

PMMA MICROFLUIDICS TECHNOLOGY: DEVELOPMENT AND CHARACTERIZATION

by

Mona Rahbar
B.Eng. Azad University of Qazvin 2004

THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF

MASTER OF APPLIED SCIENCE

In the
School of Engineering Science

© Mona Rahbar 2010
SIMON FRASER UNIVERSITY
Summer 2010

All rights reserved. However, in accordance with the Copyright Act of Canada, this work may be reproduced, without authorization, under the conditions for Fair Dealing. Therefore, limited reproduction of this work for the purposes of private study, research, criticism, review and news reporting is likely to be in accordance with the law, particularly if cited appropriately.

APPROVAL

Name: Mona Rahbar
Degree: Master of Applied Science
Title of Thesis: PMMA Microfluidics Technology: Development and Characterization

Examining Committee:

Chair:

Dr. Jie Liang, P. Eng.
Assistant Professor, School of Engineering Science

Dr. Ash Parameswaran, P. Eng.
Senior Supervisor
Professor, School of Engineering Science

Dr. Behraad Bahreyni, P. Eng.
Supervisor
Assistant Professor, School of Engineering Science

Dr. Carlo Menon, P. Eng.
Internal Examiner
Assistant Professor, School of Engineering Science

Date Defended/Approved: August 23, 2010



SIMON FRASER UNIVERSITY
LIBRARY

Declaration of Partial Copyright Licence

The author, whose copyright is declared on the title page of this work, has granted to Simon Fraser University the right to lend this thesis, project or extended essay to users of the Simon Fraser University Library, and to make partial or single copies only for such users or in response to a request from the library of any other university, or other educational institution, on its own behalf or for one of its users.

The author has further granted permission to Simon Fraser University to keep or make a digital copy for use in its circulating collection (currently available to the public at the "Institutional Repository" link of the SFU Library website <www.lib.sfu.ca> at: <<http://ir.lib.sfu.ca/handle/1892/112>>) and, without changing the content, to translate the thesis/project or extended essays, if technically possible, to any medium or format for the purpose of preservation of the digital work.

The author has further agreed that permission for multiple copying of this work for scholarly purposes may be granted by either the author or the Dean of Graduate Studies.

It is understood that copying or publication of this work for financial gain shall not be allowed without the author's written permission.

Permission for public performance, or limited permission for private scholarly use, of any multimedia materials forming part of this work, may have been granted by the author. This information may be found on the separately catalogued multimedia material and in the signed Partial Copyright Licence.

While licensing SFU to permit the above uses, the author retains copyright in the thesis, project or extended essays, including the right to change the work for subsequent purposes, including editing and publishing the work in whole or in part, and licensing other parties, as the author may desire.

The original Partial Copyright Licence attesting to these terms, and signed by this author, may be found in the original bound copy of this work, retained in the Simon Fraser University Archive.

Simon Fraser University Library
Burnaby, BC, Canada

Abstract

This thesis describes the work performed on a novel micro-fabrication method for patterning poly(methyl methacrylate) (PMMA) in an economical way. Although PMMA can be exposed using a variety of exposure sources, deep-UV radiation source at 254 nm is of interest because it is relatively inexpensive. This thesis shows that micro-structures as deep as 100 μ m can be created in commercial grade PMMA using a deep-UV light source. Additionally, to improve the fabrication process, different types of commercial PMMA and developers were characterized.

The other major focus of this thesis is the development of a new bonding method for PMMA microfluidics which combines the elements of thermally-assisted solvent bonding and microwave bonding method to produce an economical, simple and high yield bonding process for the sealing of PMMA microfluidics. This new process takes only a few minutes and produces a high strength bond.

Keywords: poly(methyl methacrylate) (PMMA) microfluidics, deep-UV (DUV) lithography, PMMA bonding, Microwave bonding, Thermally-assisted solvent bonding

Acknowledgements

I would like to offer my sincere appreciation to my supervisor Dr. M. Parameswaran for his incredible guidance and support. His patience and support has made this work possible. Additional thanks to my thesis committee members Dr. B. Bahreyni and Dr. C. Menon for their contributions. I am also deeply grateful to Dr. D. Sameoto with whom I had the pleasure of working with throughout my graduate studies. His valuable feedback and continuous support enabled me to develop an understanding of the subject.

Warm thanks go to Dr. I. Foulds and Michael Sjoerdsma for reading and commenting on my work. Special thanks to Nicholas Doyle for all his help on revisions of this manuscript and his kind support. I also would like to say thank you to Dr. R. W. Johnstone, Marius Haiducu, Sae-Won Lee, Manu Venkataram, and Suman Chhina for their support that helped bring the successful completion of this work. Thanks are extended to Bill Woods for his assistance with my cleanroom work and Dr. C. Menon for access to the laser cutter. I was delighted to interact with all my colleagues in the lab because of whom my graduate experience has been one that I will cherish forever.

My deepest gratitude goes to my parents, Fakhri and Ali, and my brothers Eric and Alireza to whom this work is dedicated. They have been a constant source of love, support, concern and strength throughout my life.

Finally, I would like to thank the NSERC for their financial support.

Table of Contents

Approval	ii
Abstract	iii
Acknowledgements	iv
List of Figures	viii
List of Tables.....	x
List of Abbreviations	xi
Chapter 1: Introduction	1
1.1 Thesis Objectives	3
Chapter 2: Micromachining of Poly(methyl methacrylate).....	5
2.1 Fabrication Process.....	8
2.1.1 <i>Process Steps</i>	9
Chapter 3: Characterizing and Improving the Fabrication Process	13
3.1 Characterization of Different Types of PMMA.....	13
3.2 Testing the Performance of Different Types of Developers	20
3.3 Collimation of Deep Ultraviolet Light Source	23
Chapter 4: Investigation of Bonding Methods Previously Attempted in the Literature	26
4.1 Thermal Fusion Bonding.....	27

4.2	Solvent-assisted Bonding	30
4.3	Microwave Bonding	32
Chapter 5: Microwave-induced, Thermal-assisted Solvent Bonding		36
5.1	Fabrication Method and Materials	36
5.2	Bonding Process	40
5.3	Testing and Results	46
5.4	Applications	59
Chapter 6: Future Works and Conclusion		65
6.1	Future Work.....	65
6.2	Contributions	67
6.3	Conclusion.....	67
Appendix A: Material Properties of Optix® PMMA Purchased from GoodFellow		69
Appendix B: Material Properties of CQ PMMA Purchased from GoodFellow		70
Appendix C: Material Properties of UVT OP-4 PMMA.....		71
Appendix D: Surface Roughness of Optix® PMMA Soaked in Ethanol for 1 Minute		72
Appendix E: Surface Roughness of Optix® PMMA Soaked in Methanol for 1 Minute		73

Appendix F: Surface Roughness of Optix® PMMA Soaked in IPA for 1 Minute	74
Appendix G: Surface Roughness of Optix® PMMA	75
Appendix H: Percentage of Bonded Surface Area for Microwave Bonding Process	76
References.....	77

List of Figures

Figure 1 : Chemical structure of PMMA	7
Figure 2: Process steps of DUV patterning of PMMA	10
Figure 3: Etch depth of UVT sample using IPA: H ₂ O developer.....	14
Figure 4: Etch depth of Cyro sample using IPA:H ₂ O developer	14
Figure 5: Etch depth of Optix® sample using IPA:H ₂ O developer.....	15
Figure 6: Etch depth of CQ sample using IPA:H ₂ O developer	15
Figure 7: Comparison of etch depth of the four types of PMMA. The samples were exposed to DUV light for 5 hours which is equivalent to 72 J/cm ² and were developed in IPA:water developer.	16
Figure 8: Comparison of etch depth of UVT sample using SU-8 developer, solid lines, and IPA:water developer, dashed lines.	22
Figure 9: Comparison of etch depth of Optix® sample using SU-8 developer, solid lines, and IPA:water developer, dashed lines.....	22
Figure 10: Illustration of the comparison between the side walls created by a) non- collimated irradiation DUV source and b) the semi-collimated irradiation source. (This schematic does not represent the actual geometry of the channels and is only for illustrating the effect of collimation on the negative angle of the sidewalls.)	24
Figure 11: A comparison between the etch rate of two 30 hours exposed Optix® samples using non-collimated and semi-collimated DUV light source and developed in IPA:H ₂ O.	25
Figure 12: A vice developed in house for pressing the two PMMA halves together during the thermal bonding procedure. Scale bar represents 2.5 cm.	28
Figure 13: Comparing the transparency of thermally bonded PMMA chip (left) to non-bonded patterned PMMA sample (right). Scale bar represents 2.5 cm.	29
Figure 14: Sample microfluidics designs for laser cut channels. Scale bar represents 2.5 cm.	37
Figure 15: Cross-section of the micro-channels created by Laser cutter (left) and DUV exposure (right). Scale bars represent 200 μm.	38
Figure 16: PMMA channel ready for microwave heating with metal clips and input tube attached. Scale bar represents 2.5 cm.	41
Figure 17: PMMA microfluidics unit with the indentations on the outer surface caused by trapped solvent between the plastic and metal clip. Scale bar represents 2.5 cm.	42
Figure 18: PMMA and water in the commercial microwave oven for the bonding	43

Figure 19: Transparent plastic clamps for testing the effect of direct microwave heating of solvent between PMMA surfaces. Scale bar represents 2.5 cm.	45
Figure 20: Optical images of cross-sections of channels created by DUV exposure (top) and laser cutter (bottom), non-bonded, (a and c), and bonded, (b and d). Scale bars represent 200 μm .	47
Figure 21: Percentage of bonded surface area for microfluidics chips using different solvents.	48
Figure 22: Close top view of a bonded laser cut channel filled with black ink showing the effectiveness of the elevated lip near the edge of the channel in sealing. Scale bar represents 200 μm .	49
Figure 23: a) Instron MicroTester model 5848, b) bonded sample used for shear stress test (the area enclosed inside the dashed square is the bonded surface area). Scale bar represents 2.5 cm.	50
Figure 24: Optical image of the PMMA samples exposed to 216 J/cm^2 of DUV after 1 min soaking in a) methanol, b) ethanol, c) IPA. Bottom half of each square was covered during the exposure; i.e. only the top halves were exposed to DUV light. Scale bar represents 2.5 cm.	53
Figure 25: Average surface roughness of PMMA for exposed (216 J/cm^2) and unexposed areas after 1 minute of soaking in different alcohols.	54
Figure 26: Hairline cracks on the edge of a micro-channel filled with opaque ink. Scale bar represents 400 μm .	55
Figure 27: Two PMMA channels created using CO ₂ laser cutter and bonded using ethanol. Top sample was annealed for duration of 12 hours and showed no hairline crack around the cut edges. Bottom sample did not undergo the annealing step and had hairline cracks near all the laser cut edges. Scale bar represents 2.5 cm.	57
Figure 28: Optical images of PEG-filled channels before (a) and immediately after (b) applications of microwave power. After 90 seconds of Microwave heating, all areas of PEG have melted. Scale bar represents 2.5 cm.	59
Figure 29: Microfluidics unit fabricated by laser ablation and microwave bonding for pinch injection test. Scale bar represents 2.5 cm.	61
Figure 30: Different steps of pinch-injection process and optical images of fluorescent dye corresponding to each step. Scale bars represent 0.25 cm	62
Figure 31: Different layers of a dual-layer microfluidics chip design for testing the reliability of the bonding process for microfluidics structures consist of more than one layer of structures. The channels are fabricated using CO ₂ Laser cutter. Scale bar represents 2.5 cm	63
Figure 32: Bonded dual-layer microfluidics chip. Scale bar represents 2.5 cm	64

List of Tables

Table 1: Material properties of PMMA. [10].....	6
Table 2: Some characteristics for four different types of PMMA	17
Table 3: Heating characteristics of metals influenced by microwaves [45]	35
Table 4: Results of the shear stress test for bonding strength	51
Table 5: Material properties of Optix® PMMA.....	69
Table 6: Material Properties of CQ PMMA.....	70
Table 7: Material properties of UVT PMMA	71
Table 8: Surface roughness of Optix® PMMA soaked in ethanol	72
Table 9: Surface roughness of Optix® PMMA soaked in methanol	73
Table 10: Surface roughness of Optix® PMMA soaked in IPA	74
Table 11: Surface roughness of Optix® PMMA without being soaked in any alcohol	75
Table 12: Percentage of bonded surface area	76

List of Abbreviations

Acronym	Description
B	Buffer
BW	Buffer Waste
CQ	Clinical Quality
DI	De-ionized
DRR	Dissolution Rate Ratio
DUV	Deep Ultraviolet
Exp	Exposure
IPA	Isopropyl Alcohol
LOC	Lab-on-a Chip
MIBK	Methyl isobutyl ketone
PC	Polycarbonate
PDMS	Poly(dimethylsiloxane)
PEG	Poly(ethylene glycol)
PET	Poly(ethylene terephthalate)
PGMEA	propylene glycol monomethyl ether acetate
PMMA	Poly(methyl methacrylate)
PPI	Pulses Per Inch
PS	Poly(styrene)
RF	Radio Frequency
S	Sample
SW	Sample waste
UV	Ultraviolet
UVT	Ultraviolet Transparent
μ -TAS	Micro-total Analysis System

Chapter 1: Introduction

Microfluidics is a technology that adapts conventional chemical and biological analysis to micrometer scales. Since the development of the first microfluidics devices in late 1970's, the field has rapidly expanded [1, 2], improving the performance of chemical and biological analysis. Microfluidics systems have several advantages, which includes a reduction in sample volume, a lowering of analysis cost and chemical waste [3, 4], and an increase in precision and accuracy [5]. Disposability, portability, and the potential for system automation are the other main advantages of miniaturization [5, 6]. Life sciences, analytical chemistry and biology, and the study of fluid transport are the main application fields of microfluidics [1, 5, 7].

Early microfluidics devices were mainly fabricated using silicon or glass because the fabrication processes, photolithography and etching technologies, for these two materials were well established in the microelectronics industry [2, 4, 6, 8]. However, each of these two materials has disadvantages that limit their usability in certain applications. Silicon is opaque in visible and UV light, and hence cannot be used where UV detection or visual monitoring is required.

Although glass is transparent, it is amorphous, which makes the etching of vertical side walls very difficult [2]. In addition, micro-devices fabricated with silicon or glass need to be manufactured in a cleanroom environment, requiring considerable processing effort and technology. Therefore, silicon or glass based microfluidics systems can be prohibitively expensive for many applications. Hence, nowadays, plastics and polymers are the materials of choice due to their much lower fabrication costs and complexity [5, 9].

Poly(methyl methacrylate) (PMMA) is a polymer commonly used for the fabrication of microfluidics chips. PMMA is a thermoplastic material and has optical transparency in visible light wavelengths [10-12]. It has been long used in LIGA¹ fabrication processes as a positive photoresist [14]. Because PMMA is also used widely in many different products as a replacement for glass, it is available in a variety of grades. Common trade names for PMMA are Acrylite, Plexiglass®, Acrylic glass, or simply Acrylic.

The work presented in this thesis characterizes new techniques for patterning PMMA as well as a bonding technique for PMMA based microfluidics chips in a cost effective and relatively quick and easy manner, which will allow the wider adaptation of microfluidics technology in exciting new applications.

¹ LIGA is a German acronym for "Lithographie, Galvanoformung, Abformung." [13] N. T. Nguyen and S. T. Wereley, *Integrated microsystem series, second edition*: Boston: Artech House, 2006.

1.1 Thesis Objectives

The work herein consists of three major parts. In the first part, a novel technology for creating of micro-structures in PMMA using DUV irradiation source at 254 nm wavelength is described and characterized. 240 nm was considered to be the upper limit of the effectiveness, but recent studies showed that 254 nm wavelength can also be used as a effective irradiation source for patterning a thin layer of PMMA [9]. This part successfully demonstrates how Plexiglas® may be accurately patterned with an inexpensive exposure system for use in microfluidics. Irradiating PMMA with 254 nm wavelength light causes main polymer chain scission, which lowers the average molecular weight of PMMA. This decrease in average molecular weight increases the polymer's solubility. In this approach, the PMMA sample is exposed through a photo-mask deposited on top surface of the samples and then photolithographically patterned. After the exposure, the exposed areas are removed in a solvent, creating microstructures on the PMMA surface.

In the second part, the focus is on the optimization of recently developed technology. Different types of commercial PMMA, with different mechanical and chemical characteristics, have been characterized in order to determine the best suited type of PMMA for different possible applications. The effect of various solvents for developing exposed PMMA has been investigated. Furthermore, to improve the newly developed technology, the effect of the collimating the light source has been investigated. This collimation of the light helps to increase the aspect ratio of the micro-scaled structures and hence the quality of the micro-

channels. As a result of this investigation, different combinations of commercial PMMA and solvents and light source collimation can be used to achieve a microfluidics unit that is desirable for a particular application based on the required specifications.

The third part, in Chapters 4 and 5, describes the different bonding methods for PMMA substrates that were investigated, and a new and reliable bonding method for encapsulating micro-channels fabricated on PMMA substrates is described. In this method, a poor solvent for PMMA is applied to the interface of the two pieces of PMMA the two parts are then clamped together using small metal clips. Excess solvent is drawn out of the channels and reservoirs prior to microwave heating, reducing channel deformation and clogging, and making the process less venerable to overheating. The clamped sample is then placed in a commercial microwave oven to heat the metal clips. This heat increases the temperature of the solvent in the interface, which dissolves and bonds the two pieces of PMMA. This new bonding method is cost effective, high yield and fast, resulting in high bond strength.

Chapter 2: Micromachining of Poly(methyl methacrylate)

Microfluidics is the key element of the micro-total-analysis-system (μ -TAS) and lab-on-a chip (LOC) technology. The recent interest in microfluidics is due to not only its ability to increase portability of devices, but also to its ability to increase the device speed and performance [8]. These devices are designed in such a way that they can process volumes of fluid on the order of microlitres down to picolitres, while the devices themselves have dimensions ranging from millimetres to micrometres. Using standard photolithographic techniques well developed in the microelectronic industry [2, 4, 6, 8], microfluidics devices originally have been fabricated on silicon (Si) and glass. The microfabrication processes to create microfluidics structures on Si and glass (e.g., Pyrex glass, soda-lime glass, fused silica) substrates are complicated and costly. In contrast, polymer based microfluidics chips that are recently replacing Si and glass based chips can be fabricated using much simpler and more cost efficient methods. In addition, polymers are available with a wide variety of chemical and mechanical

properties that can be selected to meet the requirements of each particular application. The different kinds of polymers that have been used for microfabrication of microfluidics devices include polydimethylsiloxane (PDMS), poly(methyl methacrylate) (PMMA), poly(carbonate) (PC), poly(styrene) (PS) and poly(ethylene terephthalate) (PET) [2, 15]. Some of the properties of these materials are provided in Table 1.

Table 1: Material properties of PMMA. [10]

Polymer	T _g (°C) ¹	T _m (°C) ²	Water absorption (%)	Solvent resistance	Optical transparency	
					Visible	UV
PMMA	100-122	250-260	0.3-0.6	Good	Excellent	Good ²
PC	145-148	260-270	0.12-0.34	Good	Excellent	Poor
PS	92-100	240-260	0.02-0.15	Poor	Excellent	Poor
PET	69-78	248-260	0.1-0.3	Excellent	Good	Good

¹T_g: glass transition temperature.

²T_m: melting temperature.

²The optical transparency at UV wavelength depends highly on the grade of the PMMA. There are pure PMMA grades with excellent UV transparency available as well as grades that have UV absorbing additives added to them.

Among all the above mentioned polymers, PMMA is one of the most promising for wide use in microfluidics fabrication, since it is biocompatible, has excellent optical properties [16], low cost, widely available in a variety of physical and chemical properties, and is one of the least hydrophobic polymers among the commonly used polymers [2]. The schematic of PMMA chemical structure is illustrated in Figure 1.

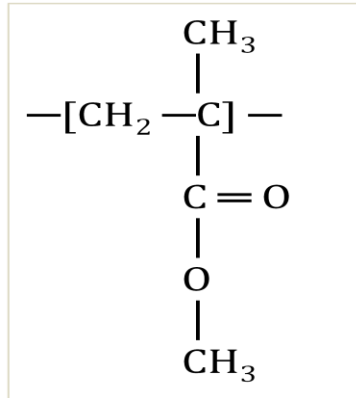


Figure 1 : Chemical structure of PMMA

Irradiated PMMA undergoes change in its chemical structure. This phenomenon is commonly known as degradation [12]. As a result of the irradiation, the molecular bonds in the polymer main chain and pendent methyl ester groups break down, reducing the average molecular weight of the polymer, and hence, increasing its solubility in certain solvents [12, 16-19]. The number of backbone scissions of polymer chain is proportional to the energy dose of the radiation source. The change in the PMMA molecular weight due to irradiation can be calculated using the following formula:

$$\frac{1}{M_{n,r}} = \frac{1}{M_{n,0}} + 1.04 \times 10^{-4} G(s)r \quad \text{Equation 2.1}$$

$M_{n,0}$ is the initial molecular weight and $M_{n,r}$ is the average molecular weight after exposure to a dose of r kGy of high energy radiation. The variable $G(s)$, often about 1 or 2, is the number of scissions per 100 ev absorbed [19].

Different radiation sources may be used to expose PMMA, such as gamma-ray [20], x-ray [21], electron [18, 22], proton [18, 23] or ion beam [24, 25], and deep-ultraviolet (DUV) with wavelengths shorter than 240 nm [9, 18, 26-28]. In most of the previously reported works on PMMA, light sources with wavelengths less than 240 nm are used, as the peak absorption of PMMA is between 190 and 260 nm [18].

Recent studies show that DUV with 254 nm wavelength can also be used to expose thin layer of PMMA [17, 26]. This wavelength is relatively inexpensive to produce using a Stratalinker 2400 exposure source (manufactured by Stratagene) [26, 28]. Nevertheless, the depth that can be exposed using DUV is only in the order of hundreds of microns, compared to the millimeters possible with X-ray. However, these depths are sufficient for creating microchannel systems that can be used in many biological or chemical applications. PMMA's desirable mechanical and optical properties [29] together with the low cost of the patterning process were the decisive factors in this project for choosing PMMA as the structural material for fabrication of microfluidics systems described later in this thesis.

2.1 Fabrication Process

A commercial grade PMMA, Plaskolite's Optix® (some of the Optix® PMMA properties is listed in Appendix A), is used as the structural material for the microfluidics devices described in this thesis. This PMMA was used because of its low cost and desirable mechanical and optical properties. The average

molecular weight for this brand of PMMA was measured to be $M_w=55.7$ kDa. A Stratalinker 2400 UV crosslinker acts as a DUV light source to expose the PMMA. The exposure chamber of Stratalinker 2400 UV crosslinker has five 15 W low pressure mercury vapor lamps and a total exposure area of $1,560 \text{ cm}^2$ inside a chamber. It provides non-collimated radiation with a nominal power of 4 mW/cm^2 and a spectrum with the strongest peak at 254 nm. Two modes of operation are available: energy mode, in which the exposure dose in $\mu\text{J/cm}^2$ is set, and time mode, in which the desired exposure time in minutes is specified. The maximum exposure dose in energy mode is too low to affect the PMMA significantly. As a result, all exposures are done in the time mode. Fifteen hours of exposure was calculated to provide 216 J/cm^2 dose of DUV radiation.

2.1.1 Process Steps

PMMA substrates can be obtained in sheets with different thicknesses and can be cut into desirable shapes and sizes. In this project, the substrates, Plaskolite's Optix[®], are originally cut into 30 cm x 30 cm by the supplier; these pieces were cut into 75 mm x 75 mm and 25 mm x 75 mm for ease of manipulation using a CO₂ laser cutter. The fabrication process steps are shown in the

Figure 2.

Chapter 2: Micromachining of Poly(methyl methacrylate)

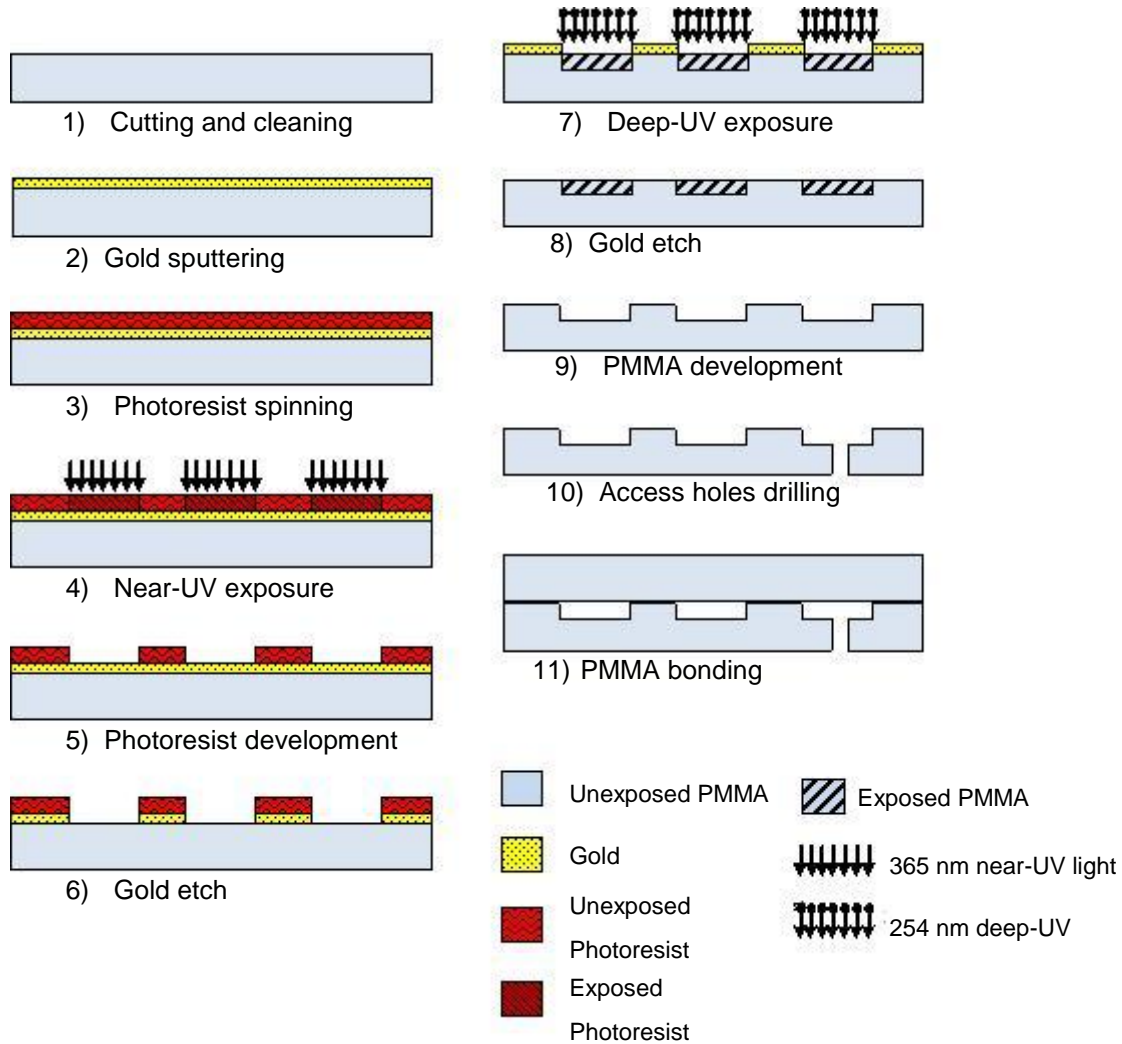


Figure 2: Process steps of DUV patterning of PMMA

After being cut, the PMMA samples were cleaned using de-ionized (DI) water and mild hand soap. They were then immersed in methanol solution for 10 minutes to remove any surface contamination. Following this step, the samples were rinsed with DI water and blow dried with N₂ gas. In the next step, a 100 nm thick layer of gold was sputtered on the PMMA surface at 80 Watts using a

Corona Vacuum System. After being photolithographically patterned, this gold layer acts as a photo-mask for the PMMA during the subsequent DUV exposure.

The gold layer was patterned using photolithography. A positive resist layer, Shipley 1813 photoresist, was spun on the gold layer at 4000 rpm for duration of 30 seconds. The photoresist was then soft baked at 75 °C for 10 minutes on a hotplate to remove the majority of the solvent. Because PMMA softening temperature is about 100 °C [29], the photoresist coated PMMA needs to be softbaked at a low enough temperature to make sure that the substrate will not deform and warp plastically. To ensure that no deformation will occur during the softbake, 75 °C was chosen for this step. During the trials, it was found that increasing the baking temperature up to 90 °C will not permanently deform the plastic. At temperatures between 75 °C to 90 °C the substrates were occasionally found to be slightly warped immediately following the bake, but after cooling down to room temperature the substrates relaxed back to their original flatness. However, temperatures above 90 °C are not recommended for the softbake, because they can lead to permanent deformation of the substrate which will effect the bonding quality.

To pattern this photoresist layer, the sample was exposed through a contact mask using an i-line source and developed in MF-319 developer. A gold etch was completed using TFA gold etchant from Transene Company, Inc. After the gold layer was patterned, the photoresist was removed using a 60 second UV exposure followed by development in MF-319. The sample was then exposed to

DUV in the Stratalinker 2400. The exposure duration depends on the desired geometry and depth of the structure. The detailed characterization of the relationship between exposure time and development depth will be discussed in Chapter 3. After DUV exposure, the gold layer was removed in TFA gold etchant and the sample was transferred to a bath containing the developer solution, a mixture of isopropyl alcohol (IPA) and DI water (IPA: H₂O, ratio of 7:3 by volume at 28 °C ± 2 °C). In order to increase the dissolution rate, a magnetic stirring rod set to 250 rpm was added to the etching system to provide a constant high agitation. In the next step, the developed samples were quenched in an ultrasonic IPA bath at room temperature (approximately 18 °C) for 15 seconds, rinsed in fresh IPA, followed by a DI water rinse and drying with N₂ gas. The ultrasonic step is needed to prevent the re-deposition of the partially dissolved PMMA, which would otherwise appear as white residue on the surface of the samples. However, the ultrasonic step can affect the small structures by physically breaking them; therefore, to prevent damaging small features, the duration of this ultrasonic bath should not be longer than 20 seconds. Finally, the input/output access ports were drilled and the channels were sealed by bonding the patterned piece of PMMA to another blank piece of PMMA.

This process can fabricate structures as small as 5 μm with a 2:1 aspect ratio.

Chapter 3: Characterizing and Improving the Fabrication Process

3.1 Characterization of Different Types of PMMA

As mentioned in the previous chapter, one advantage of choosing polymers as the structural material is that these polymers are usually available with a variety of chemical and mechanical properties. PMMA is available in many different brand names such as Plexiglass®, Lucite, Perspex and Acrylic. In order to choose the most appropriate type of PMMA for specific microfluidics applications, four PMMA brands were selected and evaluated.

The four types of PMMA selected for evaluation were: Optix® ($M_w=55.7$ kDa), Cyro ($M_w=110.5$ kDa), additive free clinical quality (CQ) grade ($M_w=628.0$ kDa), and UV-Transparent OP-4 (UVT) PMMA ($M_w=305.8$ kDa) (see appendices A, B, and C for material properties). These PMMA samples were developed by following the fabrication steps described in Section 2.1.1. The results of the characterization are shown in Figures 3 through 7.

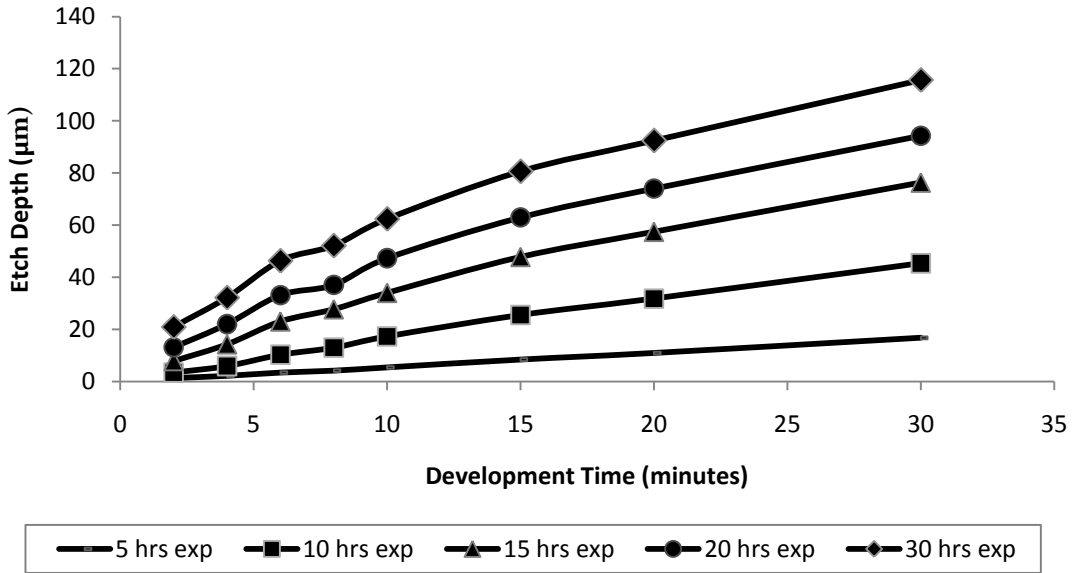


Figure 3: Etch depth of UVT sample using IPA: H₂O developer

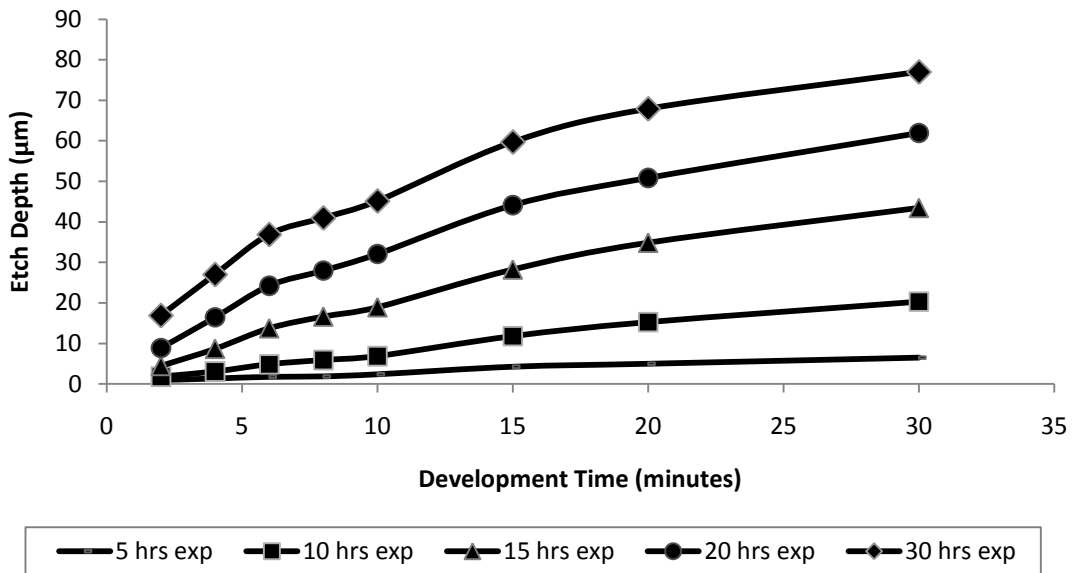


Figure 4: Etch depth of Cyro sample using IPA:H₂O developer

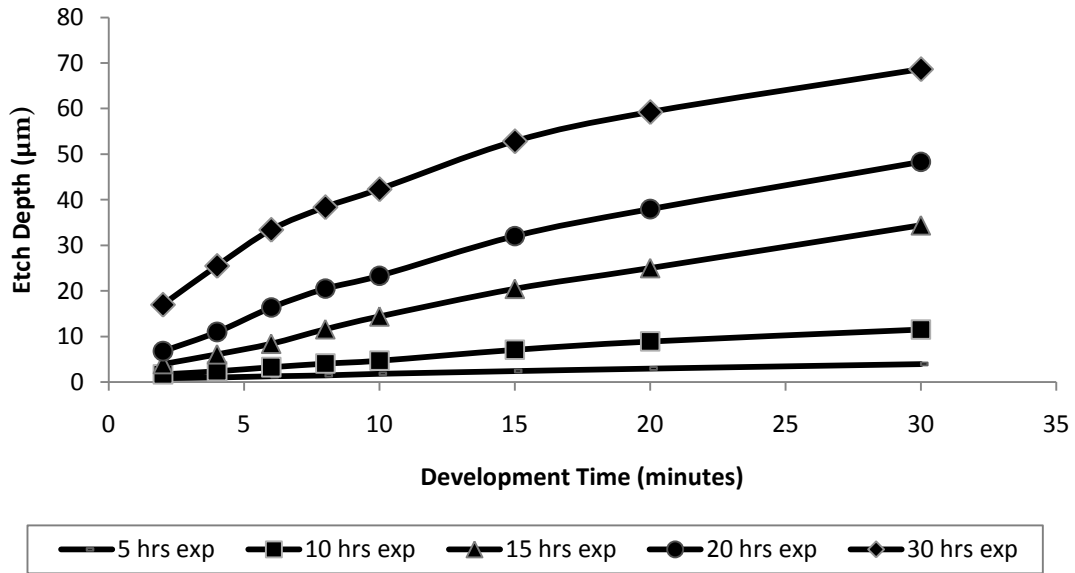


Figure 5: Etch depth of Optix® sample using IPA:H₂O developer

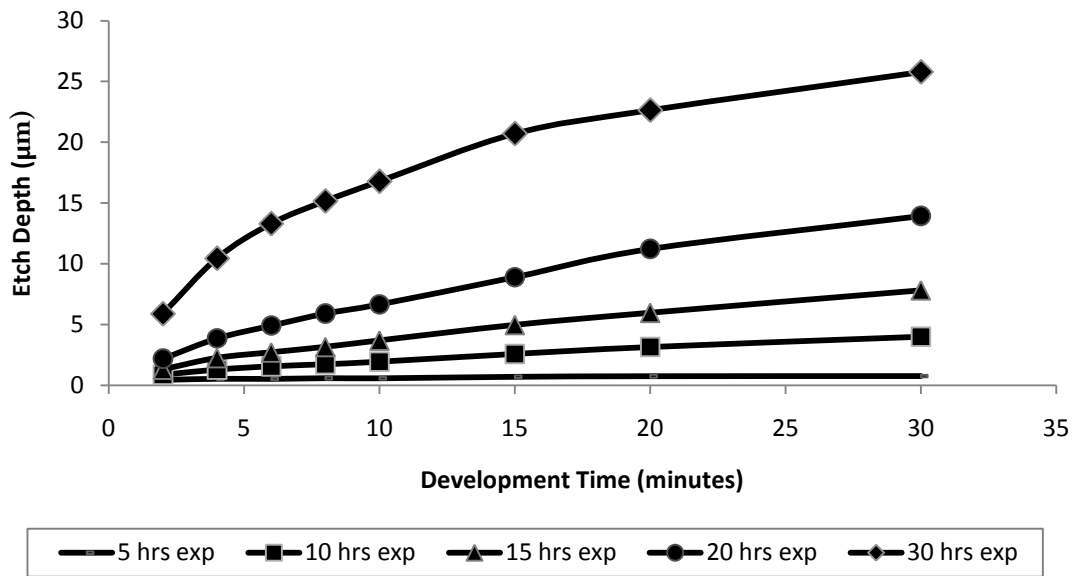


Figure 6: Etch depth of CQ sample using IPA:H₂O developer

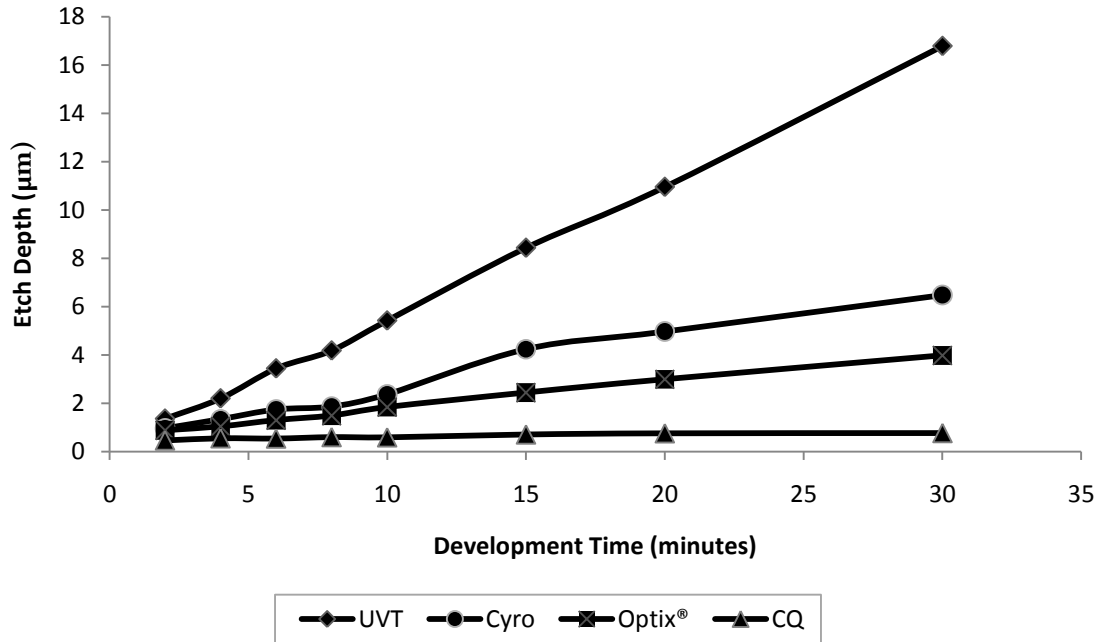


Figure 7: Comparison of etch depth of the four types of PMMA. The samples were exposed to DUV light for 5 hours which is equivalent to 72 J/cm^2 and were developed in IPA:water developer.

One of the important parameter for the fabrication process is the dissolution rate ratio (DRR): the rate at which features will be etched into the material with a given DUV exposure and etch time. DRR is defined as:

$$DRR = \frac{R_{exposed}}{R_{unexposed}} \quad \text{Equation 3.1}$$

$R_{unexposed}$ is the dissolution rate of the unexposed PMMA and $R_{exposed}$ is the dissolution rate of exposed PMMA. The dissolution rate for a given solvent can be calculated using:

$$R = R_0 + \frac{\beta}{M_f^a} \quad \text{Equation 3.2}$$

R_0 , α and β are empirical constants and M_f is the fragmented molecular weight given by:

$$M_f = \frac{M_n}{1 + \frac{g \epsilon M_n}{\rho A_0}} \quad \text{Equation 3.3}$$

In which, ρ is the resist density, A_0 is Avogadro's number and g is an efficiency factor [30].

On the other hand, considering the dissolution rate to be the slope of the etch depth versus the development time curves, it can be seen in Figure 7 that the UVT has the highest dissolution rate (i.e., its curves have the largest slope). As a result, for the equal dosages of exposure, deeper structures can be etched in a shorter period of time using UVT. Optix® and Cyro with rather similar dissolution rates are in second and third places, respectively. Finally, CQ has the lowest dissolution rate. Some of the properties of these aforementioned PMMA types are summarized in Table 2.

Table 2: Some characteristics for four different types of PMMA

Type of PMMA	cost	Anti-yellowing additives	UV-transparent	Dissolution rate	Density of the cracks as a result of development	Thickness (mm)
UVT	Medium	Yes	Yes	High	Medium	2.5
Cyro	Low	Yes	No	Medium	High	2.5
Optix®	Low	Yes	No	Medium	Medium	4.0
CQ	High	No	Yes	Low	Low	2.5

From the data in Table 2, it can be concluded that the CQ and Cyro are not desirable material for microfluidics applications due to the following reasons:

CQ:

CQ grade PMMA has a very low dissolution rate. It takes too long to fabricate a deep enough micro-structure. For 30 hours of exposure (432 J/cm^2 of DUV radiation) structures of only approximately $25 \mu\text{m}$ depth can be etched after 30 minutes of development. In addition, CQ grade PMMA is more expensive than the other types of PMMA which is not desirable for the fabrication of disposable microfluidics systems. Finally, during the exposure the CQ grade PMMA substrate's colour changes from clear to dark yellow. This change in colour due to the lack of anti-yellowing agents can be problematic, especially for applications where optical monitoring or detection of the fluid sample is required.

Cyro:

Very high density of deep cracks appeared in all of the exposed areas during the development of Cyro PMMA. The depths of these cracks were measured to be as deep or even, in some areas, deeper than the depth of the etched structure itself. These cracks can severely affect the transparency of the fluidic unit. In addition, such large scale random cracks will make it impossible to accurately determine the volume of the liquid being manipulated and act as large traps that can affect the liquid manipulation and, hence, the accuracy of the results.

Chapter 3: Characterizing and Improving the Fabrication Process

The other two types of PMMA, UVT and Optix®, were determined to be suitable for use in different microfluidics applications for the following reasons:

UVT:

- UV transparency
- Relatively low cost
- Very high dissolution rate.

Optix®:

- Very low cost
- Relatively good dissolution rate

As a result, it was determined that UVT is the best structural material out of those evaluated for the applications where UV transparency is required for the optical detection or analysis. Optix® is a good substitute for UVT in applications where UV transparency is not required, and, instead, low fabrication cost is more desirable.

One of the important problems that were noticed during the measurements was a relatively large difference in the data obtained from different locations of each sample at any given time. This inconsistency implies that power within the irradiation box varies significantly over short distances; a noticeable variation even across an individual 75 mm x 75 mm PMMA sample. In order to address this problem, a turntable was added to the test setup where the samples were mounted on top of the turntable during the exposure time in order to ensure that all the samples were receiving the same dosage of DUV energy.

3.2 Testing the Performance of Different Types of Developers

After choosing the right structural material, another set of experiments was conducted in order to determine the most appropriate developer. The developer must be chosen in such way that the solution is an ineffective solvent for unexposed PMMA but, at the same time, effectively removes the low molecular weight, exposed PMMA molecules. This solution is often a binary solvent mixture which typically consist of a solvent and a non-solvent solution [31, 32]. The most commonly used developers for PMMA mentioned in the literature are a 1:3 mixture of methyl isobutyl ketone (MIBK) and isopropanol (IPA) [30-33], G-G Developer (a mixture of 15 vol% water, 60 vol% butoxy-ethanol, 20 vol% tetrahydro-oxazine and 5 vol% aminoethanol) [34, 35], and 7:3 mixture of IPA and water [31-33]. In all of the aforementioned experiments in this thesis, the solvent that was used to remove the irradiated areas of the PMMA substrates was IPA based (a mixture of IPA:H₂O at 7:3 ratio). The 7:3, IPA:water mixture is the preferred developer compared to the G-G developer and the MIBK:IPA mixture because the G-G developer contains highly toxic chemicals [16] and the MIBK:IPA can cause swelling of PMMA, which prevents the fabrication of high aspect-ratio structures [32]. The IPA:water developer, on the other hand, provides optimal sensitivity and minimizes the swelling effect [32].

During this work, it had been found that propylene glycol monomethyl ether acetate (PGMEA), the SU-8 developer, can also be used to develop structures in PMMA.

Chapter 3: Characterizing and Improving the Fabrication Process

The main advantage of using SU-8 developer instead of an IPA-based developer is that the development using SU-8 developer is possible at room temperature, approximately 18 °C. In comparison, developing exposed PMMA using an IPA-based developer is only possible at an elevated temperature, about 28 °C, which needs to be maintained constant during the development.

Therefore, using SU-8 developed can help to decrease the fabrication time significantly while eliminating the need for hot IPA:H₂O bath preparation step. However, the disadvantage of using SU-8 developer is that it is less selective than IPA:water mixture, which means that SU-8 developer etches the non-exposed parts of PMMA in a higher rate compared to the IPA-based developer.

The two figures bellow, Figure 8 and Figure 9, compare the development depth profile of Optix® and UVT in an SU-8 developer and an IPA:water mixture. As shown in most part of the curves, the dissolution rate (the slope of the curves) for the UVT sample, Figure 8, is higher than the Optix®, Figure 9. These results are consistent with the results obtained in section 3.1. The small exceptions in some data points are most likely due to the power variation inside the irradiation source, as discussed in section 3.1.

Chapter 3: Characterizing and Improving the Fabrication Process

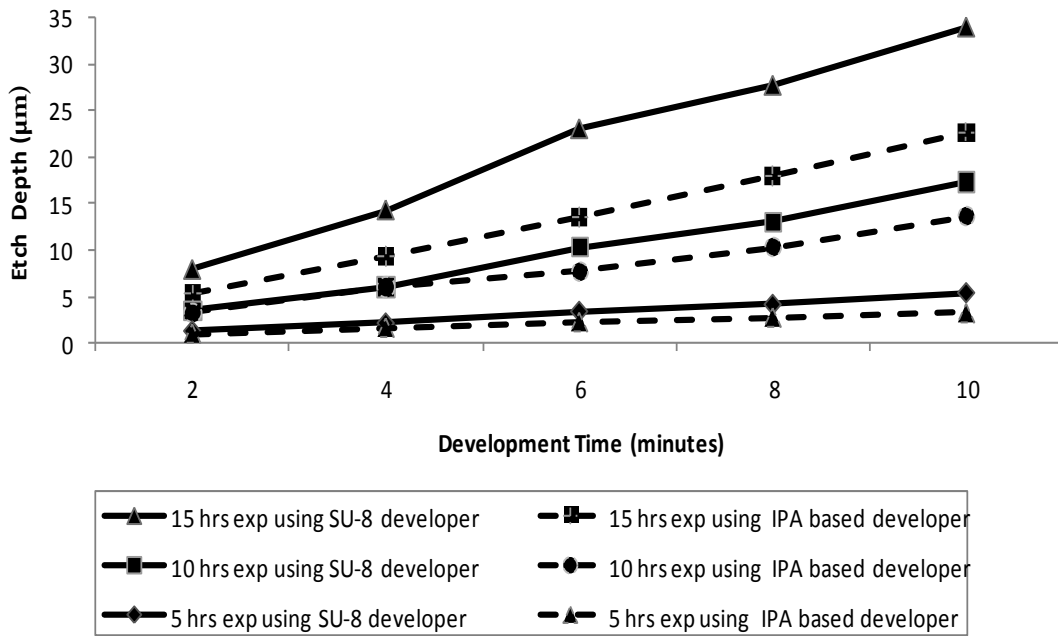


Figure 8: Comparison of etch depth of UVT sample using SU-8 developer, solid lines, and IPA:water developer, dashed lines.

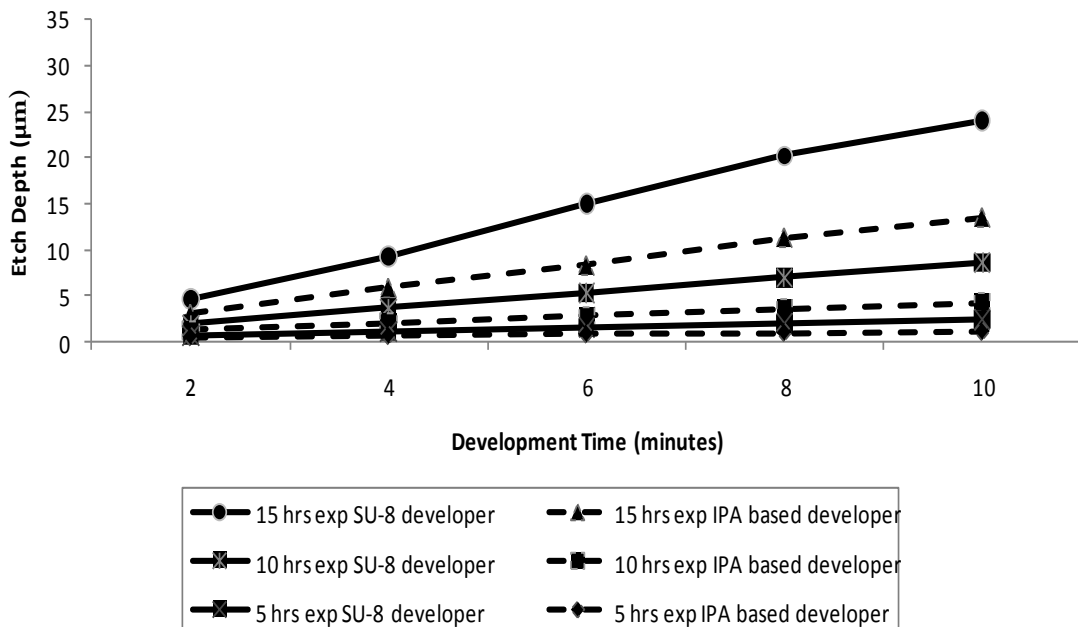


Figure 9: Comparison of etch depth of Optix® sample using SU-8 developer, solid lines, and IPA:water developer, dashed lines.

A comparison between Figure 8 and Figure 9 also shows that SU-8 developer provides a much higher dissolution rate for a given substrate compared to an IPA-based developer. These results imply that the fastest dissolution rate can be achieved by a combination of UVT as the structural material and SU-8 developer as the developer. As a result, SU-8 Developer is a good candidate as a substitute for the IPA-based developers because it not only shortens the fabrication time by elimination some preparation steps, but provides a much higher dissolution rate which also shortens the fabrication time. However, further investigation is needed to fully characterize this new developer for PMMA and determine its performance related to small features.

3.3 Collimation of Deep Ultraviolet Light Source

One of the limitations of the aforementioned fabrication process is that structures can have a maximum 2:1 aspect ratio, defined as the ratio between the depth and the width of the structures. This relatively low aspect ratio, 2:1, is partially caused by the significant negative sidewalls, which are attributed to the non-collimated nature of the DUV light source used in the process. The negative sidewalls caused by the non-collimated irradiation source causes severe undercut of the features. A schematic of the effect of non-collimated and semi-collimated irradiation source on the angle of the sidewalls of the fabricated structures is shown in Figure 10.

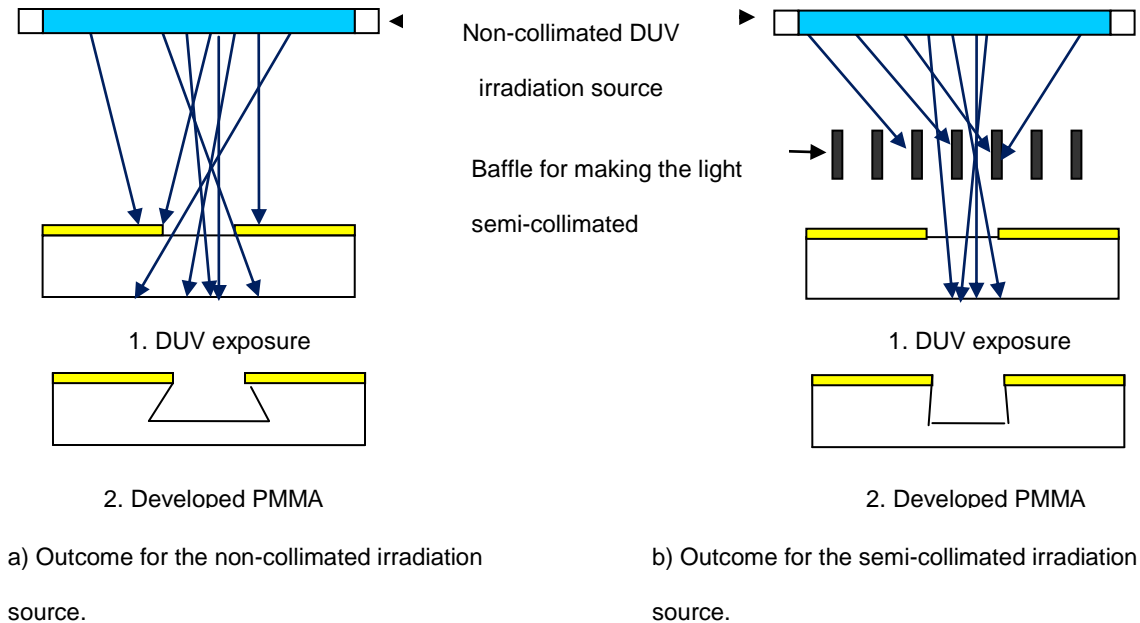


Figure 10: Illustration of the comparison between the side walls created by a) non- collimated irradiation DUV source and b) the semi-collimated irradiation source. (This schematic does not represent the actual geometry of the channels and is only for illustrating the effect of collimation on the negative angle of the sidewalls.)

One of the methods attempted to reduce the negative effect of the non-collimated light source on the aspect ratio was semi-collimating the light source. As shown in Figure 10, this is achieved by adding a baffle grid to the system between the samples and the mercury-vapor lamps to prevent light beams with sharp angles from reaching the PMMA surface. The added grid is made of 12.7 mm x 12.7 mm x 12.7 mm squares and is made out of opaque plastic. In order to eliminate the effect of shadow from the baffle on the samples and to ensure that all areas on the surface of the samples are receiving the same DUV radiation dosage, the samples need to be mounted on a turntable.

Chapter 3: Characterizing and Improving the Fabrication Process

Although samples exposed using a semi-collimated light source achieved a higher aspect ratio of 4:1, this method reduces the dissolution rate of the PMMA significantly due to a reduction in the amount of radiation energy received by the samples. Figure 11 clearly shows the effect of this collimation on the dissolution rate of PMMA. The slope of each curve shows the dissolution rate of its corresponding PMMA sample at any given time.

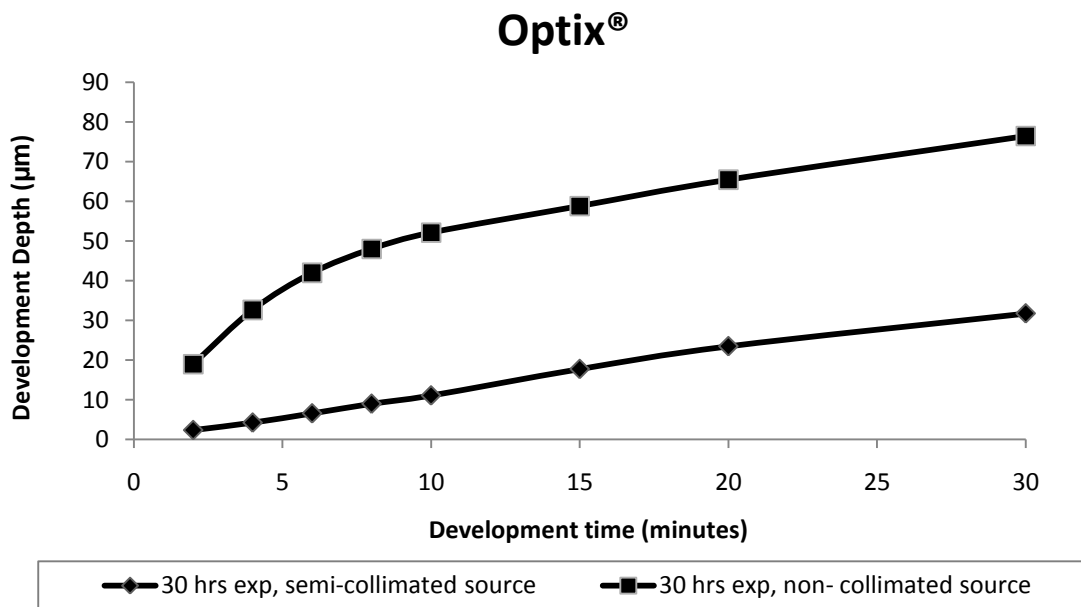


Figure 11: A comparison between the etch rate of two 30 hours exposed Optix® samples using non-collimated and semi-collimated DUV light source and developed in IPA:H₂O.

Chapter 4: Investigation of Bonding Methods Previously Attempted in the Literature

Following fabrication of the microfluidics substrate, a cover plate must be bonded to the substrate to complete the fluidic conduits. In the literature, many researchers have attempted various bonding methods: solvent-assisted bonding [10, 36, 37], thermal bonding [10, 38-40], lamination [41], PDMS intermediate layer [42], and microwave bonding [15, 43].

Currently, packaging is still a challenging issue in the fabrication of polymer based microfluidics devices because the bond must be strong enough to withstand the pressure of the fluid passing through it and parts must remain attached without delamination. In addition, the bonding cannot change or destroy the integrity of patterned microstructures. The work presented herein investigates three bonding methods: thermal bonding, solvent assisted bonding, and microwave bonding. Then, elements of these bonding methods are combined to create a new and more effective bonding method which is discussed in Chapter 5.

4.1 Thermal Fusion Bonding

In thermal fusion bonding, one or both of the substrates to be bonded are heated to a temperature close to the substrate's glass transition temperature (T_g). Then a precise amount of pressure is applied to ensure intimate contact between the substrates, while not pressing the substrate too hard and causing damage to the channels at the interface.

In the work described below, two thermal bonding procedures are used to seal the patterned channels, both methods are based on previous work described in literature [39, 44]. The two procedures are similar except in one the heating stem is done in an oven chamber at atmospheric pressure, whereas in the other, heating is done in a vacuum oven chamber at -85 KPa. The core of the process consists of three steps. In the first step, the two substrate halves were treated in a methanol bath for 10 minutes and dried using N_2 gas. In the second step, using a vice developed in-house and constructed of two thick aluminums pads, the two pieces were pressed against each other. The vice is shown in Figure 12.

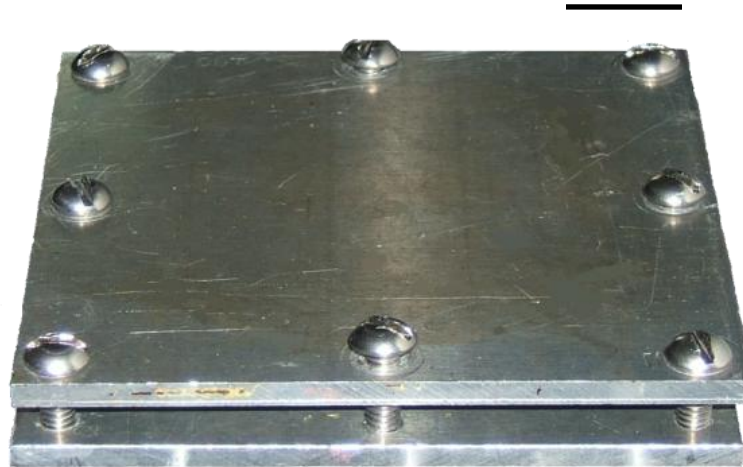


Figure 12: A vice developed in house for pressing the two PMMA halves together during the thermal bonding procedure. Scale bar represents 2.5 cm.

Finally, the setup is placed in a pre-heated oven at 90 °C for approximately 90 minutes. The ovens used for thermal bonding are the Sybron Thermolyne type 1900 for the method that required atmospheric pressure and the Forma Scientific Vacuum Oven, model 3237, for the method that required vacuum. Finally, the samples were slowly cooled down to room temperature for a period of at least 5 hours while still under pressure by the vice.

Further investigation showed that the slow cooling down step is the most crucial step because the elimination of this step results in thermal stress on the interface causing the bonding to fail, and the two halves will come apart as soon as the vice's pressure is removed. The methanol treatment does not serve a significant role in the bonding procedure and eliminating that step does not have a noticeable impact on the quality of the bonding.

Leakage testing of the thermally bonded PMMA samples was then done using Harvard syringe pump. The syringe pump was connected to the inlet hole using a polyethylen tube. The liquid flow through the channels was increased to 2 mL/min, the maximum flow rate provided by the syringe pump. At maximum flow rate the fluid pressure was measured to be 68 kPa and no leakage was observed during the test.

Figure 13 shows two 75 mm x 75 mm PMMA samples, a thermally bonded sample and a non-bonded sample.

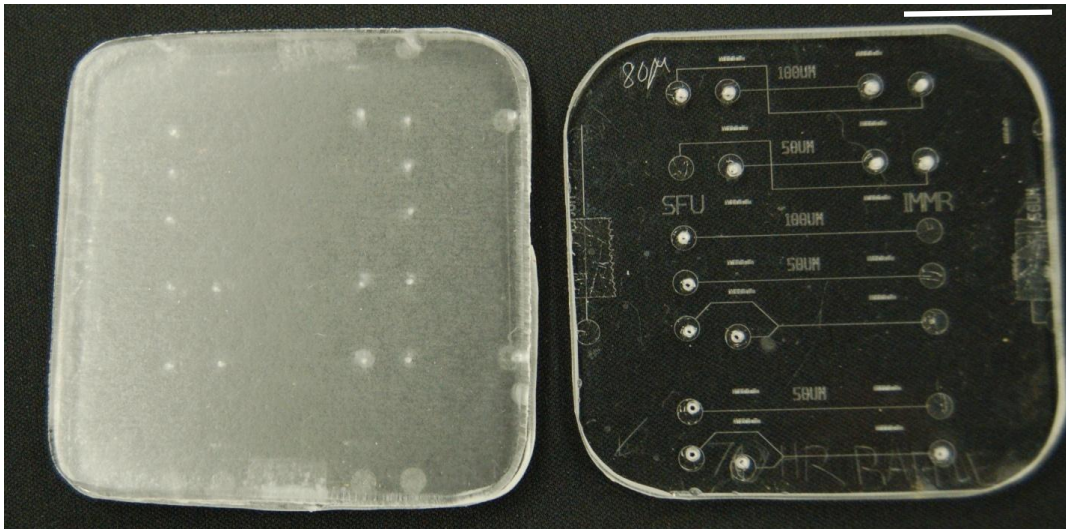


Figure 13: Comparing the transparency of thermally bonded PMMA chip (left) to non-bonded patterned PMMA sample (right). Scale bar represents 2.5 cm.

Evident in the above picture is the most important disadvantage of this bonding method: the resulting sample loses its optical transparency and clarity. This is due to the fact that the PMMA pieces were heated to a temperature near the PMMA T_g temperature, which is approximately 100°C. As a result, the polymer

softens slightly and small deformations occur in the outer surface of the PMMA, which decreases the quality of the final product. This change has an especially negative effect on devices that will be used for applications in which full transparency is required, such as optical detection or analysis. As such, chips bonded using this method cannot be used for these applications.

The other disadvantage of this method is distortion of the channels due to unoptimized temperature and pressure applied during the bonding process. To address this problem, relatively costly equipment, such as programmable hot press, commercial bonding system or a high throughput roller [10] are required to precisely control temperature and pressure throughout the entire area of the chip.

4.2 Solvent-assisted Bonding

The solvent-assisted bonding method is one of the most common bonding techniques that have been used on polymer substrates [11, 36, 37]. This technique is based on partially dissolving the bonding surfaces using a solvent to soften a thin layer of the surfaces and bringing the two surfaces in contact with each other. For bonding two PMMA substrates, different solvents, such as chlorocarbon based solvents (chloromethane, dichloromethane, and chloroform [37]) and acetone are used. However, clogging of the micro-channels frequently occurs while using these solvents due to the high solubility of PMMA in the solvents.

A common technique of solvent assisted bonding is described in general terms in this section. In this technique, the two substrates were pressed against

each other and a small amount (2 or 3 drops) of solvent is applied on the edges where the two halves are in contact. Capillary force draws the solvent into the space between the two polymeric substrates and bonds them together. However, in order to prevent the solvent from entering the channels, and hence destroying the micro-scale structures, the two PMMA substrates need to be in a very intimate contact, meaning that a high and uniform pressure is required to keep the two substrates together. To be able to apply such pressure, an embossing or a hot press machine is required, both of which are expensive pieces of equipment. Because one of the main goals of this project based on DUV irradiation is decreasing the fabrication costs by using simple and cost effective equipment, an alternative method was necessary. Therefore, instead of using an embossing machine, the in-house vice shown in Figure 12 was tested for its suitability for applying the required pressure. Unfortunately, the outcomes were not satisfying. Using this vice, it was not possible to apply a uniform pressure across the whole contact area, and the two substrates were only bonded in some small regions. In addition, a precise control of the applied pressure was required to prevent the solvent from entering into the channels, which was not achievable using the in-house vice.

Another important disadvantage of the solvent bonding method is that for samples larger than 25 mm x 25 mm it was nearly impossible to insert enough solvent into the interface that covers the entire surface area, because the solvent applied to the edge of clamped pieces bonds the edge area almost immediately,

which prevents the insertion of enough solvent into the interface, and the middle parts of large samples remain unbonded.

4.3 Microwave Bonding

Microwave-based polymer bonding is one promising technique for sealing PMMA based microfluidics units. This method uses microwave radiation and a thin film of metal or conductive polymers (e.g. polyaniline) to provide local heating at the bonding interface to fuse the two pieces together [15, 43].

Microwave heating originated from radio frequency (RF) heating research conducted during the 1940s which was introduced into industrial applications after World War II [45]. Currently, it is being widely used in chemical synthesis and the food industry [45-48], as well as for metal sintering [37, 49, 50]. The first commercial microwave ovens were used for food processing applications and were introduced by Raytheon in 1947. Shortly after that, researchers realized that there are many other potential applications for microwave heating for the processing of different materials [45]. Microwave heating can be applied to a vast variety of materials, such as polymers, metals, semiconductors, glass, and biomaterials [45, 47].

Packaging in the food industry is one of the main applications for using ferrite materials in microwave ovens. To provide high quality food that can resemble the ones prepared using traditional ovens, food companies created packaging that avoids directly heating the food [45]. Using susceptors in the packaging prevents the microwaves from heating the food directly. Instead, the

microwave energy produces enough eddy current in the susceptor or ferrite thin film material to heat it up. Hence, the microwave energy will be transferred into heat, which conducts or radiates the food surface. A review of different food packaging technique using metals is summarized by Datta *et al.* [47].

Microwave heating uses the interaction of high frequency microwave energy with microwave absorbing materials, such as metals or conductive polymers. Microwave heating is widely used for the curing of polymer composites and polymers. In general, polymers have very low thermal conductivity; therefore, curing of polymers using conventional heating methods requires a relatively long process time. The use of microwave heating allows for a rapid and uniform temperature increase during the curing of polymers. Thermosetting polymers absorb microwaves efficiently during polymerization, but this absorption decreases after the curing is completed. However, thermoplastic materials, such as PMMA, are fully polymerized materials and are difficult to heat using a microwave [45].

The power needed to increase the temperature of a mass, m kg, of material from an initial temperature, T_0 , to the final temperature, T , in the interval of t seconds is calculated by:

$$\text{Power absorbed} = \frac{mc_p(T-T_0)}{t} \quad \text{Equation 4.1}$$

The rate of heating, dT/dt , for the material influenced by an electric field is calculated using:

$$\frac{dT}{dt} = \frac{\text{Power absorbed}}{\rho C_p} = \frac{2\pi\epsilon_0\epsilon'' f E^2}{\rho C_p} \quad \text{Equation 4.2}$$

As evident in Equation 4.2, the rate of heating is directly proportional to the loss factor, ϵ'' , frequency, f , and the square of the applied electric field, E . Also, the rate of heating is inversely proportional to the density, ρ , and the material's heat capacity, C_p , where ϵ_0 is the permittivity of free space [15].

To date, most literature about the effects of microwaves on different substances focuses on food, ceramic, and dielectric materials. Although most metals cannot be easily heated by microwaves because they are reflective to the magnetic component of microwaves, ferromagnetic materials will interact with the magnetic field component of the RF radiation and heat up [51, 52]. The high degree of absorption leads to a significant rise in thermal energy of the ferrites. In 1988, Walkiewicz *et al.* showed that pure metal powders can couple effectively with microwaves and heat up to an elevated temperature above 700 °C in a few minutes [53].

The main concern about heating metals in microwave oven is the fear of fire and damaging the microwave due to arcing. However, the arcing only applies to bulk metals, so this phenomenon is not a concern while heating small metallic particles and powders [45].

Ferrites are particularly suitable for microwave heating applications because of their Curie temperature. Ferromagnetic materials heat up in microwave ovens only up to their Curie point, at which point they lose their magnetic moment and, therefore, magnetic loss. Ferrites with low-dielectric-loss

will only heat to a limited temperature which is determined by the material's Curie temperature, and the rate of heat loss to surroundings. Finally, the material and its surroundings will reach a thermal equilibrium in which the heat lost due to convection will be equal to the energy absorbed by magnetic loss, and the material's temperature no longer increases [52]. The heating characteristics of some metals are shown in Table 3.

Table 3: Heating characteristics of metals influenced by microwaves [45]

metal	Temp (°C)	Time (min)	Rate (°C/min)
Al	577	6.00	96.2
Cu	228	7.00	32.6
Fe	768	7.00	109.7
W	690	6.25	110.4

The main disadvantage of all the previously reported microwave bonding approaches is that, in those techniques, a low power customized microwave is required. The need for customized and rather costly equipment is not desired for the low cost fabrication process that is the goal of this thesis.

Chapter 5: Microwave-induced, Thermal-assisted Solvent Bonding

To achieve strong, reliable and relatively fast bonding, a combination of elements of several bonding methods was used. By combining features of solvent-assisted bonding, thermal fusion bonding, and microwave bonding, a new bonding procedure was introduced that is effective and does not require expensive and specialized equipment.

5.1 Fabrication Method and Materials

For the bonding trials, commercial grade PMMA (Optix® obtained from Plaskolite) was used, which was available in 30 cm x 30 cm sheets with a 1.6 mm thickness. To use the polymer sheet as a substrate, it was cut into 75 mm x 75 mm or 25 mm x 75 mm using a VersaLaser 3.60 CO₂ laser cutter with 80% power at 20% speed and 500 pulses per inch (PPI), of the laser cutter's arbitrary maximum setting [54].

The new bonding technique was tested on microfluidics chips created using two different methods in order to determine the most suitable fabrication-bonding combination. Bigger samples 75 cm x 75 cm, were exposed and developed using the novel DUV patterning technique described in Chapter 2 and Chapter 3. After developing the samples, through holes were drilled in the reservoirs using a drill press to act as inlet and outlet ports for the microfluidics structure. Finally, the samples were cut into desirable sizes before the bonding. The smaller, 25 cm x 75 cm, substrates were directly patterned using the VersaLaser CO₂ laser cutter. The settings of the laser cutter for these samples were 36% power, 30% speed and 500 PPI for the channels, and 80% power, 20% speed and 500 PPI for the access holes. As shown in Figure 14, several basic microfluidics designs were used, including simple straight channels with various lengths, T-shape channels used in electrophoresis and pinch injection, and various meandering channels.

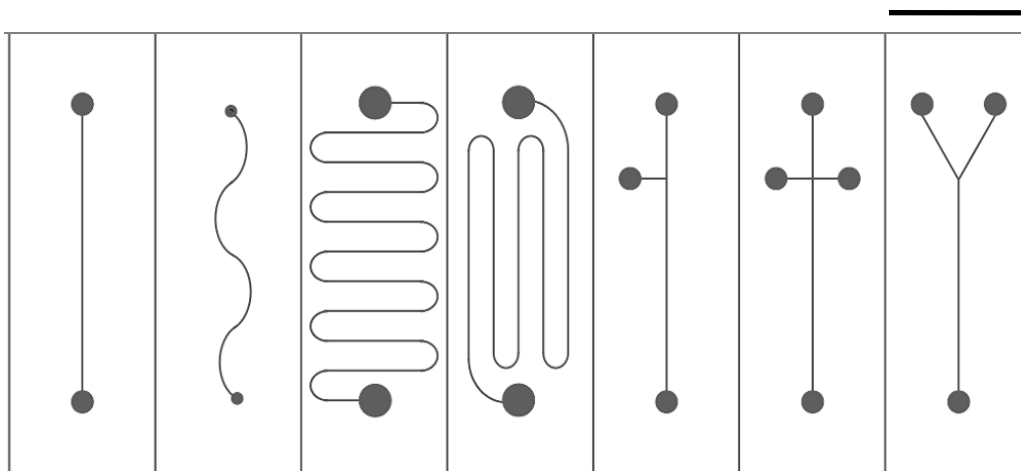


Figure 14: Sample microfluidics designs for laser cut channels. Scale bar represents 2.5 cm.

Blank pieces of PMMA were also cut into appropriate sizes using the laser cutter to be used as the cover side for bonding with both DUV patterned and laser cut channels. Optical images of the cross-section of channels created using DUV exposure and the laser cutter are shown in Figure 15.

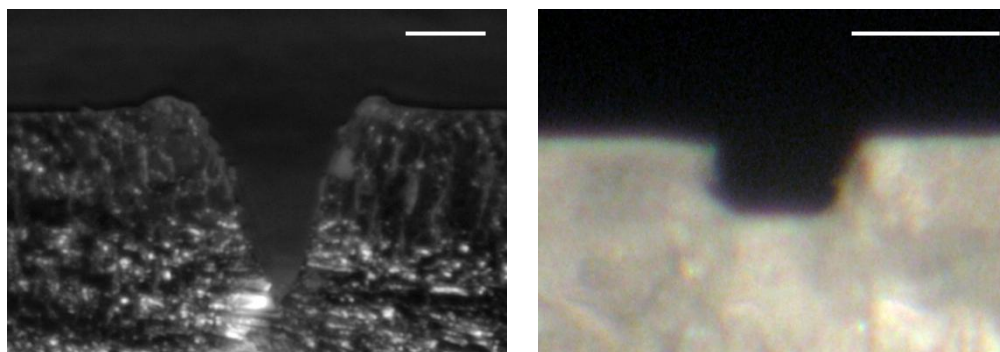


Figure 15: Cross-section of the micro-channels created by Laser cutter (left) and DUV exposure (right). Scale bars represent 200 μm .

In the case of the channels fabricated using the laser cutter, the dimensions of the channels were 400 ± 20 μm wide at the surface, 450 ± 20 μm deep with a Gaussian profile, and a 10 ± 1 μm high lip at the perimeter of the channel. The dimensions of the channels created by DUV patterning technique were 200 μm wide and 100 μm deep, with no lip at the perimeter of the channel. In case of the laser cut channels, more variation in the dimensions of the channels across a single sample was observed. In contrast, the dimensions of the DUV patterned samples were almost perfectly constant throughout a single substrate. After all the micro-channels and access holes were created, the samples were cleaned in an ultrasonic bath in DI water for 20 seconds to remove

any possible small particles and debris from the channels. Finally, the samples were blow dried using a nitrogen gun.

In the solvent bonding methods mentioned in Chapter 4, a strong solvent such as acetone was used to dissolve the interface of the two PMMA substrates and fuse them together. As mentioned previously, it is very difficult to keep the solvent out of the channels while still introducing enough solvent to bond the entire surface area. The fact that the solvents react strongly to PMMA and dissolve the surfaces immediately contributes to the large failure rate of this method, where as soon as the smallest amount of the solvent enters the channels, the channel deforms and clogs. Therefore, the bonding between the channels and the cover will fail and the entire chip will be useless. In contrast, in the new bonding method, microwave-induced, thermal-assisted solvent bonding, the solvent used is a poor solvent for PMMA at room temperature and only becomes effective at higher temperatures.

Introducing a poor solvent at the beginning gives the operator enough time to remove excess solvent from the channels via capillary action or by suction to guarantee that the channels are empty, and hence, will remain intact during heating. In addition, it becomes easier for the operator to align the two halves that are to be bonded which is particularly important when bonding microfluidics chips with more than one layer of channels. Furthermore, the effectiveness of the solvent on PMMA can be controlled by controlling the temperature. Therefore, the process can be easily optimized to reduce the channel deformation or increase the bonding strength [5].

5.2 Bonding Process

In the previously reported microwave bonding methods, a very thin layer of metal or conductive polymer was used to absorb energy and heat up the PMMA interface. This method promotes localized melting of the PMMA and fuses the two substrates together [15, 43]. To achieve the desired temperature, a low power microwave source, 10-30 W, as well as a relatively high precision equipment to apply pressure between the two substrates was required [43].

In the new bonding method, after creating and cleaning the channels, the interface surfaces were fully covered with solvent to ensure that no air bubbles were present in the interface. Any air bubble in the solvent during the microwave heating prevents bonding at that spot and creates a void which can cause leakage if it intersects with the edge of the channels. The voids become more of concern when dealing with structures with higher density of channels or a higher design complexity.

To find the most effective solvent that has the strongest bonding result and the minimal channel distortion, three different solvents were tested: methyl alcohol, ethyl alcohol, and IPA. The reason for selecting these alcohols for bonding is that all of them are poor solvents for PMMA at room temperature but are effective solvents for PMMA bonding at temperatures above 60 °C [5]. After flooding the two pieces with the solvents, they were brought together and the outer surface was dried using absorbing cloths. After the outer surface area was dried, the channel and cover slide were clamped together using miniature bulldog clips, 3/5" size, around the perimeter of the chips. For the 25 mm x 75 mm size

samples, eight clips were used to provide enough and relatively uniform pressure throughout the entire contact area (Figure 16).



Figure 16: PMMA channel ready for microwave heating with metal clips and input tube attached. Scale bar represents 2.5 cm.

Drying the outer surface is important because if the solvent remains on the outer surface of the chip during the microwave heating, it will heat up wherever it is in direct contact with the metal clips and will cause melting on the surface, leaving an indentation on the outer surface. Although these indentations are not deep enough to distort or block the channels, they can reduce clarity and transparency of the final chip, and they are visually unattractive. Figure 17 shows a sample with the indentation caused by the solvent trapped between the outer surface of PMMA and metal clips.

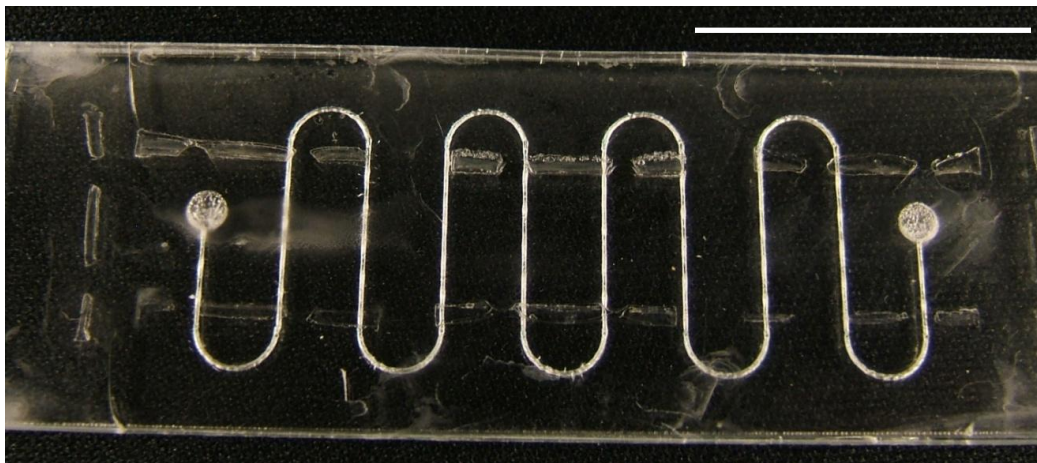


Figure 17: PMMA microfluidics unit with the indentations on the outer surface caused by trapped solvent between the plastic and metal clip. Scale bar represents 2.5 cm.

Note that the bigger size clips are not recommended for this method, because as mentioned in Chapter 4, large metallic objects can cause arching when exposed to microwave radiation. In addition, two adjacent small size clips must not touch each other, because arching may also occur.

At this stage, the excess solvent can be drawn out of the channels using capillary action and an absorbent material or by suction. A piece of tightly rolled Kimwipe can be inserted into the reservoirs through the access hole to suck out the solvents by capillary action. The channels can be visually inspected to ensure that no solvent is blocking them. Also, for channel designs that have more than one layer of structures, crude alignment can be performed in this step. One of the advantages of this method is that, at this stage, the two PMMA pieces are still intact. Therefore, any failure in the above mentioned steps can be redone. The samples can be unclamped and dried, and the bonding process can be repeated

on the same sample. This rework in the production reduces the overall failure rate of this bonding method.

After visual inspection, the samples were placed in a commercial microwave oven for different intervals between 30 seconds and 90 seconds. The set up for bonding process is shown in Figure 18.

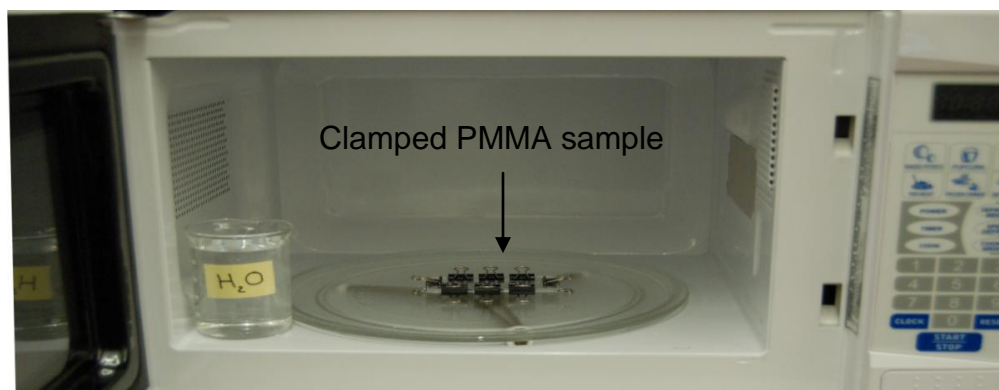


Figure 18: PMMA and water in the commercial microwave oven for the bonding

To increase the safety of the process, the microwave oven was placed inside a fume hood, and the process was continuously monitored during the microwave heating. Also, to avoid any damage to the microwave oven, a small amount of water, approximately 100 mL, was placed inside the oven to absorb any excess microwave energy. The water was replaced with room temperature fresh water after each trial to avoid overheating of the water.

In the previously reported microwave bonding methods, a specialized low power microwave was used; whereas, in this method, a commercial microwave

oven was operated at its full power of 700 W to heat the metal clips. Using off-the-shelf equipment reduces the cost and complexity of the bonding process.

Although PMMA as a thermoplastic and fully polymerized material is almost invisible to microwave radiation and most of the bulk metals are reflective to the microwaves, enough microwave energy will be absorbed by the surface of small metal clips to create the Eddy current and heat up the metal. The composition of the small bulldog clips is spring steel, which is a ferromagnetic material. The heat caused by microwave radiation absorbance on the metal surface will be conducted to the solvent and will warm up the alcohol to an effective temperature.

At the 2.45 GHz frequency used in commercial microwave ovens, alcohols absorb microwaves more effectively than water [5]. In the case of this process, the volume of alcohol in the interface is not large enough to absorb enough energy to heat the alcohol to a temperature above 60 °C. For the 25 mm x 75 mm samples created using the laser cutter, this volume is estimated to be 1.875 x 10 mL which is calculated using following dimensions: 25 mm in width, 75 mm in length and 0.01 mm in height of the lip around the channels. For the DUV patterned samples, this volume should be even less due to nonexistence of the lip around the channels. Because, microwave heating is volumetric, the small volume of solvent cannot heat up faster than it loses heat to the environment. Therefore, direct absorption of microwave radiation by the alcohol cannot be used to increase the alcohol temperature.

Chapter 5: Microwave-induced Thermal-assisted Solvent Bonding

In order to demonstrate that conducted heat created by absorption of microwaves by the metal clips, is the main contributor to the increase in solvent temperature, a plastic press was fabricated that did not require any metal clips. As shown in Figure 19, the press was constructed from two pieces of PMMA, with a thickness of 0.5 cm, tightened together using nylon screws. All trials using these plastic clamps with methanol, ethanol or IPA in different durations up to 90 seconds failed and no visible change in the interface surface of substrates were observed. This lack of bonding indicates that the solvent temperature never exceeded 60 °C and implies that the solvents alone cannot absorb enough energy to become effective for the bonding.

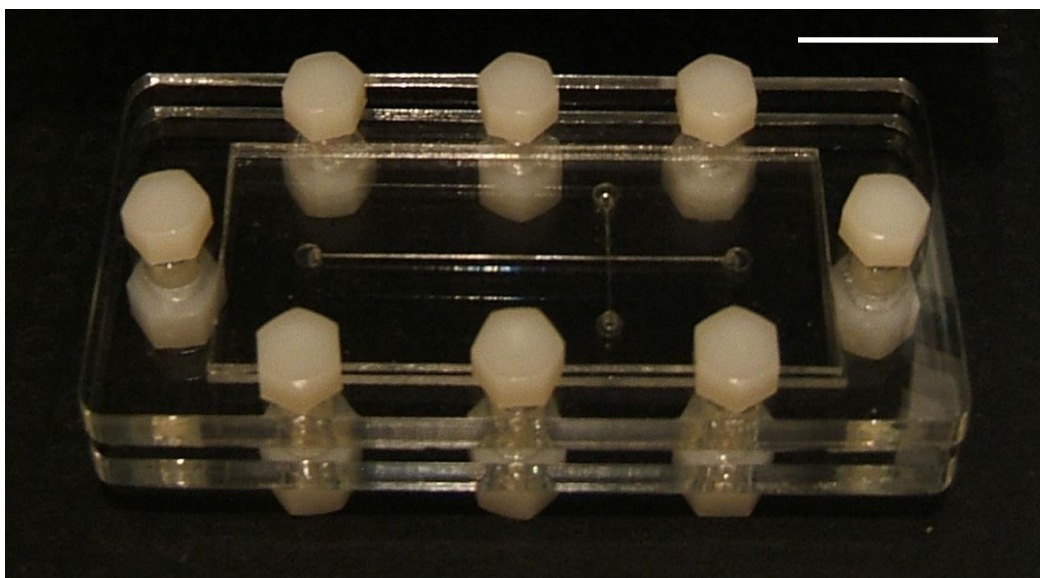


Figure 19: Transparent plastic clamps for testing the effect of direct microwave heating of solvent between PMMA surfaces. Scale bar represents 2.5 cm.

5.3 Testing and Results

To evaluate the quality and success rate of the new bonding technique, one of the tests conducted was a leakage/clogging test. Polyethylene tubes with a diameter of 1.27 mm were connected to the access holes and fixed in place using Loctite 495 superglue. The inlet tube was then connected to a syringe pump. Dark blue dye was pumped at the pump's maximum flow rate, 2 mL/min, through the channels. At this flow rate, the measured pressure was calculated to be 68 kPa. Visual inspection during the leakage test showed no leakage in the bonded area of the channels. Leakage only occurred in areas where visible void in bonding overlapped the channels. The only leakage observed during testing was the occasional failure of the glue that was used to attach the tubes to the inlet holes or the syringe pump.

Test for clogging was done visually as well. In all the chips that were properly bonded around the channels (with no visible void in the bonding overlapping the channels), the channels were found unclogged. Because this method uses a weak solvent that does not affect PMMA at room temperature, the method is less vulnerable to overheating during the microwave bonding. The alcohol will not cause any deformation of channels before the microwave step; by removing all the excess alcohol from the channels via capillary action, the interior part of the channels will remain cool enough during the microwave heating. Hence, no major deformation or channel distortion will occur and the channels will remain unplugged. Figure 20 shows the intersection of bonded and unbonded channels created by laser cutter and DUV patterning methods.

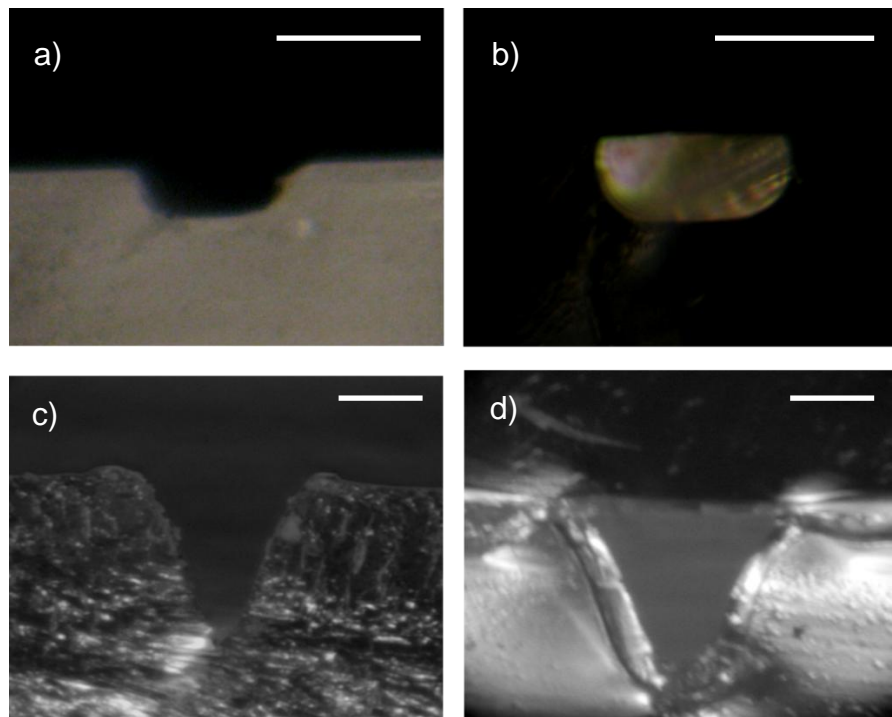


Figure 20: Optical images of cross-sections of channels created by DUV exposure (top) and laser cutter (bottom), non-bonded, (a and c), and bonded, (b and d). Scale bars represent 200 μm .

The other factor visually tested to evaluate the bonding quality was the percentage of bonded surface area. Simple straight channel structures were used for this evaluation. Three different alcohols (methanol, ethanol, and IPA) were used for these bonding trials. Samples for each alcohol were heated up in the microwave for 5 durations between 30 to 90 seconds. Because, after 90 seconds, the majority of the alcohol in the interface is already absorbed or evaporated and in longer heating periods the metal clips may overheat, 90 seconds was chosen as the maximum effective bonding time. Figure 21 shows the average bonding area percentage for these different trials. Details of the data collected for the diagram is summarized in Appendix H. As shown in Figure 21,

ethanol has the best performance, whereas IPA yields the lowest percentage of bonded surface area. These results are consistent with the results reported in earlier works by Hsu *et al.* [5].

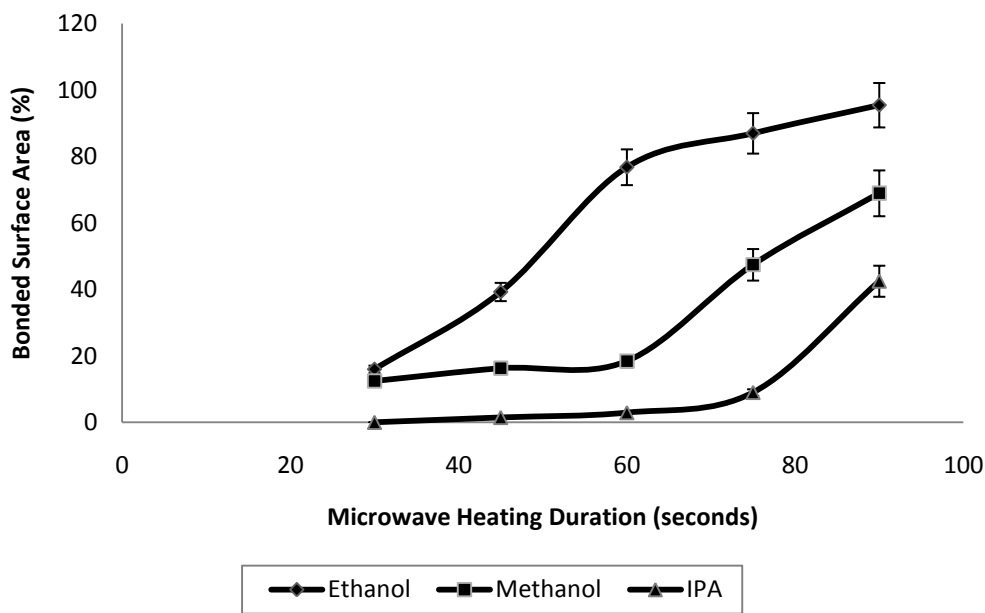


Figure 21: Percentage of bonded surface area for microfluidics chips using different solvents

From Figure 21, note that the percentage bonded area is a nonlinear function of microwave heating time because of the poor thermal conductivity of the bulk PMMA substrate, and the initial time needed for the metal clips to heat up to 60 °C (approximately 30 seconds).

Initially, it was hypothesized that the approximately 10 μm tall lip around the edges of the channels fabricated using laser cutter would negatively affect the bonding quality. However, further investigations showed that the lip actually has beneficial effects on the process because the elevated area just next to the

channel edge receives higher pressure and it will be the first area that bonds before the solvent begins to evaporate. As a result, the lip can improve the quality and success rate of the bonding (Figure 22).

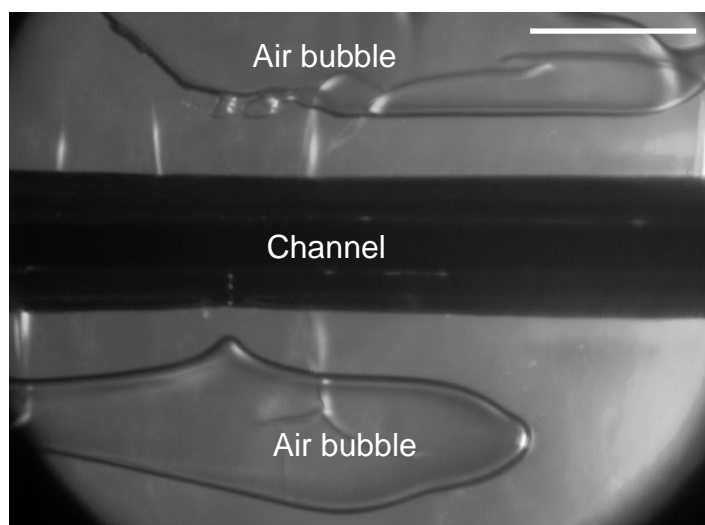


Figure 22: Close top view of a bonded laser cut channel filled with black ink showing the effectiveness of the elevated lip near the edge of the channel in sealing. Scale bar represents 200 μm .

The laser cut samples were evaluated, and chips with overall bonded surface area as low as 50% were properly functional and leakage free. However, if the cut produced lips at the edge much taller than 10 μm , or if it caused the bulk substrate to warp, the quality of bonding decreased, resulting in a reduction of the bonding success rate. The negative effect of warping usually appear in the areas where two or more channels overlapped to creates a junction or in large reservoirs where the laser cuts many times in close proximity, which leads to the melting of a large area of substrate and, hence, deformation and warping.

Chapter 5: Microwave-induced Thermal-assisted Solvent Bonding

Among all the different attempted bonding methods reported in literature, solvent bonding provides the strongest bonds [10]. To test the bond strength of the microwave-induced, solvent-assisted bonding method, PMMA samples were cut and bonded using ethanol in such a way that the bonded surface area was approximately 1cm^2 , as shown in Figure 23-b. The samples were bonded by heating in the microwave oven for intervals of 30, 45, 50, 75, and 90 seconds. Then, a Instron MicroTester model 5848 machine, manufactured by Instron corporation, was used to apply shear stress to the samples. As shown in Table 4, in all cases the bulk PMMA substrate started to fracture before the bonded area delaminated.

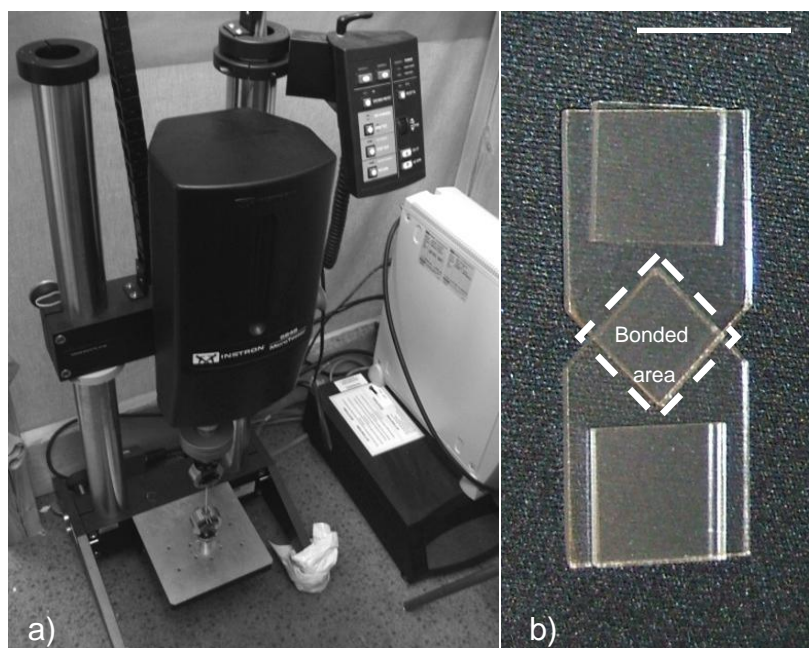


Figure 23: a) Instron MicroTester model 5848, b) bonded sample used for shear stress test (the area enclosed inside the dashed square is the bonded surface area). Scale bar represents 2.5 cm.

Table 4: Results of the shear stress test for bonding strength

Microwave heating duration (sec)	Bonded surface area (cm ²)	F _{max} applied before bulk PMMA fracture (N)	Applied shear stress (MPa)	Delamination of bonding
30	1.1	215	1.95	No
45	1.0	300	3.00	No
60	1.3	360	2.77	No
75	1.0	260	2.60	No
90	1.2	340	2.83	No

Considering the results, it is expected that the new bonding method has a similar bonding strength and quality as the ones reported in literature for solvent bonding. Based on the earlier studies by Hsu *et al.* [5] on the bonding strength of thermally assisted solvent bonding using ethanol, it was expected that the strength of the new bonding method would exceed 3 MPa.

The other important factor that needed to be determined was channel deformation caused by the solvent. As the majority of the solvent was taken out of the channel prior to microwave heating and the solvents used for bonding do not affect unexposed PMMA at room temperature, the channel deformation was not a major concern for the samples fabricated using the laser cutter. However, in the case of samples created using the 254 nm DUV exposure method, channel deformation could be a significant problem during bonding. As explained in Chapter 3, areas of the PMMA chip that are exposed to DUV radiation will have a lower molecular weight than the unexposed bulk PMMA due to bond cleavage in

the main polymer chain. Cleavage in the main chain will lower the molecular weight and, hence, increases the solubility of PMMA in solvents.

For the samples fabricated using the DUV exposure method, the channels areas were exposed to DUV light and, therefore, they had a lower average molecular weight. During the development process, the majority of these lower molecular weight polymer chains dissolved and were washed away from the channels, but it was still expected that all channels and reservoirs to have a slightly lower average molecular weight than the bulk area. As a result, it is expected that the channels would be affected by the solvent even before the microwave heating step, resulting in greater channel deformation. An experiment was designed in order to determine the damage and deformation caused by the alcohol in the channels before they were dried out.

In this experiment, four PMMA substrates were cut into 75 mm x 75 mm squares. Half of the surface area of each substrate was covered by aluminum foil to block the DUV light. The samples were then exposed to 216 J/cm² of DUV light, equivalent to 15 hrs exposure in the Stratlinker 2400. Samples were then soaked in three different solvents (methanol, ethanol, and IPA) for one minute at room temperature, 18 °C. On average the operator needed one minute to remove the solvent from the channels after applying the solvent and before starting the microwave heating. The samples were finally dried using a nitrogen gun without any extra rinsing step by fresh solvent. No rinsing was performed in order to simulate the worst bonding condition. In the case of the micro-channels, most of the low molecular weight polymer chains were already removed during the 30

minutes development period. However, for the samples soaked in solvents, almost all the low molecular weight PMMA layer was still in place because of the very short development time of 1 minute. The average surface roughness of these samples was measured before and after contact with the solvents. For all three different alcohol types, cracking occurred in all the exposed areas, as shown in Figure 24. The cracking phenomenon only appears in the very large exposed surface areas and is not a common problem while dealing with small dimensions, such as micro-channels. Therefore, while measuring the surface roughness, the cracks were avoided. Appendixes D, E, F, and G present the measurements of these tests.

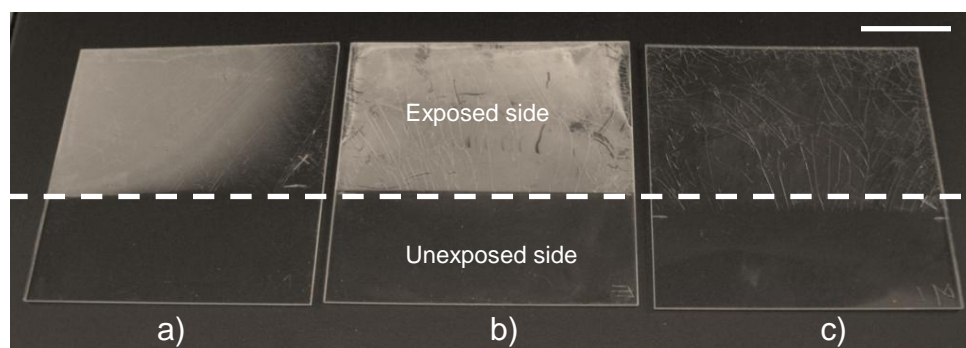


Figure 24: Optical image of the PMMA samples exposed to 216 J/cm^2 of DUV after 1 min soaking in a) methanol, b) ethanol, c) IPA. Bottom half of each square was covered during the exposure; i.e. only the top halves were exposed to DUV light. Scale bar represents 2.5 cm.

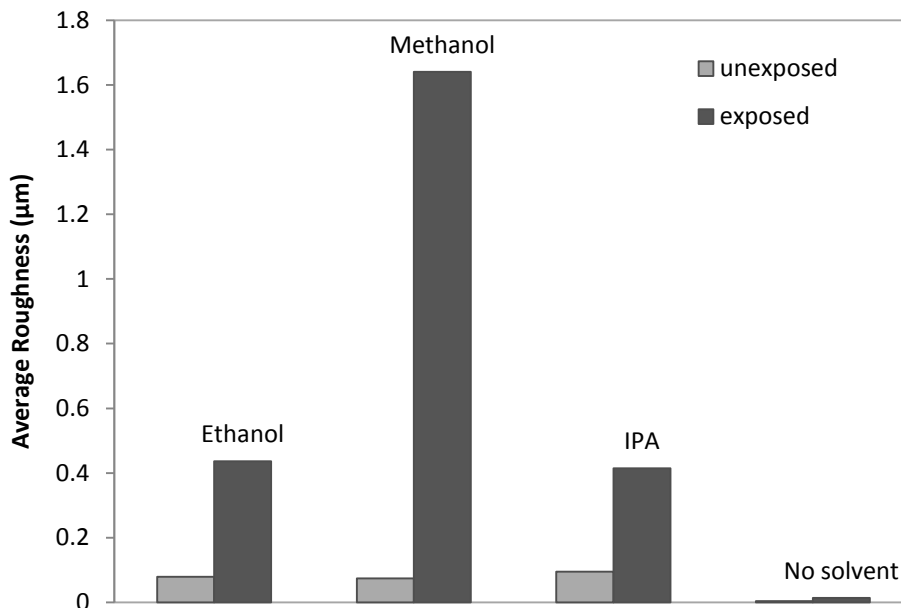


Figure 25: Average surface roughness of PMMA for exposed (216 J/cm^2) and unexposed areas after 1 minute of soaking in different alcohols.

From Figure 25, IPA has the least surface roughness and leftover white ‘scum’. Ethanol created the same surface roughness as ethanol, but it had more white ‘scum’ left over, indicating a higher dissolution rate compared to IPA. Finally, methanol had the most severe surface roughness as well as largest variation across a single sample. However, all of the measured surface roughness values were considerably less than the values measured for the samples created using the laser cutter. Also, the quality and transparency of the channels were much higher even in the case of the worst alcohol, methanol.

Because of the lower dissolution rate of PMMA in IPA and methanol, the percentage of bonded surface area was smaller for these alcohols.

One of the issues that affects the success rate of this bonding method is that during the microwave heating, the alcohol starts to evaporate before the bonding process begins. This effect was most prominent on the outer edge of the chips where the evaporated alcohol can escape most easily. Leaving enough space between the edge of the chips and the channels, approximately 5 mm, reduced the effect of this phenomenon and improved the overall bonding success of the channels. Therefore, although some parts of the chip's surface area near the edge might remain un-bonded, most of the chips were still functional and leakage free.

As mentioned previously, one of the disadvantages of solvent-assisted bonding of PMMA is the appearance of hairline cracks on the laser cut PMMA after contact with alcohols. This phenomenon is illustrated in Figure 26.

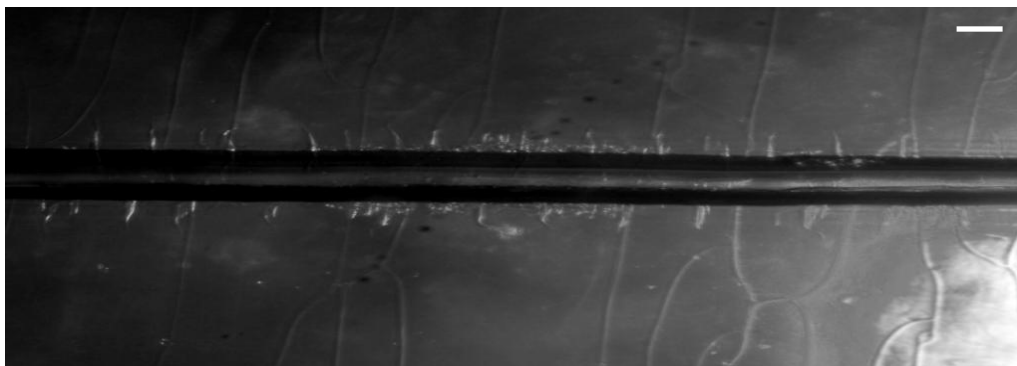


Figure 26: Hairline cracks on the edge of a micro-channel filled with opaque ink. Scale bar represents 400 μm .

The cracking phenomenon normally does not appear in the samples fabricated by using DUV exposure method. However, the samples created using the CO₂ laser cutter are more vulnerable to the cracking. Although the hairline cracks are small enough so they do not cause a leakage problem, it can be visually unattractive. In addition, in some extreme cases, these cracks were deep enough to affect the overall strength of the bulk PMMA and it became more vulnerable to major cracks that could occasionally break the chip into pieces. While fabricating channels using the laser cutter, the laser beam vapourizes the plastic causing rapid melting and cooling of PMMA in close proximity to the evaporated area. This vapourization introduces a high internal stress in the areas near the evaporated channels. These areas are highly vulnerable to damage, and the cracking always begins in these areas after contact with alcohols. The cracking problem was addressed by annealing the samples for over 12 hours at 80 °C after the cutting step (Figure 27).

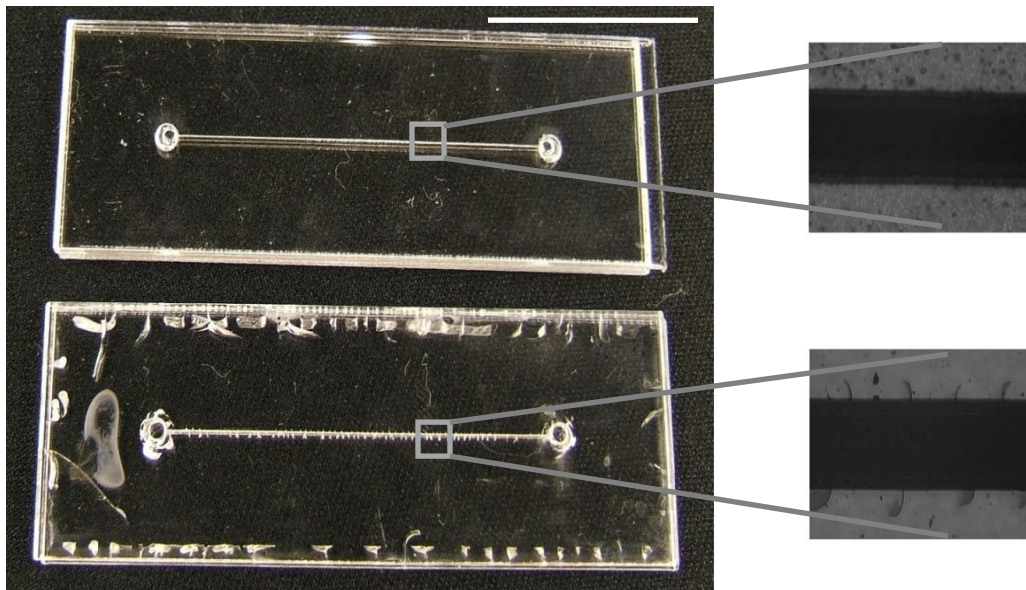


Figure 27: Two PMMA channels created using CO₂ laser cutter and bonded using ethanol. Top sample was annealed for duration of 12 hours and showed no hairline crack around the cut edges. Bottom sample did not undergo the annealing step and had hairline cracks near all the laser cut edges. Scale bar represents 2.5 cm.

Another one of the challenges of this bonding method was the accurate measurement of the temperature. Due to the use of a microwave for the rapid heating, using a metallic thermocouple or liquid thermometer was not possible. Also, due to the low thermal capacity of the miniature clips used in this bonding method, it was not practical to accurately measure the temperature immediately after the microwave heating step. Therefore, to be able to estimate the temperature of the interface of the two PMMA pieces that were to be bonded, the channels were filled with poly(ethylene glycol) (PEG), which has an average molecular weight (M_n) of 4400-4800 and a melting point of 60 °C. This product was purchased from Sigma Aldrich and was in a white crystalline flakes or powder form in its solid form. PEG is a water soluble wax like material that

changes from white in its solid form to transparent in its melted form. This way, it can visually be determined when the temperature reached 60 °C. This temperature is important because ethanol become an effective solvent for unexposed PMMA at a temperature above 60 °C [5].

For this experiment, the channels were cut in a meandering shape and the access holes were not cut into reservoirs in order to create a completely closed channel. Prior to bonding, the channels were filled with melted PEG. The excess PEG was removed using the edge of a glass cover slide and the surface was cleaned with a wet kimwipe paper. A blank PMMA slide was then bonded to the channel side to create a completely closed structure filled with PEG. Because the PEG is trapped in the core of the chip, it will need a relatively long time to cool down compared to the cooling time of the metal clips. Therefore, the sample can be inspected immediately after the microwave heating in order to evaluate when the interior surface reaches 60 °C. It was observed that the PEG in the areas directly underneath the metal clips start to melt after 30 seconds, but the entire surface area reached 60 °C after 90 seconds (Figure 28). Although the overall temperature of the metal clips never elevated high enough to melt the PMMA surface during the trials described in this project, it is recommended that the sample be continuously monitored while heated in the microwave to ensure the clips do not overheat.

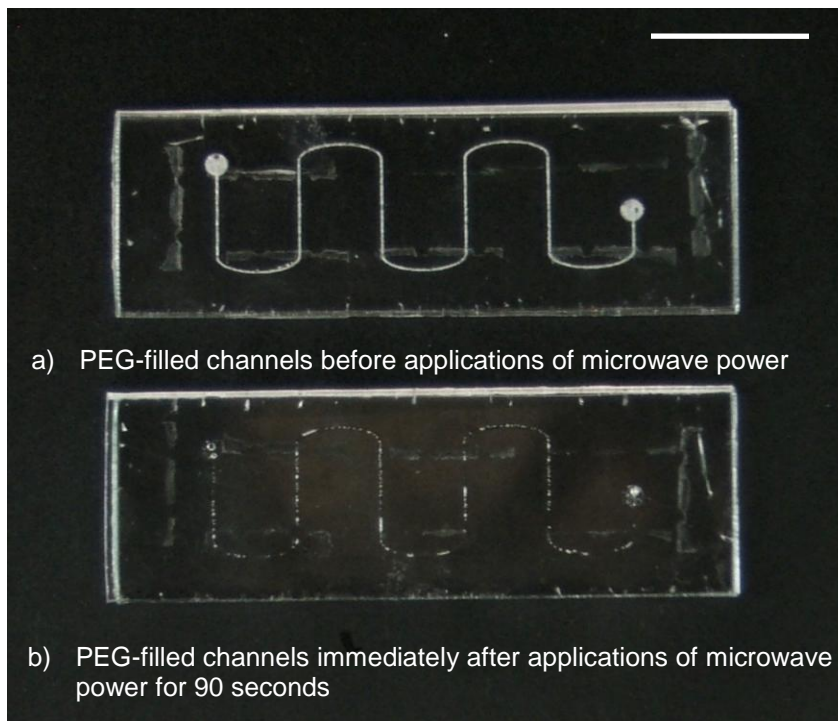


Figure 28: Optical images of PEG-filled channels before (a) and immediately after (b) applications of microwave power. After 90 seconds of Microwave heating, all areas of PEG have melted. Scale bar represents 2.5 cm.

5.4 Applications

After creating the microfluidics unit, several tests were conducted to show that the micro-channels fabricated using the DUV method or the laser cutter combine with microwave-induced, thermally assisted solvent bonding was compatible with common microfluidics processes. One of the tests that were conducted on these chips was electrophoretic pinch injection.

Electrophoresis is a technique by which charged particles can be separated under the influence of an electric field. In this method, particles suspended in a medium are separated by their velocity in an electric field. The

particles' velocity depends on their electric charge and size. This technique is widely used for the separation of large bio-molecules, such as proteins and DNA fragments from a suspension mixture. Historically, the process has been carried out on materials such as starch gel and paper. Poly acrylamide and agarose gels are the media currently in common use for electrophoresis. Due to recent advancements in the field of microfluidics, the separation can now be conducted using a microfluidics structure. Electrophoretic pinch injection was tested and compared on channels created by the two fabrication methods described earlier in this thesis, CO₂ laser cutter and DUV exposure.

In both methods, T-shape electrophoresis channels were created on PMMA (Optix®) substrates. The T-shape channel consisted of a 5.00 cm long horizontal and 1.25 cm long vertical channel, as shown in Figure 29.

After fabricating the channels, the samples were bonded to a blank cover side using ethanol as the solvent and using microwave heating for duration of 90 seconds. To increase the volume of reservoirs, 4 mm thick o-rings made of PMMA were glued to the top surface by Locatite superglue (Figure 29).

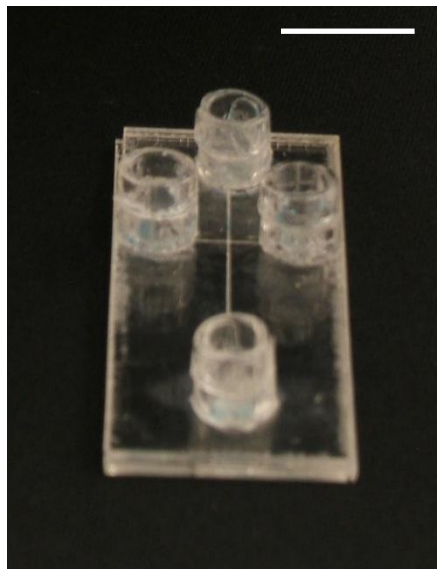


Figure 29: Microfluidics unit fabricated by laser ablation and microwave bonding for pinch injection test. Scale bar represents 2.5 cm.

The sample solution used for the electrophoresis separation consisted of sodium borate mixed with a fluorescent dye, and sodium borate Ph 9.5 mM was used as the buffer solution. Flow of the fluid through the channels was controlled by the potentials applied to the reservoirs, as shown in Figure 30. To perform the pinch injection, the applied voltages were switched between two modes, loading and dispensing modes. During the loading mode, the sample solution was moved from sample (S), 0 V, to sample waste (SW), 300 V, reservoirs. In the dispensing mode, the buffer solution was moved from buffer (B), 0 V, to buffer waste (BW), 692 V, reservoirs. The dispensing step pushed a plug of sample into the horizontal channel and moved it from B to BW. In dispensing step the S and SW were given a 477 V potential to prevent the back flow of buffer into sample channel and create a sample plug. Separation was achieved through this

operation. The demonstrated performance showed that the micro-channel units created by the process presented in the previous chapters are compatible with chemical and biological applications that require electro osmotic flow.

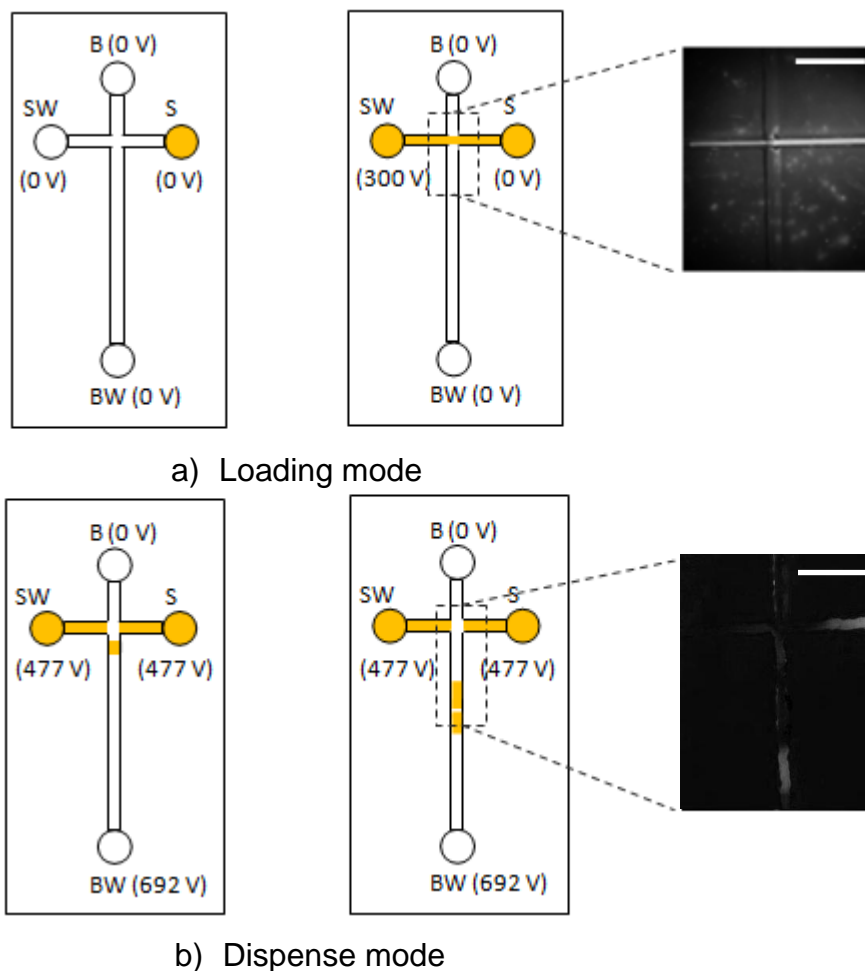


Figure 30: Different steps of pinch-injection process and optical images of fluorescent dye corresponding to each step. Scale bars represent 0.25 cm

Chapter 5: Microwave-induced Thermal-assisted Solvent Bonding

Another structure fabricated to test the reliability and functionality of the fabrication and bonding process was a two layer structure, consisting of a simple straight channel layer and a meandering channel layer (Figure 31).

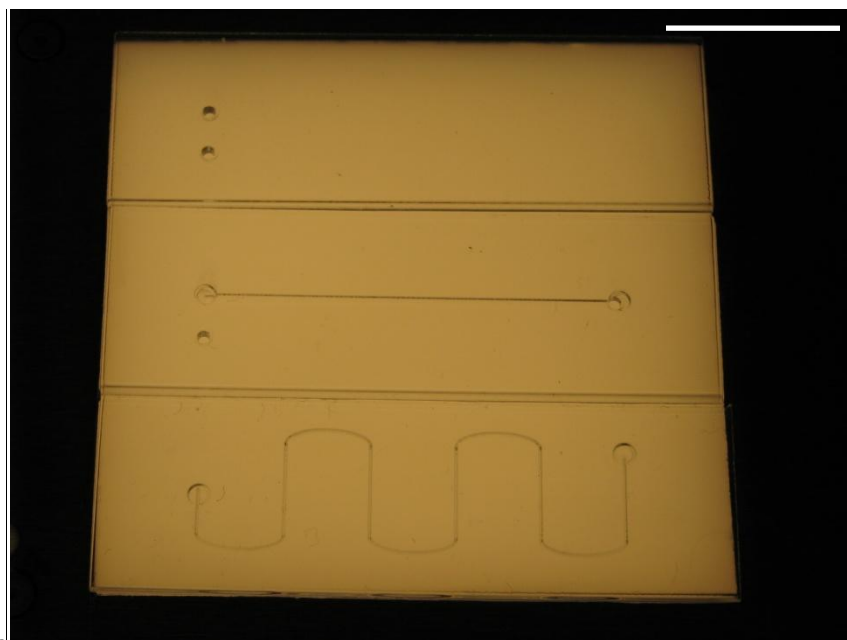


Figure 31: Different layers of a dual-layer microfluidics chip design for testing the reliability of the bonding process for microfluidics structures consist of more than one layer of structures. The channels are fabricated using CO₂ Laser cutter. Scale bar represents 2.5 cm

The layers shown in Figure 31 were bonded using ethanol and 90 seconds of microwave heating for each layer. After the bonding inlet and outlet tubes were attached using Loctite superglue and dark blue dye was run through the channels for blockage and leakage testing (Figure 32).

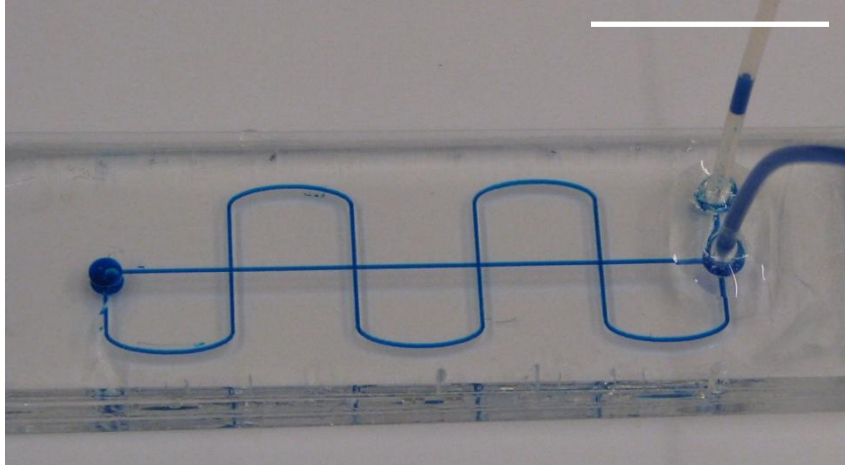


Figure 32: Bonded dual-layer microfluidics chip. Scale bar represents 2.5 cm

The dual-layer channels fabricated by using the fabrication techniques presented in this thesis did not fail the pressure-induced flow test and were fully functional, indicating that the process is reliable for producing multilayer microfluidics structures.

Chapter 6: Future Works and Conclusion

6.1 Future Work

In the course of the work described in this thesis, several possible process improvements were identified for future work. Future improvements can be separated into two main categories: improvements related to the DUV PMMA patterning process and improvements related to the bonding process.

In keeping with the philosophy of fabricating simple, low cost and disposable microfluidics units, one modification is to avoid the gold layer deposition. In the fabrication process described in Chapter 2, a thin, 100 nm, layer of gold was used as photo-mask layer during the DUV exposure step. This method requires access to high-cost equipment for metal deposition, patterning and etching, as well as a need for access to cleanroom environment for the required deposition and patterning steps. Instead of the gold photo-mask layer, a layer of UV opaque ink can be used to selectively block the DUV light during the

exposure. In this new approach, a silkscreen can be utilized to produce the required photo-mask [55].

Silkscreen printing techniques have been long used for transferring images to fabrics in the textile industry. This technique has also been employed for printing circuit boards in electronic industry. This technique was adopted to minimize the need for access for the specialized and costly lab facilities, and it can be employed to pattern a UV opaque photo mask layer on PMMA surface for DVU exposure. Replacing the gold layer with an UV opaque layer will make the 254 nm patterning technique accessible for low budget laboratories and fabrication facilities as well as research laboratories. It also makes PMMA a great candidate for rapid and cost effective substrate for prototyping microfluidics designs

Another way to improve the manufacturing process is to design and fabricate customized clips rather than using off-the-shelf bulldog clips. Depending on the approach chosen for the fabrication of these clips, the fabrication of clips may add to the overall cost of the fabrication method. However, it will provide the ability to selectively heat up the desired locations of the microfluidics surface unit instead of bonding the entire interface area. This selective bonding method can provide an advantage if there are other structural components in the design, such as electrodes, that need to be placed closed to the channels and inside the interface. Additionally, one of the limitations of the current bonding method is that it can only be applied on small size samples, because as the size of the PMMA chip increases, it becomes more difficult to heat up and bond the middle part of

the chip due to the small size of metal clips. Customized bonding clips can be designed in such a way that provides enough heat for bonding samples of different shape and size.

6.2 Contributions

This work has resulted in a number of publications. Development of the DUV patterning method was published in *Journal of Micromechanics and Microengineering (JMM)* [9]. Because of the novelty of the work, the research committee selected the paper to be part of *JMM Highlights of 2008*. Development of the microwave-induced, thermal-assisted solvent bonding method was also published in *JMM* [56].

6.3 Conclusion

This thesis has described a fabrication process that allows manufacture of plastic microfluidics components in an economical manner. The novel techniques described in this work can produce fully functional microfluidics components easily and in a highly reproducible manner at a low cost. It was also successfully demonstrated that both the microchannel fabrication technique and bonding method are compatible with common applications of microfluidics units. Through conducting characterization tests, the process was optimized for reliability and consistency. In addition, by eliminating the need for toxic and corrosive chemicals commonly used during traditional microfabrication methods, the technique represents a safe and environmental friendly alternative. Adopting the processes described in this thesis will help hasten the shift from expensive glass and silicon

Chapter 6: Future Works and Conclusion

based processes to low-cost, biocompatible plastic designs, opening the field of microfluidics to new, novel applications.

Appendix A: Material Properties of Optix® PMMA Purchased from GoodFellow

Table 5: Material properties of Optix® PMMA [29].

Properties	Units	Values
Physical		
Specific Gravity	-	1.19
Optical Refractive Index	-	1.49
Light Transmittance Total/Haze	%	92/2
Sound Transmission	db	27
Water Absorption	% By Weight	0.40
Shrinkage	% Shrinkage	<5%
Mechanical		
Tensile Strength - Max	psi	11,030
Tensile Elongation - Max	%	5.8
Tensile Modulus of Elasticity	psi	490,000
Flexural Strength - Max	psi	17,000
Flexural Modulus of Elasticity	psi	490,000
Thermal		
Maximum Recommended	°F	170-190
Softening Temperature	°F	210-220
Melting Temperature	°F	300-315
Coefficient of Thermal Expansion -	In/(in-°F)	3.0
Thermal Conductivity	BTU-ft/	0.075
Chemical		
Resistance to Stress-Critical	psi	900

Appendix B: Material Properties of CQ PMMA Purchased from GoodFellow

Table 6: Material Properties of CQ PMMA [58].

Properties	Units	Values
Physical		
Density	g/cm ³	1.19
Limiting Oxygen Index	%	17 - 20
Reflective Index	-	1.49
Resistance to UV	-	good
Water Absorption (24h)	%	0.2
Mechanical		
Hardness - Rockwell	-	M92 - 100
Poisson's Ratio	-	0.35 - 0.40
Tensile Modulus	GPa	2.4 - 3.3
Tensile Strength	MPa	80
Thermal		
Specific Heat	-	70 – 77
Coefficient of Thermal Expansion –	J/(KxKg)	1400 -1500
Thermal Conductivity @ 23 °C	W/(mxK)	0.17 – 0.19

Appendix C: Material Properties of UVT OP-4 PMMA

Table 7: Material properties of UVT PMMA [57]

Properties	Units	Values
Physical		
Specific Gravity	-	1.19
Optical Refractive Index	-	1.49
Light Transmittance Total/Haze	%	92/>1
Water Absorption (24h @ 73 °F)	%	0.2
Water Absorption (long term)	%	0.5
Mechanical		
Tensile Strength	MPa	69
Tensile Elongation	%	4.2
Tensile Modulus of Elasticity	MPa	2,800
Flexural Strength	MPa	114
Flexural Modulus of Elasticity	MPa	3,300
Thermal		
Specific Heat @ 77°F	J/kgxK	1470
Coefficient of Thermal Conductivity	w/mK	0.19
Coefficient of Linear Thermal	In/in/°F	0.00004

Appendix D: Surface Roughness of Optix® PMMA Soaked in Ethanol for 1 Minute

Table 8: Surface roughness of Optix® PMMA soaked in ethanol

Ethanol		
Measurement points	Exposure dose 216 (J/cm ²)	Exposure dose 0 (J/cm ²)
	Roughness (μm)	Roughness (μm)
Point 1	0.407	0.105
Point 2	0.526	0.020
Point 3	0.415	0.032
Point 4	0.420	0.082
Point 5	0.412	0.178
Point 6	0.407	0.010
Point 7	0.357	0.214
Point 8	0.601	0.032
Point 9	0.422	0.013
Point 10	0.389	0.096
Average	0.435	0.078

Appendix E: Surface Roughness of Optix® PMMA Soaked in Methanol for 1 Minute

Table 9: Surface roughness of Optix® PMMA soaked in methanol

Methanol		
Measurement points	Exposure dose 216 (J/cm ²)	Exposure dose 0 (J/cm ²)
	Roughness (µm)	Roughness (µm)
Point 1	0.109	0.058
Point 2	1.608	0.092
Point 3	1.857	0.032
Point 4	1.365	0.090
Point 5	8.237*	0.057
Point 6	6.252*	0.042
Point 7	9.075*	0.098
Point 8	1.055	0.095
Point 9	0.710	0.030
Point 10	4.785	0.140
Average	1.641	0.073

*These data points are not used in the calculation of average surface roughness

Appendix F: Surface Roughness of Optix® PMMA Soaked in IPA for 1 Minute

Table 10: Surface roughness of Optix® PMMA soaked in IPA

Isopropyl alcohol		
Measurement points	Exposure dose 216 (J/cm ²)	Exposure dose 0 (J/cm ²)
	Roughness (μm)	Roughness (μm)
Point 1	0.390	0.115
Point 2	0.390	0.017
Point 3	0.825	0.030
Point 4	0.277	0.067
Point 5	0.337	0.120
Point 6	0.210	0.102
Point 7	0.340	0.025
Point 8	0.275	0.050
Point 9	0.285	0.075
Point 10	0.817	0.092
Average	0.414	0.069

Appendix G: Surface Roughness of Optix® PMMA

Table 11: Surface roughness of Optix® PMMA without being soaked in any alcohol

No solvent		
Measurement points	Exposure dose 216 (J/cm ²)	Exposure dose 0 (J/cm ²)
	Roughness (µm)	Roughness (µm)
Point 1	0.010	0.004
Point 2	0.019	0.003
Point 3	0.011	0.002
Point 4	0.016	0.002
Point 5	0.008	0.004
Point 6	0.018	0.006
Point 7	0.011	0.005
Point 8	0.009	0.004
Point 9	0.016	0.005
Point 10	0.021	0.003
Average	0.013	0.004

Appendix H: Percentage of Bonded Surface Area for Microwave Bonding Process

Table 12: Percentage of bonded surface area

Ethanol						
Bonding time (sec)	try1 (%)	try2 (%)	try3 (%)	try4 (%)	try5 (%)	Average (%)
30	10	14	18	23	12	15
45	35	45	38	39	36	39
60	72	85	66	81	77	76
75	85	93	85	81	88	86
90	97	97	98	83	98	95
Methanol						
Bonding time (sec)	try1 (%)	try2 (%)	try3 (%)	try4 (%)	try5 (%)	Average (%)
30	21	8	15	6	10	12
45	18	14	17	14	16	16
60	17	12	24	16	20	18
75	66	50	42	37	40	47
90	73	70	68	60	71	68
Isopropyl alcohol						
Bonding time (sec)	try1 (%)	try2 (%)	try3 (%)	try4 (%)	try5 (%)	Average (%)
30	0	0	0	0	0	0
45	4	1	0	2	0	1
60	3	2	3	2	3	2
75	18	6	7	5	7	8
90	36	49	38	43	44	42

References

- [1] N. T. Nguyen and S. T. Wereley, *Integrated microsystem series, second edition*, Boston: Artech House, 2006.
- [2] K.S. Yung and E. Yoon, "Fabrication of complex multilevel microchannels in PDMS by using three-dimensional photoresist masters," *Lab on a Chip*, vol. 8, no. 2, p. 5, 2008.
- [3] P. M. Martina, D. W. Matsona, w. D. Bennetta, and D. J. Hammerstrom, "Fabrication of plastic microfluidic components," *Proceedings of the SPIE - The International Society for Optical Engineering*, vol. 3515, p. 5, 1998 1998.
- [4] P. C. H. Li, "Microfluidic lab-on-a-chip for chemical and biological analysis and discovery," Boca Raton, Fla: Taylor & Francis Group, 2006.
- [5] Yi-Chu Hsu and T.-Y. Chen, "Applying Taguchi methods for solvent-assisted PMMA bonding technique for static and dynamic μ -TAS devices," *Biomed Microdevices*, vol. 9, p. 10, 2007.
- [6] M. Haiducu, "Low-cost microfluidics on commercial grade Poly(Methyl Methacrylate) (PMMA) using deepUV patterning " in *Applied science*. vol. Master of applied science Burnaby: Simon Fraser University, 2009, p. 85.
- [7] O. B. Bakajin, T. A. J. Duke, C. F. Chou, S. S. Chan, R. H. Austin, and E. C. Cox, "Electrohydrodynamic Stretching of DNA in Confined Environments," *Physical Review Letters*, vol. 80, no. 12, p. 2737, 1998.
- [8] Yun Chen, Luyan Zhang, and G. Chen, "Fabrication, modification, and application of poly(methyl methacrylate) microfluidic chips," *Wiley interscience*, vol. 29, no. 9, p. 14, 2008.
- [9] M. Haiducu, M. Rahbar, I. G. Foulds, R. W. Johnstone, D. Sameoto, and M. Parameswaran, "Deep-UV patterning of commercial grade PMMA for low cost, large-scale microfluidics," *Journal of Micromechanics and Microengineering*, vol. 18, no. 11, p. 8, 2008 2008.
- [10] Chia-Wen Tsao and D. L. DeVoe, "Bonding of thermoplastic polymer microfluidics," *Microfluid Nanofluid*, vol. 6, no. 1, p. 16, 2009.
- [11] H. Becker, "Hot embossing as a method for the fabrication of polymer high aspect ratio structures," *Sensors and Actuators*, vol. 83, no. 1, p. 6, 2000.
- [12] R. Truckenmuller, P. Henzi, and V. W. K. Saile, "A new bonding process for polymer micro- and nanostructures based on near-surface degradation," *17th IEEE International Conference on Micro Electro Mechanical Systems. Maastricht MEMS 2004 Technical Digest (IEEE Cat. No.04CH37517)*, p. 4, 2004.
- [13] N. T. Nguyen and S. T. Wereley, *Integrated microsystem series, second edition*: Boston: Artech House, 2006.

- [14] C.G. Willson, *Introduction to Microlithography, in Introduction to Microlithography*, 2nd ed.: American Chemical Society: Washington, D.C., 1994.
- [15] A. A. Yussuf, I. Sbarski, J. P. Hayes, M. Solomon, and N. Tran, "Microwave welding of polymeric-microfluidic devices," *J. Micromech. Microeng.*, vol. 15, p. 8, 2005.
- [16] R. w. Johnstone, "Self-sacrificial surface-micromachining using poly(methyl methacrylate)," in *Engineering Science*. vol. Doctor of Philosophy Burnaby: Simon Fraser University, 2008, p. 172.
- [17] R. Truckenmuller, P. Henzi, D. Herrmann, V. Saile, and W. K. Schomburg, "Bonding of polymer microstructures by UV irradiation and subsequent welding at low temperatures," *Microsystem Technologies*, vol. 10, no. 5, p. 3, 2004.
- [18] J. O. Choi, J. A. Moore, J. C. Carelli, J. P. Silverman, and H. Bakhru, "Degradation of poly(methylmethacrylate) by deep ultraviolet, x-ray, electron beam, and proton beam irradiations," *Journal of Vacuum Science and Technology B*, vol. 6, no. 6, p. 4, 1988.
- [19] O. Güven, "Crosslinking and scission in polymers," in *Proceedings of the NATO Advanced Study Institute on Advanced Methods of Determination of Crosslinking and Scission in Polymers and their Effects on Mechanical Properties*, Kemer-Antalya, Turkey, 1988.
- [20] H. Hiraoka, "Radiation-Chemistry of Poly(methacrylates)," *IBM Journal of Research and Development*, vol. 21, no. 2, p. 10, 1977.
- [21] P. Meyer, A. El-Kholi, and J. Schulz, "Investigations of the Development Rate of Irradiated PMMA Microstructures in Deep X-Ray Lithography," *Microelectronic Engineering*, vol. 63, no. 4, p. 10, 2002.
- [22] M. Bolorizadeh and D.C. Joy, "Low Voltage Electron Beam Lithography in PMMA," *Technical Proceedings of the 2004 NSTI Nanotechnology Conference and Trade Show*, p. 4, 2005.
- [23] K. A. Mahabadi, I. Rodriguez, S. C. Haur, J. A. V. Kan, A. A. Bettiol, and F. Watt, "Fabrication of PMMA Micro- and Nanofluidic Channels by Proton Beam Writing: Electrokinetic and Morphological Characterization," *Journal of Micromechanics and Microengineering*, vol. 16, no. 7, p. 8, 2006.
- [24] Y. Gonin, F. Munnik, F. Benninger, and S. Mikhaïlov, "Creating Sub-Surface Channels in PMMA with Ion Beam Lithography in Only One Ste," *Applied Surface Science*, vol. 217, no. 1-4, p. 5, 2003.
- [25] M. B. H. Breese, G. W. Grime, F. Watt, and D. Williams, "MeV Ion Beam Lithography of PMMA," *Nuclear Instruments and Methods in Physics Research B*, vol. 77, no. 1-4, p. 6, 1993.
- [26] R. W. Johnstone, I. G. Foulds, and M. Parameswaran, "Deep-uv exposure of poly(methyl methacrylate) at 254 nm using low-pressure mercury

- vapour lamps," *Journal of Vacuum Science and Technology B*, vol. 26, no. 2, p. 4, 2008.
- [27] M. J. Madou, "Fundamentals of Microfabrication: The Science of Miniaturization," 2002: CRC.p. 752.
- [28] M. Haiducu, "Low-cost microfluidics on commercial grade Poly(Methyl Methacrylate) (PMMA) using deepUV patterning " in *Applied science*. vol. Master of applied science Burnaby: Simon Fraser University, 2009, p. 85.
- [29] Optix® Acrylic Sheet. Available: <http://www.plaskolite.com/productguide/04.pdf>. Access date: March, 10, 2010
- [30] J. S. Greeneich, "Solubility rate of poly-(methyl methacrylate), PMMA, electron-resist " *Journal of the Electrochemical Society* vol. 121, no. 12, p. 3, 1974.
- [31] S. Yasin, D.G. Hasko, and H. Ahmed, "Comparison of MIBK/IPA and water/IPA as PMMA developers for electron beam nanolithography," *microelectronics engineering*, vol. 61-62, p. 9, 2002.
- [32] T.N. Lo, Y. T. Chen, C. W. Chiu, C. J. Liu, S. R. Wu, I. K. Lin, C. I. Su, W. D. Chang, Y. Hwu, B. Y. Shew, C. C. Chiang, J. H. Je, and G. Margaritondo, "E-beam lithography and electrodeposition fabrication of thick nanostructured devices," *JOURNAL OF PHYSICS D: APPLIED PHYSICS*, vol. 40, p. 5, 2007.
- [33] Y. T. Chen, T. N. Lo, C. W. Chiu, J. Y. Wang, C. L. Wang, C. J. Liu, S. R. Wu, S. T. Jeng, C. C. Yang, J. Shiue, C. H. Chen, Y. Hwu, G. C. Yin, H. M. Lin, J. H. Jef, and G. Margaritondog, "Fabrication of high-aspect-ratio Fresnel zone plates by e-beam lithography and electroplating," *Synchrotron Radiation*, vol. 15, p. 6, 2008.
- [34] C. K. Malek and S. Yajamanyam, "Evaluation of alternative development process for high-aspect-ratio poly(methylmethacrylate) microstructures in deep x-ray lithography," *J. Vac. Sci. Technol. B*, vol. 18, no. 6, 2000.
- [35] C. M. Cheng and R. H. Chen, "Development behaviours and microstructure quality of downward-development in deep x-ray lithography," *Journal of Micromechanics and Microengineering*, vol. 11, p. 692, 2001.
- [36] L. Brown, T. Koerner, J. H. Horton, and R. D. Oleschuk, "Fabrication and characterization of poly(methylmethacrylate) microfluidic devices bonded using surface modifications and solvents," *Lab on a Chip*, vol. 6, no. 1, p. 8, 2006.
- [37] C. H. Lin, L.M. Fu, C. H. Tsai, C. H. Chao, and C. W. Lan, "Low azeotropic Solvent sealing of PMMA Microfluidic Devices," *Transducers'05, the 13th International Conference on Solid-State Sensors, Actuators and Microsystemsn, Seoul, Korea*, p. 4, 2005.

- [38] J. H. Li, D. Chen, and G. Chen, "Low-Temperature Thermal Bonding of PMMA Microfluidic Chips," *Analytical Letters*, vol. 38, no. 7, p. 10, 2005.
- [39] Y. Sun, Y. C. Kwok, and N. T. Nguyen, "Low-Pressure, High-Temperature Thermal Bonding of Polymeric Microfluidic Devices and Their Applications for Electrophoretic Separation," *Journal of Micromechanics and Microengineering*, vol. 16, no. 8, pp. 1681-1688, 2006.
- [40] X. Zhu, G. Liu, Y. Guo, and Y. Tian, "Study of PMMA thermal Bonding," *Microsystem Technologies*, vol. 13, no. 3, p. 5, 2007.
- [41] J. S. Rossier, A. Schwarz, F. Reymond, R. Ferrigno, F. Bianchi, and H.H Girault, "Microchannel networks for electrophoretic separations," *Electrophoresis*, vol. 20, 1999.
- [42] W. W. Y. Cjow, K. F. Lei, G. Shi, W. J. Li, and Q. Huang, "Channel fabrication by PDMS-interface bonding," *Smart Materials and Structures*, vol. 15, no. 1, p. 5, 2006.
- [43] Kin Fong Lei, Syed Ahsan, Nasser Budraa, Wen J. Li a, and J. D. Maib, "Microwave bonding of polymer-based substrates for potential encapsulated micro/nanofluidic device fabrication," *Sensors and Actuators A*, vol. 114, no. 2-3, p. 7, 2004 2004.
- [44] J. H. Li, D. Chen, and G. Chen, "Low-Temperature Thermal Bonding of PMMA Microfluidic Chips," *Analytical Letters*, vol. 38, no. 7, pp. 1127-1136, 2005.
- [45] Manoj Gupta, Wong Wai Leong, and Eugene, *Microwaves and Metals*. Singapore: John Wiley & Sons, 2007.
- [46] S. Caddick, "Microwave Assisted Organic Reactions," *Tetrahedron*, vol. 51, p. 29, 1995.
- [47] Ashim K. Datta and R. C. Anantheswaran, *Handbook of Microwave Technology for Food Applications*. New York: Marcel Dekker, Inc., 2001.
- [48] Antonio de la Hoz, A´ngel Di´az-Ortiz, and A. s. Moreno, "Microwaves in organic synthesis. Thermal and non-thermal microwave effects," *Chemical Society Reviews*, vol. 34, p. 14, 2005.
- [49] R. Roy, D. Agrawal, J. Cheng, and S. Gedevarishvili, "Full sintering of powdered-metal bodies in a microwave field," *Nature*, vol. 399, p. 3, 1999.
- [50] K. Saitou, "Microwave sintering of iron, cobalt, nickel, copper and stainless steel powders," *Scripta Materialia*, vol. 54, p. 5, 2006.
- [51] D. A. Copson, "Heating method and apparatus," US Patent 2830162, 1958.
- [52] E. A. Maguire and D. W. Readey, "Microwave-Absorbing ferrite-Dielectric composites," *Journal of the American Ceramic Society*, vol. 59, no. 9-10, p. 4, 2005.

- [53] J. W. Walkiewicz, G. Kazonich, and S. L. McGill, "Microwave heating characteristics of selected mineralas and compounds," *Miner. Metall. Proc.*, vol. 5, p. 4, 1988.
- [54] http://www.ulsinc.com/versalaser/english/PDFs/VLS_Platform_0608.pdf, "Universal Laser Systems," 2009.
- [55] A. A. Kaleibar, M. Rahbar, M. Haiducu, and M. Parameswaran, "Patterning of PMMA microfluidic parts using screen printing process," *SPIE.*, vol. 7593, DOI: 10.1117/12.841345, 2010.
- [56] M. Rahbar, D. Sameoto, and M. Parameswaran, "Microwave-induced, thermally assisted solvent bonding for low-cost PMMA microfluidic devices " *Journal of Micromechanics and Microengineering*, vol. 20, no. 1, 2010.
- [57] C. A. O.-A. s.
<http://www.sdplastics.com/acryliteliterature/1354BOP4UVtransTECHDATA.pdf>, "CYRO Acrylite OP-4 Acrylic sheet. ," 2010.