Fabrication of 3D Microfluidic Devices by Thermal Bonding of Thin Poly(methyl methacrylate) Films

Thesis by
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ABSTRACT

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Paul Said Ehrlich Pérez

The use of thin-film techniques for the fabrication of microfluidic devices has gained attention over the last decade, particularly for three-dimensional channel structures. The reasons for this include effective use of chip volume, mechanical flexibility, dead volume reduction, enhanced design capabilities, integration of passive elements, and scalability. Several fabrication techniques have been adapted for use on thin films: laser ablation and hot embossing are popular for channel fabrication, and lamination is widely used for channel enclosure. However, none of the previous studies have been able to achieve a strong bond that is reliable under moderate positive pressures. The present work aims to develop a thin-film process that provides design versatility, speed, channel profile homogeneity, and the reliability that others fail to achieve.

The three building blocks of the proposed baseline were fifty-micron poly(methyl methacrylate) thin films as substrates, channel patterning by laser ablation, and device assembly by thermal-fusion bonding. Channel fabrication was characterized and tuned to produce the desired dimensions and surface roughness. Thermal bonding
was performed using an adapted mechanical testing device and optimized to produce the maximum bonding strength without significant channel deformation. Bonding multilayered devices, incorporating conduction lines, and integrating various types of membranes as passive elements demonstrated the versatility of the process. Finally, this baseline was used to fabricate a droplet generator and a DNA detection chip based on micro-bead agglomeration.

It was found that a combination of low laser power and scanning speed produced channel surfaces with better uniformity than those obtained with higher values. In addition, the implemented bonding technique provided the process with the most reliable bond strength reported, so far, for thin-film microfluidics. Overall, the present work proved to be versatile, reliable, and fast, making it a good candidate to reproduce several on-chip functions. Future work includes implementing thick-substrate bonding techniques to further improve the process and decrease energy requirements.
ACKNOWLEDGEMENTS

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I also want to express my gratitude to Tayyab Ahmed, at the Central Workshop, who was always helpful with the mechanical design and fabrication of elements for the bonding system. Research scientists at the Advanced Nanofabrication Imaging and Characterization Core Lab, and staff at the KAUST Library also have my appreciation. I’m also obliged to Antonia Forshaw, my Graduate Program Coordinator, for her advice and patience throughout this process.

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## ACRONYMS

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>CA</td>
<td>Cellulose Acetate</td>
</tr>
<tr>
<td>CE</td>
<td>Cellulose Ester</td>
</tr>
<tr>
<td>COC</td>
<td>Cyclic Olefin Copolymer</td>
</tr>
<tr>
<td>CTE</td>
<td>Coefficient of Thermal Expansion</td>
</tr>
<tr>
<td>EMPIRe</td>
<td>Electromechanical Microsystems and Polymer Integration Research</td>
</tr>
<tr>
<td>EOF</td>
<td>Electro-Osmotic Flow</td>
</tr>
<tr>
<td>HAA</td>
<td>Heat Activated Adhesive</td>
</tr>
<tr>
<td>IPA</td>
<td>Iso-Propyl Alcohol</td>
</tr>
<tr>
<td>MEMS</td>
<td>Micro Electro-Mechanical Systems</td>
</tr>
<tr>
<td>MMA</td>
<td>Methyl Methacrylate</td>
</tr>
<tr>
<td>PC</td>
<td>Polycarbonate</td>
</tr>
<tr>
<td>PEEK</td>
<td>Poly EtherEther Ketone</td>
</tr>
<tr>
<td>PDMS</td>
<td>Poly Di-Methyl Siloxane</td>
</tr>
<tr>
<td>PMMA</td>
<td>Poly Methyl Methacrylate</td>
</tr>
<tr>
<td>POC</td>
<td>Point Of Care</td>
</tr>
<tr>
<td>PPI</td>
<td>Points Per Inch</td>
</tr>
<tr>
<td>PSA</td>
<td>Pressure Sensitive Adhesive</td>
</tr>
<tr>
<td>SAR</td>
<td>Split And Recombine</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>μTAS</td>
<td>micro- Total Analysis System</td>
</tr>
</tbody>
</table>
LIST OF SYMBOLS

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO₂</td>
<td>Carbon dioxide</td>
</tr>
<tr>
<td>KOH</td>
<td>Potassium Hydroxide</td>
</tr>
<tr>
<td>P</td>
<td>Laser power</td>
</tr>
<tr>
<td>Pᵇ</td>
<td>Bonding pressure</td>
</tr>
<tr>
<td>s</td>
<td>Laser speed</td>
</tr>
<tr>
<td>tᵇ</td>
<td>Bonding time</td>
</tr>
<tr>
<td>tᶜ</td>
<td>Cooling time</td>
</tr>
<tr>
<td>tₖₑₚ</td>
<td>Compression time</td>
</tr>
<tr>
<td>tʰ</td>
<td>Heating time</td>
</tr>
<tr>
<td>Tᴬ</td>
<td>Ambient or room temperature</td>
</tr>
<tr>
<td>Tᵇ</td>
<td>Bonding temperature</td>
</tr>
<tr>
<td>T₉</td>
<td>Glass transition temperature</td>
</tr>
<tr>
<td>Tₘ</td>
<td>Melting temperature</td>
</tr>
<tr>
<td>z</td>
<td>Honeycomb panel vertical position</td>
</tr>
</tbody>
</table>
Chapter 1

Introduction

The first microfluidic device ever built was a gas chromatograph, which was presented in 1979 by Terry et al. [1]. Although an unparalleled accomplishment, this work was largely ignored until over a decade later. In 1990, Manz et al. [2] described scaling laws and the benefits obtained from miniaturizing systems, which gave rise to microfluidics as an independent field. As of June 2012, There are 239 companies that specialize in microfluidics[3]. These include companies that specialize in point-of-care (POC) diagnostics, foundries, bioMEMS components, or tool fabrication for the life sciences.

Microfluidics, nowadays, is still a broad, disperse and fast-changing field. During the last decade research has focused on both the fabrication and application of microfluidic devices. There is not one fabrication process that is superior; all of them have advantages and disadvantages that make them more suitable for some applications than others. New materials are constantly being implemented, and techniques are being developed, that enhance functionality or reduce device fabrication costs.

The first approach to fabricating microfluid handling devices came from borrow-
ing techniques from the micro-fabrication industry. Silicon and glass were the primary substrates used, but researchers were quick to recognize the need for different materials. Microfluidics aims to solve problems where fast and inexpensive fabrication processes are required, hence, the use of lithography, associated techniques, and cleanroom facilities are not appropriate. A high percentage of research in microfluidics is focused on developing fast and cheap devices for POC diagnostics, although other applications such as sensing, reducing reagent consumption, increasing assay throughput, and DNA replication obtain significant attention as well.

Plastics are the obvious substrate choice for prototyping as well as for mass production of microfluidic chips. Soper et al. [4] summarize the reasons for which plastics are the best suited for this application. The material must be machinable, annealable, transparent, inert to assay conditions, have good thermal and electrical properties, and have a modifiable surface.

1.1 Polymer microfluidics

There are many reasons for choosing polymers over glass or silicon based substrates. Mechanical characteristics make polymeric substrates resistant to rough handling, in contrast to silicon or glass, which easily brake. The more fundamental reasons for choosing plastics are the low cost, ease of fabrication, and biocompatibility. Silicon and glass based microfluidic chips require expensive facilities, highly trained personnel, and handling of toxic substances [5]. These factors add to the cost of manufacturing microfluidic devices. Because it is desirable for chips in biological applications to be disposable, substrate cost is an important consideration [6].
Beside substrate costs being significantly lower, fabrication techniques for polymers tend to be less expensive as well. At first, cleanroom-based fabrication processes were used on polymeric materials, but now various off-cleanroom methods have been developed and improved to reduce costs. Plastics can be molded which gives them an advantage over crystalline substrates, it also enables the use of replication methods. By implementing such methods, production in series can be achieved. It is widely understood that commercial success is defined by a low cost and reliable fabrication baseline [7].

Polymers are commonly divided in two groups: thermoplastics and thermosets. Thermosets can be molten only once and shaped by using a mold; once it cools down it becomes solid and its form cannot be modified any further. On the other hand, thermoplastics can be molded indefinite times, making them the preferred material for microfluidic device fabrication [7].

On different grounds, silicon is not appropriate for some applications due to protein or DNA binding. This can be avoided by coating the channels, but this adds another step and, hence, increases the cost of fabrication [7]. Polymeric materials don’t suffer from this problem to the same extent. Moreover, polymeric materials can be easily functionalized to change properties like protein binding, and hydrophobicity amongst others. There is an extensive variety of polymers, each with different mechanical, optical, electrical, thermal and chemical characteristics. In other words, given the application, there is a polymeric material suitable for it.
1.2 PMMA over other polymers

Many polymers have been researched and used in the fabrication of microfluidic devices. Some examples are poly(dimethylsiloxane) (PDMS), poly(carbonate) (PC), poly(ester) (PE), poly(styrene) (PS), poly(ethylene terephthalate glycol) (PETG), poly(etheretherketone) (PEEK), cyclic olefin polymer (COP), poly(olefins) (PO), poly(imide) (PI), and poly-methyl methacrylate (PMMA). Out of all these materials, PMMA has received attention due to its unique properties. Up till 2008, PMMA was the second most used material in research.

Physical properties

Poly-methyl methacrylate is a thermoplastic, which means it can be molded and shaped many times without losing structural integrity. The physical phenomenon underlying this behavior is known as glass transition. Thermoplastics can reversibly change from a hard and brittle state to a soft and rubbery composition when their temperature is increased. The temperature at which this occurs is known as the glass transition temperature and denoted by $T_g$. The glass transition temperature for PMMA ranges between 105 and 120°C, depending on molecular weight and additives incorporated during polymerization. This characteristic makes thermoplastic materials adequate for molding and replication techniques. In addition, PMMA can decompose into the methyl methacrylate monomer enabling it for reuse, reducing the environmental footprint.

PMMA is the least hydrophobic out of the common plastic materials used, which means water will show the least resistance to flow through small channels when compared to other polymers. PMMA has excellent optical transparency in the visible
and shows only minor absorbance in the UV spectrum\textsuperscript{[9]}. Good optical quality enables the use of optical detection measuring methods, and the potential of UV curing of substances inside the chip is another application.

Thermal behavior of PMMA is optimal for laser ablation. PMMA’s low heat capacity and low heat conductance guarantee a rapidly rise in temperature in a localized manner \textsuperscript{[11]}. It has been reported that PMMA (out of PEEK, COC, PC and PDMS) offers the best laser machining characteristics \textsuperscript{[12]}. A comparison between the physical characteristics of PMMA and other polymers is presented in Table \textsuperscript{[1]}.

Table 1.1: Physical properties of some thermoplastic materials \textsuperscript{[1]}

<table>
<thead>
<tr>
<th>Material</th>
<th>$T_g$ (°C)</th>
<th>$T_m$ (°C)</th>
<th>$CTE$ ($10^{-6}$°C$^{-1}$)</th>
<th>Water absorption (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PMMA</td>
<td>100-122</td>
<td>250-260</td>
<td>70-150</td>
<td>0.3-0.6</td>
</tr>
<tr>
<td>PC</td>
<td>145-148</td>
<td>260-270</td>
<td>60-70</td>
<td>0.12-0.34</td>
</tr>
<tr>
<td>PS</td>
<td>92-100</td>
<td>240-260</td>
<td>10-150</td>
<td>0.02-0.15</td>
</tr>
<tr>
<td>PET</td>
<td>69-78</td>
<td>248-260</td>
<td>48-78</td>
<td>0.1-0.3</td>
</tr>
<tr>
<td>PEEK</td>
<td>147-158</td>
<td>340-350</td>
<td>47-54</td>
<td>0.1-0.5</td>
</tr>
</tbody>
</table>

$T_g$ = glass transition temperature, $T_m$ = melting temperature

$CTE$ = Coefficient of thermal expansion

**Chemical properties**

Organic solvents, particularly acetone, cannot be used to clean PMMA, or used as working fluids in the channels \textsuperscript{[12]}, because they damage the material in a few seconds. PMMA is soluble in ethyl acetate, ethylene dichloride, trichloroethylene, chloroform and toluene \textsuperscript{[13]}. One good use of this characteristic is the ability to bond two pieces of PMMA together by dissolving their adjacent surfaces together.

Surface modifications can overcome intrinsic limitations of PMMA. Such limitations include hydrophobicity and poor electro osmotic flow (EOF) and separation
performance [8]. Chemical characteristics of some polymers, including PMMA, are presented in Table 1.2.

Table 1.2: Chemical properties of some thermoplastic materials [9]

<table>
<thead>
<tr>
<th>Material</th>
<th>Solvent resistance</th>
<th>Acid/base resistance</th>
<th>Visible&lt;sup&gt;a&lt;/sup&gt;</th>
<th>UV&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>PMMA</td>
<td>Good</td>
<td>Good</td>
<td>Excellent</td>
<td>Good</td>
</tr>
<tr>
<td>PC</td>
<td>Good</td>
<td>Good</td>
<td>Excellent</td>
<td>Poor</td>
</tr>
<tr>
<td>PS</td>
<td>Poor</td>
<td>Good</td>
<td>Excellent</td>
<td>Poor</td>
</tr>
<tr>
<td>PET</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Good</td>
<td>Good</td>
</tr>
<tr>
<td>PEEK</td>
<td>Excellent</td>
<td>Good</td>
<td>Poor</td>
<td>Poor</td>
</tr>
</tbody>
</table>

<sup>a</sup> Optical transmissivity

**Biocompatibility**

PMMA substrates are highly biocompatible. As a non-porous amorphous solid, biomolecule adsorption in channel surfaces (hence contamination) is reduced [14]. Moreover, hydrolysis does not occur on poly(methyl methacrylate), and its surface does not react in neutral aqueous solutions. PMMA is also highly biocompatible for long term implantation. All of these make PMMA suitable for working with biological reagents in point-of-care devices.

### 1.3 Methods for micro channel fabrication in polymer substrates

There are two main steps when fabricating microfluidic devices: creating the channel or groove in a substrate, and covering it afterwards. Several methods for performing both steps are discussed in the literature. A brief overview of these methods is dis-
cussed below.

1.3.1 Groove fabrication

Fabrication of microfluidic channels in crystalline substrates was performed with the use of lithographic tools. The transition to polymer substrates consisted of using lithography’s high resolution to create a silicon master, which was then used to transfer the machined pattern onto softened polymeric material; these are termed “replication” methods. Later on, off-cleanroom techniques have been implemented in order to reduce fabrication costs; these are know as “direct” methods.

Replication methods

These technologies are based in the fabrication of a master, which will be used to transfer the pattern to multiple polymer substrates. Lithographic techniques followed by an electroplating step are commonly used to prolong master’s life. Because the expensive step in the fabrication baseline is only performed once, replication technologies offer a low-cost alternative for microfluidic chip fabrication. Surface roughness and surface chemistry impact the de-molding process. Roughnesses under 100 nm RMS and inert surfaces are ideal for good replication [7].

There are design restrictions on the master that are inherent to the process; undercut structures cannot be achieved. Another drawback is that high aspect ratio structures are difficult to achieve with lithographic methods, which contributes to an increase in cost. A limited numbers of substrates can be fabricated with the same master. Kelly et al. [15] reported the production by hot embossing of up to 65 parts with the same silicon master. Direct technologies do not share this impediment.
The basic replication technologies discussed in the literature are injection molding, hot embossing, and casting. The first one is a technique borrowed from the macroscopic world where molten plastic is injected into a mold kept at the melt temperature of the polymer (Table 1.1). The mold is then cooled and the patterned pieces taken out. Casting consists of a similar approach: the master is covered with the liquid form of an elastomer material, commonly PDMS. The material is cured depending on its characteristics such as concentration of polymerizing agent, and molecular weight. Finally, hot embossing is performed by heating the plastic substrate and pressing the master against it. Temperatures above \( T_g \) are required and pressure values depend on the materials elasticity. A summary of general characteristics of replication technologies is presented in Table 1.3, and more extensive description of these methods are present in [7, 8, 10, 11].

Table 1.3: Important details of various molding technologies. Taken from [10].

<table>
<thead>
<tr>
<th>Process</th>
<th>Hot embossing</th>
<th>Injection molding</th>
<th>Casting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Materials</td>
<td>Thermoplastics</td>
<td>Thermoplastics</td>
<td>Elastomers</td>
</tr>
<tr>
<td></td>
<td>Duroplastics</td>
<td>Duroplastics</td>
<td>and epoxies</td>
</tr>
<tr>
<td>Tool costs</td>
<td>Low-medium</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Cycle time</td>
<td>3-10 min</td>
<td>0.3-3 min</td>
<td>min-h</td>
</tr>
<tr>
<td>Forces</td>
<td>High (kN)</td>
<td>High</td>
<td>No forces</td>
</tr>
<tr>
<td>Temperatures</td>
<td>( T_g ) (100-200°C)</td>
<td>&gt; ( T_m ) (150-400°C)</td>
<td>( T_A )-80°C</td>
</tr>
<tr>
<td>Automation</td>
<td>Little</td>
<td>Yes</td>
<td>Little</td>
</tr>
<tr>
<td>Geometry</td>
<td>Planar</td>
<td>Bulk, spherical</td>
<td>Planar</td>
</tr>
<tr>
<td>Min. feature</td>
<td>nm</td>
<td>Some 10 ( \mu m )</td>
<td>nm</td>
</tr>
<tr>
<td>Aspect ratios</td>
<td>5-50</td>
<td>5-50</td>
<td>Around 1</td>
</tr>
</tbody>
</table>
Direct methods

Direct machining methods have gained popularity in the last decade, mainly for prototyping and proof-of-concept device fabrication. These methods are attractive because of short turn-over times, and no requirement of special facilities, but mostly because of their low-cost nature. The only expenses associated with these methods are tool and polymeric substrate costs.

Mechanical computer numerical controlled (CNC) milling has been used to fabricate channels in polymeric substrates. Churski et al. [17] used this method to drill 400-micron (and larger) channels on a five millimeter PET substrate. Feature sizes depend on the diameter of the available drill bits. Another simple approach was presented by Yuen et al. [18] where a desktop craft cutter was used (Xurography) to pattern a double sided pressure sensitive adhesive tape to create the channels. The minimum good quality feature obtained with this process was 200 μm in size. The most attractive feature of the craft cutter method is its low cost. However, this method is not suited for large production schemes.

The most expensive option in direct micromachining is lithographic patterning of resists, such as SU-8. The ease to spin thick resins in one single step and to create high-aspect ratio structures (UV-LIGA), make for a good combination for fabricating microfluidic structures [9]. A 3D microfluidic fabrication baseline consisting of successive lithographic steps was developed by Abgrall et al. [19]. High cost and specialized equipment and manufacturing techniques are required, which represents a drawback for generating low-cost devices.

Laser ablation is one of the preferred methods for direct micromachining of polymer substrates. It is attractive due to its maskless, low cost and ease of fabrication
characteristics. On the other end, it lacks the resolution of methods that make use of lithography’s precision. Feature sizes depend on the diameter of the focused laser beam; they are generally on the 100-micron range although they can be reduced (40 μm) by implementing masks [20].

There are doubts in the scientific community on whether laser machining is a suitable technology for commercial-level production. Cheng et al. and Sun et al. both support using a laser platforms as a tool for scalable manufacturing [14, 21]. From another point of view, using a commercial laser platform has the disadvantage of producing parts in a sequential way limiting its ability for production in series [3, 11].

1.3.2 Groove enclosure

Similarly to channel fabrication, there are two types of bonding: direct and indirect. Indirect methods use an adhesive or some kind of different material to hold two substrates together; they rely on electric interactions. On the other hand, direct methods bring surfaces of the same material into contact and are based on intermolecular entanglement [9]. Reproducing a reliable, efficient and scalable method is one of the reasons mass production of PMMA microfluidic devices is not a reality yet [8, 22].

There are many factors that should be considered prior to selecting the type of bonding for a specific application. The first and most crucial characteristic is bond strength; the finished device should be able to withstand the liquid pressures it has been designed for. Other factors include the chemical stability of the bond, optical transparency (in the case of using adhesives between layers), uniformity and homogeneity of channel profile, and degree of manufacturing complexity [4].
Indirect methods

One of the most popular indirect methods used in the literature is lamination, due to its low tool cost, scalability and high throughput \cite{9}. Some examples are discussed in Roberts et al. \cite{20}, and Klank et al. \cite{12}. A laminator is a device that compresses polymer films between two rotating rods while heating them. Furthermore, there are many available types of lamination films; they are made from different polymers, coated with pressure sensitive adhesives or heat activated adhesives, and also come in various thicknesses.

Other types of adhesive-based bonding techniques exist where multiple step processes are followed to avoid clogging microfluidic channels. Sealing approaches that include other materials, be it adhesive or else, have undesired effects like Taylor dispersion and reduced separation efficiency. This is important when performing capillary electrophoresis \cite{23}. Sun et al. \cite{21} suggested that the use of lamination techniques had undesirable effects such as band broadening caused by the inhomogeneities in the zeta potential. Various research groups \cite{12, 15} agreed that thermal, chemical and electrical properties should be uniform inside the channel to ensure better performance. For example, if the material is the same around the channel profile, stresses due to thermal expansion are homogeneous, as is electroosmotic mobility.

Direct methods

One of the most important advantages of direct thermal bonding is that it produces devices where the channels have homogeneous surface properties. This makes thermal bonding and its variations a desirable method \cite{9, 12}.

Direct surface to surface bonding is not achieved as easily as using adhesives.
First of all, there is no commercial solution to perform bonding processes. Second, temperatures have to be elevated above the glass transition temperature of the substrates, and pressure applied to ensure close contact between surfaces. This type of bonding has to be controlled in a precise way to prevent deformations to the channel profile and in the most extreme cases channel blockage. Many variations for thermal bonding processes have been described in the literature (Kelly et al. [15], Chen et al. [23], Sun et al. [21], Truckenmuller et al. [22], amongst others). A detailed review of thermal bonding studies of the past is presented in Section 1.3. A summary of generalized characteristics concerning different bonding methods are summarized in Table 1.4.

<table>
<thead>
<tr>
<th>Method</th>
<th>Bonding Strength</th>
<th>Bonding quality</th>
<th>Process complexity</th>
<th>Bonding time</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive interstitial bonding</td>
<td>High</td>
<td>Fair</td>
<td>Medium-high</td>
<td>Short-medium</td>
<td>Low-medium</td>
</tr>
<tr>
<td>PDMS-interface bonding</td>
<td>Medium</td>
<td>Low</td>
<td>Medium</td>
<td>Long</td>
<td>Medium</td>
</tr>
<tr>
<td>Lamination film bonding</td>
<td>Medium</td>
<td>Fair</td>
<td>Low</td>
<td>Short</td>
<td>Low</td>
</tr>
<tr>
<td>Thermal fusion bonding</td>
<td>Medium</td>
<td>Fair</td>
<td>Low</td>
<td>Long</td>
<td>Low-medium</td>
</tr>
<tr>
<td>Solvent bonding</td>
<td>High</td>
<td>Fair</td>
<td>Low</td>
<td>Low</td>
<td>Low</td>
</tr>
<tr>
<td>Solvent bonding with sacrificial material</td>
<td>High</td>
<td>Good</td>
<td>High</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Localized welding</td>
<td>Medium</td>
<td>Fair</td>
<td>Medium-high</td>
<td>Medium</td>
<td>Medium</td>
</tr>
<tr>
<td>Surface treatment bonding</td>
<td>Medium-High</td>
<td>Good</td>
<td>Medium-high</td>
<td>Medium</td>
<td>Medium-High</td>
</tr>
</tbody>
</table>
1.4 Laser machining of polymer substrates

Since the 80’s and early 90’s, interactions between laser radiation and organic polymers have been of interest to researchers \cite{24, 25}. It was not until 1997 that Roberts et al. \cite{20} first implemented a UV pulsed excimer laser in the fabrication of microfluidic devices. Since then several research groups have explored the potential of laser ablation by using different wavelengths on various types of polymers with the objective of achieving a rapid, low-cost, and reliable fabrication baseline.

The first approach to understanding laser ablation is to study the physical phenomena that take place during the process. Exposing a polymer to such radiation creates a visible change in its surface that depends mostly on laser and material characteristics. A fundamental aspect of laser radiation is its high intensity, which comes from a high number of same-energy photons concentrated on a small area. The energy associated with each photon is given by its frequency (or wavelength), which also determines the nature of the interaction between polymer molecules and radiation.

Most of laser ablation studies in the literature deal with UV lasers with wavelengths of 248 and 193 nm \cite{20, 24, 25}. However, the most recent trend is to use CO$_2$ lasers due to their lower cost and higher commercial availability \cite{12}. Photoablation mechanisms concerning these two lasers differ phenomenologically. Shorter wavelengths have enough associated photon energy to break bonds between carboxylic groups and the polymer chain \cite{12}; this is called a photochemical ablation. Radiation emitted from a carbon dioxide laser (infrared, 10.6 $\mu$m) cannot break bonds in the same way. The working mechanism of CO$_2$ laser machining is know as photo-thermal ablation, where bonds are broken by the elevated temperature produced by emitted radiation.
The focused CO$_2$ laser output shined on the material surface rapidly rises its temperature [26]. It has been reported that for a 248 nm pulsed laser temperature ranges between 177 and 477°C [27]. PMMA decomposes in three phases which are accessible at different temperatures. These are defined by the three types of bonds formed in synthesizing the poly-methyl methacrylate molecule. The polymer chain is formed by adding the MMA monomer units by a head to tail mechanism. The termination of the macromolecule takes place in one of two ways: head to head coupling, or by disproportionation. In the first method, carbon atoms (head) with a free electron form a covalent bond. On the second, a head is left with a double bond and the other head bonds to a hydrogen atom.

At a threshold temperature of 180°C the head to head bonds break. The second stage of decomposition is reached at 250°C where the unsaturated end groups start “unzipping”. Finally, at 300°C a phenomenon called “random chain scission” takes place (head-tail bonds break) producing significant damage to the polymer’s structure [28]. Products released during the ablation process are the methyl methacrylate (MMA) monomer, carbon dioxide (CO$_2$), and low molecular weight polymeric material (PMMA). Srinivasan et al. [25] also observed that only 18% of the ablated material is MMA, most of it being large chunks of non-fully-decomposed material [25]. It was also found that the amount of MMA produced depends on the frequency of the laser used.

Pulsed [20, 11, 29, 21] and continuous [12, 26] lasers have been used successfully in the fabrication of microfluidic channels. By emitting pulses, the material is allowed to cool down before it is irradiated again, keeping the heat affected zone smaller. It has been shown, however, that pulsed lasers can leave wavy patterns on ablated surfaces [16].
Mathematical models of laser ablation in PMMA

Modeling laser ablation of organic polymers is not a trivial problem and a large set of assumptions need to be made because the underlying physics are of nonlinear nature. This is due to interdependence of variables that are neglected to ease mathematical analysis. Heat loss and dispersion being dependent on beam speed and input power, evaporation rate as a function of temperature, and energy loss by conduction and radiation are all disregarded \cite{11,26}. Assumptions made in the literature include the following:

- Evaporated material is assumed to not interfere with the ablation process.
- The amount of evaporated material is directly proportional to the available heat.
- The laser is programmed to scan the material at a fixed velocity and power specified beforehand.
- The intensity of the laser beam has a Gaussian distribution.

Despite these assumptions and physical dependencies being unaccounted for, the following models obtain results that agree with empirical observations. The first model, developed by Snakenborg et al. \cite{11}, relates channel depth with velocity, power, and the number of laser passes over the same area. Their final equation is:

\[ D = c_1 \frac{P}{v} - c_2 \]  

(1.1)

with

\[ c_1 = \frac{k\alpha}{\rho a}, \quad c_2 = \frac{kQ_{th}}{\rho a^2} \]

where \( P \) is the laser power, \( v \) is the speed, \( \alpha \) is the absorptance, \( \rho \) is the density, \( Q_{th} \) is the heat threshold, \( a \) is related to the spot size, and \( k \) is related to the chemical
binding energy of the polymer.

A different model, proposed by Yuan et al. [26], obtains an expression for both channel profile and depth. The relations is:

\[ D(y) = D(0)e^{-y^2/R^2} \] (1.2)

where \( y \) is the direction perpendicular to the motion of the laser, \( R \) is the radius of the laser at its waist, and \( D(0) \) represents the depth, which is given by:

\[ D(0) = \frac{\alpha P}{\sqrt{\pi}Rv\rho[L + c_p(T_v - T_0)]} \] (1.3)

where \( c_p \) is the specific heat, \( R \) is the laser beam radius at focal waist, \( L \) is the latent heat of decomposition, \( T_0 \) the room temperature, and \( T_v \) the decomposition temperature.

Both models describe the dimensions of the groove obtained by ablating PMMA in terms of the power, speed and physical constants. In the following section the findings concerning channel dimensions and characteristics in relation to these parameters are presented.

**Channel dimensions and their dependence on user defined laser-cutting parameters**

Lasers used for cutting or engraving materials are distributed as a fully integrated platform controlled by a computer. A software interface is usually provided, which permits the modification of the laser output through variables such as: power, speed,
resolution (pulses per inch), number of passes and focal distance. The channel’s physical dimensions and characteristics are dependent on these parameters.

Depth is commonly the dimension that most research groups focused on. Both Snakenborg et al. \cite{11} and Yuan et al. \cite{26} performed experiments to test the accuracy of their mathematical models. It was observed that equation 1.1 predicts depth with a standard deviation of $4 \mu m$ in the ranges of laser speeds and powers of 300 and 1000 mm/s, and 2.6 and 33 W. Similarly, equation 1.3 predicts the linear correlation found experimentally by varying laser power for constant speeds and beam diameter. Other experimental studies found the same relationship between power and depth \cite{21, 12, 30}.

Roberts et al. \cite{20} fabricated channels on different polymers using an excimer UV pulsed laser. Their findings show that the channel depth is proportional to the number of laser pulses. Cheng et al. \cite{14} found that this relationship is not linear and preserves width, in contrast to the relationship between depth and increasing power (which is linear and increases width). Srinivasan et al. \cite{25} found that depth has a linear relationship to laser fluence, which is not a parameter modifiable by users in many systems, but is related to power as well.

Channel width does not get as much attention from the research community as channel depth. Snakenborg et al. \cite{11} observed that channel width can be influenced only to a limited extent. As a final experiment on their work, channels were ablated by using different powers and adjusting the number of passes accordingly. For example, the first channel was etched with $P = 26 W$ and $N = 1$ (number of passes), and another one with $P = 7.5 W$ and $N = 4$, keeping the “total power” constant. Channels fabricated with the lowest powers and highest number of passes showed the
narrowest channels. A study by Wang et al. [30] showed that there is a maximum width value that can be fabricated when varying power. Moreover, the relationship between channel width and power is not linear and resembles a curve that quickly approaches a horizontal asymptote.

The model derived by Yuan et al. [26] obtains a mathematical expression for the channel profile; they concluded the profile has a gaussian shape. A comparison between the model and experimental data was also provided, showing a maximum deviation of 5%. The findings also reveal that channel width is directly proportional to the quotient of laser power and speed.

The gaussian profile of the laser-ablated groove has been verified by several studies [12, 21, 29, 30]. Klank et al. [12] reported that width changed from 150 to 250 μm and in a nonlinear fashion. They concluded that this behavior is due to the low thermal diffusivity of plastic materials. Furthermore, multiple laser passes increased channel width in a linear fashion.

A study performed by Sun et al. [21], found that channel width is inversely proportional to the laser’s scanning speed. They suggested that high scanning speeds mean that each point along the laser’s path is irradiated for a shorter time. When speed is low, every point receives more “radiation time” and hence more energy.

**Bump formation**

It has been observed that laser ablation often produces a bump or bulge around the channels [11, 12, 13]. Several mechanisms have been proposed, including re-solidifying liquid material ejected from inside the groove [11] and re-deposited low
molecular weight polymeric matter. Another proposed mechanism suggests that the temperature of the plastic will increase rapidly when radiation is absorbed. If not enough energy is supplied in order to raise the material’s temperature above the decomposition threshold, only thermal expansion occurs. If the energy is withdrawn rapidly (as it is when using a pulsed laser) the material cools down maintaining the expanded shape \[29\].

In the experiment performed by Snakenborg et al.\[11\] described in the previous section, they found that channel width decreases and bump size is augmented. Material density on the bump should be lower due to breakage of polymer chains without evaporation.

Klank et al.\[12\] suggested that a bigger bump forms at lower speeds due to gas pressure having more time to eject material from inside the groove. The bump is specific to the material and has less density than the unmodified polymer, hence it is softer and easily compressible.

**Roughness of the laser-ablated surface**

It is widely known that laser ablated channels have rougher surfaces than imprinted or molded ones. Particularly, the degree of roughness is highly dependent on the absorption of the polymer at the radiation wavelength \[16\]. Another important factor that contributes to the generation of rough surfaces is bubble development in heated PMMA \[11\].

Klank et al.\[12\] looked at the roughness with a cross section SEM and determined it in the range of 1 to 2 μm when ignoring “solidified protrusions” (ejected material that landed on the channel surface). Excimer UV laser ablation produced rugosities
on the order of 0.13 \mu m for channels deeper than 20 \mu m according to Roberts et al. [20]. The measured value for the unablated polymer’s surface is 0.01 \mu m.

With the objective of correcting rough surfaces, Cheng et al. [14] fabricated a groove using a CO\textsubscript{2} laser and proposed annealing the substrate at high temperatures. After laser ablation, surface roughness is as large as 20 \mu m and after annealing at 170\degree C it has a value of 2.04 nm. It could be expected that PMMA would flow above its \( T_g \), however, polymerization is extensive enough to hold the material in place.

1.5 Thermal bonding

Thermoplastics are the type of polymers (amongst three) that are known to experience changes in their mechanical characteristics above a threshold temperature (\( T_g \)). Upon cooling, they return to their original state without suffering any chemical changes [9]. This behavior makes thermoplastics ideal for molding, embossing and bonding applications, which makes them a good selection for microfluidic applications. In this section different approaches and variants to thermal fusion bonding of thermoplastic substrates for enclosure of microfluidic channels are reviewed.

In order to reproduce thermal fusion bonding between two thermoplastic substrates, heat and pressure need to be applied. Increasing temperature makes the material soft and moldable (above \( T_g \)); applying pressure has the purpose of forcing intimate contact between the two surfaces. Diffusion and interlocking of molecular chains will guarantee a strong bonding if the parameters are chosen correctly [1]. Optimization of bonding processes is necessary and non-trivial to achieve a reliable and reproducible fabrication baseline. Failure to do so can have detrimental consequences.
Too much pressure or heat will close channels or even make them disappear; on the other hand, insufficient values of the mentioned parameters will render the bond as weak and unreliable.

Nanochannels, such as the ones fabricated by Abgrall et al. [31] are particularly shallow and, thus, in great risk of disappearing under an unoptimized bonding process [4]. Accurate control of bonding pressure, time and temperature are key to secure bonding strength as well as acceptable limits to channel deformation. Chen et al. [23] reported channel deformation to be $0.3 \pm 0.07 \, \mu m$. Kelly and Woolley reported a decrease in channel depth of $0.2 \, \mu m$ with their water bonding process. Channel deformation is a risk with un-optimized processes.

So far thermal bonding has only been related to temperature and pressure variables. The time a substrate is exposed to the heat source and pressure is another variable that needs optimization [9]. Parameters used on various thermal bonding techniques found in the literature are presented in Table 1.5. It is highly convenient to have access to a programmable hot press or any adapted device that is controlled by a computer.

**Variations to the thermal fusion bonding technique**

Straightforward thermal fusion bonding has been the most used variation of direct bonding methods. However, there are studies that aim to loosen the requirements to generate strong and reliable bonds. In order to overcome the challenges associated with channel deformation, a trend to lower bonding temperature gained momentum. Experiments performed by Kelly and Woolley [15], and by Chen et al. [23] stand out.
Table 1.5: Parameters used on various thermal bonding experiments found in the literature

<table>
<thead>
<tr>
<th>Group</th>
<th>Method</th>
<th>$P$</th>
<th>$T_b$ (°C)</th>
<th>$t_b$ (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chen et al. [23]</td>
<td>Vacuum</td>
<td></td>
<td>112</td>
<td>60</td>
</tr>
<tr>
<td>Kelly et al. [15]</td>
<td>Water</td>
<td></td>
<td>100</td>
<td>60</td>
</tr>
<tr>
<td>Ford et al. [32]</td>
<td></td>
<td>50 lb weight</td>
<td>150</td>
<td>120</td>
</tr>
<tr>
<td>Sun et al. [21]</td>
<td>Plain</td>
<td>20 kPa</td>
<td>165</td>
<td>30</td>
</tr>
<tr>
<td>Klank et al. [12]</td>
<td>Solvent</td>
<td></td>
<td>85</td>
<td>90</td>
</tr>
<tr>
<td>Truckenmuller et al. [22]</td>
<td>UV</td>
<td>3 MPa</td>
<td>135</td>
<td>10</td>
</tr>
<tr>
<td>Liu et al. [33]</td>
<td>UV</td>
<td>500 psi</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>Zhu et al. [34]</td>
<td>MMA</td>
<td>0.5 - 3 MPa</td>
<td>95-99</td>
<td></td>
</tr>
<tr>
<td>Abgrall et al. [31]</td>
<td>O$_2$ plasma</td>
<td>8.9 bar</td>
<td>85</td>
<td>30</td>
</tr>
<tr>
<td>Cheng et al. [14]</td>
<td>Plain</td>
<td></td>
<td>120</td>
<td>30</td>
</tr>
</tbody>
</table>

The work by Kelly and Woolley proposes conducting the bonding by submerging the clamped substrates in a bath of boiling water. After hot embossing a pattern in a PMMA substrate, the surface was wetted and clamped to a blank substrate; this helped eliminate any air residue present between the two layers. The layers were then submerged in boiling water for one hour after which the devices were strongly bonded.

Chen et al. presented a technique that incorporates vacuum to the standard thermal fusion bonding process. The first advantage of this method is the elimination of air bubbles between substrates which can lower the bonding quality. As a consequence, the pressure needed to bring the surfaces into close contact is no longer needed; Chen et al. achieved a strong bonding without applying any extra pressure at a vacuum pressure of 10 mbar.

A study by Sun et al. [21] proposes the opposite approach to prevent channel deformation during bonding. They demonstrated that low pressures, on the order of 20 kPa, should be used in order to avoid channel or feature deformation. Instead, high temperatures (165 °C) should be implemented to obtain desired bonding strength. This process has been used to fabricate applications such as agent sarin detection in
A slightly different approach was proposed by Ford et al. [32] where the substrates’ surface is heated up to 150°C on a hot plate for five or ten minutes. This method prevents the formation of bubbles in the bulk. After the substrates were removed they were positioned in contact with each other and a 50 lb copper weight was placed on top. The sample was left to cool down at a controlled temperature. The obtained bond was strong enough to endure the operation of the micro pump fabricated in this study.

The use of solvents as a bonding aid is greatly beneficial to bonding strength [9]. When cohesive forces between the molecules of the polymer and the solvent are similar, the dissolution of one in the other is possible. When the surface of a polymer substrate has been in contact with a solvent, the molecules become free and can easily diffuse into another solvated layer. Application of solvents on polymer surfaces should be done very carefully because channels might be blocked. Klank et al. [12] submerged two PMMA substrates in ethanol for 10 mins and compressed them at a temperature of 85°C for 90 minutes. Care was taken to dry the substrates before they were placed in the press to avoid channel clogging.

Surface treatments are becoming widely used to improve bonding quality and also to loosen its requirements. Increasing the surface energy of a polymer substrate helps attain stronger bonds by facilitating interdiffusion of polymer chains on top of enhanced mechanical interlocking. Several surface treatments have been implemented: O₂ plasma [31], UV exposure [22], and MMA monomer [33].

Oxygen plasma and UV treatments work in the same way. Both sources are ca-
pable of breaking polymer chains on thermoplastic surfaces [22]. Degradation of the surface means that a layer of a certain thickness has a lower glass transition temperature (as much as 60K, Truckenmuller 2004, [22]). For this reason, bonding can be achieved at lower temperatures. In addition, the broken chains can mechanically interlock with the other surfaces’ chains or diffuse into the lower density material, increasing bonding strength. Surface modification with the MMA monomer prior to bonding was proved to increase the bond strength in a study conducted by Zhu et al. [34]. They determined that bond strength is proportional to temperature, and pressure.

**Bonding strength**

Ideal conditions lead to a bond that can be as strong as the bulk material [9]. Strength and uniformity are the most critical parameters for judging the quality of the bond. Bond strengths obtained by various research groups in the literature are summarized in Table 1.6. The magnitude of the bond strength can be characterized by three methods: normal stress, sheer stress, and rupture pressure.

**Table 1.6:** Variations of a thermal bonding process and achieved bonding strength

<table>
<thead>
<tr>
<th>Source</th>
<th>Bonding technique</th>
<th>Bonding strength (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chen et al. [23]</td>
<td>vacuum bonding</td>
<td>153</td>
</tr>
<tr>
<td>Kelly et al. [15]</td>
<td>water bonding</td>
<td>130</td>
</tr>
<tr>
<td>Sun et al. [21]</td>
<td>thermal bonding</td>
<td>2150</td>
</tr>
<tr>
<td>Zhu et al. [34]</td>
<td>MMA surface modification</td>
<td>2000</td>
</tr>
<tr>
<td>Cheng et al. [44]</td>
<td>thermal bonding</td>
<td>2068</td>
</tr>
</tbody>
</table>

The first method consists on attaching a fixture to each of the bonded layers and pulling them in an orthogonal fashion. The resulting stress will be calculated as:
\[ P = \frac{F}{A} \quad (1.4) \]

where \( P \) is the pressure or bond strength, \( F \) is the maximum supported load or force before separation of the substrates, and \( A \) is the bonded area. Implementation of a mechanical testing device is preferable \[23\] but not necessary; an alternative used by Kelly and Woolley \[15\] was to bond (solvent assisted) fixtures to top and bottom layers, suspend the device on one end and hang weights on the other end until the device breaks. The sheer stress method very similar but applies forces in a parallel way, (Sun et al. \[21\]). The third test characterizes devices by measuring the maximum pressure a liquid going through it, can have before the bond fails.

### 1.6 Thin-film microfluidics

Thin polymer films have gained popularity in both research and industry as a substrate for microfluidic device fabrication. Polymer foils provide manufacturers with more design freedom and enable new forms of interaction between the inside and the outside of the chip. For example, due to the low heat capacity of polymer substrates, temperature controlled on-chip biologic reactions (such as PCR) are not possible when using a thick substrate. Films with thicknesses in the order of fifty micrometers permit accurate control of temperature in microfluidic channels. Pumps and valves will also benefit from thin-film based fabrication processes; mechanically deformable materials help fulfill design requirements for these type of active elements. Thin films are also pierceable substrates; mass transfer between the device and the user is made possible. Overall, one of the best characteristics provided by using polymer foils is their smaller volume, which means substrate costs are reduced making them good candidates for fabricating disposable devices. In addition, the usage of thin films pro-
duce flexible chips and permit the integration of many on-chip functional elements, unlike planar devices. These advantages are thoroughly detailed in the review paper by Focke et al. [36].

Chen et al. [8] stated that channels are usually fabricated by creating grooves on PMMA substrates and covering them afterwards. Thin-film processes slightly diverge from this idea. Channels are cut through each layer of material and two additional substrates are bonded to its top and bottom; channel depth is defined by the film thickness. This way it is possible for channels to interact with others in adjacent layers through an extended area. This topology is used by several other groups [18, 37, 38, 39, 40, 41].

Previous work

The combination of various fabrication techniques gives rise to fabrication methods that differ from every other with regards to functionality, applications, durability and cost. An overview of the most significant fabrication methods is described below.

The first thin foil fabrication baseline was reported in the literature by Martin et al. [37]. Channels were laser ablated out of a 125-micron polyimide substrate and covered by using intermediate layers of heat-activated adhesive (Monokote). Metal lines were also incorporated to the fabrication scheme providing measuring capabilities. The fabrication of a 15-layer chromium detector and a three-layer micro-dialysis chip using their process was demonstrated.

Wang et al. [30] used a similar approach but they patterned a biocompatible adhesive layer instead. They demonstrated three-dimensional microfluidic structures
by bonding three of the adhesive layers together and covering top and bottom with two blank polymer films. Excluding the design, the rest of the fabrication steps can be carried out in less than half an hour.

A different method for patterning thin films is using an automated blade or digital craft cutter. This device is analogous to a printer or to a laser cutting platform in the way it communicates with the controlling computer. Yuen et al. [18] cut channels and through holes on transparencies and pressure sensitive adhesive (PSA) layers that are positioned alternatingly one on the top of the other. The channels walls were formed by the thickness of the adhesive layer, and top and bottom, of transparency material. Demonstration of the technique was achieved by fabricating serpentine channels and a concentration gradient generator. Bartholomeusz et al. [41] fabricated channels using the same mechanism but enclosure was performed by lamination. No three-dimensional characteristics were presented in this study.

Malek et al. [40] used a Ti:Zaf femtosecond laser to cut features in a three layer stack of SU-8 polystyrene and polyethylene. The layers were laminated together at 65°C and at a pressure of one bar. The obtained bond was weak, which is a great disadvantage, but the fabrication of 3D structures was possible.

Weigl et al. [38] uses nearly the same adhesive-between-layers approach as [37]. The material chosen was Mylar, which was laser ablated with a CO₂ laser. The stack was formed by alternating plain Mylar and adhesive coated layers, and was compressed between two rollers to perform bonding.

The use of adhesives or other materials for bonding is important to the functionality of the device. Mechanically, different residual stresses may induce bending of
the substrate, specially because the used substrates are thin films. In addition, inhomogeneities of the chemical, optical and electrical kind are important [3]. Unless a particular application requires the adhesive material for additional purposes than holding films together, thermal bonding is a better alternative.

The method proposed by Wang et al. [39] is based on printing a wax mask onto a poly(amide) substrate and chemically (KOH) etch the open areas. The achieved features are lines of 170 µm in width and a minimum separation of 70 µm between features. The implemented method is capable of printing and both sides of the polymer substrate with an alignment error of less than 100 µm. The maximum aspect ratio that can be fabricated with this method is three. Layers were bonded by thermal fusion bonding of deposited wax at 80°C for three minutes. A maximum bond strength in the order of 10 kPa was reported. Because the device is unable to withstand high pressures, the driving method for liquid is negative pressure applied at the output of the chip. This device cannot be used in high temperature application because wax would melt, ruin the bonding and maybe clog the channels.

A technique that uses lithography was presented by Abgrall et al. [19]. The process starts with a glass or silicon wafer where a multilayered lithography process is performed as the first step. If the application requires it, depositing metal lines is a possibility. The next step was to laminate a photopatternable dry SU-8 film. This last layer can be exposed and developed, which will define the next layer in the process. Repetition of the last step can be performed as many times as desired to fabricate a 3D microfluidic structure. From all the thin film processes, this is the most accurate and has the best resolution; it can reproduce ten micron features, produce high aspect ratio structures, and has the potential for good alignment. Other methods usually deal with larger features so alignment is not a concern. The approach suggested
by Bartholomeusz et al. where alignment pins are used to prevent layers from moving is a possible solution to this problem. Cost, time, and process control and uniformity are some of the main drawbacks of the present technique.

Roll embossing of COC and PMMA was performed to fabricate channels in films by Metwally et al. Two different sealing methods were implemented. A thin SU-8 dry film was laminated on top of the open channel structure. The bonding parameters were 65-75 °C, a pressure of 2 bar and a speed of 1 m/min. Strength of the bond was not mentioned.

One of the most successful methods on scaling up microfluidic devices is the fabrication of paper-based chips. The process is to submerge pieces of paper in SU-8 (or wax) which are then exposed and developed. This leaves only paper where the resin is removed. The wicking force of the paper is sufficient to transport the liquid through the channels and perform the designed assay. An alternative method to SU-8 is to use a wax printer, and do everything else the same.

The patterning/deposition of electrodes mentioned by Wang et al. greatly increases the applications of any of the fabrication baselines described here. Several techniques are available, starting from the most basic ones: sputter deposition, inkjet printing, laser cutting of a thin metallized foil, and using conductive ink mixed with elastomeric material. Another way to add to the versatility is to recur to heterogeneous material integration. Due to the topologies achievable by stacking thin films, adding different elements to the chip is a possibility.
1.7 Objectives and Contributions

The most prominent methods found in the literature for 3D thin-film microfluidic device fabrication were described in Section 1.6. The present work aims to establish and characterize a fabrication baseline by using CO₂ laser ablation and direct and unassisted thermal fusion bonding of 50-μm thick poly(methyl methacrylate) films. Based on a detailed literature review, no other research group uses the exact same fabrication process as proposed in this work. The concise objectives of this project are enlisted below.

- Characterize the laser-cutting process of thin poly(methyl methacrylate) films
- Demonstrate that using different combinations of laser power and speed, similar channel dimensions are obtained but surface roughness can be improved.
- To establish the use of Instron’s mechanical testing device as a platform for thermal bonding of polymer substrates.
- Optimize the bonding process and characterize the bond between thin poly(methyl methacrylate) films.
- Fabricate working 3D microfluidic devices using the proposed fabrication baseline
- Exemplify the versatility of the fabrication process by incorporating non-PMMA materials as passive elements
Chapter 2

Fabrication of Microfluidic Channels on Thin Poly(methyl methacrylate) Films

Polymer thin-film gaskets were fabricated using a PLS6.75 platform from Universal Laser Systems, Arizona, USA. It operates a carbon dioxide (CO$_2$) laser whose position is defined by mirror-guided lasers. The workspace is a rectangular area of 813 by 457 mm supported by a honeycomb panel that can be displaced up to 64 mm along the direction perpendicular to the ground. The pulsed CO$_2$ laser has a wavelength of 10.6 $\mu$m, a spot size of 127 $\mu$m and a gaussian profile. The laser head is controlled by proprietary software that allows the user to modify the output power ($P$), speed ($s$), Points Per Inch ($PPI$) and height ($z$) of the work area. The operating ranges of these parameters are shown in Table 2.1. Power and speed are defined as a percentage of the maximum values through the software. From now on these quantities will be represented in such way for ease of presenting data and its discussion.

The platform is calibrated such that the focal point of the laser optics is on the
Table 2.1: Operating ranges of modifiable parameters of the PLS6.75

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Operating range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power $(P)$</td>
<td>0 to 75W</td>
</tr>
<tr>
<td>Speed $(s)$</td>
<td>0 to 300mm/s</td>
</tr>
<tr>
<td>Height $(z)$</td>
<td>0 to 64mm</td>
</tr>
<tr>
<td>Points Per Inch (PPI)</td>
<td>10 to 1000</td>
</tr>
</tbody>
</table>

plane defined by the honeycomb panel when its position is $z = 0.0$ mm. Changes in the $z$ direction allow for introducing objects of different thicknesses and/or to de-focus the laser. If out of focus, the energy density that acts on the surface of the material is lower, hence, changing the characteristics of the ablated surface. A mathematical approach that relates the user-modifiable parameters and the outcome of the laser-cutting routine was presented by Snakenborg et al. [11] and Yuan et al. [26]. From their work it can be identified that an increase in power increases the energy received by a portion of the material, and conversely, increasing speed cause such quantity to decrease.

Universal Systems’ laser platform works analogously to a printer. It is connected to a computer equipped with CAD design or drawing solutions. Once the patterns are drawn, they can be transferred to the laser-cutter by the "Print" command. After automatically importing the drawing, the software permits repositioning in addition to defining the laser output parameters.

50 μm-films of impact modied poly(methyl methacrylate) which contains a small percentage of elastomer material were purchased as 600x2000 mm single foils from Goodfellow Cambridge Limited, Cambridge, UK. As preparation, thin films were cleaned with compressed air and taped onto the honeycomb panel to avoid movement during the cutting process. Failure to place the film in a completely flat fashion
caused the ablated channels to have different characteristics along their length due to part of the film moving out of the focus range.

2.1 Experiments

In order to find the parameters most appropriate to fabricate channels in PMMA thin films, it was necessary to characterize the channels generated by different laser powers and speeds. Throughout the experiments the number of pulses per inch \((PPI = 1000)\) and vertical position \((z = 0.0 \text{ mm})\) were kept constant. The justification of the latter is that this number is the closest to the film’s thickness \((0.05 \text{ mm})\) and the laser should be focused on its surface. Regarding the constrained pulses per inch, the objective was to fabricate a channel with straight walls and a smooth wall surface. The 127 \(\mu\text{m}\) spot fits 200 times in one inch, which is the minimum \(PPI\) value that generated a continuous array of orifices: a channel. When higher numbers were used, the pulses overlapped each other reducing the number and size of dents creating a straight wall. Hence, \(PPI = 1000\) (the permitted maximum), was used for channel fabrication.

As a first approach laser speed was kept at 100% while the laser power was varied through almost the complete range allowed by the software. Setting the speed at 100% is aligned with the objective of achieving short prototyping times. The set of laser-power values tested can be divided in three groups defined by the cut-through threshold. Laser powers below 17% didn’t cut through the film properly (at 100% speed); this defined the first interval. The second interval ranged from 18 to 25% where power is increased by 1%. Higher resolution was needed in this interval because it was expected that the target width would be obtained within such values. Finally, the third interval ranged from 30 to 100% with a ten percent step per channel.
Channel widths produced by higher powers was measured for completeness. A total of 23 channels were fabricated.

A second experiment was performed where both power and speed were changed while maintaining a constant ratio between their assigned values. According to both Snakenborg et al. [11], and Yuan et al. [26], the output energy is directly proportional to $P/s$. As long as this quotient is kept the same, the energy supplied by the laser is the same. This experiment was similar to the one conducted by Snakenborg et al., [11]; where they conserved the same overall power by adjusting laser power and number of passes. Three ratios were chosen: 1, 0.5 and 0.25. Four channels per ratio were fabricated. The expectation for this experiment was to obtain similar channel widths but to observe a change in the quality of the surface generated by the ablation process. Lower speeds should distribute energy more uniformly and, thus, produce smoother surfaces. Fabricated channels were observed by Scanning Electron Microscopy (SEM) and optical microscopy.

To complete the characterization of both sets of fabricated channels the physical dimensions of the bumps were measured. This was done using the profile meter, as in the first experiment, and analyzing images obtained through optical microscopy and SEM.

### 2.2 Characterization of channel profile

All the measurements were performed using a profile meter by Veeco, New York, USA, model Dektak 150. The parameters used to obtain the data are shown in Table 2.2. The profiler output of a laser-ablated channel and the place where the cursors
were placed to conduct the measurement are illustrated in Figure 2.1. Definition of top and bottom channel width is shown in Figure 2.1a. It is important to note that the values obtained for bottom width are affected by the physical characteristics of the profiler’s stylus: slope and/or tip diameter. The determined spots for measuring bump size are shown in Figure 2.1b. First the outer cursor (with respect to the channel) is placed on the boundary of the heat-affected zone. Secondly, the inner cursor is placed on the other side of the bump where the profiler marks the minimum vertical difference; the horizontal difference is the bump width. For the height, the outer cursor remains in its position and the inner one is moved to the highest vertical difference.

Table 2.2: Profiler parameters used to characterize laser-cut channels

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measuring range</td>
<td>524 μm</td>
</tr>
<tr>
<td>Stylus force</td>
<td>to 3.0 mg</td>
</tr>
<tr>
<td>Profile</td>
<td>Hills and Valleys</td>
</tr>
<tr>
<td>Stylus type</td>
<td>12.5 μm</td>
</tr>
<tr>
<td>Scan resolution</td>
<td>0.444 μm/sample</td>
</tr>
</tbody>
</table>

The uncertainty associated with the profilers measurement is of no importance to this study because it is much smaller than the error associated with placing the cursors in the right location. Channel width was measured four times at different points along it and an average was calculated. These results are shown in the following sections.

Results

Data obtained for top and bottom widths of laser ablated channels at constant speed and increasing power is illustrated in Figure 2.2. It can be recognized that the relation
between channel width and power is not linear. The behavior expected from the data is that of a curve approaching an asymptote, which is related to the diameter of the laser and the characteristics of the material that define the heat-affected zone. These results are phenomenologically similar to those observed by Sun et al. [21], Klank et al. [12], and Wang et al. [31].

The maximum width obtained was 243.7\(\mu\text{m}\) by using 100\% power. The minimum power that generated a completely cut-through channel was 19\% and the top width associated with it is 180\(\mu\text{m}\). Data points obtained for powers between 20 and 30\% stray away from the general behavior of the rest. The measurements for these channels were performed close to the extremes of the groove, where the energy per unit length is higher due to acceleration (and deceleration) of the scanning head. A detailed explanation is presented in appendix A.

The same relationship between bottom width and laser power is illustrated in Figure 2.2b. Due to the geometry of the profilers stylus’ tip, measurements performed on bottom width do not represent the real dimension. The stylus has a 12.5 \(\mu\text{m}\)
Figure 2.2: Width of the channels (a) top, and (b) bottom, generated by different values of laser power at a 100% speed radius and a 45° slope. For a 50 \( \mu \text{m} \) thickness only values greater than 100 \( \mu \text{m} \) can be measured. Film thickness was also measured using the profiler and its value is 68.0 \( \mu \text{m} \). This is the reason for the bottom width showing a null value for powers of 11% and under.

2.3 Effect of laser speed on channel smoothness

The values assigned for power and speed to fabricate the different channels are summarized in Table 2.3. Twelve channels were fabricated overall, four channels per power to speed ratio. Channels were characterized using two methods. First, their dimensions were measured using the same method described above to verify that they don’t diverge. Second, SEM imaging was used to have an accurate image of channel roughness.

It was found that for every ratio the lowest channel width is generated by the lowest power/speed pair. For example, \( P = 100\% \) and \( s = 100\% \) yield a top width of 302 \( \mu \text{m} \) while \( P = 1\% \) and \( s = 1\% \) yield only 249 \( \mu \text{m} \); there is a 53 \( \mu \text{m} \) difference. For
ratio $P/s = 0.5$ this difference is $22 \, \mu m$, and for $P/s = 0.25$, $10 \, \mu m$. The complete information regarding channel size characterization is illustrated in Figure 2.3.

Table 2.3: Power and speed pairs chosen...

<table>
<thead>
<tr>
<th>Ratio</th>
<th>Power (%)</th>
<th>Speed (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>12.5</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>0.25</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>12.5</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>

Figure 2.3: (a) top and (b) bottom channel width generated by different values of laser power at a 100% speed

SEM images of all twelve channels, previously coated with a five-nanometer layer of gold, were generated at KAUST’s Core Lab facilities. Magnifications of 250, 500 and 1000 were used. All obtained images are shown in appendix C. The channel wall
generated with with $P = 100\%$ and $s = 100\%$ is shown in Figure 2.4. It can be noticed that besides little empty pockets seen on the surface there are large ”craters” measuring around 50 $\mu$m.

![SEM image of channel wall ablated with $P = 100\%$ and $s = 100\%$](image)

**Figure 2.4:** SEM image of channel wall ablated with $P = 100\%$ and $s = 100\%$

It was found that the number of large “craters” decreased with power/speed ratios. The density of small holes is also decreased. The wall of a channel ablated with $P = 1\%$ and $s = 1\%$ is depicted in Figure 2.5. Such is the case for the channels in every group; the lower power/speed ratios generated the least number of big holes or surface defects. A comparison between ratio 1 ($P = 100\%, s = 100\%$), ratio
0.5 \((P = 50\%, s = 100\%)\) and ratio 0.25 \((P = 25\%, s = 100\%)\) is shown in Figure 2.6.

![SEM image of the channel wall ablated with \(P = 1\%\) and \(s = 1\%\)](image)

**Figure 2.5:** SEM image of the channel wall ablated with \(P = 1\%\) and \(s = 1\%\)

On the lower end of output energies used is the channel ablated with \(P = 1\%\) and \(s = 4\%\), which belongs to the 0.25-ratio group, there were no large holes or craters found on the surface, as can be seen in Figure 2.7. The largest holes measured 10 \(\mu m\). For all fabricated channels the lowest surface roughness was obtained by the lowest power/speed ratios.

When power is delivered to the material in short time intervals, temperature is
Figure 2.6: SEM images of channel walls ablated with different power to speed ratios

(a) P: 100% s: 100%
(b) P: 50% s: 100%
(c) P: 25% s: 100%

Figure 2.7: SEM image of the channel wall ablated with $P = 1\%$ and $s = 4\%$
risen abruptly and can cause large chunks of lower weight polymeric material to be ripped from the polymer and ejected [23]. This could be the cause of the large craters appearing in Figure 2.4. When the scanning speed is low each zone in the polymer is given enough time and a stable supply of energy to raise the temperature and slowly decompose (thus avoiding large craters). Bubbles generated in the material might account for the smaller porosity observed in all the samples [11]. Although surface roughness values obtained here are worse than those found in the literature, more information regarding the ablation process was gained.

Optical microscopy was also used to analyze channel surfaces. It was found that the lowest laser speed produces straighter walls. Channels generated with 100% speed versus 1% for P/s ratios of 1 and 0.25 are compared in Figure 2.8. For (a) and (c) it can be seen that channel walls are not straight as in channels (b) and (d) that were ablated at 1% speed. In general, channels fabricated with high speeds tended to have jagged edges and protrusions on its walls, whereas lower speeds produced grooves with higher uniformity and better surface roughness.

The evidence gathered from Figure 2.8 proves that not only do lower speeds produce softer surfaces, they also result in better removal of polymeric material. Both surface roughness and wall structural homogeneity are factors that can impact a device’s performance; jagged edges may act mechanically as traps for molecules or beads, and surface roughness plays a role in surface wettability.
2.4 Bump formation and characterization

The bump generated on the side of the channels was measured using the same technique explained in Section 2.2. The points were cursors were placed to measure bump height and width are illustrated in Figure 2.11.

The dimensions of the bump depended on the values assigned to laser power and scanning speed. The height and width of the bump measured on the same channels as the first experiment are shown in Figure 2.9. Both dimensions decrease with decreasing power although for lower powers the results were not very consistent, specially for channel width.
SEM images of channels (500x magnification) revealed some information on the structure of the bump. The inside channel wall and the bump generated on the opposite side are shown in Figure 2.10. The bump structure in (a) is not smooth; the width is not consistent and a high number of craters can be seen. On the other hand, the bump in (b) is more uniform and there are no big holes on it. The difference between these channels was the power and speed at which they were fabricated: for (a) P = 100% and s = 100% were used, for (b) P = 1% and s = 4%.

The presence of craters in the bump area indicates that material was ablated from that region, which contradicts the theory of it being formed by resolidified ejected material [11, 12]. Li et al., [29] suggested that the bump is generated by thermal expansion of the material caused by temperatures below the decomposition temperature. On the outer edge of the gaussian intensity distribution, temperatures will not be elevated as much as in the center. Particularly, with a low scanning speed the bump showed the same results as in the channel walls, no big craters but a more uniform surface and width. The bump surrounding the channel should be generated
Figure 2.10: SEM images showing the channel wall and the generated bump for different values of power and speed

in the same way Li et al. [29] fabricate polystyrene molds.

Figure 2.11: [a] height and [b] width of bump generated by different power to speed ratios

Bump characteristics were also measured on the channels used for the second (surface roughness) experiment. It was found that height and width become larger when decreasing speed and readjusting the power to maintain the same $P/s$ ratio. The maximum height value was found to be greater than 20 $\mu$m for all three ratios fabri-
cated. Regarding the width, the bump for $P/s = 1$ at the lowest speed tested, had a value of 115 $\mu$m, 86 and 78 $\mu$m corresponded to $P/s = 0.5$ and 0.25, respectively. These results are in agreement with the hypothesis presented in [29] that a slow supply of energy causes the material to expand without vaporizing. The material later cools down without completely loosing its increase in size but compromising density and, hence, mechanical integrity.

2.5 Summary

Channels ablated with $s=100\%$ and powers covering the full operational range of the Universal Laser Systems platform were characterized. On a second experiment, it was demonstrated that smoother surfaces can be fabricated by reducing laser power and scanning speed. The size and characteristics of the bump were measured and its relation to laser parameters, analyzed. Optimal parameters for fabrication of microfluidic channels on thin PMMA films were determined to be $P=1\%, s=4\%, PPI=1000$, and $z=0.0$ (for a single layer).
Chapter 3

Thermal Bonding of Thin Poly(methyl methacrylate) Films

Thermal fusion bonding is one of the pillars of the fabrication baseline proposed in this work. It provides the final device with a uniform cross section and high bond strength, which leads to device reliability. The establishment of a method that includes plain thermal fusion bonding is only the first step in the thin-film processing techniques at the EMPIRe Laboratory, KAUST; further improvements will be made to increase the quality of manufactured devices, without relaxing the requirement for low costs. This chapter describes the steps taken to optimize the thermal bonding process and characterized the bonding produced between thin poly(methyl methacrylate) films.

Apparatus description

The device used to apply pressure on the films is the 5966 Electromechanical testing suite by Instron, USA. The instrument features a crosshead that is able to move vertically with an accuracy in the micrometer range. Attached to this crosshead is the
load cell, responsible for gauging forces. Different load cells can be used depending on the desired measurement range. Overall, the system has a capacity of exerting 10kN on a sample. The physical dimensions of the testing space are 1756 by 400 mm. A temperature controlled chamber provides the capability of performing tests at various temperatures. The model owned by the EMPIRe Laboratory is the 3119-506 “Heatwave”. It’s operating range is from -70 to 350°C.

The 5966 is equipped with a panel for manual control although its main operation is through a software solution provided by the vendor. Programs can be customized for the specific kind of test that will be performed. Unfortunately, the furnace is operated through a different controller.

The mechanical testing suite does not include proper add ons to perform compression of the film stack. The required pieces were designed and fabricated in KAUST’s mechanical shop. The only requirements are that they have a flat surface and that, together, are longer than the furnace’s height. A picture of the fabricated compression rods and the ready-to-bond system is displayed in Figure 3.4a.

3.1 Important considerations and preparations

One of the important characteristics of PMMA is its good optical quality. It is rare that machined metal has a smooth enough finish that preserves the polymers’ see-through surface. Two glass slides were cut to match the size of the films and placed on both sides of the stack, preventing any possible pattern transfer onto the outer layers of the stack.
Even though the load cell is not inside the furnace during the bonding process, its temperature rises due to heat conduction on the metal rod that compresses the films. Changes in temperature have an effect in the load cell’s measurements. Compression load will increase with rising temperature and the inverse is also true. Two mechanisms are responsible for this measurement variation: temperature dependent mechanical characteristics of the measuring element and thermal expansion of the metal rod that compresses the film stack. The change in load measurement with no real load applied on the system is illustrated in Figure 3.1a.

![Figure 3.1:](image)

(a) Load vs temperature  
(b) Chamber temperature vs substrate temperature

**Figure 3.1:** (a) shows the dependence of load measurements on temperature while no real load is applied. (b) shows the difference between PMMA temperature and chamber temperature.

Another important consideration is that the temperature measured by the furnace’s controller accounts for air temperature only. Polymers and, particularly PMMA, have low heat conductivity and capacity. PMMA temperature will not rise as high or at the same rate as chamber temperature during the bonding process. Two pieces of thick (2.4 mm) PMMA were engraved with a cavity in the middle where a thermocouple was placed (silver ink was added to ensure appropriate measurement). Such polymer stack was subject to a standard bonding process. The difference between
the two measured quantities is shown in Figure 3.10. None of the studies cited in this work’s literature review (summarized in Table 1.5) mention this phenomenon. The temperature they achieved on the plastic depended on the method used to heat it.

### 3.2 Bonding process

#### Cleaning

To obtain good bonding quality in terms of uniformity and hence, strength and durability, the films that compose the final chip must be pristine clean. After the laser cutting process dust particles, grease and re-deposited PMMA rest on the film’s surface. Out of the common solvents used in micro-fabrication, acetone quickly damages the material unlike isopropyl alcohol (IPA) or ethanol. IPA was chosen because it evaporates faster.

The films were submerged in IPA in an ultrasonic bath for ten seconds after which they were left to dry between two pieces of lint-free laboratory cloth. Although clean-room facilities are not necessary to ensure acceptable bonding standards, special attention should be put on preventing dust particles from landing on the films. Too many particles on the films will lead to poor contact between layers which will surely translate into a weaker bonding and leakage spots.

#### Bonding recipe

The bonding process consisted of four steps: pre-heating, heating, bonding, and cooldown. The parameters controlled by the user were temperature \((T)\), defined through the furnace’s controller, compression time \((t_{comp})\) and load \((L)\), defined through the
software. This bonding recipe is very similar to the ones found in [33] with the exception that in our process temperature was not constant at any moment.

**Pre-heating** The furnace was heated to a temperature of 45°C for ten minutes. This is mostly to maintain uniformity between the bonding processes when they were performed one after the other.

**Heating** Temperature is raised to the desired set point ($T_b$). Compression should not take place before heating to avoid bubbles caused by trapped expanding air between the films.

**Bonding** The film stack was compressed according to the parameters set in the control software ($P$ and $t_{comp}$).

**Cool-down** Heating was stopped and a blower connected to the furnace was turned on. The sample was cooled down still under compression to avoid thermal stress defects. The sample was removed when the chamber temperature was 50 °C.

![Figure 3.2: Temperature and pressure profiles of the bonding process](image)

The ten kilonewton load cell was used for all bonding runs together with top and bottom metal rods and a thick slab of PDMS that helped distribute pressure evenly.
Instron’s control software records load, extension and time measurements. The displayed information can be customized; in the default configuration load vs. extension and load vs. time charts are displayed.

### 3.3 Optimization of the bonding parameters

Temperature, load and bonding time are the three parameters that need to be optimized to achieve the best quality of bonding and the least damage to structures present in each of the films. The procedure to find the best value of those three variables is to bond thin polymer films varying their value each time; the bonding strength will be tested afterwards revealing which of them produced better results.

Two thin PMMA films were bonded to each other using different values of temperature, load and bonding time. The films were laser cut with the pattern shown in Figure 3.3. The rectangles measured five centimeters in width by seven and a half centimeters in height. The film was divided in three section joint at the bottom; only the middle fringe, which was 2.5 cm in width, was peeled off. A test pattern was laser cut into the film. The circles measured one and five millimeters in diameter, respectively. The height of the vertical line was three centimeters. One of the glass slides (top) used to compress the films was modified from its rectangular shape; it’s shape is shown in Figure 3.3a. The objective of including the mentioned features is to observe any possible deformations caused by the process. Large deformations could define the upper bound of bonding parameters. When the bonded area is curved, the resistance offered to the peeling force augments gradually (because of the increase in area). This prevents film from breaking at the bonding interface.
Figure 3.3: (a) Layout of laser-cut layers that were bonded with different values of $L$, $T_b$, and $t_b$ to perform a 90° peel test, and (b) modified glass slide used to define the bonded area.

In the first experiment load ($P$) and bonding time ($t_b$) were kept at values of 850 N and 30 min, respectively. Temperature varied from 125 to 170°C in increments of 5°C (165°C was omitted). Results are presented in Figure 3.7a. Bonding time varied from 15 to 40 minutes in 5 minute increments. The values of the temperature and load were kept constant at 155°C and 850 N. Finally, bonding time was kept constant at 35 minutes and three (at different temperatures) different ”load” batches were produced. Loads used were 200, 350, 500, 700 and 850 N, all at temperatures of 145, 150 and 155°C.

During the testing of the first fabricated devices it was evident that shear stresses were not an issue for the produced bond; compact and thin devices are not usually subject to shear stress. However, it was noticeable that it was rather easy to peel the films off of each other. A ninety degree peel test was the selected method to gauge bonding quality.

The peel test setup is depicted in Figure 3.4b. The one kN load cell is used in order to reduce instrument uncertainty in measurements. This uncertainty was declared to be 0.05% of the measurement and 1/500th of the total cell capacity by the
manufacturer. Attached to the load cell is a 500 N pneumatic grip by Instron, USA, and on the bottom the 90° peel fixture. The latter consists of a one-degree-of-freedom horizontal surface held in place by a metal pin in the bottom, which is able to slide back and forth in a direction parallel to the ground. This surface holds the sample in place while part of it is peeled away. A metal cable (blue) constrains the motion of the metal plate to the vertical displacement of the crosshead. This constraint ensures the peel angle is always ninety degrees.
Results

An example of the plots obtained during the 90° peel test are shown in Figure 3.5. On the left, load versus peel extension is plotted for a bonded pair where the middle fringe was completely peeled off. Two depressions are observed: the first one between 26 and 28 mm, and the second between 44 and 49 mm. These account for the two circular patterns cut out of the bonded film. The plot to the right (Figure 3.5b) belongs to a sample that was not completely peeled off due to high bonding strength. When the crosshead tried to separate the films they resisted this motion, and in the end the piece of PMMA held by the grip broke off. The bonded region stayed intact as shown in Figure: 3.6c. After this point the measured load was null. The loads obtained during the tests remained in the lower part of the load cell’s operating range, they did not exceed 30 N. According to the manufacturer the uncertainty for all of them is 2 N.

![Plots of load vs peel extension](image)

(a) $L=700$ N, $T_b=145^\circ$C, $t_b=30$ min 
(b) $L=500$ N, $T_b=155^\circ$C, $t_b=30$ min

Figure 3.5: Sample of peel test plots, load vs peel extension. [a] shows the type of plot obtained when film is completely peeled off, and [b] shows the plot of a film so strongly bonded that breaks instead of de-bonding.

Pictures taken of samples in the temperature batch are displayed in Figure 3.6. In subfigure [a] the peeled middle fringes for every sample, organized in order of ascending $T_b$ are shown. The opaqueness of the bonded area increased with higher
Figure 3.6: (a) Peeled middle fringes for all temperatures (b) Bottom film after peel test. $T=145^\circ C$ (c) Bonded films after peel test. $T=155^\circ C$

**Figure 3.6:** (a) Peeled samples of the temperature batch. Films from the whole interval are shown. (b) shows the peeled base of the $145^\circ C$ sample. (c) $155^\circ C$ sample after peel test
temperatures is, indicating a better contact and adhesion with the bottom layer. The bottom layer of the sample bonded at 145°C is shown in Figure 3.6b. The bonding area can be distinguished by the opaque area. The sides of the bonded films clearly show which are the bonded and not bonded areas. The bonded area looks completely transparent while the non-bonded one is not completely flat and thus reflect some light. The sample bonded at 155°C did not peel off properly; the bonding was too strong. The bonded films post peel test are shown in subfigure (c). Because the films never separated, the grip pulled the sample (not completely) out of the holding brackets, down causing it to bend, prior to breaking.

After peel tests were performed for all samples, the maximum load recorded was plotted as a function of their respective varying parameter. Obtained results are shown in Figure 3.7. The first graph shows that bond is stronger for higher temperatures. The last two data points correspond to samples that didn’t peel off and broke instead. It was found that bonding time (subfigure (b)) relates to bond strength similarly to temperature. Samples bonded at 35 and 40 minutes did not peel off correctly and broke.

As mentioned before, three batches of load samples were fabricated, each one of them at a different temperature. Figure 3.7c confirms the relationship between bond strength and bonding temperature: higher temperature means stronger bonding. It also reveals that increasing bonding pressure produces a stronger bonds as well. The data point corresponding to a load of 700 N and 155°C failed to peel off and broke. There are two inconsistencies in the mentioned plots: in (a) the sample bonded at 150°C, and in (c) the sample at 350 N and 155°C. Both samples broke before the peeling started. This could indicate either a very strong bond or prior film mechanical fatigue.
Overall, the results indicate that bond strength is directly proportional to a power (> 1) of \( t_b \), \( T_b \), and \( P \), which coincides with test found in the literature([8, 9, 34]). Mechanical failure of the film prior to de-lamination was considered as the threshold for acceptable bonding.
3.4 Further characterization

In order to compare results with other thermal bonding processes presented in the literature, three different techniques were implemented: measurement of bond strength by applying shear stress \[21\], measurement of the maximum pressure a device can withstand \[14\], and measurement of the maximum amount of load (orthogonal to the surface) that the bond resists before de-laminating \[13, 23, 34\].

Shear bond strength

Two PMMA films were bonded to each other using the optimal parameters found in section \[3.3\]. The mechanical testing suite was equipped with two grips, one on the bottom and one on the top. The two layers were held by the grips, pulled apart, and the maximum strength was recorded.

Figure 3.8: \(a\) Maximum load obtained on the shear stress test. \(b\) fractured samples of (top) single PMMA film, and (bottom) two thermally bonded films

Three bonded samples and a single piece of fifty-micron PMMA film were tested and the results obtained are plotted in Figure 3.8. The breaking point of all bonded samples occurred at very similar times and loads. At the beginning of the test, the
load measurement increases until a maximum of 84.3 N ± 3.7% and then decreases slowly. Bonded films fractured shortly after reaching the maximum while the single film was mechanical deformed without breaking until it was elongated for 18.6 mm (Figure 3.8d). All the bonded samples presented a clean cut on the border of the bonded area, which suggests that the material weakens in that boundary. The bonded area was 9 cm$^2$ and consequently the maximum shear stress was 93.6 kPa. However, these measurements are not representative of the bonding strength; films did not delaminate.

**Rupture pressure**

Devices composed of three layers were patterned by laser ablation and bonded. The top layer included only an inlet hole and the bottom layer was completely blank. The middle layer contained a micro channel and a circular reservoir of five mm in diameter (figure 3.9). This means that the input pressure will act over the surface area of the mentioned reservoir.

![Laser-cut layers that make up the chip](image1.png)  ![Delaminated chip](image2.png)

**Figure 3.9:** Detail of the chip used to test the maximum pressure withstood by the bonding process, and example of delaminated chip

Four samples were fabricated and tested by injecting pressure driven flow into the
channel. At approximately 1.7 bar the air inside the channel was completely compressed and appeared only as a bubble in the liquid inside it. Top and bottom films bowed out taking a larger amount of liquid. At 2 bar (30 psi), de-lamination of the samples occurred, relieving the pressure in the channel. The test performed by Cheng et al. [14] concluded that their bonding can withstand liquid pressures of 20.6 bar. Their result is impressive given the fact that their bonding process is performed at 130°C. They perform a post-bonding anneal process with the objective of improving channel smoothness. This extra step has an effect on bond quality and strength as demonstrated by Zhu et al. [34]

Orthogonal pull

Two PMMA films were thermally bonded using \( L=700 \text{ N}, \ t_b=35 \text{ min}, \) and \( T_b=155^\circ \text{C}. \) Two six-millimeter thick substrates were laser cut in five by seven and a half centimeter rectangles (same size as the bonded films) with a circular depression and a through-hole in the center. A screw was secured through this hole using nuts and washers. Both surfaces of the bonded films and the thick PMMA substrates were cleaned with isopropyl alcohol and left to dry. All three were glued together using chloroform as illustrated in Figure 3.10b. This technique assumes that the chloroform bond is stronger than the thermal bond.

The ensemble was held in place by Instron’s grips and consequently pulled apart until separated. The maximum load before separation was 719 N, which is equivalent to a bond strength of 190 kPa. It was verified that films were separated, meaning that the chloroform bond is, indeed, stronger.
Channel deformation

The samples fabricated to perform the peel test had a bonded and a non-bonded area as depicted in Figure 3.11a. This channel was used to measure the differences on the channel produced by the bonding process.

Channels were measured as described in section 2.2. The definition of top channel width presented in Figure 2.1a does not apply to bonded channels because the bump disappears. In these channels, width is determined by measuring from one edge to the other. A sample profiler output obtained from the \( t_b = 25 \) min of the bonding time batch is illustrated in Figure 3.11b. To account for bump loss, width provided by the
bump is subtracted from the original not bonded channel width and then compared to bonded channel width. For the lowest values of the used parameters, it was found that channel width decreased by only one percent. On the other hand, the largest deformation was found to be 29% and produced by the highest values given to $t_b$, $T_b$, and $L$. Width reduction for the optimal parameters found in section 3.3 has an upper bound of 20%. The channels were open for each of the bonded samples, i.e. there were no obstructed channels in the process.

Another effect of the bonding process is a change in the surface roughness of the laser ablated channels. It is demonstrated in Figure 3.12 that porosity in channel walls is considerably diminished. The channel in subfigure (a) was ablated with a laser power of 100% and 100% speed, and the one in subfigure (c) with 1% power and 4% speed. On the right, SEM images reveal a smoother wall surface for both channels. The porosity decreased and the holes were less than one micrometer in diameter, in contrast to the ten micron upper bound in pre-annealed channels. It was reported by Cheng et al. [14] that annealing chips at 170°C after bonding makes channel surfaces smoother [13]. Appendix C contains more images detailing the change of surface smoothness in these channels.
Figure 3.12: Annealing effect on laser ablated surface roughness. Subfigures \(a\) and \(b\) are the pre and post anneal channels fabricated with 100% power and 100% speed, \(c\) and \(d\) were fabricated with 1% power and 4% speed.

3.5 Summary

A direct thermal bonding process using a mechanical testing device was established and characterized for thin PMMA bonding. The optimal parameters were found by
performing a peel test with the same testing platform; they are $P = 160$ kPa, $T_b = 150^\circ$C, and $t_b = 30$ min. Bond strength was tested with three different methods: shear stress, orthogonal stress and rupture pressure tests. The results place the present work above those of Chen et al. [23] and Kelly et al. [15] and below the studies of Sun et al. [21], Cheng et al. [14], and Zhu et al. [34]. As mentioned before, the development of this platform is the first step in the evolution of the thin film process being developed at the EMPIRe Laboratory. Incorporation of other techniques is underway with the aim of reducing bonding temperature and providing the same quality of bond. A comparison between the present work and those described in the literature is summarized in Table 3.1.

Table 3.1: Bond strength comparison between this work and those described in the literature.

<table>
<thead>
<tr>
<th>Source</th>
<th>Technique</th>
<th>Substrate</th>
<th>Strength (kPa)</th>
<th>$T_b$(°C)</th>
<th>Anneal (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chen et al. [23]</td>
<td>vacuum</td>
<td>thick</td>
<td>153</td>
<td>112</td>
<td>N</td>
</tr>
<tr>
<td>Kelly et al. [15]</td>
<td>water</td>
<td>thick</td>
<td>130</td>
<td>100</td>
<td>N</td>
</tr>
<tr>
<td>Sun et al. [21]</td>
<td>plain</td>
<td>thick</td>
<td>2150</td>
<td>165</td>
<td>N</td>
</tr>
<tr>
<td>Zhu et al. [34]</td>
<td>MMA</td>
<td>thick</td>
<td>2000</td>
<td>95</td>
<td>50</td>
</tr>
<tr>
<td>Cheng et al. [14]</td>
<td>plain</td>
<td>thick</td>
<td>2068</td>
<td>120</td>
<td>170</td>
</tr>
<tr>
<td>Present work</td>
<td>plain</td>
<td>thin-film</td>
<td>190</td>
<td>133</td>
<td>N</td>
</tr>
</tbody>
</table>
Chapter 4

Thin-Film 3D Microfluidic devices

Fabrication methods of any microfluidic device are defined by mainly three things: material selection, channel formation, and channel enclosure. The present work proposes laser ablation of 50-micron-thick films of poly-methylmethacrylate, which are thermally bonded to each other for channel enclosure. Besides a detailed description of the fabrication process, this chapter discusses the performed procedures and findings while fabricating a 3D microfluidic device by using the proposed process. Design rules arising from empirical observation are enlisted in Appendix B. Prior to submerging into the intricacies of design and fabrication, a discussion on the integration of material selection and implemented methods are presented in Section 4.1.

4.1 Integration

The first building block of the fabrication method is the choice of material. Section 1.2 discusses some of the mechanical, electrical, optical and chemical characteristics of poly(methyl methacrylate) although deciding to use thin films rather than thick substrates comes from a different set of reasons. The first and most evident being that overall volume of the final device is greatly reduced. Moreover, substrate stiff-
ness decreases along with total thickness enabling the fabrication of flexible chips. An example is illustrated in Figure 4.1 where both chips reproduce the same 3D channel structure but one is fabricated with thick substrates and the second with thin films. Changes in layer thickness impact the overall device size and dead-volume at interconnection between layers; every inter-layer connection reduces its height. The use of thin films increases the potential of the fabrication baseline by enabling the addition of layers and, hence, incorporating more features/functions on the same chip without compromising manageability of the device.

There is an important topological difference between fabrication in thick and thin substrates. In the thick one, channels are grooves, which are then covered by another thick substrate. In thin films, channels are generated by cutting through them and covering top and bottom for enclosure; this means channel depth is exactly the film’s thickness. Because of this, the structure in Figure 4.1 is fabricated with four thick substrates or seven thin films. This provides another degree of freedom in the design of microfluidic chips. Liquid in channels from different layers can interact between them as illustrated by the split and recombine (SAR) mixer in Section 4.3.

Channel fabrication in thin films was accomplished by laser ablation with a platform operating a CO$_2$ source, by Universal Systems, USA. The laser ablation process and its relation to user-controlled variables is described in Chapter 2. Bump for-
mation around channels is of particular importance to the integration of proposed
techniques for device fabrication. It has been demonstrated here and in other studies
[26], that different laser parameters have an effect on bump characteristics. This is
due to the lower density polymeric material that composes the protrusion. Several
studies, including this one, have proved that the bump does not affect the quality
of the bonding achieved. Klank et al. suggested that the bump acts as a built-in
seal around micro channels and conclude that even 30-micron-tall bumps are not an
impediment to obtain reliable bonding [12]. The bumps observed in the present study
ranged from five to twenty-five micrometers in height and did not represent and issue
for bonding.

Because the bump is made of low-molecular-weight polymeric material it becomes
liquid at lower temperatures compared to the bulk; the material will rearrange because
of applied pressure during the bonding process. This phenomenon is responsible for
channel blockage in particular circumstances that depend on the geometry and layout
of channels in adjacent layers, i.e. regular enclosure of channels is reliably achieved.
Mentioned circumstances are discussed in Appendix B, which also discusses the design
rules associated to the process. Examples of open and closed channels are presented
in Figure 4.2. On the left figure, the horizontal channel is clearly open. The black line
that appears to be blocking the vertical channel is the edge of the circular feature;
the vertical channel is in the layer below and it is open. The structures in (b) and (c)
show blockage in the vertical channels; it can be recognized by noting a discontinuity
in the parallel lines that define the channel.

Thermal bonding has a positive consequence: annealing the laser ablated chan-
nels was favorable for the surface roughness of its walls, as mentioned in Chapter 3.
This is useful because it improves hydrophilicity and helps prevent particle entrap-
Figure 4.2: Examples of open and blocked channels due to bump reflow

ment in channel walls. The aftermath of the bonding process also includes channel
width reduction with minor deformation. Although the thermal bonding process is
robust, wide channels or large areas covered by a single layer of fifty-micron PMMA
may collapse (more detail in Appendix B). Such phenomenon is easily controlled in
lamination methods [41, 40, 38, 37] and its not an issue for other types of adhesive [18].

One of the reasons for developing a fusion-bonding baseline was obtaining a homo-
geneous channel profile, which results in uniformity of its physical characteristics like
hydrophobicity, thermo-mechanical stresses, electric, and optical properties amongst
others [9, 12]. The second reason was the desire for a stronger bond, which is where
other studies fail [39, 41, 42].

4.2 Fabrication baseline

The overall fabrication process consists of six steps: design, laser-cutting, cleaning,
aligning, thermal bonding, and adding the world-to-device interface. Each of these
steps is on its own simple, fast and inexpensive.
Design

Microfluidic channels can be drawn using any CAD solution available on the market. For its simplicity, the software used in this work was Oracle’s Open Office ‘Draw’. This software offers basic drawing tools that allow the user to define feature dimensions. Two form factors were used: a square with a one-inch side, and a rectangle that measures two by three inches. The latter was the preferred size for fabricating devices and characterizing the bonding process.

Devices were made up of top, middle and bottom layers. Every thin film was patterned with a circular hole in every corner, for alignment purposes. The bottom layer usually has no features except these holes, the topmost one includes the input and output orifices of the device. These openings were cut with a fixed diameter of one millimeter (trying to keep the dead volume as low as possible). This can be changed based on application requirements.

Channels and microfluidic structures were laser-ablated on the middle layer(s). It was found that specific features cannot be combined in adjacent films. Systematical channel blockage was observed for some of them, which gave form to Appendix B where design rules for fabrication of multilayered (3D) microfluidic devices are described. This design step can take as little as five minutes and as much time as is required by the application.

Laser-cutting

The first step in laser cutting thin films was correctly placing them on the honeycomb platform, inside the laser cutter’s workspace. The films were taped down on the corners to avoid movement during the process. After importing the design into
the laser’s control software, the features were relocated to the real film’s position and
the parameters were set.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>1%</td>
</tr>
<tr>
<td>Speed</td>
<td>4%</td>
</tr>
<tr>
<td>PPI</td>
<td>1000</td>
</tr>
<tr>
<td>z</td>
<td>0.0 mm</td>
</tr>
<tr>
<td>Top width</td>
<td>235.1 μm ± 3%</td>
</tr>
<tr>
<td>Bump height</td>
<td>20.6 μm ± 20%</td>
</tr>
<tr>
<td>Bump width</td>
<td>84.9 μm ± 10%</td>
</tr>
</tbody>
</table>

Figure 4.3: Laser parameters used, and profile of the outcome channel

According to the discussion presented in Chapter 2, surface roughness is minimal
when scanning speed and laser power were kept to minimum values. The parameters
chosen as well as channel profile are presented in Figure 4.3. Even when reducing
speed to four percent (12 mm/s), the total process time per layer is less than thirty
seconds.

Cleaning

Thin films were cleaned by immersion in isopropanol for 30 seconds while being
slightly agitated. They were also rinsed with the same solvent immediately after
being removed from the bath. Finally, they were placed between two lint-free wipes
to dry. Isopropanol was selected because it doesn’t attack this polymer’s surface (at
room temperature) and evaporates quickly. The total cleaning time was approxi-
mately twenty minutes.
Aligning

Although for simple designs aligning is not necessary it might be the case for complex multilayer designs; one example is the four-layer SAR (Split And Recombine) mixer shown in Figure 4.7. The most straightforward way to maintain alignment is to include structures for this purpose in the laser-cut design. Alignment holes, measuring one millimeter in diameter, were cut on each corner of every layer.

Two different approaches were used. The first secures alignment by introducing short metal pins in the corner holes. The appropriate diameter for metal pins was chosen so it fits tightly into the holes, maintaining the position of the films. This approach required using thick PMMA substrates as top and bottom layers. Metal pins cannot be used with thin films alone because they should not exceed the total thickness of the stack. If they did, contact between glass slides and polymer layers would not be uniform, compromising the bonding quality.

The second approach, implemented only with the one inch squared form factor, was the fabrication of an aligning fixture that consisted of two compression plates. The bottom compression plate contained four alignment pins where the layers forming the chip were placed. The pressure needed when using this add-on was higher due to the friction between alignment pins and top compression plate. The alignment fixture is shown in Figure 4.4. Neither of the two methods required more than five minutes.

Thermal bonding

The thermal bonding process was performed just as described in Chapter 3. After aligning, two glass slides were placed on top and bottom of the film stack to prevent
transfer of the metal surface pattern to the polymer. When the sample was ready, the steps described in Section 3.2 were carried out. The bonding recipe is summarized in Table 4.1. Heating up took between twelve and thirteen minutes. The overall time for bonding was approximately 70 minutes.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Preheat temperature</td>
<td>45°C</td>
</tr>
<tr>
<td>Preheat time</td>
<td>10 min</td>
</tr>
<tr>
<td>Bonding temperature ($T_b$)</td>
<td>155°C</td>
</tr>
<tr>
<td>Bonding pressure ($P$)</td>
<td>160-200 kPa</td>
</tr>
<tr>
<td>Bonding time ($t_b$)</td>
<td>30 min</td>
</tr>
<tr>
<td>Cooling time ($t_c$)</td>
<td>20 min</td>
</tr>
</tbody>
</table>

**World-to-device interface**

An important part of the fabrication process is providing a way for the chip to interact with the user, or surrounding analysis elements. When using thin films it is impossible to insert tubes as suggested by Wang et al. [30], hence, the need for an adapter between input and output orifices and normal tubing.
Figure 4.5: PMMA laser-cut tube adapters \( \text{(a)} \) design, and \( \text{(b)} \) sample.

Tube adapters were designed, and fabricated using the laser cutter on two-millimeter-thick PMMA. The design of these adapters is illustrated in Figure 4.5a. The outer and inner diameter measured eight and two millimeters, respectively. The grey circumference indicates a groove of arbitrary depth that has the function of spreading and allocating excess solvent injected through any of the orbiting holes. Such holes have a quarter millimeter radius. Finally, the grey circle indicates a raster scan that aims to separate the tube from channel input to avoid blockage with loctite glue.

The adapters were placed on the surface of the top layer and chloroform was injected through the small holes. Chloroform spread in between the adapter and the device due to capillary forces. The device was left to dry for five minutes. Plastic tubing with outer and inner diameters of two and 0.7 millimeters, respectively, was placed inside the center hole and Loctite 430 glue was added. Ten minutes later the device was ready to be tested. Liquid pressures up to thirty pound per square inch (2 bar) can be easily tolerated by the adapter; delamination of the device occurred before adapter leakage.
4.3 First designs

Because of the nature of the process’ topology, the simplest device is made up of three layers. An example of such device is a “Y” channel and is shown in Figure 4.6. In the microfluidic regime mixing of two liquids is not an easy task as demonstrated in Figure 4.6b. When two liquids come together they don’t instantaneously mix, on the contrary, a flow with two phases is formed leaving diffusion as the only mixing mechanism. These types of devices are used to separate particles based on their diffusivities.

![Empty Y-channel](image1) ![Laminar flow in a Y-channel](image2)

**Figure 4.6:** Laminar flow in a Y-channel

The next example is made up of four layers and its purpose is to mix two different reagents coming together at an intersection. An example of the split and recombine (SAR) mixer is presented in Figure 4.7. Mixing action takes place in the middle two layers. At the start the channel in both films followed the same path. On the first divergence, channels in middle-top and middle-bottom go in separate directions, splitting the flow vertically. When channels recombine at the next intersection there are four liquid layers instead of two. Adding more iterations of this structure helps mixing by decreasing liquid layer width until a regime where diffusion can easily happen.
These type of devices are a good example of the virtues of using thin films; channel flow in adjacent layers can interact between them. It is also a good example of a device that requires good alignment. It can be seen in Figure 4.7b that the alignment could be improved. However, even with non-perfect matching of the channels, the device accomplished mixing of the two input flows.

4.4 Versatility of the fabrication process

Fabricating microfluidic devices using this method can be taken even further. Due to the fact that this technique uses thin films and thermal bonding, it is extremely versatile. Multilayer bonding, using layers of different thicknesses, deposition of metal lines and contacts, and the integration of non-PMMA elements are some of the advantages of the proposed fabrication baseline.
Multilayer bonding

Being able to bond multiple layers provides an extra degree of freedom to chip design. The more layers the more features that can be added in the same chip. Even a device with twenty layers would result in a device with a thickness of one millimeter, because the PMMA films used are only fifty microns thick. Up to this point it has been possible to bond twelve layers in the same bonding process and up to eighteen layers in three steps. This represents a time advantage on layer-by-layer methods (lamination based \[12\] and SU-8 dry film \[19\]), although those methods have the advantage in aligning.

Different thickness layers

Channels and other features are not limited to fifty micrometers in height. The proposed method can combine PMMA layers of different thicknesses, increasing the aspect ratio (AR). For example, if the fabrication of a square channel is desired, three fifty-micron layers can be bonded together and laser cut afterwards. Finally, this channel will be integrated to other elements in the overall design, top and bottom layers at least. Performing the bonding step prior to laser cutting eliminates the need for alignment for these layers.

Microfluidic channels created by laser ablation have a gaussian profile. However, when laser-machining channels in thin PMMA films (50 μm), its walls are almost vertical. When multiple layers are stacked and laser-cut, this consideration has to be revisited. If straight channel walls are desired, a good approach was demonstrated by Cheng et al.\[14\], where multiple passes of the laser increased depth but didn’t have a significant effect on width.
**Metal lines**

Adding metal patterns to the devices requires only a simple process and opens a new realm of possibilities. Having electrodes in the system doesn’t only mean measurements can be performed, but also enables back and forth interaction between the system and surrounding electronics, or the user. This is a trade that is common to most of thin-film based microfluidic devices in the literature [36, 37, 19]. There are many ways that metal lines can be added to a microfluidic device fabricated with the present method. One option is buying and laser machining metal-coated film, for further integration in the device. Another option is to use inkjet printing of metal inks, or sputter deposition. The later was used to demonstrate that this can be achieved.

**Table 4.2:** Sputter recipe used to deposit gold lines on thin PMMA films

<table>
<thead>
<tr>
<th>DC Power</th>
<th>Ar flow</th>
<th>Pressure</th>
<th>Height</th>
<th>Pre-Sput. time</th>
<th>Sput. time</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 W</td>
<td>25 sccm</td>
<td>5 mT</td>
<td>160 cm</td>
<td>15 s</td>
<td>300 s</td>
</tr>
</tbody>
</table>

A mask was created using the same laser-cutting procedure. It was taped together with a patterned thin PMMA film and sputtered with 250 nm of gold using the recipe shown in Table 4.2. The resulting features are shown in Figure 4.8b and 4.16b. A positive outcome of sputtering is that channel walls are also covered with gold, allowing for uniform electric fields through the channel. The cleaning step had no effect on deposited gold. Furthermore, an attempt made to remove gold by rubbing it with a cloth soaked in solvent (IPA); gold started going away after a minute of continuous rubbing. A peel test using adhesive tape was also performed, metal lines remained intact.

Metal lines should not be in contact with air inside the chamber, i.e. the sputtered patterns should not reach the borders of the films. If lines or contact pads are
exposed to hot air their temperature will rise faster than that of the substrate causing only the polymer close to sputtered material to melt. This can produce as little as slight deformations, or as much as complete channel blockage. In order to avoid this and provide contact to metal lines after the bonding process a thick PMMA layer was used as the top layer. The backside of such layer was raster scanned by the laser with the objective of forming an empty chamber, as illustrated in Figure 4.8c. After bonding, a hole was drilled and silver ink followed by a metal pin were introduced. The pin was held in place using epoxy glue.

A test was performed where oil and water were injected into the chip through its two input orifices. A current source was connected to the metal pins and set to two μA. Voltage was measured using a Tektronix oscilloscope connected in parallel, also at the pins. The voltage observed is shown in Figure 4.9 together with a picture of the water and oil phases in the channel. When the water phase is between the metal
lines, a field is established between them and current flows in the circuit. Oil opens
the circuit and forces the power supply to increase voltage in order to maintain the
set current. Peaks were observed in the oscilloscope every time oil passed between
the metal lines.

**Non-PMMA elements**

The layer by layer structure presented here permitted the integration of non-PMMA
elements with the objective of performing different on-chip functions. This technique
has also been implemented by Weigl et al. [38]. A variety of different materials can
be potentially used including filter paper, plastic membranes, and thin layers of other
polymeric materials. The present work only tested this feature by integrating differ-
et types of filters and membranes to the fabrication process.

A microfluidic chip was fabricated to test if the filters withstand the process with-
out loosing their structural characteristics. The device consisted of ten layers out of
which four hold the filter in place. The chip cross section is illustrated in Figure 4.10a.
and the assembled parts prior to being bonded are shown in (b). Five different filters were laser machined to the appropriate size using $P = 5\%$, $s = 100\%$, $PPI = 1000$, and $z = 0.0 \text{ mm}$. Some characteristics of selected filters are summarized in Table 4.3. The chip was bonded with the same procedure described in Section 4.2.

### Table 4.3: Characteristics of different types of filters incorporated to the fabrication process

<table>
<thead>
<tr>
<th>Material</th>
<th>pore size (μm)</th>
<th>Thickness (μm)</th>
<th>Vendor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polycarbonate (PC)</td>
<td>1.2</td>
<td>20-25</td>
<td>Millipore</td>
</tr>
<tr>
<td>Polycarbonate (PC)</td>
<td>10</td>
<td>17.8</td>
<td>Millipore</td>
</tr>
<tr>
<td>Cellulose ester (CE)</td>
<td>5</td>
<td>135</td>
<td>Millipore</td>
</tr>
<tr>
<td>Nylon</td>
<td>40</td>
<td>60</td>
<td>BD</td>
</tr>
<tr>
<td>Cellulose (CA)</td>
<td>11</td>
<td>180</td>
<td>Whatman</td>
</tr>
</tbody>
</table>

Visual inspection of the filters indicated that none of them suffered damage during the process (Figure 4.11). One concern expressed by Kelly et al. [15], is that filters tend to swell when hydrated; this effect could cause device de-lamination. The filters were wetted first with phosphate buffered saline solution (PBS), then with 0.8 molar sodium chloride solution. Images of dry and hydrated filters were captured with a Canon 5D mark II camera and a 10X objective; these are shown in Figure 4.12. Dry filters are in the top row, and the corresponding wetted ones in the bottom; no
Figure 4.11: Different filters tested on-chip. Top row: filters in place after bonding, bottom: filters before bonding

Figure 4.12: Different filters tested on-chip. Top row: dry filters, bottom: hydrated with PBS solution

de-lamination or any type of failure was observed. Both solutions were previously prepared at the EMPIRe Laboratory.

Magnetic beads functionalized with oligonucleotides were injected into the channel with the objective of testing binding between them and the filters; this was only done for three of the filters (PC 1.2 μm, CE 5 μm, and CA 11 μm). The beads
used measure 2.8 \( \mu m \) in overall diameter, which includes an iron core and polystyrene coating. The beads were injected in a medium of the same 0.8 molar sodium chloride solution used to wet the filters. Obtained images are presented in Figure 4.13. Beads were completely trapped in the 1.2 \( \mu m \) filter and they passed through the other two, more easily so for the nylon one. Images taken after injecting 300 \( \mu l \) are shown in the bottom row.

The last experiment performed on the filters was injecting agglomerated beads and observing their interaction with the 11-micron cellulose, and the 40-micron nylon filters. Bead types 23 and 24 were mixed with binding DNA causing them to agglomerate in a medium of 0.8 molar solution of NaCl. Insertion of the beads into the channel was done carefully to prevent breaking the bonds between agglomerated beads. Images showing the accumulation of agglomerated beads at the nylon filter were captured and are displayed in Figure 4.14. Agglomerated beads were also stopped by the cellulose filter, which can be appreciated in Figure 4.15. The intricate
fiber structure is detrimental to visibility and also causes the agglomerates not to move easily.

Figure 4.14: Nylon filter test with agglomerated beads. This sequence shows the entrapment of the agglomerates from when they first reach the filter (a), and 300 μl later, (h).

Figure 4.15: Cellulose filter test with agglomerated beads

4.5 Applications

There are numerous potential applications for the proposed fabrication baseline. This method’s versatility provides enough freedom of design, to achieve high complexity
3D structures. This section describes two basic applications that prove the capabilities of thin film microfluidics, and this fabrication baseline, in particular.

**Droplet generation**

A microfluidic droplet generator was fabricated using three thin PMMA films and 2 thick substrates. The layout of the device is illustrated in Figure 4.16a. Three films were bonded together and laser cut with the design shown in the left most layer. This resulted in a channel of approximately 150 μm, in depth, by 250 μm wide. Top and bottom covers were fabricated with two-millimeter thick PMMA. The rightmost layer represents the bottom, and top is depicted in the middle.

![Droplet generator's layout and finished device](image)

**Figure 4.16:** Droplet generator composed of five layers with both fluid and electrical connections added.

Prior to bonding the complete stack, a thin gold layer was sputtered onto the thin films providing a line that was only separated at the channel (see Figure 4.16a for a detail on the mask used). Grey squares on the top layer represent a raster scan that does not cut through the entire thickness of the material. Their purpose is to provide a separation between the back side and the middle layers, in order to make drilling possible without damaging the metal lines. This process was described in Section 4.4.
A cross section of the device is illustrated in Figure 4.8c.

(a) 5:5  (b) 5:4  (c) 5:3
(d) 5:2  (e) 5:1  (f) 5:0.5

Figure 4.17: Droplet generation at different water flow rates.

Mineral oil with 3% of Span 80, surfactant bought from Sigma-Aldrich, was used as the continuous phase. It was injected into the straight micro channel using a syringe pump at a rate of 5 \( \mu \text{l} \) per minute. Water-based food coloring was injected at different rates into the curved channel. Results for different flow rates are shown in Figure 4.16b. The droplet generation process was captured in video and played frame by frame to calculate its frequency. A five \( \mu \text{l} \) water flow produced sixteen droplets per second. Taking into account the displaced volume per minute and the number of droplets it was divided in, the calculated volume was 5.2 nl. For a 0.5 \( \mu \text{l} \) the frequency was eight hertz, and the volume per droplet was one nanoliter.

Generation of oil-in-water droplets cannot be achieved with this device due to the hydrophobicity of PMMA. However, it was possible to generate separated phases of oil and water, avoiding laminar flow in the channel. The resulting flow was used to
Agglomeration detection

The detection of biological compounds such as proteins or DNA is of the utmost important for applications like diagnostics, drug development, and defense. A simple method based on microbead agglomeration was recently demonstrated by Kodzius et al. [43]. Micron size beads that are functionalized with oligonucleotides that do not bind to each other are kept in a solution. If the right kind of DNA is added, it will bind to both the oligonucleotides and keep the beads attached as illustrated in Figure 4.18. After an incubation period, micro beads are binded to one or more other beads forming a cluster (or agglomerate) that can be optically detected. A device capable of performing the agglomeration reaction and detection on-chip was designed and fabricated. To reproduce the agglomeration experiment, Dynabeads M-270 Streptavidin were acquired from Life Technologies, USA. The surface of the beads was functionalized with Streptavidin, which is single stranded DNA of 23 or 24 bases. The target DNA used to bind the micro-beads together was 3-47SRY.

Figure 4.18: Bead agglomeration mechanism: bead A, and bead B held together by a DNA fragment

An agglomeration detection system was fabricated in several steps. The layout of the devices is shown in Figure 4.19. First, two stacks of three fifty-micron films were bonded. Of those, one was patterned with the input channels and the other with the output ones (top left and top right in Figure 4.19a, respectively). The fabricated
input channels were bonded between two films and the channel connecting the two filter reservoirs was laser machined afterwards (top middle). This way, input channels direct fluid on top of the filters and output drain liquid from the bottom of them. The selected filters were the 1.2-micron polycarbonate based and the 40-micron nylon based membranes because of their performance, demonstrated in Section 4.4. They were placed in the space defined by the remaining machined layers (bottom) and bonded between the other two previously bonded stacks. A total of fourteen layers compose the device, taking top and bottom thick substrates into consideration.

![Figure 4.19: Layout for agglomeration detection device. Layers including input and output channels are shown in the top row, in the bottom are the films that keep the filters in place.](image)

The device was first filled with 0.8 molar sodium chloride solution. Next, magnetic beads types 23 and 24 were injected through input one while orifices two and four are kept closed (this was done by fixing a syringe with a blocked plunger to the corresponding adapters). A solution containing the binding DNA was injected next,
taking care not to push it further than the reservoir where the beads are. The device was left to incubate for 35 minutes and was observed on the microscope afterwards. Pressure was applied on output three while inputs one and two were closed. Beads were then transported through the thick channel to the second filter. Observing the second filter, revealed that agglomeration was achieved. Captured images are displayed in Figure 4.20. The produced agglomerates are not as large as the ones tested in Section 4.4. A consequence of the reduced size is that some agglomerates can travel through the membrane and not be stopped and, hence, go undetected. Nonetheless, some bead clusters were stopped and detected. By increasing the pressure, some agglomerates were forced to move through the membrane and into the output.

Figure 4.20: Production and detection of bead agglomeration on chip.
4.6 Summary

Successful integration of laser ablation and thermal-fusion bonding of thin PMMA films permitted the fabrication of three-dimensional microfluidic devices. The fabrication baseline was thoroughly described, and the fabrication of various types of devices, demonstrated.

Throughout the present study, it was found that the proposed process is advantageous and disadvantageous in some areas when compared to others presented in the literature. The strongest trait of this fabrication method was the use of thermal-fusion bonding. Although not many research groups working with thin-film devices have provided a detailed characterization of their bonding method, it is accepted that optimal thermal bonding is stronger and better than using adhesives [9]. Another advantage is that a high number of films can be bonded in the same step.

Fabrication costs of all the cited studies are very low with exception of those using lithography in every step [19], followed by those that use it on only once [42]. Fabrication times are also considerably longer for those processes using lithography. However, they have superior resolution, and channel surface smoothness.

The present process was demonstrated to be versatile by bonding multiple layers at a time, by the use of various layer thicknesses, and by the integration of metal lines and passive elements. In theory, most of the thin film based microfluidic devices are capable of this with the exception of devices that cannot withstand high pressured liquids [39] or whose adhesive interlayer doesn’t permit. A comparison of the different fabrication baselines is summarized in Table 4.4.
Table 4.4: Comparison between different thin film technologies

<table>
<thead>
<tr>
<th>Source</th>
<th>Channel fab.</th>
<th>Resolution $\mu m$</th>
<th>Bonding</th>
<th>Bond strength</th>
<th>Time (h)</th>
<th>Cost</th>
<th>Substrates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bartholomeusz et al. [41]</td>
<td>Xurography</td>
<td>20</td>
<td>Lamination</td>
<td></td>
<td>0.5</td>
<td>Low</td>
<td>polymer films</td>
</tr>
<tr>
<td>Yuen et al. [18]</td>
<td>Xurography</td>
<td>200</td>
<td>PSA</td>
<td></td>
<td>&lt; 1</td>
<td>Low</td>
<td>polymer films</td>
</tr>
<tr>
<td>Metwally et al. [42]</td>
<td>Roll embossing</td>
<td>Lithography</td>
<td>SU-8 dry film laminationPDMS coating</td>
<td>weak</td>
<td>24</td>
<td>Higher</td>
<td>PMMA</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>weak</td>
<td></td>
<td></td>
<td>COC</td>
</tr>
<tr>
<td>Abgrall et al. [19]</td>
<td>Lithography</td>
<td>10-500</td>
<td>SU-8 dry film lamination</td>
<td></td>
<td>24</td>
<td>Higher</td>
<td>SU-8</td>
</tr>
<tr>
<td>Malek et al. [40]</td>
<td>Laser ablation</td>
<td>50</td>
<td>Lamination (epoxy adhesive)</td>
<td>weak</td>
<td>&lt; 2</td>
<td>Low</td>
<td>Polyester + SU-8 + Polyethylene</td>
</tr>
<tr>
<td>Wang et al. [39]</td>
<td>Wax mask + chemical etch</td>
<td>170</td>
<td>Wax bonding</td>
<td>weak</td>
<td></td>
<td>Low</td>
<td>Polyimide</td>
</tr>
<tr>
<td>Weigl et al. [38]</td>
<td>Laser ablation</td>
<td>100</td>
<td>Lamination</td>
<td>Medium</td>
<td>24</td>
<td>Low</td>
<td>Polyester</td>
</tr>
<tr>
<td>Martin et al. [37]</td>
<td>Laser ablation</td>
<td>$\leq 100$</td>
<td>HAA</td>
<td>Medium</td>
<td></td>
<td>Low</td>
<td>Polyimide</td>
</tr>
<tr>
<td>Present work</td>
<td>Laser ablation</td>
<td>150</td>
<td>Thermal-fusion bonding</td>
<td>Strong</td>
<td>$\leq 2$</td>
<td>Low</td>
<td>PMMA</td>
</tr>
</tbody>
</table>
Chapter 5

Conclusions and Future Work

The thin-film microfluidics process developed throughout this work is by no means mature. In order to access the full potential of the proposed technique, it is necessary to implement additional techniques and to extend research for achieving both additional functionality, and lower fabrication time.

Channels

The present study demonstrated that the surface roughnesses produced depend on the selection of laser power and scanning speed parameters. The effect of the bonding process and/or plain annealing reduces roughness even further. The smoothness of the surface should impact its interaction with water or any other fluid that could be used for chip operation. This is a feature that is yet to be characterized. Chemical surface modification can be implemented to change PMMA’s hydrophilic behavior, which can enable the fabrication of devices for different applications.
**Bonding**

The present study involves straight-forward thermal-fusion bonding of thin PMMA films. A high number of different techniques that aim to reduce the temperature and pressure required to produce adequate bond strengths are described in the literature. Enhancing the bonding method established at the EMPIRE laboratory is fundamental to the advancement, and the success of this fabrication baseline. Surface modification via oxygen plasma [31], UV degradation [33] and MMA assisted bonding [34] are techniques of particular interest. Work is underway for enabling the system to perform thermal fusion bonding in vacuum conditions.

**Devices and applications**

This work can be enhanced by the implementation of different types of materials. For example, the flexibility provided by PDMS films can be used for applications like valving and finger actuated pumps. Paper can be used as a wicking force for generating flow, generating a stand-alone device [38]. Electric and magnetic interactions could be used for detection, droplet sorting, and electro-coalescence. Once steps are taken to improve the bonding process and to produce hydrophilic surfaces, improvement of the technique will become specific to the application.

**Conclusions**

Thin-film 3D microfluidics devices have gained attention over the last decade as they have proved themselves to be cost effective, fast, and reliable. The present work proposed the fabrication of three-dimensional microfluidic devices by laser ablation and thermal bonding of thin PMMA films.
• The laser ablation process was characterized and optimized for producing straighter and smoother channel surfaces. Choosing lower values for laser power and scanning speed produce softer textures and reduce the occurrence of jagged edges.

• Optimization of the thermal-fusion bonding process yielded a bond stronger than those presented in the literature (for thin films). A method for characterizing bond strength between thin films (peel test) was presented. Utilizing different techniques, it was demonstrated that the produced bond is comparable to that in thick-substrate experiments. The pressure tolerance of fabricated devices is greater than other thin-film baselines.

• The versatility and characteristics attained with this fabrication baseline are unrivaled. The present work is the only thin-film method that provides a homogeneous channel profile. Implementation of on-chip metal electrodes and non-polymeric materials was demonstrated and used for detection purposes.

• The proposed fabrication baseline is fast, reliable, inexpensive, and fit for many applications. A high number of layers, including sputtered electrodes and non-polymeric materials were bonded at the same time. As a consequence, this process is placed within the fastest and most reliable due to its bond strength. The access to multiple applications is given by PMMA, which has been widely implemented in the literature.


Correct operation of the laser cutting system is fundamental to producing the desired features and guarantee functionality of a device. A set of considerations was derived from the experience gained throughout the duration of this work. These are divided in two groups: Interpretation of design characteristics by the laser cutting software, and operation of the laser cutting platform.

A.1 Operation of the laser cutting platform

The laser cutting platform is composed of a honeycomb horizontal panel (where the sample is placed), and a moving head whose position is determined by electric actuators. The function of the head is to redirect the laser from the source to any coordinate point over the honeycomb panel. There are four parameters that can be
modified through the control software: laser power \(P\), scanning speed \(s\), points per inch \(PPI\) and the panel’s height \(z\). A description of the effect of changing these parameters is described below.

**Power** This quantity is directly related to the energy output of the laser. An increase in power signifies greater depth and width in the pattern generated. It was demonstrated on Chapter 2 of this work, that high values produced rough surfaces when compared to low ones.

**Speed** The scanning speed is that of the laser head, and has an inversely proportional relationship with the output energy. Low values result in deeper and wider grooves when compared to high ones. A slow scanning speed allows for better uniformity of the bump around the channel and smoother surfaces. An often disregarded consideration is the head’s acceleration and deceleration at the start or end of a patterned line. This translates into a deeper and wider groove. Besides the extremes of lines, sharp corners produce the same effect.

**PPI** The number of points per inch can be adjusted to a minimum of ten or a maximum of one thousand. This quantity defines the pulses the laser will emit per traveled inch. The spot of the focused laser beam measures 127 \(\mu m\), which means that 200 pulses would produce a line of continuous dots. When higher numbers are selected, overlapping of the pulses occurs, which favors smoother edges.

**Height** This number defines the vertical position of the honeycomb panel. Ideally, the laser should be focused on the surface of the material that will be patterned. When it’s value is equal to zero, the beam is focused on the surface of the honeycomb panel. Changing the height allows to engrave the surface of thicker
A.2 Design features and their output

This section presents a brief description of how design features translate to the laser cutter’s software. Connected or unconnected lines, sharp edges, color, and the direction a line was drawn are characteristics commonly overlooked when sketching a design. However, all of the above have an effect on the procedure the laser cutting platform follows to generate the results. The different aspects of drawing that have an effect on the outcome are listed below.

**Color** The laser cutter’s software can recognize up to eight colors. To ensure correct translation of features from the CAD tool of preference to the laser cutter’s software, color RGB values should match between them. The choosing of color also affects the order in which features will be drawn. Through the software, each color can be assigned its own $P$, $s$, $PPI$, and $z$ values.

**Direction** The direction a line is drawn is directly translated to the laser cutter’s software. Then, the same direction lines are drawn is the direction the laser head will follow during the ablation process.

**Connected lines** Two lines, even if the second starts where the first ends, will be interpreted as two separate features. This will make the head stop and start again, producing a wider feature at the intersection. If lines are "connected" on the CAD tool, the laser cutter will render the feature in only one pass.

**Filled areas** The moving laser head performs two types of scan: vector and raster. Filled areas will always be rastered. Lines should be drawn with "hairline"
thickness or else they will be treated as filled areas. \textit{PPI} is not considered on raster scans. The resolution of the feature is defined by the imported design.

\textbf{Machining order} The order in which features will be laser-cut depends firstly on its scan type (vector or raster), and secondly on its color. The order of the colors is as specified by the software. Raster scans will be performed according to color order. Vector scans will be produced after raster scans, according to their color as well. Within features drawn on the same color, the order in which they were drawn is the same in which they will be machined by the laser.
Appendix B

Design Rules

Throughout the curse of several months, various design techniques were implemented in the fabrication of microfluidic chips. Some of these were successful and others were not. The result of these observations is a summary of "safe" design practices that have the purpose of preventing failure of devices by channel blockage. This phenomenon occurs mostly due to the bump generated by the laser ablation process. Another reason, but not as common, is channel collapse.

There are three ways to avoid bump channel blockage. The first is to anneal and compress each film individually. This reincorporates the bump material into the bulk rendering it harmless for the later assembly of the device. Even though a large stack of alternating films and glass slides can be put through the same process run, it doubles de fabrication time. Secondly, bump material can be removed using a scalpel. This is just as effective but is not reproducible nor efficient. The third way is to implement design features that prevent bump material from being pushed into the channels and, thus, blocking them.

The following are a set of rules that aim to prevent the situation described above
and lay down the basic rules for fabrication of microfluidic devices using the EMPIRe Lab’s thin-film baseline.

**Drawing** The capability of using various colors in the design permits the assignment of a machining sequence of the features. The following aspects should be followed to guarantee the best results.

1. **Outlining chip area.** The line defining the area where the features are contained should be left last, that is, drawn in the last color. The laser-cutting platform makes use of an air extractor to remove debris and evaporated material. This air is capable of making films move and vibrate. Channels and other features will be better transferred if the area is held on tightly, i.e. the chip’s area is not yet delimited and is still part of the entire polymer foil.

2. **Connect lines.** When the laser head starts or ends its movement it must accelerate or decelerate. This represents a change in speed and such will represent non uniform patterning. If a single channel is composed of two line segments, they should be 'connected’ through the preferred CAD tool.

3. **Avoid sharp angles.** Every time the laser head makes a sharp turn, it has to change its speed. A change in speed represents exposure time increase for the corners of the design. The patterning will not be uniform.

4. **Line intersections.** The order and direction in which intersecting lines are cut through the polymer has an important role on the outcome. The beginning and ending of a line always leave some debris behind. A “T” junction is the perfect case to exemplify this phenomenon. The correct way of cutting the material is shown in Figure 11.1. The same observations were done by Snakenborg et al. [11].
**Closed loops** No closed patterns can be machined in the same layer. Doing so removes the inner part leaving a large open area rather than a closed loop channel. Any desired closed loops require interconnected channels in two or more layers.

**Low aspect ratio features** Although not impossible, very low aspect ratio features are difficult to accomplish. Care should be taken in not designing features wider than necessary. If such structures are too wide, the top layer may collapse, taking away from the device’s functionality.

**Layer-to-layer connection** Interconnections between layers can be used for two purposes: prevent the ablation-created bump from blocking the channels, and to facilitate alignment. The easiest way to do this is by adding a circular feature in the top or intermediate level. This circular feature prevents contact between the bottom layer bump, and the top layer channel. The intermediate orifice should be smaller, but close in size to the top level, to prevent collapse.

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**Figure B.1:** The correct order of cutting is shown in subfigure (a) along with the order that the features should be machined in. The use of different colors is illustrated in subfigure (b). This design also shows the use of interconnecting holes and how to fabricate a closed loop.
Appendix C

Additional Images

The images presented here have the objective of complementing the information presented in previous chapters (2 and 3), regarding the surface roughness produced by the laser ablation process and the effect that high temperature anneal has on it.

Scanning Electron Microscopy was used to observe the surface roughness of twelve channels produced with three different power to speed ratios, each channel with different magnitudes such quantities. Three images per row are shown; each of them illustrates a channel at different magnifications. The smallest (250X) shows the density of large holes and craters. The middle one (500X) depicts the bump’s structure and offers a closer view on the big holes. Finally, the highest magnification (1000X) illustrates the porosity produced in the polymeric material. Captured images are displayed in Figures C.1, C.2, and C.3.

The same technique was used to analyze the effect that thermal annealing has on surface roughness. The same samples were put through the thermal annealing process described in Chapter 3. The results are displayed in Figure C.4.
Figure C.1: SEM images of channel walls ablated with a power to speed ratio of 1.
Figure C.2: SEM images of channel walls ablated with a power to speed ratio of 0.5
Figure C.3: SEM images of channel walls ablated with a power to speed ratio of 0.25
Figure C.4: SEM images of machined channels with a power to speed ratio of 1 (first two rows), and 0.25 (last two rows) after being annealed.