15% Pluronic flow. The data from this test is presented in Figure 13 shows that the results were quite different for these two cases. To reduce experimental uncertainty, the experiment was modified to remove variables.
2.2.5.2.2 Test 2: Hotplate heating

Instead of heating the slide with the on-slide heaters, we used a Fisher Isotemp hotplate. This hotplate had an integral temperature display but its resolution was ±5°C and was an element temperature, rather than a hotplate surface temperature. To determine the temperature more accurately, we used the Omega HH23 thermocouple reader and J-type thermocouple measuring the slide temperature. The hotplate temperature was then varied and the thermocouple temperature and RTD resistance were measured for three cases:

- Bare heater
- Covered heater, no fluid
- Covered heater, fluid flowing at 5psi.

The apparatus is shown in Figure 14; the results are shown in Figure 15.

Figure 13 – RTD resistance vs heater voltage for the bare and covered heater cases

![Graph showing RTD resistance vs heater voltage for the bare and covered heater cases.](image-url)
2.2.5.3 Analysis

2.2.5.4 Analysis

The goal of the data analysis is to come up with a relationship that allows us to determine the temperature of any RTD we will be using in the future based on its resistance.

The relationship between resistance and temperature for an RTD is:

\[ R(T) = R_0 \times (1 + \alpha \times (T - T_0)) \]
Where $R_0$ is the resistance at the temperature $T_0$ and $\alpha$ is a material property at the temperature $T_0$. 

As is obvious from the above equation, the relation between resistance and temperature is linear, in agreement with our experimental results. Thus a linear relation has been fitted to the data. The results are:

Dry channel: $R(T) = 0.0011960T + 0.3724058$ [\Omega]

Wet Channel: $R(T) = 0.0013129T + 0.3784029$ [\Omega]

Bare Heater: $R(T) = 0.0016149T + 0.5567747$ [\Omega]

As mentioned above, $R_0$ and $\alpha$ need to be specified at a certain temperature. For simplicity, this temperature $T_0$ will be 20°C, as most tabulated results are at this temperature. The resulting relationship is:

Dry channel: $R(T) = 0.4116058\Omega \times (1 + 0.004761838/°C \times (T - 20°C))$

Wet Channel: $R(T) = 0.4046609\Omega \times (1 + 0.003244444/°C \times (T - 20°C))$

Bare Heater: $R(T) = 0.5890727\Omega \times (1 + 0.002741427/°C \times (T - 20°C))$

The resistances at 20°C, $R_0$ are quite variable due to variations that result from the manufacturing of the devices. The evaporation and lift-off of the RTD gold traces is not always reproducible and sometimes even results in unusable RTD’s. However, for our purposes it is not necessary to have exactly the same resistance in all the RTD’s that we produce because we can always measure the 20°C resistance in order to be able to take temperature measurements with our RTD device.

The more important constant is $\alpha$, also known as the temperature coefficient of resistance. Theoretically, $\alpha$ should be a material property and should not be affected by the state of the channel (dry/wet/bare). However, according to our results $\alpha$ varies in between experiments. Therefore in order to use the RTD to measure temperature we need to conduct more hotplate experiments and determine the average value of $\alpha$. As we improve our experimental technique, we may be getting more and more consistent results. The tabulated value for the temperature coefficient of resistance of gold is 0.003715/°C which is quite close to the values that we found. However our RTD is not made entirely out of gold; rather it is a 50nm layer of chromium and a 60nm layer of gold.

### 2.2.6 Electrolysis Test

#### 2.2.6.1 Introduction

The unusual results of RTD Test 1 caused us to ask if electrolysis was causing the strange results. Furthermore, throughout the characterizations of devices dating back even to the series 3 fluidic chips, when we have applied in excess of 10V, we have seen the formation of bubbles. Since
electrolysis had never been seen in past work by Bazargan et. al\textsuperscript{15}, it was not initially suspected and instead, the effect was attributed to overheating of the fluid to the point of boiling. In theory, electrolysis would happen whenever a potential of 1.23V is created in water. In our experiments, we typically explored voltages between 0-9V and did not believe any electrolysis occurred.

### 2.2.6.2 Methods

To determine at what voltage electrolysis will occur, a power supply was attached to the heater set. Since the magenta side is separate from the blue one, any current would pass through the fluid, allowing for electrolysis. To investigate if the location of electrolysis in the heating zone varies, 3 geometries were tested as shown in Figure 16. For all experiments, the power was varied from 0-6V with 15% Pluronic containing fluorescent beads being pushed through the control channel at 5psi.

![Figure 16](image)

**Figure 16 – Schematic of the electrolysis test apparatus; a) crossed leads; b) parallel leads; c) conventional connection**

### 2.2.6.3 Results

In both apparatus setups (a) and (b), the electrolysis would occur at random locations within the heating zone. Throughout the entire experiment, the current flow would remain constant at 0.045mA. Electrolysis was seen as small bubbles approximately 5 microns in diameter being formed in the control channel at a heater trace. These would first appear at 2.89V. As the voltage was increased, the bubbles would appear more in more locations within the heating zone and with greater diameters. Only above 4V are the bubbles large and widespread enough to be seen under UV illumination. From 2.89-4V, the only way to see the electrolysis is under visible light, which is not typically when characterizing fluidic devices.

Figure 17 – Electrolysis seen when the device is connected as in Figure 16 (a); a) before the application of 4V; b) t=2s after power applied; c) t=7s d) t=14s. Recorded using 10x lens.

2.2.6.4 Conclusions
Throughout our device characterizations, we have applied voltages far above the electrolysis threshold. Because this effect is not visible under UV light until 4V, we have considered voltages below this level to be safe; however, it is not apparent that the application of greater than 2.89V will cause electrolysis. Since this is not high enough to cause reach the gelation temperature of 15% Pluronic, the heater will need to be electrically isolated or redesigned with lower resistance to keep the applied voltage low.
3 Project Deliverables

3.1 Deliverables

3.2 As presented in our Project Charter (Appendix B - Recipe for 10 um SPR220-7.0 Mold for 4-inch Si Wafers)

Preparation Steps
Wafer is cleaned with Acetone, then Methanol, then Isopropanol Alcohol, and then gently blown with N2 gas.
Wafer is baked for dehydration for 20 minutes at 200°C, then cooled to room temperature for 10 min.

Spin Coating
The wafer is centered on the spinner chuck and vacuum sealed.
HMDS (hexamethyldisilazan) drops are placed on the wafer using a dropper until 50% of the wafer is covered.
HMDS is spun at 3500 rpm for 35 seconds. Let the wafer sit for 1 minute on the spinner before pouring photoresist.
SPR220-7.0 photoresist is poured over the wafer straight from the bottle, covering roughly half the area. The bottle should be cleaned thoroughly with a clean wipe before and after pouring. The wafer is spun for 5 sec at 500 rpm and 40 sec at 1500 rpm.
The wafer is let sit on the spinner for 1-2 minutes.

Photoresist Soft bake
The wafer is gradually warmed to 90 C, by using a layer of aluminum foil or wipe on the hotplate. After 1 minute transfer it directly to the 90 C hotplate, let it sit there for 2 min, then transfer to another hotplate at 115 C, let it sit for 3 min.
The wafer is slowly cooled to room temperature for 10 min

UV-Light Exposure
The wafer is loaded into the Canon PLA-501F double-side 100mm mask aligner.
The printed mask on a transparent sheet is attached to a thick glass plate and is loaded as the photomask into the mask aligner.
UV-light is exposed to the layer for 70 sec.
The wafer is let sit on the mask aligner for 1 min.
The wafer is cooled down at the room temperature 21°C for 30 min for dehydration.

Photoresist Develop
An MF 319 bath and a DI-water bath are prepared.
The wafer is placed into the MF-319 for 5 min and visually checked. If the developments looks completed, the wafer is placed into the water bath for 1 min and is rinsed with DI water and dried with a N2 gun.
The pattern is checked under the microscope and especially the corners and posts are examined for complete development. The developing process can be repeated if additional development is needed.
The thickness of the pattern then is measured using the Wyko NT1100 interferometer.

Reflow Process
The wafer is placed on the hot plate for 2 min at 90°C.
The hotplate is set for 140 C and ramps up for 5 minutes, then the hotplate is shut off.
The wafer remains on the hotplate for 5 minutes for gradual cooling, then taken off and allowed to cool to room temperature.
The shape and the thickness of the pattern then are measured using Wyko NT1100 optical.
Appendix C), the following deliverables were agreed upon at the start of the project:

1. A hydrogel actuated microvalve that has a response time of less than 3 seconds.
2. A portable device that allows the use of the fluidic chip independent of fixed power and air systems.
3. A peristaltic pump composed of hydrogel actuated valves capable of moving a cell in a loop.

3.2.1 Deliverable 1: Microvalve with 3 second response time
The design of the new Series 4 fluidic chips was completed. Due to ongoing fabrication and Pluronic issues, the performance has not been characterized. At present, we are able to form gel using the PDMS-coated chips, but these devices clog rapidly to allow for sustained characterization.

3.2.2 Deliverable 2: Portable device
The hardware for this device is complete. The code that drives the PWM heater controls has been written but due to the unavailability of functioning fluidic chips, it has not been validated.

3.2.3 Deliverable 3: Peristaltic pump
Due to ongoing fabrication and Pluronic issues, we have not been able to actuate valves with any degree of reliability. The requirement for 2 adjacent valves to function predictably has not been seen, causing us to not achieve this deliverable.

3.3 Financial Summary
3.3.1 Macro Components
The cost of the macroscopic components is as follows:

<table>
<thead>
<tr>
<th>Description</th>
<th>Qty</th>
<th>Vendor</th>
<th>Cost Per</th>
<th>Total Cost</th>
<th>Purchased by</th>
<th>Funded by</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arduino Mini</td>
<td>1</td>
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<td>28.16</td>
<td>Project Lab</td>
<td>Stoeber Lab</td>
</tr>
<tr>
<td>RB-Ard-02</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
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<td>Stoeber Lab</td>
</tr>
<tr>
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<td>London Drugs</td>
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<td>6.49</td>
<td>Project Lab</td>
<td>Stoeber Lab</td>
</tr>
<tr>
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<td>Stoeber Lab</td>
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<tr>
<td>Pressure Gauge</td>
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<td>Project Lab</td>
<td>Stoeber Lab</td>
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</table>

Table 2 - Macro Component Breakdown
3.3.2 Micro Components

<table>
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<th>Vendor</th>
<th>Cost Per</th>
<th>Total Cost</th>
<th>Purchased by</th>
<th>Funded by</th>
</tr>
</thead>
<tbody>
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<td>Cleanroom time</td>
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<tr>
<td>Transparency</td>
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<td>Gold Evaporation</td>
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<td>UBC</td>
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<td>180</td>
<td>Stoeber Lab</td>
<td>Stoeber Lab</td>
</tr>
</tbody>
</table>

Table 3- Micro Component Breakdown

3.4 Ongoing Commitments by Team Members
The team will continue to collectively put in a sum of 10 hours per week in an effort to complete the items as outlined in Section 5.1.
4 Conclusion

4.1 Important Results
Below is a list of important discoveries made during the project.

1. Parylene-parylene bonding does not work for PDMS microfluidic devices because the microfluidic devices collapse under the pressure applied to the two PDMS pieces to activate parylene bonding between them.
2. Diffusion of water through the PDMS, and resulting increase in Pluronic concentration in the microfluidic device is not an issue. We never saw the gel stop flowing of its own accord.
3. Spin-coating our glass slides with uncured PDMS at 8000rpm results in a 10um PDMS thickness and eliminates leakage.
4. We can form gel in our devices with 17% Pluronic. 15% Pluronic and below are not good candidates for gel formation.
5. It is possible to achieve heaters with 80nm thickness. Previously, Vahid used heaters with 250nm and there was concern that imperfections would have a dominant role below 250nm and make the heater non-functional..

4.2 Project Review
We completed two of our three objectives. We complete objective #1, eliminate leakage and objective #3, production of portable microfluidic device. We did not complete objective #2, microvalves with response time of <3 seconds incorporated into a micropump. We did not complete objective #2 because there were many unforeseen challenges in the fabrication and gel activation process. Four assumptions we made from the outset of the project were:

1. Gel formation could easily be predicted, achieved and reproduced.
2. There would be no electrolysis in the device.
3. We were confident that we had appropriate steps and contingency plans to quickly eliminate leakage in our device.
4. PDMS spun onto a glass slide at high rpm (>1000rpm) would have extremely high porosity due to stretching of the polymer.

4.2.1 Gel formation of Pluronic could easily be predicted, achieved and reproduced
We had inconsistent Pluronic behaviour throughout our experiments at both the macroscopic and microscopic level. Pluronic is supposed to reproducibly form a gel at a concentration dependent gel point. We calculated approximately how much power we needed from our heaters to heat up the fluid in our microchannels beyond the gel point. However, the Pluronic seemed to only sometime form a gel despite application of identical parameters. The following three paragraphs describe a situation in which the gel formation seemed to be lost over the period of two hours.

In early October we used design iteration 3 and 15% Pluronic to show that after we initially put Pluronic in our devices that we could get formation in both the snake design and dense column
designs. We observed that the snake design had better gel formation and pressure holding abilities and concluded that the more surface area the better the gel holding capability of the device. We tested the snake design to 13psi and the gel held for the entire test, the dense columns failed at 7psi.

When the snake design device was first connected and the control channel pressure inlet pumped at 10psi we observed gel formation and complete stoppage of flow within one second of turning on the heater at 490mW of power. Within five seconds of turning off the heater there was full liquefaction of the gel and fluid flow had recovered to its previous high velocity. The device was left in place with Pluronic flowing through the device while other experiments were performed. After two hours had gone by we tried the same experiment with identical pressure and power parameters and could not get gel formation. It was only at a pressure of one psi that we could get gel formation again. We still do not understand why the gel behaviour changed over the course of the two hour experiment. Pluronic from the same bottle was continually cycled through the device and the resistance of the heater and power applied was the same.

Inconsistent Pluronic behaviour was also observed at the macroscopic level as well. For example, when we initially placed 15% Pluronic (batch #2) in an oven we observed that from 35C to 45C the Pluronic was a solid gel, we could flip the Pluronic bottle upside down and the Pluronic stayed in place. At 50C the Pluronic became a liquid again. The Pluronic was kept in a tightly sealed bottle overnight and the following day when the same bottle was placed at 40C the Pluronic did not respond as before. It became more viscous but it still continually flowed to the bottom of the bottle. We made more Pluronic mixtures but none of them had the same response we originally observed.

When we could not reproducibly get gel formation by heating the Pluronic in the microfluidic channels or by heating the big bottle of Pluronic in the lab oven we started to suspect the Pluronic itself so we proceeded to do viscometry measurements with a viscometer. The viscometer persuasively showed that the 15% Pluronic had a sharp viscosity increase between 32C and 34C.

We still cannot explain the inconsistent Pluronic behaviour in which we had gel formation and then a few hours later with identical parameters could not reproduce the same gel formation. Further, we cannot explain why the viscometry shows a sudden spike in viscosity and why the Pluronic in the microfluidic device did not have the same behaviour. The most likely explanation is that the range of gel formation for 15% Pluronic is small and we consistently overheat the Pluronic past the gel formation point. However, this is an unsatisfactory explanation because we have carefully tested the full range of power that can be applied to the heaters.

4.2.2 There would be no electrolysis in the device.
Bazargan et al. undertook a similar project in 2008 which included creating a valve with two heaters in a microfluidic device. He heated the Pluronic by applying 25 mW of power (4.29 volts
and 5.83mA) across a 250nm thick gold heater. Vahid did not observe electrolysis so we did not expect to have electrolysis. However, we did observe electrolysis and the electrolysis may have contributed to our poor gel performance.

4.2.3 We were confident that we had appropriate steps and contingency plans to quickly eliminate leakage in our device.

We could not eliminate leakage in our device by either parylene-parylene bonding or varying plasma treatment parameters for PDMS to glass bonding. Our final solution is to spin on uncured PDMS on a glass slide at 8000 rpm which gave a thickness of 10um and a PDMS to PDMS bonding surface that can hold pressure up to 20 psi and does not have any leakage. We did not consider spinning on PDMS in our initial project proposal because Vahid told us that PDMS spun on at (>1000rpm) would be porous and prone to extensive diffusion.

In section 3.4 of our project proposal we proposed two new techniques by which we could eliminate leakage.

1) Collaboration with Dr. Eric Lagally to implement Dr. Lagally’s recently published peptide bonding technique\textsuperscript{16}. We planned to treat the PDMS with (3-aminopropyl)-trimethoxysilane (APTMS; 97%) and the glass and gold with 10% TMS-EDTA. The treatment results in peptide bond formation between the PDMS and glass and PDMS and gold. Philip did one round of unsuccessful tests in late August and we did not choose to pursue this option.

2) Collaboration with Dr. Carl Hansen to implement parylene-parylene bonding. This technique was pursued by Honza and it was declared unsuccessful on November 19\textsuperscript{th}, 2010.

We predicted that if either of the two techniques did not work we could further optimize the direct glass to PDMS bonding by reducing the gold heater thickness, modifying the plasma treatment time, modifying the bake time and adjusting the weights we placed on the PDMS after bonding. However, despite an exhaustive round of testing with the PECVD plasma machine we could not get consistent PDMS to glass bonds. Sometimes we had too much collapse and other times we had leakage everywhere. On December 13\textsuperscript{th}, 2010 Mario Beaudoin, UBC Cleanroom Manager, sent an email with attached picture to the UBC cleanroom noting that the inside of the PECVD machine was very dirty. We were never taught how to clean the internal chamber of the PECVD and most of our tests were done before December 13\textsuperscript{th}, 2010 so the dirtiness of the PECVD may have contributed to our poor bonding results.
4.2.4 PDMS spun onto a glass slide at high rpm (>1000rpm) would have extremely high porosity due to stretching of the polymer. Vahid told us that PDMS spun on at (>1000rpm) would be porous and prone to extensive diffusion. Thus, we did not consider spinning on PDMS onto our glass slide until early January, 2011 when it was obvious that parylene-parylene bonding and plasma treatment bonding parameter modification had not worked.
5 Recommendations

The recommendation section is broken into two sub-sections. The first sub-section is the specific recommendations that are directly related to the ongoing project goal of developing a micropump. The second sub-section is more general and includes retrospective recommendations about work flow and other project management strategies.

5.1 Specific Recommendations

1. Increase PDMS spin-on speed. Uncured PDMS is currently spun onto the glass slide at 8000rpm which results in a 10um thickness. 8000rpm is the upper bound of the spin speed of the spinner that is available to us in Dr. Stoeber’s lab. Finding a spinner capable of faster spin speeds will allow us to further reduce the PDMS thickness and increase response time.

2. Test adhesion promoter GE SS4120 in hopes of increasing the pressure that the triple layer PDMS devices can withstand.

3. Measure response time of individual valves.

4. Measure pumping rate and pumping pressure of final micropump.

5. Explore the discrepancy in the viscometry results between the 15% Pluronic that do and do not have fluorescent beads.

6. Repeat viscometry measurements for 17% Pluronic and compare to 15% Pluronic results.

5.2 General Recommendations

1. Recreate Vahid’s results. Use the identical microfluidic and heater design and identical project parameters to recreate Vahid’s results. If we had done this at the outset of the project we would quickly have identified that we needed 17% Pluronic instead of 15% Pluronic. Recreating Vahid’s device will help to establish whether our ongoing poor results are due to a poor design or something that is wrong with the Pluronic solution.

2. Optimization and large scale production of multi-layer microfluidic chips. We should have made three identical control channel wafers and three polyurethane molds so that we could make 12 microfluidic multi-layer chips at once. We only made microfluidic chips in batches of 4 which proved to be very time consuming.
References


6 Appendices

6.1 Appendix A - AZ 5214E Photoresist Datasheet
AZ 5214 E

Image Reversal Photoresist
GENERAL INFORMATION

This special photoresist is intended for lift-off-techniques which call for a negative wall profile. Although they are positive photoresists (and may even be used in that way) comprised of a novolak resin and naphthoquinone diazide as photoactive compound (PAC) they are capable of image reversal (IR) resulting in a negative pattern of the mask. In fact AZ 5214E is almost exclusively used in the IR-mode.

The image reversal capability is obtained by a special crosslinking agent in the resist formulation which becomes active at temperatures above 110°C and - what is even more important - only in exposed areas of the resist. The crosslinking agent together with exposed PAC leads to an almost insoluble (in developer) and no longer light sensitive substance, while the unexposed areas still behave like a normal unexposed positive photoresist. After a flood exposure (no mask required) this areas are dissolved in standard developer for positive photoresist, the crosslinked areas remain. The overall result is a negative image of the mask pattern.

As everybody knows a positive photoresist profile has a positive slope of 75 - 85° depending on the process conditions and the performance of the exposure equipment (only submicron-resists get close to 90°). This is mainly due to the absorption of the PAC which attenuates the light when penetrating through the resist layer (so called bulk effect). The result is a higher dissolution rate at the top and a lower rate at the bottom of the resist. When AZ 5214E is processed in the IR-mode this is reversed as higher exposed areas will be crosslinked to a higher degree than those with lower dose, dissolution rates accordingly. The final result will be a negative wall profile ideally suited for lift-off.

The most critical parameter of the IR-process is reversal-bake temperature, once optimised it must be kept constant within ± 1°C to maintain a consistent process. This temperature also has to be optimised individually. In any case it will fall within the range from 115 to 125°C. If IR-temperature is chosen too high (>130°C) the resist will thermally crosslink also in the unexposed areas, giving no pattern. To find out the suitable temperature following procedure is suggested:

Coat and prebake a few substrates with resist. Without exposing them to UV-light subject them to different reversal-bake temperatures, i.e. 115°, 120°, 125° and 130°C. Now apply a flood exposure of > 200mJ/cm² and afterwards immerse them into a standard developer make up, i.e. AZ 351B, 1:4 diluted, or AZ 726 MIF for 1 minute. From a part of the substrates the resist will be removed, another part (those exposed to a too high temperature) will remain with the resist thermally crosslinked on it. Optimum RB-temperature now is 5° to 10°C below the temperature where crosslinking starts.

The flood exposure is absolutely uncritical as long as sufficient energy is applied to make the unexposed areas soluble. 200 mJ/cm² is a good choice, but 150 - 500 mJ/cm² will have no major influence on the performance.

Finally it should be noted that the imagewise exposure energy is lower than with normal positive processes, generally only half of that. So a good rule of thumb is: compared to a standard positive resist process, imagewise exposure dose should be half of that, flood exposure energy double of that for AZ 5214E IR-processing.

Once understanding and being familiar with this IR-procedure it is quite simple to set up a different process for lift-off. A T-shaped profile can be achieved by the following process sequence:

The prebaked AZ 5214E photoresist is flood exposed (no mask) with a small amount of UV energy, just to generate some exposed PAC at the surface. Now the reversal-bake is performed to partially crosslink this top areas. By this treatment a top layer with a lowered dissolution rate compared to the bulk material is generated. After this the resist is treated like a normal positive photoresist (imagewise exposure and development) to generate a positive image! Due to the lower dissolution rate in the top layer a T-shaped profile with overhanging lips will be the result.
PHYSICAL and CHEMICAL PROPERTIES

<table>
<thead>
<tr>
<th></th>
<th>AZ 5214E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids content [%]</td>
<td>28.3</td>
</tr>
<tr>
<td>Viscosity [cSt at 25°C]</td>
<td>24.0</td>
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<tr>
<td>Absorptivity [l/g*cm] at 377nm</td>
<td>0.76</td>
</tr>
<tr>
<td>Solvent</td>
<td>methoxy-propyl acetate (PGMEA)</td>
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<tr>
<td>Max. water content [%]</td>
<td>0.50</td>
</tr>
<tr>
<td>Spectral sensitivity</td>
<td>310 - 420 nm</td>
</tr>
<tr>
<td>Coating characteristic</td>
<td>striation free</td>
</tr>
<tr>
<td>Filtration [µm absolute]</td>
<td>0.1</td>
</tr>
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</table>

FILM THICKNESS [µm] as FUNCTION of SPIN SPEED (characteristically)

<table>
<thead>
<tr>
<th>spin speed [rpm]</th>
<th>2000</th>
<th>3000</th>
<th>4000</th>
<th>5000</th>
<th>6000</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ 5214E</td>
<td>1.98</td>
<td>1.62</td>
<td>1.40</td>
<td>1.25</td>
<td>1.14</td>
</tr>
</tbody>
</table>

PROCESSING GUIDELINES

Dilution and edge bead removal: AZ EBR Solvent
Prebake: 110°C, 50”, hotplate
Exposure: broadband and monochromatic h- and i-line
Reversal bake: 120°C, 2 min., hotplate (most critical step)
Flood exposure: > 200 mJ/cm² (uncritical)
Development: AZ 351B, 1:4 (tank, spray) or AZ 726 (puddle)
Postbake: 120°C, 50s hotplate (optional)
Removal: AZ 100 Remover, conc.

HANDLING ADVISES

Consult the Material Safety Data Sheets provided by us or your local agent! This AZ Photoresists are made up with our patented safer solvent PGMEA. They are flammable liquids and should be kept away from oxidants, sparks and open flames. Protect from light and heat and store in sealed original containers between 0°C and 25°C, exceeding this range to -5°C or +30°C for 24 hours does not adversely affect the properties. Shelf life is limited and depends on the resist series. The expiration date is printed on the label of every bottle below the batch number and coded as [year/month/day]. AZ Photoresists are compatible with most commercially available wafer processing equipment. Recommended materials include PTFE, stainless steel and high-density poly-ethylene and -propylene.
The information contained herein is, to the best of our knowledge, true and accurate, but all recommendations are made without guarantee because the conditions of use are beyond our control. There is no implied warranty of merchantability or fitness for purpose of the product or products described here. In submitting this information, no liability is assumed or license or other rights expressed or implied given with respect to any existing or pending patent, patent application, or trademarks. The observance of all regulations and patents is the responsibility of the user.

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6.2 Appendix B - Recipe for 10 um SPR220-7.0 Mold for 4-inch Si Wafers

Preparation Steps
Wafer is cleaned with Acetone, then Methanol, then Isopropanol Alcohol, and then gently blown with N2 gas.
Wafer is baked for dehydration for 20 minutes at 200°C, then cooled to room temperature for 10 min.

Spin Coating
The wafer is centered on the spinner chuck and vacuum sealed.
HMDS (hexamethyldisilazan) drops are placed on the wafer using a dropper until 50% of the wafer is covered.
HMDS is spun at 3500 rpm for 35 seconds. Let the wafer sit for 1 minute on the spinner before pouring photoresist.
SPR220-7.0 photoresist is poured over the wafer straight from the bottle, covering roughly half the area. The bottle should be cleaned thoroughly with a clean wipe before and after pouring. The wafer is spun for 5 sec at 500 rpm and 40 sec at 1500 rpm.
The wafer is let sit on the spinner for 1-2 minutes.

Photoresist Soft bake
The wafer is gradually warmed to 90 C, by using a layer of aluminum foil or wipe on the hotplate. After 1 minute transfer it directly to the 90 C hotplate, let it sit there for 2 min, then transfer to another hotplate at 115 C, let it sit for 3 min.
The wafer is slowly cooled to room temperature for 10 min

UV-Light Exposure
The wafer is loaded into the Canon PLA-501F double-side 100mm mask aligner.
The printed mask on a transparent sheet is attached to a thick glass plate and is loaded as the photomask into the mask aligner.
UV-light is exposed to the layer for 70 sec.
The wafer is let sit on the mask aligner for 1 min.
The wafer is cooled down at the room temperature 21°C for 30 min for dehydration.

Photoresist Develop
An MF 319 bath and a DI-water bath are prepared.
The wafer is placed into the MF-319 for 5 min and visually checked. If the developments looks completed, the wafer is placed into the water bath for 1 min and is rinsed with DI water and dried with a N2 gun.
The pattern is checked under the microscope and especially the corners and posts are examined for complete development. The developing process can be repeated if additional development is needed.
The thickness of the pattern then is measured using the Wyko NT1100 interferometer.

Reflow Process
The wafer is placed on the hot plate for 2 min at 90°C.
The hotplate is set for 140 C and ramps up for 5 minutes, then the hotplate is shut off. The wafer remains on the hotplate for 5 minutes for gradual cooling, then taken off and allowed to cool to room temperature.
The shape and the thickness of the pattern then are measured using Wyko NT1100 optical.
Project Number, Title: 1071, A Micropump Using Thermally Activating Hydrogels

Project Summary:
The aim of this project is to design and fabricate a thermally activated peristaltic micropump in a monolithic multi-layer polydimethylsiloxane (PDMS) device. This project will incorporate the newly designed micropump into a fully portable microfluidic device that can be used to pump fluid and cells through the chip. The goal is to design thermally activated valves with a response time of <3 seconds and micropumps that can pump fluid at 0.1 nL/sec.

Start Date: September 27, 2010                     End Date: December 14, 2010

Statement of Deliverables:
- A hydrogel actuated microvalve that has a response time of less than 3 seconds.
- A portable device that allows the use of the fluidic chip independent of fixed power and air systems.
- A peristaltic pump composed of hydrogel actuated valves capable of moving a cell in a loop.

Criteria for Success:
- A hydrogel actuated microvalve that has a response time of less than 3 seconds.
- A portable device that allows the use of the fluidic chip independent of fixed power and air systems.
- A peristaltic pump composed of hydrogel actuated valves capable of moving a cell in a loop.
- Team members become familiar with state of the art fabrication, bonding and microfluidic device characterization

Initial Budget Estimate and Source of Funds:
Total expected costs: $928.
Source of Funds: Dr. Stoeber and Granting Agencies.
For details, see proposal.
<table>
<thead>
<tr>
<th>Project Scope - Activities in Scope</th>
<th>Activities out of Scope</th>
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</thead>
<tbody>
<tr>
<td>o Design, fabrication,</td>
<td>o None.</td>
</tr>
<tr>
<td>characterization of all</td>
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<tr>
<td>microfluidic devices supporting</td>
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<tr>
<td>portable systems.</td>
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<table>
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<th>Assumptions and Anticipated Risks (for detailed analysis, see proposal)</th>
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<tr>
<td>o Peptide bonding and parylene bonding does not result in a leak-free</td>
</tr>
<tr>
<td>bonding</td>
</tr>
<tr>
<td>o Eric Lagally Unavailable</td>
</tr>
<tr>
<td>o PIV does not work</td>
</tr>
<tr>
<td>o Peristaltic valve kills cells</td>
</tr>
<tr>
<td>o Water jet cutter is taken out of service</td>
</tr>
<tr>
<td>o Heating is not consistent across heater</td>
</tr>
<tr>
<td>o Cannot get thermally activated micropump to work.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Stakeholders:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Project Sponsor: Boris Stoeber</td>
</tr>
<tr>
<td>Team Members: Philip Edgcumbe, Jun Wei Fu, Jan Vohradsky</td>
</tr>
<tr>
<td>Project Lab: Jon Nakane, Chris Waltham, Bernhard Zender</td>
</tr>
<tr>
<td>Advisor: Vahid Bazargan</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Communication and Meeting Schedule:</th>
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</thead>
<tbody>
<tr>
<td>The Team Members will provide written weekly reports on Monday.</td>
</tr>
<tr>
<td>The Team Members will meet with the Project Sponsor weekly on</td>
</tr>
<tr>
<td>Tuesday. Other communication with stakeholders and resources by</td>
</tr>
<tr>
<td>email and telephone as needed.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Other Issues:</th>
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<tbody>
<tr>
<td>None.</td>
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<table>
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<th>Project Charter Sign-Off</th>
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<table>
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<th>Name/Date</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Project Sponsor</td>
<td>Project Sponsor</td>
</tr>
<tr>
<td>Name/Date</td>
<td>Name/Date</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td>Team Member 1</td>
<td>Team Member 2</td>
</tr>
</tbody>
</table>
6.4 Appendix D - Team Member's Time Contributions

Presented in Figure 19 is a graph of the team's time contributions to the project per week. The cumulative hours contributed is compared to the required 10 hours per week per team member.

Figure 19 – Team member’s time contributions vs. time
6.9 Appendix I - Series 4 Lithography Masks
6.10 Appendix J - Cleanroom Wafer Fabrication Record
<table>
<thead>
<tr>
<th>Date</th>
<th>Time at start of process (24:00)</th>
<th>HMDS spin (length of time @ speed-rpm)</th>
<th>Time that wafer is left on spinner after HMDS spin (min)</th>
<th>SPR 220-7.0 spin (length of time @ speed-rpm)</th>
<th>Time that wafer is left on spinner after SPR spin</th>
<th>Soft bake step 1: length of time (min) @ temperature (celcius)</th>
<th>Soft bake step 2: length of time @ temperature (celcius)</th>
<th>Time cooling before exposure (min)</th>
<th>Feature height (microns)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>23/06</td>
<td>8:00:00</td>
<td>35 @ 3500</td>
<td>2</td>
<td>500</td>
<td>2 @ 90</td>
<td>3 @ 115</td>
<td>10 yes</td>
<td>75</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>24/06</td>
<td>7:30:00</td>
<td>35 @ 3500</td>
<td>2</td>
<td>500</td>
<td>2 @ 90</td>
<td>3 @ 115</td>
<td>10 yes</td>
<td>60</td>
<td>1:30:00</td>
<td>15:00:00</td>
</tr>
<tr>
<td>25/06</td>
<td>7:30:00</td>
<td>35 @ 3500</td>
<td>2</td>
<td>500</td>
<td>2 @ 90</td>
<td>3 @ 115</td>
<td>10 yes</td>
<td>60</td>
<td>2:30:00</td>
<td>11:00:00</td>
</tr>
</tbody>
</table>

I fabricated this design with Patrick and we both used the same program… yet his features were 8 microns. The only difference is that I developed my wafer four hours after exposure. I didn't think this would be an issue because Sarah had told me that some groups wait an entire day before doing development.

Maybe bubbles are from heat shock? Should we perhaps transfer the wafers to a hot plate that is at 60 degrees before transferring it to a 90 degree hot plate? Put aluminum foil on hot plate to try to get rid of the bubbles. We will try this in the future.

I did not think that it would be an issue that the glass slide sucked very strongly to the table today. This is likely because Patrick had cleaned the glass plate before we used it today.

Exposure time (sec)

Developme

time (time when glass slide or wafer removed from developer)

Feature height (microns) Comments Questions
I mixed up H23 and H29 because...
<table>
<thead>
<tr>
<th>Channel</th>
<th>Heater Type</th>
<th>Treatment</th>
<th>Time</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wide A</td>
<td>Heater A</td>
<td>30/07 12:30:00</td>
<td>50</td>
<td>yes 1:50:00</td>
</tr>
<tr>
<td>Snake C</td>
<td>Heater C</td>
<td>30/07 15:00:00</td>
<td>25</td>
<td>yes 25 10s 2</td>
</tr>
<tr>
<td>Wide A</td>
<td>Heater A</td>
<td>30/07 15:00:00</td>
<td>53</td>
<td>yes 53 10s 2</td>
</tr>
<tr>
<td>Snake B</td>
<td>Heater B</td>
<td>30/07 15:00:00</td>
<td>40</td>
<td>yes 40 10s 2</td>
</tr>
<tr>
<td>Snake C</td>
<td>Heater C</td>
<td>30/07 15:00:00</td>
<td>60</td>
<td>yes 60 10s 2</td>
</tr>
</tbody>
</table>

- **Notes:**
  - Started development 40 mins after exposure.
  - Bake time questionable - a bit over 2 min.
  - Spun on HMDS at 4000rpm by mistake.
  - Waited 35 mins after exposure before development.
  - Bottom left heater = good.
  - Centre left heater = ok.
  - Centre right heater = good.
  - Top three heaters are all merged together.
  - Only waited 10 secs before removing glass slide from mask aligner.
  - 40 mins between exposure and development.
  - Top three are good, bottom three are merged... passable but far from ideal.

- **additional notes:**
  - 3 to 7 evaporated gold onto this but I placed it upside down in the evaporator so the gold was evaporated onto the wrong side so I can't use it.
turned on UV light at 4:20pm

had long spr 220-

16/8/2

10@500

them less than ideal- 40 mins sitting out between exposure and development

spr 220-

7.0

16/8/2

010 17:30:00

40 @ 3500 1

40@4000 1 2@115 60 min yes 50 1:40:00 - all good

h60

7.0

010 17:30:00

40 @ 3500 1

40@4000 1 2@115 60 min yes 50 2:00:00

h61

spr 220-

10@500

10@500

- bottom three are good

7.0

010 17:30:00

40 @ 3500 1

40@4000 1 2@115 60 min yes 50 1:40:00

- one of three of top one are good

spr 220-

H4S

H4L

two specs after spinning.- - generally good after development.  Slightly concern that the glass and photoresists seem quite dirt
g

can see that vacuum is on though 65 40sec 6min

can't open the spr container and spent a lot of time cleaning the lid.- when I took wafer out of spinner after 55 20 secs 2 min

two spots after spinning

ditto above

ditto above

ditto above

ditto above

ditto above

spr 220-

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115 20

three more spots appeared during heating- saw a spec of dust on it when we were about to do exposure... not sure where the dust came from.

H63

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115 10+

no -

no -

ditto above 55 20 secs 2 min

ght off the wafer also chan
ged while it w
can see vacuum is on (main vacuum display) 65 1 7

5 secs

19

5 secs

17

5 secs

7.0 ##### 17:10:00

5 secs

18

5 secs

7.0 ##### 16:10:00

.  Lots of 5 micron specs.  Onl

heat
ey.  11 has a piece of spec big enough that makes me think it might not form a full heater.

H64

7.0 ##### 21:00:00

5 secs

19

5 secs

7.0 ##### 17:10:00

5 secs

18

5 secs

7.0 ##### 16:10:00

-23 min after exposure

H65

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115

ditto above 55 7um

Didn't take the slide off the paper towl on the oven for 2 minutes.  Bak
H66

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115 10+

no -

no -

H67

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115

no -

no -

ditto above 55 2

no -

H68

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115

H72

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115

H71

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115

H70

7.0 ##### 21:00:00

40 @ 3500 1

40@4000 1 2@115
As per Ben Mustin's suggestion I cleaned the wafer with acetone, IPA, and methanol, blow dried it, and let it sit for 45 minutes on the hot plate at 200°C and then cool down at room temp at 200°C. Ben suggested that the wafer came from a box of wafers in the bioMEMS lab. The box was no longer sealed and left out in the bioMEMS lab, so there are specs of dust on the wafer. We used the cleanroom microscope and found that most of the wafers did not have this problem. However, wafers 17, 18, and 19 did not have this problem and they come from the box of wafers that were kept in the cleanroom.

The wafer was then exposed to UV light at various times and with different exposure times. The exposure times and conditions were as follows:

- 5 secs @ 500 rpm
- 40 secs @ 1500 rpm

These conditions were repeated for each wafer. After exposure, the wafers were immersed in developer solutions at various times and with different exposure times. The developer times and conditions were as follows:

- 5 secs @ 500 rpm
- 40 secs @ 3500 rpm

These conditions were repeated for each wafer. After development, the wafers were examined with a microscope and the results were recorded. The results showed that the design looks very good, and the heater trace is sharp. However, there was one part with slightly rough edges in wafer 1. This is strange, and it is suspected that the marker may have dissolving. I should use a fine tip marker to minimize this problem.

Overall, the process was successful, and the glass slides were clean. I found that it is slightly overdeveloped, and I worry that the gold traces may be affected. I decided to go ahead with the development, but I stopped developing a little before the last of the developer came off. The features are much more sharp, and the gold traces will be spaced farther apart. I stopped developing a little before the last of the developer came off.

Generally good except for wafers 4, 5, 8, and 12. All the bad ones have dirt on them. Maybe this is because I didn't use a new developer. I re-used the developer bath from H81, H82 - batch 2. After development, wafers 17, 18, and 19 were all good. This was interesting because I did not use permanent marker. I suspect that permanent marker contributes to a dirty glass slide. Surprisingly, wafers 1-6 look much better, whereas wafers 7-12 look really good. It's hard to understand how one half of a slide can be over-developed. - batch 3

After washing the wafers with water, it was found that the glass slide was cleanest by far. This is interesting because I did not use permanent marker. I suspect that permanent marker contributes to a dirty glass slide. - batch 4.
6.11 Appendix K - Glass Slide Record
<table>
<thead>
<tr>
<th>Heater #</th>
<th>PDMS design</th>
<th>Date that glass and PDMS bonded together</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Single (S) or double (D) layer PDMS - if D include the thin layer spin speed</td>
<td>Date that glass and PDMS bonded together</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Post-bonding treatment. Cooking time (CT) and Weight (W) on device</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pressure where leakage occurs across gold (psi)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Comments</td>
</tr>
</tbody>
</table>

**20/7/2010**
- **SD-D**
  - **D**
  - **20/7/2010**
  - **B**
  - **- very dirty surface => long strands of hair.**
  - **- right valve has channel collapse and the membrane bonding to the glass slides.**
  - **- cannot use this chip.**
  - **- I suspect that there are two slide 9s**

**22/7/2010**
- **CT: 48 hours in the oven**
- **W: Cleanroom phone**
- **C: Vahid recipe. Glass aligning (GA), wait time (WT)**
  - **#1: No GA, no WT**
  - **#2: No GA, yes WT**
  - **#3: Yes GA, yes WT**
  - **#4: Yes GA, yes WT**

**10/8/2010**
- **Vahid's old heater**
- **Two good snake designs cut into two pieces each**
- **D - 2700rpm**

**17/8/2010**
- **Leakage and very dirty wit lots of hair on it. When I was hole punching the device I leaned over the exposed devices and maybe that's when some hair fell down?**

**10-12psi 25+**

**12/8/2010**
- **H46**
- **3A (ok to bad allignment) and 3B (good allignment)**
- **D - 2700rpm**

**17/8/2010**
- **W: ?**

**10-12psi 25+**

**20/8/2010**
- **W: two scrap aluminum blocks in bioMEMS**
- **CT: 4 hours in bioMEMS oven**
- **B - direct placement (between 30secs and 1 min) - 45 seconds of plasma**
  - **- Looks dirty. This is frustrating because I used tape to remove dust from PDMS and I used compressed air to remove dust from the heaters before plasma treatment so I expected the bond to be clean.**

**8/10/2010**
- **H41**
- **Snake Design C 2000**
- **D- 3000rpm**

**23/8/2010**
- **H62**
- **3A and 3B**
- **D- 3000rpm**

<table>
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</table>
33

- Collapse everywhere. I could only get flow in one of the three valves because there was so much collapse in the pressure inlet.

- Tested to 20 psi leakage in top left area.
- Tested and saw no leakage. Never saw leakage at 25 psi. - Difficult to tell where leakage occurs.
- Pressure where fluid left the channel to another area. - Only collapse that occurred after 24 hr in oven was in the coolant channel.

CT: 24 hours in oven
W: 8 lb
metal block

- Tested to 20 psi leakage in top left area.
- Tested and saw no leakage. Never saw leakage at 25 psi. - Difficult to tell where leakage occurs.
- Pressure where fluid left the channel to another area. - Only collapse that occurred after 24 hr in oven was in the coolant channel.

CT: 24 hours in oven
W: 0 lb
bioMEMS 65C

- Tested to 20 psi leakage in top left area.
- Tested and saw no leakage. Never saw leakage at 25 psi. - Difficult to tell where leakage occurs.
- Pressure where fluid left the channel to another area. - Only collapse that occurred after 24 hr in oven was in the coolant channel.

CT: 24 hours in oven
W: 8 lb
In the experiment, the PDMS design was tested on a blank glass slide with different layers and treatments. The multi-layer H52 and 3C and 3D devices were tested for leakage at various pressures. The following results were observed:

- **Slide 3A:**
  - Complete collapse on both PDMS chips at 30 psi
  - None of the solution can leak out from the part of the pressure output which has been cut too small so the pressure outlets are not fully enclosed
  - Flawless, it has no collapse and holds pressure to 30 psi

- **Slide 3B:**
  - This slide has two multi-layer chips on it. 3B and 3D. 3D is a single layer.
  - Tested to 10 psi
  - No collapse anywhere
  - I confirmed that the valves are working
  - I applied 10 psi at the junction test and saw no leakage
  - I saw no leakage in the coolant channel and it did not leak

- **Slide 3C:**
  - No collapse anywhere
  - There is some collapse in the coolant channel and in the column areas near the collapsed pressure

- **Slide 3D:**
  - Complete collapse on both PDMS chips. At 30 psi, could not push any solution into the chip.
  - This was a very thin multi-layer device. I tested it at 5 psi and however, there was lots of leakage. It flowed out of the channels.
  - Complete collapse on 11/12 pressure inlets so that at 30 psi we could not push any solution into the chip.

- **Slide 3E:**
  - Glass slide cracked when I was placing weight on top of it so I could only test two of the three valves.
  - Complete collapse on all pressure inlets across gold (psi)

- **Slide 3F:**
  - Pressure where leakage occurs
  - After I saw 5 psi failure on the gold heater I set up a T junction to try and see how much pressure the PDMS to glass boundary could take
  - After 24 hours in CT I confirmed that the valves are working
  - I applied 10 psi at the junction test and saw no leakage
  - I saw no leakage in the coolant channel and it did not leak
  - I saw no leakage in the coolant channel and it did not leak

Overall, the PDMS design performed well with no collapse and held pressure to 30 psi. Further testing and analysis are needed to determine the optimum conditions for bonding and adhesion fail.
Jan.5: Thin membrane was ruined so I poured 50 grams of PDMS and let it cure at 65°C.
- Put in 65°C oven at 14:50.
- heater 5 hairy but flow is good.
- 9/12 toast
- 3/6 blocked and the heaters are bad.
- On Dec 30 Philip spun on a thin layer of PDMS at 8000 rpm.

**Cooking time (CT)**: 26hrs
**Weight (W)**: 4lb
- No finger pressing.

Philip writes on Jan. 9:
- Only info I could find was in work record
- plasmatreated to clean inside of PECVD like Jonas
- see video M94
- note: i did not plasma treat to clean inside of PECVD like jonas
- see video M94
- Vahid used this device 2 years ago for his research. We want to see if we can recreate the same results that he got.

In one of the chips, there are two double-layer and one double-layer chips.

- Height of gold traces is 400nm
- For the rest of the process, Vahid's recipe: power: 30, pressure: 500, temp: 25, O2: 100, time: 15secs
- No failure until 12 psi. Minor collapse but generally happy. Valve 4 exhibited significant collapse.

There is some collapse but I am able to pump water through the channel. The middle channel seems OK when I am running water through it.

Philip writes on Jan. 9:
- acetone, ipa and water clean
- spun at 3500 repm 8 min after mixing
- air clean only
- spun at 7000 rpm 5 min after mixing
- air clean only
- spun at 3500 rpm 1 min after mixing
- acetone, ethyl alcohol, water, hot plate clean
- spun at 3500 repm 8 min after mixing
- acetone, ipa and water clean
- 115C for 5 mins before plasma bonding
- PDMS for this chip was only cured at 65°C for 4 hours before plasma bonding
- no significant collapse.

This might be because the PDMS only had a four hour bake time.

Philip writes on Jan. 9:
- acetone, ipa and water clean and 115C for 5 mins before plasma bonding
- pdms for this chip was cured at 65C for 24+ hours before plasma bonding
- no failure up to 20psi.

On Dec 30 Philip spun on a thin layer of PDMS at 8000 rpm.
- Valve 4 exhibited significant collapse.
- The 7-9 valves on this chip appeared to be viable, but the PDMS collapsed such that fluid could not be pumped through.

Valves 1-3 were not properly bonded, causing a widespread rupture when fluid was pushed in.

Philip writes on Jan. 9:
- Pdms prepared on Nov. 21 and only had 4 hours in oven for curing on Nov. 21st. It set out at room temp from Nov. 21st to Nov. 24th.
- extensive channel collapse. Could barely push any fluid through.

This probably had to be trimmed.

Plasmatreated to clean inside of PECVD like Jonas
- see video M94
- Vahid gave us
- no high pressure leakage, although no apparent leakage occurs between PDMS and glass (psi)
- pressure where bonding results.
- valve closes around 12 psi. Minor collapse but generally happy. Valve 4 exhibited significant collapse.

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<td>1) heater 1 jumpered 2) heater 3 has broken trace 3) instant rupture when 5 psi applied to 7,8 and 9. 1,2,3,10,11 and 12 were ruptured from surgery to clear pads. 4) pushing from side 5, I could not get it through the area 5 zig zags at 15 psi. Attempts to massage the chip failed. The device ruptured at 20 psi. 5) pushing from side 4, the flow is sluggish and needs &gt;10 psi. There is leakage to the fluidic layer such that the fluid prefers to fill the fluidic channel instead of entering the area 1 zig zags.</td>
</tr>
<tr>
<td>1/7/2011</td>
<td>1) Valve 2-5 leaked at 10 psi 2) Valve 1-4 leaked at 5 psi after I lifted the needle out of the PDMS and that caused the PDMS to come up. The needle was really stuck in so I don’t think this means there was bad PDMS-glass bonding. 3) Jan. 5: I cleaned heater 72 with acetone, IPA, and water and 115°C for 5 minutes and poured 10:1 PDMS (3:30 mixing and 2:30 defoaming) onto it and spun it at 8000 rpm and placed it in the oven. Tomorrow it will be ready to bond to the last two of the multi-layer PDMS chips I made today.</td>
</tr>
<tr>
<td>1/5/2011</td>
<td><strong>H4W</strong> 1) 4A (covers valve 7-12) and 4D (covers valve 1-6). 2) Double layer, spin speed of 2000 rpm, 80 minutes before alignment. 3) January 5: I cleaned heater 72 with acetone, IPA, and water and 115°C for 5 minutes and poured 10:1 PDMS (3:30 mixing and 2:30 defoaming) onto it and spun it at 8000 rpm and placed it in the oven. Tomorrow it will be ready to bond to the last two of the multi-layer PDMS chips I made today. 4) Plasma cleaning treatment. BioMEMS (B) or cleanroom (C). 5) December 17: I cleaned heater 72 with acetone, IPA, and water and 115°C for 5 minutes and poured 10:1 PDMS (3:30 mixing and 2:30 defoaming) onto it and spun it at 8000 rpm and placed it in the oven. Tomorrow it will be ready to bond to the last two of the multi-layer PDMS chips I made today. 6) Heater 1 jumpered 7) heater 3 has broken trace 8) instant rupture when 5 psi applied to 7,8 and 9. 1,2,3,10,11 and 12 were ruptured from surgery to clear pads. 9) pushing fluid from side 5, I could not get it through the area 5 zig zags at 15 psi. Attempts to massage the chip failed. The device ruptured at 20 psi. 10) pushing from side 4, the flow is sluggish and needs &gt;10 psi. There is leakage to the fluidic layer such that the fluid prefers to fill the fluidic channel instead of entering the area 1 zig zags.</td>
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<td>1/3/2011</td>
<td><strong>H4W</strong> 1) 4A (covers valve 7-12) and 4D (covers valve 1-6). 2) Double layer, spin speed of 2000 rpm, 80 minutes before alignment. 3) January 5: I cleaned heater 72 with acetone, IPA, and water and 115°C for 5 minutes and poured 10:1 PDMS (3:30 mixing and 2:30 defoaming) onto it and spun it at 8000 rpm and placed it in the oven. Tomorrow it will be ready to bond to the last two of the multi-layer PDMS chips I made today. 4) Plasma cleaning treatment. BioMEMS (B) or cleanroom (C). 5) December 17: I cleaned heater 72 with acetone, IPA, and water and 115°C for 5 minutes and poured 10:1 PDMS (3:30 mixing and 2:30 defoaming) onto it and spun it at 8000 rpm and placed it in the oven. Tomorrow it will be ready to bond to the last two of the multi-layer PDMS chips I made today. 6) Heater 1 jumpered 7) heater 3 has broken trace 8) instant rupture when 5 psi applied to 7,8 and 9. 1,2,3,10,11 and 12 were ruptured from surgery to clear pads. 9) pushing fluid from side 5, I could not get it through the area 5 zig zags at 15 psi. Attempts to massage the chip failed. The device ruptured at 20 psi. 10) pushing from side 4, the flow is sluggish and needs &gt;10 psi. There is leakage to the fluidic layer such that the fluid prefers to fill the fluidic channel instead of entering the area 1 zig zags.</td>
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6.12 Appendix L - Macro Assembly