LOW-COST POLYMER-BASED MICROFLUIDIC SYSTEMS

by

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Abstract

Microfluidics technology shows a wide variety of applications in multiple research areas. Up to now, most of the microfluidic devices are fabricated in the cleanroom, using the highly sophisticated instruments. With the desire to make the microfluidic systems more accessible to chemical or biological researchers, a series of low-cost fabrication technologies for polymer-based microfluidic systems will be introduced in this research. With the help of these proposed low-cost and cleanroom-free fabrication methods for polymer-based microfluidic devices, users could easily fabricate their own microfluidic devices based on their usage.

These proposed low-cost fabrication technologies covered all three steps of the microfluidic systems fabrication processes, which are microchannel fabrication, microchannel sealing (bonding) and integration technologies. For microchannel fabrication, different low-cost methods such as laser ablation and deep-UV patterning will be introduced in this research. For microchannel sealing method, the thermocompression bonding technology will be investigated in detail. For integration technologies in microfluidic systems, various kinds of low-cost integration technologies for microlenses, metal electrodes and wafer dicing will be introduced.

In this research, the introduced low-cost fabrication technologies for microfluidic systems are based on different polymer materials such as poly(methyl methacrylate) (PMMA), polystyrene (PS) and polydimethylsiloxane (PDMS). The fabrication, bonding and integration methods introduced in this research can be widely adapted to different polymer materials.

This research offers a flexible choice of polymer materials and fabrication technologies to maximize the utility for researchers, and could be beneficial to lower the access of microfluidics technologies in chemical and biological fields.
Preface

The contents in chapter 2 have been published.

- The 2.1.1 has been published: [Yiqiang Fan], Liu, Y., Li, H. & Foulds, I.G. Printed wax masks for 254 nm deep-UV patterning of PMMA-based microfluidics. Journal of Micromechanics and Microengineering 22, 027001 (2012). I conducted the manuscript writing and most of the experimental work.

- The 2.1.2 has been published: Li, H., [Yiqiang Fan] & Foulds, I. Deep-UV-LIGA PROCESS USING A 254 NM LIGHT SOURCE. IEEE APCOT2012 (2012). I conducted part of the experimental work (pattern design, mask fabrication and data collection) and part of the manuscript writing.

- The 2.2.1 has been published: Li, H., [Yiqiang Fan], Kodzius, R. & Foulds, I.G. Fabrication of polystyrene microfluidic devices using a pulsed CO₂ laser system. Microsystem Technologies 18, 373-379 (2011). I conducted part of the experimental work (profile measurement and testing) and part of the manuscript writing.

- The 2.2.2 has been published: Li, H., [Yiqiang Fan], Conchouso, D. & Foulds, I.G. CO₂ laser-induced bump formation and growth on polystyrene for multi-depth soft lithography molds. Journal of Micromechanics and Microengineering 22, 115037 (2012). I conducted part of the experimental work (laser ablation and profile measurement) and part of the manuscript writing.

- The 2.2.3 has been published: [Yiqiang Fan], Huawei, L., Ying, Y. & Foulds, I.G. Low-cost rapid prototyping of flexible plastic paper based microfluidic devices. in Nano/Micro Engineered and Molecular Systems (NEMS), 2013 8th IEEE International Conference on 175-178 (2013). I conducted the manuscript writing and most of the experimental work.

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Technologies, 1-6 (2013). I conducted the manuscript writing and most of the experimental work.

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<tbody>
<tr>
<td>µ-TAS</td>
<td>Micro Total Analysis System</td>
</tr>
<tr>
<td>ABS</td>
<td>Acrylonitrile butadiene styrene</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscope</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer-aided Design</td>
</tr>
<tr>
<td>COC</td>
<td>Cyclic Olefin Copolymer</td>
</tr>
<tr>
<td>COP</td>
<td>Cyclic Olefin Polymers</td>
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<tr>
<td>DI</td>
<td>Deionized</td>
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<tr>
<td>DRIE</td>
<td>Deep Reactive Ion Etching</td>
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<tr>
<td>DUV</td>
<td>Deep Ultraviolet</td>
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<tr>
<td>HDPE</td>
<td>High-density Poly(ethylene)</td>
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<td>HEMA</td>
<td>Hydroxyethylmethacrylate</td>
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<td>IPA</td>
<td>Isopropanol</td>
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<tr>
<td>LIGA</td>
<td>Lithography, Electroplating and Molding</td>
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<tr>
<td>LOC</td>
<td>Lab on a Chip</td>
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<tr>
<td>MEMS</td>
<td>Microelectromechanical Systems</td>
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<td>MIBK</td>
<td>Methyl Isobutyl Ketone</td>
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<td>MLA</td>
<td>Microlens Array</td>
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<td>PC</td>
<td>Poly(carbonate)</td>
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<td>PCR</td>
<td>Polymerase Chain Reaction</td>
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<td>PDMS</td>
<td>Poly(dimethyl siloxane)</td>
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<td>PE</td>
<td>Poly(ethylene)</td>
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<td>PEG</td>
<td>Poly (ethylene glycol)</td>
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<td>PEGDA</td>
<td>Poly(ethylene glycol) diacrylate</td>
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<td>PLA</td>
<td>Poly(lactic acid)</td>
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<td>PMMA</td>
<td>Poly(methyl methacrylate)</td>
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<td>PP</td>
<td>Poly(propylene)</td>
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<td>PPI</td>
<td>Point Per Inch</td>
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Finally, my thanks would goes to my beloved parents and my fiancée for their unconditional love and encouragement for all these years.
Dedication

Dedicated to my parents
Chapter 1 Introduction

1.1 Goal of This Dissertation
In order to ensure that following work is read in the intended context, the purpose and scope of this dissertation will be introduced before any background is given.

The goal of this dissertation is to increase access to microfluidic technology by providing a set of fabrication processes that will enable researchers in the biological and chemical sciences, who do not have microfabrication experience or access to cleanroom facilities, to fabricate microfluidic devices using common laboratory equipment based on a variety of polymer materials.

1.2 Introduction to Microfluidics
Microfluidics refers to the process, control and manipulation of a small amount of fluids (usually, in micro or nano liter scale) in microfluidic devices. In the late 1970s, with the rapid development of miniaturized electronic devices, researchers began to fabricate the micro-mechanical devices, which later became known as microelectromechanical systems (MEMS). The idea of MEMS systems is now much more developed with the integration of fluidic and optical components. In the 1980s, with the development of MEMS techniques, researchers started to develop a series of fluids related micro-components such as the flow sensor (Benecke, 1989), micropumps (Richter et al., 1990), microvalves (Esashi et al., 1989) and micromixers (Bajpai & Reuss, 1982). The concept of the microfluidic systems, also known as the micro total analysis system (µTAS) or lab-on-a-chip (LOC), was first presented in Manz's paper in 1990 (MANZ & GRABER, 1990) which proposed a potential of processing the whole chemical analysis and detections on a single chip. After more than 20 years' development, the microfluidic techniques have now found wide applications in the mechanical, chemical and biological fields and attracted the interests of many researchers from traditional engineering fields.

The advantages of microfluidics come from the scaling laws: the miniaturizing of the whole system is beneficial from many aspects. First, the miniaturized system enables the ability to conduct chemical or biological analysis with a very small amount of sample and reagents (usually less than 1 µL), which is very helpful to lower the cost and promote the
technologies. One of the most frequently quoted examples is the breakthrough of the DNA microarray technology (Laurell & Nilsson, 2005). Another advantage is carrying out the analysis or chemical reaction with rapid speed and high sensitivity. For example, the hybridization time in the DNA analysis process has been reduced with the help of microfluidic devices from 1-2 hours to 5 minutes (Erickson & Liu, 2004) with the sensitivity down to 1 pM (Zimmermann & Delamarche, 2005). Besides these advantages, low-cost is another significant attraction for researchers and users. Low-cost refers to both materials and fabrication process. The microfluidic devices can be fabricated on low-cost materials such as polymers (Becker & Gärtner, 2007; Quaglio et al., 2008; Roy et al., 2010), glasses (Grover et al., 2003; Chen et al., 2011; Gundabala et al., 2013) or even paper (Lu et al., 2009a; Lei et al., 2012; Li et al., 2012). Some of these chips have been commercialized and are able to conduct the on-site chemical or biological analyses with very low-cost and in a disposable format (Bu et al., 2013).

1.3 Polymer Materials Used in Microfluidic Devices

In the early stages of the development for microfluidic systems in 1990s, glass (Harrison & Fluri, 1993) and silicon (Wilding & Pfahler, 1994) were the most commonly used materials. At that time, these two materials have been widely used in the semiconductor industry, and their properties were well studied. Later on, with the development of the microfluidics technology, glass and silicon showed some disadvantages such as complicated fabrication procedure, high cost, brittle and limited surface properties. Because of the disadvantages on using glass and silicon in microfluidics, researchers were trying to use polymers for the fabrication of microfluidic devices. Compared with glass and silicon materials, polymers give a much wider choice of mechanical, optical and surface properties, and users can choose different types of polymer materials based on their applications. Further, compared with glass and silicon materials, the cost of fabrication for polymer-based microfluidic devices is much lower and fabrication can be performed in much less time. In fact, polymers have become the most promising material for microfluidic devices with many different fabrication techniques, such as laser ablation (Klank et al., 2002a), injection molding (Attia et al., 2009), hot embossing (Becker &
Dietz, 1998) and casting (Sethu & Mastrangelo, 2004), these fabrication techniques will be briefly introduced in the later sections in this chapter.

**1.3.1 General Properties of Polymer Materials**

In general, polymer is a large molecule composed of many repeated units, which are known as monomers. The molecular weight of the polymers is ranged from 10,000 to 100,000 Da, consisting of more than 1000 monomer units. Polymer can be classified as natural and synthetic. All polymers, both natural and synthetic, are established by the polymerization of monomers (Becker & Gartner, 2000).

The selection of polymer material for microfluidic systems depends on the usage of the microfluidic devices and the available fabrication techniques. There are several parameters that play an important role when choosing a polymer material. The first is the glass transition temperature, \( T_g \). That is the transition temperature of most polymers from a liquid to solid-like state during the cooling process. When the polymer is heated above \( T_g \), the energy from the motion of the polymer chain will overcome the intramolecular friction. This will lead to the free movement of the polymer molecules and a softening of the material. Below \( T_g \), the polymers are usually a rigid and solid material. The glass transition temperature heavily influences the choice of polymer materials for microfluidic devices. For instance, during the thermal molding of polymers, polymers only start molding above \( T_g \) and demolding after the temperature is lower than \( T_g \). Some other parameters are also very important according to the applications of the microfluidic devices, such as auto-fluorescence (when using optical detection), gas permeability (for living cells or gas and liquid separation) and chemical resistance (if using acid or caustic fluidics) (Becker & Locascio, 2002).

**1.3.2 Polymer Materials Used in Microfluidic Systems**

A broad variety of polymer materials have been used in the microfluidic systems. Some of the most commonly used polymers include following: polycarbonate (PC), cyclic olefin polymers (COP), cyclic olefin copolymers (COC), polymethylmethacrylate (PMMA), polypropylene (PP), polystyrene (PS) and polydimethylsiloxane (PDMS).
PMMA, historically has been often called acrylic glass, is particularly useful for disposable microfluidic chips because of its low price, rigid mechanical property, excellent optical transparency, and compatibility to electrophoresis (Vourdas et al., 2008). PDMS has also attracted lots of interest from researchers for the simple and high resolution casting process used to transfer the pattern from a master mold with the sub-0.1 μm fidelity (Anderson & Whitesides, 2000). Additionally, the chemical and physical properties (i.e. gas permeable) of PDMS make it possible to fabricate devices with more useful functionality. PS is one of the most widely used plastics with a high transmission of the visible light. PS provides some features such as biocompatibility, inert and resistivity again alcohols which could be very beneficial as the material for microfluidic devices. Table 1.1 shows the summary of polymer materials properties that are commonly used in microfluidic systems.

Table 1.1 Physical properties for commonly used polymers in microfluidic devices (Tsao & DeVoe, 2008).

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Acronym</th>
<th>T&lt;sub&gt;m&lt;/sub&gt; (°C)</th>
<th>T&lt;sub&gt;m&lt;/sub&gt; (°C)</th>
<th>CTE (10&lt;sup&gt;-5&lt;/sup&gt; °C&lt;sup&gt;-1&lt;/sup&gt;)</th>
<th>Water absorption (%)</th>
<th>Solvent resistance</th>
<th>Acid/base resistance</th>
<th>Optical Transmittance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclic olefin (co) polymer</td>
<td>COC</td>
<td>70-155</td>
<td>150-320</td>
<td>60-80</td>
<td>0.01</td>
<td>Excellent</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polymethylmethacrylate</td>
<td>PMMA</td>
<td>100-122</td>
<td>250-260</td>
<td>70-150</td>
<td>0.3-0.6</td>
<td>Good</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>PC</td>
<td>145-148</td>
<td>260-270</td>
<td>60-70</td>
<td>0.12-0.34</td>
<td>Good</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polystyrene</td>
<td>PS</td>
<td>92-100</td>
<td>240-260</td>
<td>10-150</td>
<td>0.02-0.15</td>
<td>Poor</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polypropylene</td>
<td>PP</td>
<td>-20</td>
<td>160</td>
<td>18-185</td>
<td>0.10</td>
<td>Good</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Polyetheretherketone</td>
<td>PEEK</td>
<td>147-158</td>
<td>340-350</td>
<td>47-54</td>
<td>0.1-0.5</td>
<td>Excellent</td>
<td>Good</td>
<td>Poor</td>
</tr>
<tr>
<td>Polyethylene terephthalate</td>
<td>PET</td>
<td>69-78</td>
<td>248-260</td>
<td>48-78</td>
<td>0.1-0.3</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Good</td>
</tr>
<tr>
<td>Polyethylene</td>
<td>PE</td>
<td>-30</td>
<td>120-130</td>
<td>180-230</td>
<td>0.01</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Fair</td>
</tr>
<tr>
<td>Polyvinylidene chloride</td>
<td>PVDC</td>
<td>0</td>
<td>76</td>
<td>190</td>
<td>0.1</td>
<td>Good</td>
<td>Good</td>
<td>Good</td>
</tr>
<tr>
<td>Polyvinyl chloride</td>
<td>PVC</td>
<td>80</td>
<td>180-210</td>
<td>50</td>
<td>0.04-0.4</td>
<td>Good</td>
<td>Excellent</td>
<td>Good</td>
</tr>
<tr>
<td>Polysulfone</td>
<td>PSU</td>
<td>170-187</td>
<td>180-190</td>
<td>55-60</td>
<td>0.3-0.4</td>
<td>Fair</td>
<td>Good</td>
<td>Fair</td>
</tr>
</tbody>
</table>

T<sub>m</sub>: melting temperature, CTE: coefficient of thermal expansion

Polymers offer a promising alternative to glass and silicon as the materials for microfluidic devices. Polymer materials can provide material properties in a wide range, low material cost as well as rapid and flexible fabrication processes. Polymer-based
microfluidic devices are disposable and can avoid the risk of sample contamination, comparing with glass, silicon or quartz microfluidic devices. Fabrication methods for the polymer materials are more flexible and low-cost, comparing with the highly sophisticated instruments in the cleanroom for the fabrication of silicon or glass based microfluidic devices.

However, compared with glass or silicon, the polymer materials have some drawbacks such as lower resistance to chemical solvents, greater aging, and lower mechanical and UV stability. It is also worth to mention that polymers have an increased fluorescence at the wavelength shorter than 400 nm compared with glass, which is a drawback when using the laser-induced fluorescence detection (LIF) method for the investigation of the fluid flow inside the microchannels.

1.4 Fabrication Techniques for Polymer-based Microfluidic Devices
1.4.1 General Introduction to Fabrication Techniques
Fabrication techniques for microfluidic devices started with the experience inherited from microelectronics field. At the early stage, the most commonly used materials were glass and silicon, fabricated using the photolithography method inherited from silicon microelectronics. Then, with the development of microfluidics technology, more and more applications were found in the chemical analysis and biological fields which require low-cost and disposable devices to lower the cost and avoid cross-contamination. Instead of using glass or silicon and their complex fabrication techniques, polymers proved to be the more suitable choice for the low-cost rapid fabrication methods. Additionally, polymer materials (PS, PE, PMMA, etc.) have been used in laboratory tools (beakers, containers, flasks) for a long time, users are very familiar with them and the material properties have been well studied and characterized.

With more than 20 years' development after the first conceptual suggestion of using microfluidic devices for chemical analysis in 1990s, a wide variety of fabrication methods for polymer-based microfluidic devices have been invented. These fabrication methods for polymer-based microfluidic devices will be briefly introduced in the following sections in this chapter.
1.4.2 Soft Lithography

The concept of soft lithography refers to a group of techniques for the fabrication or replication of structures using replica molding (REM), micro-contact printing (µCP), micro-transfer molding (µTM) and some other micro molding methods. The method is called “soft” because instead of using the rigid mask for the regular photolithography process, all the above mentioned soft photolithography methods use polymers as the stamp, mold or mask to generate the micropatterns and microstructures.

PDMS is one of the most commonly used polymers for the soft lithography process as the replica material due to its suitable properties: low surface interfacial free energy, transparent (down to 300nm of wavelength), biocompatibility, gas permeable and stable thermal behaviors. Figure 1.1 displays a typical fabrication process of microfluidic device using PDMS stamp (Xia & Whitesides, 1998). The process starts with spin coating photoresist (SU-8) on a silicon wafer, then the SU-8 layer was patterned by UV exposure using a mask (the mask could be fabricated with various low-cost methods). Then, the unexposed SU-8 photoresist was developed using chemicals, leaving the exposed areas on silicon wafer. After that, the PDMS oligomer and cross-linking agent are mixed with the ratio of 10:1 and casted on the silicon wafer. After 1 hour bake at 60 °C and demolding (gently peeling off from the wafer), the PDMS replica was ready for use. The patterned SU-8 master mold on silicon wafer could be reused for several times.

However, there are also some drawbacks of PDMS: the PDMS structures will shrink (~1 %) during the curing process.
Generally, the greatest advantages of soft lithography compared with the conventional photolithography method are the rapid fabrication process and low-cost of the materials. With the soft lithography techniques, it is possible to design, fabricate the master and start to produce the replicated structures within one day. Unlike the regular photolithography process, the soft lithography process is applicable to most of the polymer materials. With the help of soft lithography, users can have a much wider choice of materials to meet their requirements than direct lithographic methods. In the followed chapters in this dissertation, soft lithography will also be used for low-cost fabrication of microchannels and metal structures for polymer-based microfluidics.

### 1.4.3 Hot Embossing

Hot embossing is one of the most efficient methods for the replica fabrication of microfluidic devices (Yang & DeVoe, 2013). In a typical hot embossing process, a polymer substrate is aligned and put in direct contact with the master mold which is made of more rigid materials like silicon, glass and SU-8. After that, this polymer material (usually thermoplastics like PMMA or PC) is heated up to the glass transition
temperature and pressure is applied between the master mold and polymers, transferring the desired patterns to the surface of the polymer material.

Figure 1.2 Hot embossing method for fabricate microfluidic devices for cell culture on COC. (a): Patterning SU-8 on silicon substrate. (b): PDMS molding (c): Molding of epoxy using PDMS mold. (d) (e): Using the epoxy as the master mold for the hot embossing on COC. (f)(g)(h): Cutting, drilling for inlet and outlet ports and sealing the microchannels (Jeon et al., 2010).

Figure 1.2 shows a commonly used hot embossing procedure to fabricate a microfluidics device which was used for cell culture (Jeon et al., 2010). This process started with patterning SU-8 photoresist on a silicon substrate (figure 1.2a) with an ordinary photolithography process. Then, the PDMS was poured on the substrate and cured (figure 1.2b). After that, a more durable epoxy master mold was patterned using the PDMS mold (figure 1.2c). This epoxy master mold was then employed to the hot embossing process of COC at 100kPa with the temperature of 120 °C for an hour. After the hot embossing process, the COC polymer was demolded at 60 °C. Finally, the inlet and outlet holes were drilled, and the microchannels were sealed by bonding another layer of COC. The SEM images are provided in figure 1.3 for the silicon wafer, PDMS mold, epoxy master mold and the COC microfluidic devices fabricated after hot embossing process.
With the development of hot embossing method for fabricating microfluidic devices, a high efficiency and high speed replication technique called “hot roller embossing” attracted lots of interests from researchers recently. Ten et al. first reported the roller nanoimprint lithography in 1998 (Tan, 1998). After that, more and more researches have conducted hot roller embossing experiment and reached the imprinting speed of 1000 mm/s for the polymer foil with the width of 2 m. The main drawback of the roller hot embossing is the aspect ratio of the features, ratio of 1:1 is easily to replicate, 5:1 needs more care and it is very difficult to reach 10:1 (Ng & Wang, 2008).

Figure 1.4 shows a typical hot roller embossing process. The upper roller was made of steel and covered with nickel film which was patterned by electroplating. The lower roller was also made of steel and covered with rubber. Heat and pressure were applied by these two rollers during the hot embossing process. A 1.5 mm thick PMMA sheet was insert between the two rollers, a typical system settings were: temperature from 80-160°C, pressure ranges from 1 to 6 bar and linear speed of 1.5-35 mm/s. Result showed the depth could reached up to 30 µm with the feature size of 100 µm (Ng & Wang, 2008).
1.4.4 Injection Molding

Up to now, injection molding is still the most widely used fabrication method for polymer materials in the macroscale fabrication field in industry. However, injection molding has not yet been widely used in microfabrication of polymer-based microfluidic devices, due to high instrument cost and complicated fabrication process.

A schematic of an injection molding instrument is shown in figure 1.5, the polymer materials (in powder) were fed into the hopper and then driven by hydraulic pressure through a heated barrel. The temperature in the chamber can reached as high as 200-350 °C depending on the different polymer materials used, in order to melt the polymers into a liquid state. The liquid polymer was then injected into the mold with a high pressure through a sprue (typically at 600 to 1000 bar). The pressure was held for a while for the complete filling of the mold and polymer solidification (cooling), followed by demolding. Demolding included a series of complicated processes, which required accurate control of temperature and pressure matched to each different polymer and the aspect ratio of the
microstructures to avoid air bubble formation or damage to the structure. Figure 1.6 shows a typical injection molding process of fabricating a microfluidic devices introduced in (Heckele & Schomburg, 2004). The injection molding process can then be repeated with typical cycle times from a few seconds to a few minutes.

Figure 1.5 Schematic of the cross-section of injection molding instrument (Tosello & Hansen, 2010).

Figure 1.6 Typical process for the injection molding. (a): Mold is sealed and heated up. (b): Polymer materials were injected. (c): Cooling down lower than the glass transition temperature and demolding (Heckele & Schomburg, 2004).

The advantages of injection molding are obvious, it can fabricate a large quantity of microfluidics chips within a short time, the fabrication process can be fully automated, and replication of the shape is accurate and allows for a flexible choice of polymer materials. However, there are also some drawbacks of injection molding for microfluidics chips: instrument cost is very high compared with other low-cost fabrication methods, the
mold needs to be refabricated with the change of microfluidics chip design, and the lack of the metal electrode integration ability. Primarily, injection molding is a high volume production tool and is prohibitively expensive for the type of research setting at which the work in this thesis is aimed.

1.4.5 Laser Ablation

With the increasing demand for low-cost and rapid fabrication of polymer-based microfluidic devices, lasers have been found to be a useful tool in microfabrication in recent years. Lasers have been widely used in many areas in industry from product design to production, the properties of the different kind of lasers have been well characterized (Khan Malek, 2006). The relatively high expense of laser systems used to prevent their wide application in microfabrication in the past years. However, with the falling of the cost for laser systems, nowadays more and more researchers are starting to use various kinds of laser systems for the fabrication of polymer microfluidic systems.

Generally, the lasers used in the polymer-based microfluidics fabrication have a wide range of wavelength from extreme UV (ultraviolet) (157 nm) to infrared laser (IF) (10 μm). These lasers can be classified into two groups based on their wavelength. The first group is the laser systems with short wavelength (below 500 nm), which includes UV laser and excimer laser. Another group is the laser systems with relatively longer wavelength, such as Nd:YAG laser and CO2 laser. Table 1.2 compares the wavelength, system cost and minimum feature the between the UV laser, CO2 laser and Nd:YAG laser used in microfabrication for polymer materials. It can be easily observed from Table 1.2 that the cost of the UV laser system is about 6 times higher than the CO2 laser system, while the minimum feature fabricated by a UV laser (~6 μm) is much smaller than that of a CO2 laser (~80 μm). Since most of the microchannels fabricated on polymers in microfluidics are in the range from 50 μm to 500 μm, CO2 lasers seems to be an optimum choice for most of applications in microfluidics.

For all kinds of laser systems, laser ablation can be utilized for the fabrication of microchannels on polymer materials. During the laser ablation process, polymer materials absorb the strong laser illumination, leading the polymers to heat up and causing removal of the material after a certain threshold. The laser ablation mechanism is very
complicated, for the UV laser, the photon absorption of from UV laser can directly break the polymer bonds directly, as well as thermal breakdown (Klank et al., 2002b). For the CO₂ laser, the ablation process is purely thermal, the materials melt, thermally decompose and vaporized. Therefore, the wavelength of the laser plays an important role during the ablation process. Generally, the shorter the wavelength is, the smaller microstructure feature size that can reach. Besides wavelength, the duration of the laser pulse will also seriously influence the minimum feature size and surface qualities. The ultra-short-pulse laser, which is also called femtosecond laser, can reach the sub-micron feature size. Ke (Ke & Hasselbrink, 2005) proposed a femtosecond pulse laser system (wavelength 527 nm, pulse time 800 fs) with a repetition rate of 1.5 kHz that can fabricate channels on a glass substrate with a diameter as narrow as 700 nm.

Table 1.2 Comparison between UV laser and CO₂ laser for microfabrication.

<table>
<thead>
<tr>
<th>Type of laser</th>
<th>Wavelength</th>
<th>System cost</th>
<th>Operation time</th>
<th>Minimum feature</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV laser</td>
<td>266 nm</td>
<td>About US$ 100,000</td>
<td>1000 hours for crystal replacement</td>
<td>About 6 µm</td>
</tr>
<tr>
<td></td>
<td>355 nm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CO₂ laser</td>
<td>10.6 µm</td>
<td>About US$ 15,000</td>
<td>1000 hours for laser cartridge replacement</td>
<td>About 80 µm</td>
</tr>
<tr>
<td>Nd:YAG laser</td>
<td>1064 nm</td>
<td>About US$ 60,000</td>
<td>8,000 to 10,000 hours for crystal replacement</td>
<td>About 100 µm (on glass)</td>
</tr>
</tbody>
</table>

Figure 1.7 compared the system setup for UV laser and CO₂ laser system. The UV laser has a frequency quadrupled Nd:YAG laser source of the wavelength at 266 nm and adjustable pulse duration down to 10 ns with the maximum power output at 1.5 W (Cheng et al., 2005). A CNC stage was used for control the laser to direct write on glass substrate. The microchannel fabricated with this direct UV laser writing system is shown in figure 1.8a with a depth around 5 µm.
The system setup for CO$_2$ laser is very similar to the UV laser system except the wavelength of the laser source is $10.6 \mu$m with the maximum power output of 12 W (Hong et al., 2010). Figure 1.8b shows the scanning electron microscope (SEM) image of the CO$_2$ laser ablated microchannel on PMMA. The laser power was set at 4 W with a scanning speed of 120 mm/s. The Gaussian profile can be easily observed from figure 1.8b. Figure 1.9 shows the formation process of this Gaussian like profile. When the CO$_2$ laser scanning on the surface of PMMA, a molten pool is formed, some of the material vaporizes from the molten pool, while some material remains and will re-solidify. The profile of the cross-section depends on the thermal diffusivity of the PMMA substrate (Klank et al., 2002b). Since the thermal diffusivity of PMMA as well as most of other
polymers is relatively low, the intensity distribution will decide the profile of the channel cross section. Thus, the Gaussian like profile is formed on the PMMA substrate after CO₂ laser ablation.

Figure 1.9 Schematic of the laser ablated microchannel cross-section on PMMA substrate (Klank et al., 2002b).

For the research conducted this dissertation, the CO₂ laser ablation will be widely used for the processing of polymer materials in the low-cost polymer-based microfluidics system, the detailed experimental process and result will be introduced in chapter 2.

1.5 Surface Modification for Polymer-based Microfluidic Devices
Due to the wide applications of polymer materials in microfluidic systems, changing the surface properties of the polymers after the fabrication of microchannels has attracted a lot of research interest. Researchers were trying to modify the surface properties in many ways: lowering the surface roughness to increase optical quality (Ogilvie et al., 2010), increasing hydrophilicity of microchannels (Soper et al., 2002), enhancing biological compatibility (Goddard & Hotchkiss, 2007) and reducing surface roughness after laser treatment (Cheng et al., 2004). In the following sections, some commonly used surface modification methods will be presented.
1.5.1 Surface Modification to Lower the Surface Roughness.

A lot of research has been conducted to lower the roughness of the polymer surface (Prokhorova & Kopyshev, 2003; Ogilvie et al., 2010; Lin & Hosseini, 2012). Polymer materials like PMMA or COC are commonly patterned by injection molding or hot embossing. The surface roughness is in the order of 100 nm (Ogilvie et al., 2010), which is acceptable at most of the time, but surface modification will be needed if application requires an enhanced optical quality. Some of the commonly used methods include chemical vapor treatment (Ogilvie et al., 2010; Lin & Hosseini, 2012) and thermal recycling (K & Z-G, 2002). Figure 1.10 shows the surface modification of PMMA and COC. Microchannels on PMMA and COC were fabricated using micro-milling, the surface roughness after fabrication was 100-200 nm, after the chemical vapor treatment and heat recycling, the surface roughness has been reduced to less than 15 nm (Ogilvie et al., 2010).

![Figure 1.10 Images for the surface modification of PMMA and COC. (a): PMMA microchannels after milling. (b): PMMA microchannels after 4 minutes chloroform vapor treatment and 30 minutes heat recycling at 60 °C. (c): COC after milling. (d): COC microchannels after 4 minutes cyclohexane vapor treatment and 30 minutes for heat cycling treatment at 60 °C (Ogilvie et al., 2010).](image-url)
1.5.2 Surface Modification to Enhance the Biological Compatibility

The polymer-based microfluidic devices have been widely used in the biological field. There are many studies conducted to enhance the biological compatibility of the polymer surface in order to attach the bio-compounds (cells, antibody, DNA, etc.) to the polymer substrate (Henry & Tutt, 2000; Chu & Chen, 2002; Makamba et al., 2003; Sui & Wang, 2006; Goddard & Hotchkiss, 2007). Due to the inert nature of most of the polymer materials, the surface needs to be functionalized before the attachment of bio-compounds.

Figure 1.11 shows a typical surface functionalization process for bio-compounds attachment. After choosing and cleaning polymer substrate, a layer of polyfunctional agent was grafted on the surface of polymer material. Then, another layer of spacer molecule was attached to further enhance the bioactivity and spacing the bio-compound that attached to the hydrophobic surface of the polymer. Finally, the biomaterial (i.e. Enzyme, Peptide, Antibody) was attached (Goddard & Hotchkiss, 2007).

1.5.3 Increase the Hydrophilicity of Microchannels

As mentioned before, PDMS is the most widely used polymer for the bio-related microfluidic devices. The advantages of using the PDMS in bio-related microfluidic devices include the following: excellent optical properties with transparency down to 200 nm, non-toxic and gas permeable. However, the biggest drawback is the hydrophobic surface properties of PDMS after the curing process, which makes the microchannels hard to fill with aqueous liquids. Surface modification needs to be performed to overcome this drawback in some applications.

The surface of PDMS can be plasma (Chaudhury & Whitesides, 1991) or UV (Efimenko et al., 2002) treated to increase hydrophilicity. Figure 1.12 shows the AFM
images before and after PDMS surface treatment using oxygen plasma (Sung et al., 2006). However, the hydrophobic nature of PDMS will return in time, which is called the recovery effect (Makamba et al., 2003). The recovery effect is mainly due to the low molar mass chains from bulk material migrating to the surface (Hillborg et al., 2000). In order to reduce the recovery of hydrophobicity after surface treatment, researchers are trying to coat the poly (ethylene glycol) (PEG) on the surface of PDMS immediately after plasma or UV treatment (Klenkler et al., 2008; Mikhail et al., 2010). Up to now, the recovery of the PDMS after treatment has not been fully solved. It is worth to mention that, besides PDMS, other polymers like PMMA, PS, PE also have a hydrophobic surface quality. Surface modification may be needed for the microfluidic devices fabricated on these polymers depending on the intended application.

Figure 1.12 AFM images for the surface of PDMS before (a) and after (b) surface treatment using oxygen plasma (Sung et al., 2006).

1.5.4 Reduce the Surface Roughness after Laser Ablation on Polymer Materials
Using laser ablation for microchannel fabrication on polymers has been introduced in the previous sections. As mentioned, during the laser ablation process, polymer will be heated up due to photon absorption, which generates thermal energy. For CO₂ laser ablation, the laser ablation process is purely thermal (Klank et al., 2002b). Polymers are melted and vaporized in a very short time, this violent phase change leaves a very rough surface after CO₂ laser ablation (figure 1.13a). Researchers have to reduce the surface roughness by thermal annealing (170 °C for 30 minutes) of the polymers after laser ablation (Cheng et al., 2004; Bartnik et al., 2009) or pre-heat the substrate (between 80 °C
to 90 °C) before laser ablation to prevent the formation of air bubbles (Huang et al., 2010).

Figure 1.13 CO₂ laser ablated microchannels before and after thermal annealing. (a): The CO₂ laser ablated microchannels. (b): Same microchannels after thermal annealing process, insert shows the AFM (atomic force microscope) topography (Cheng et al., 2004).

Figure 1.13 compares the microchannels fabricated by CO₂ laser ablation on PMMA substrate with and without the post thermal annealing process. Authors in this research claimed that the roughness has been reduced from 5-10 µm to less than 2 nm (Cheng et al., 2004).

1.6 Bonding Techniques for Polymer-based Microfluidic Systems

After the fabrication of microchannels on polymer substrates, regardless which fabrication method has been used, we need to enclose (seal) the fluid channels. For polymer-based microfluidics, the most commonly used method is to bond another layer of polymer onto the substrate to enclose the microchannel. The polymer material for this layer may or may not be the same material as the substrate layer with the pre-fabricated fluid channels.

Bonding is the most important part of the back-end processing for microfluidics chip fabrication. There are several parameters that need to be taken into consideration to achieve proper bonding. First and foremost, the bond strength. In order to safely seal the fluid channels without leaking, we need to make sure the bond strength is high enough and has enough tolerance to the changes in the environment (temperature, chemical and
solvent). Some other parameters, like the optical properties, surface chemistry of the bonding interface, and smoothness of the channel sidewall also need to be taken into consideration when choosing the bonding method. It is worth mentioning that, on choosing the bonding method and bonding parameters, we need to make sure that the bonding will not cause significant chip deformation or collapse and blockages of the microchannel, which are the two most commonly seen bonding failures.

Generally, bonding methods could be classified as either indirect or direct bonding. These two methods will be briefly introduced in the following sections.

1.6.1 Indirect Bonding of Polymer Materials

Adhesive bonding has been widely used as the indirect bonding method for the polymer-based microfluidic devices. Adhesive bonding is usually a simple process without the special requirement of the instruments. Generally, the adhesive bonding is achieved by adding a layer of adhesive high viscosity liquid between the two surfaces that need to be bonded. Then, this layer of adhesive is cured either by UV exposure (Schlautmann & Besselink, 2003; Tuomikoski & Franssila, 2005; Pan & Yang, 2006) or thermal treatment (Yu et al., 2006; Samel et al., 2007).

A typical bonding process by curing the adhesive layer using thermal treatment is shown in figure 1.14 (Samel et al., 2007). After the PDMS microstructures with microchannels was fabricated by replica molding, the curing agent (Sylgard 184) of PDMS was spin coated on a silicon wafer with the rotation speed of 6000 rpm (figure 1.14b). After that, the pre-fabricated PDMS microstructure and silicon wafer coated with curing agent were attached (figure 1.14c) and carefully baked at 50 °C for 24 hours (figure 1.14d) to avoid the delamination caused by the trapped air bubbles. Author claimed that the bonding strength could reach as high as 531 kPa using this adhesive bonding method.
Figure 1.14 Schematic of the adhesive bonding process of PDMS on silicon substrate (Samel et al., 2007). (a): Replica molding of PDMS structures. (b): Spin coating of curing agent on silicon wafer. (c): Attach the PDMS structures with the curing agent spin coated on silicon wafer. (d): Baking for 24 hours to achieve the bonding.

Figure 1.15 shows a method of indirect bonding using UV curable resin (Lai et al., 2004). The fabricated PMMA microfluidics platform with the lid plate were encapsulated in a chip holder, then hydroxyethylmethacrylate (HEMA) and surface modification agent were injected through the inlet port to fill the gap between the platform and lid. High pressure nitrogen was then used to pump the resin out from the channels. The resin left in the gap between the platform and lid was then cured by a UV radiation source. The resin (HEMA) could also be thermally cured in this case.

Figure 1.15 Procedure of resin-gas injection bonding (Lai et al., 2004).
The advantage of the indirect bonding (adhesive bonding) is the low requirement for equipment, usually without the need for any sophisticated instrument. On the other hand, the disadvantage is obvious: the residuals of the adhesives left inside the microchannel may dissolve in the fluid inside the microchannel, which could contaminate the fluid and influence the experimental result.

1.6.2 Direct Bonding of Polymer Materials

1.6.2.1 Thermocompression Bonding

Thermocompression bonding is also called the thermal fusion bonding. The substrate and covering layer are heated near the glass transition temperature \(T_g\), then pressure is applied between the contacting surfaces. The mechanism behind the thermocompression bonding is the molecular entanglement of the two attached polymer surfaces, which under high temperature and high contacting pressure will lead to a very strong bonding force.

Thermocompression bonding has been widely reported on various kinds of polymers such as PMMA (Kelly & Woolley, 2003; Zhu et al., 2006), PS (polystyrene) (Li et al., 2011a), PC (polycarbonate) (Ogończyk et al., 2010), COC (Shim et al., 2010; Roy et al., 2011) and COP (Pemg et al., 2010). Besides bonding the polymers of the same material, the bonding between different polymers (ie. PMMA-COC (Kim et al., 2007; Tsao et al., 2007b), PMMA-PS (Fan et al., 2013b)) were also reported.

Although the thermocompression bonding process is simple and effective, bonding failure due to sidewall collapse and blockage of microchannels is common. The reason for this bonding failure is having either the temperature or the bonding pressures too high. To achieve proper bonding, we need to accurately control the bonding temperature within the range from \(T_g\) to the melting point, as well as the amount of compression pressure.

A simple system setup for the thermocompression bonding is shown in figure 1.16. Two stainless steel plates were connected by four screws, the parylene coated glass (up) and parylene coated silicon (down) wafers were sandwiched between two steel plates. The two parylene layers are the structure layers fabricated on the two glass wafers. By fastening the four screws, pressure will accumulate on the contact surfaces of the
parylene layers. During the thermocompression bonding process, this bonding system will be placed in a convection oven at 160 °C with a pressure between two plates of 4.9 MPa for 30 minutes to achieve the desired bonding strength.

Figure 1.16 System setup for parylene thermocompression bonding (Ziegler et al., 2006).

1.6.2.2 Solvent Bonding

Solvent bonding is a simple and rapid process for the bonding of polymer materials and has been widely used (Lin et al., 2007; Ng et al., 2007; Sun et al., 2007). For solvent bonding, organic solvent is applied between the two contact surfaces of polymers, the polymer chains become mobilized, and result in entanglement of the polymer chains across the interface.

The solubility of polymers in organic solvent can be easily found out with the help of Hildebrand solubility parameter ($\delta$). This parameter provides a numerical estimation of the degree of interaction between polymers, in our case, can be a useful indication of solubility of polymer materials in organic solvent. Table 1.3 provided the solubility parameter of the commonly used polymers and organic solvent in microfabrication. In this table, each polymer materials and organic solvents has a solubility parameter $\delta$. Polymers easily dissolve in the organic solvent which has a similar solubility parameter. For example, according to Table 1.3, PMMA ($\delta=20.1$) is easily dissolved in acetone ($\delta=20.4$), which has a similar $\delta$, but dissolves much slower in ethanol ($\delta=26.0$).

A typical solvent bonding process is illustrated in figure 1.17 (Lin et al., 2007). The solvent (DE-20) used for bonding was a mixture of two organic solvents: 20 % of 1,2-dichloroethane and 80 % of ethanol. Author claims the 1,2-dichloroethane helps to glue
the surfaces while the ethanol was used for protecting the microchannels. The bonding process starts with cleaning the PMMA substrate in acetone for 10 s. Then the DE-20 was applied to the contact surfaces of PMMA. Finally, the PMMA substrate and cover plate were attached and clamped with a pressure of 10 N/cm² for 5 minutes at room temperature. We may notice that, in this process, some solvent residue left inside the microchannels, which needs to be cleaned before use. Due to the chemical reactions between PMMA and the bonding agent, the bonding agent residue inside the microchannel can hardly be completely cleaned.

Table 1.3 Solubility properties of the commonly used polymer materials in microfluidic devices and organic solvents (Tsao & DeVoe, 2008).

<table>
<thead>
<tr>
<th>Polymer</th>
<th>δ (J/cm³)</th>
<th>Solvent</th>
<th>δ (J/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PTFE</td>
<td>12.6</td>
<td>Cyclohexane</td>
<td>16.7</td>
</tr>
<tr>
<td>PE</td>
<td>16.3</td>
<td>Methylene dichloride</td>
<td>19.8</td>
</tr>
<tr>
<td>PP</td>
<td>16.3</td>
<td>Ethylene dichloride</td>
<td>20.0</td>
</tr>
<tr>
<td>COC</td>
<td>17.7</td>
<td>Acetone</td>
<td>20.4</td>
</tr>
<tr>
<td>PMMA</td>
<td>20.1</td>
<td>n-Hexanol</td>
<td>21.8</td>
</tr>
<tr>
<td>PS</td>
<td>18.7</td>
<td>Isopropanol</td>
<td>23.4</td>
</tr>
<tr>
<td>PVC</td>
<td>19.4</td>
<td>Acetonitrile</td>
<td>25.1</td>
</tr>
<tr>
<td>PC</td>
<td>19.4</td>
<td>Ethanol</td>
<td>26.0</td>
</tr>
<tr>
<td>PET</td>
<td>21.8</td>
<td>Dimethyl sulfoxide</td>
<td>26.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Methanol</td>
<td>29.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Water</td>
<td>47.7</td>
</tr>
</tbody>
</table>
Since the microchannels are easily clogged by the organic solvent, the choice of solvent in the solvent bonding process is critical. The composition of the solvent should be strong enough to dissolve the polymer surface without damaging the microchannels. Also the bonding temperature and pressure need to be taken into consideration carefully to achieve a secure bonding without damaging the microchannels.

**1.7 Research Motivation and Objective**

In recent years, microfluidics technologies have found wide applications and significant advantages in the biological and chemical fields. At the same time, polymers are replacing the glass and silicon as the major materials being used for the fabrication of microfluidic devices, which lowers the material cost and simplifies the fabrication process (Developpement, 2013).

However, as introduced in the previous sections, fabrication technologies for polymer-based microfluidics are inherited from MEMS (microelectromechanical systems), and usually require highly sophisticated instruments in the cleanroom and professional knowledge of microfabrication. Because of this, lack of instruments or lack
of microfabrication knowledge can be a large barrier for biologists and chemists to benefit from the advantages of microfluidics technologies.

As illustrated in figure 1.18, the common microfluidics system’s fabrication process usually contains three steps: the first step is microchannel fabrication, where microchannels with desired dimensions are fabricated on the surface of the polymer substrate; the second step is to enclose (seal) the microchannels, usually by bonding another layer of polymer material on top of the substrate; the third step is integration, based on users’ application, some components such as metal electrodes (for heating or detection), microlenses (for easier observation) and “world-to-chip” connectors could be integrated in the microfluidic systems.

Figure 1.18 Schematic diagram of the general methodology for fabricating microfluidic systems.

This research is aiming to provide a series of cleanroom free and low-cost fabrication technologies for polymer-based microfluidic systems, cut down the technical barrier, making the microfluidics technologies more accessible for researchers in biological and chemical fields.

The fabrication methods proposed in this research covered all the above mentioned three steps in the fabrication process for polymer-based microfluidics. Instead of using highly sophisticated instruments in the cleanroom, the fabrication methods provided in this research only require some common laboratory instruments and do not require a cleanroom environment. These fabrication technologies have a wide adaptability to various kinds of low-cost commonly used polymer materials. By cutting the cost of instruments and materials, these proposed fabrication technologies could significantly lower the fabrication cost for polymer-based microfluidic systems in a research setting.
1.8 Chapter Outline

There are five chapters in this dissertation. Chapter 1 is the general introduction for the polymer-based microfluidics, motivation and goal of this research. The following three chapters will be introduced as the same order of microfluidic systems fabrication process: microchannel fabrication, microchannel sealing and integration technologies. Chapter 2 will propose various kinds of low-cost microchannel fabrication technologies on polymers using laser processing, deep-UV exposure, etc. Chapter 3 will discuss sealing the microchannels fabricated on different polymer materials using a thermocompression bonding method. Chapter 4 will introduce some low-cost integration technologies for microfluidic system. Components such as metal electrodes and microlens are integrated into microfluidic systems with various low-cost methods. Some other low-cost back-end processing techniques after microfluidics chip fabrication like wafer scribing and a chip holder setup are also introduced in chapter 4. Chapter 5 is the conclusion and future directions for this research.
Chapter 2  Low-cost Polymer-based Microchannel Fabrication Technologies

As introduced in chapter 1, there are various different methods for the fabrication of polymer-based microfluidic devices. These fabrication methods included: thick resist lithography (X-Ray LIGA), polymeric surface micromachining, soft lithography, microstereo lithography and injection molding. However, most of these fabrication processes are conducted in the cleanroom with highly sophisticated microfabrication instruments. For LIGA process, the cost of the X-Ray source is very high. For soft lithography, usually the silicon wafer is used as the master mold with the expensive and complicated microfabrication process in the cleanroom. Microstereo lithography and injection molding both need the highly sophisticated instruments to perform the microfabrication process.

In this chapter, instead of using these high-priced fabrication methods in the cleanroom, a series of low-cost fabrication methods for polymer-based microfluidic systems without the requirement of the cleanroom environment will be introduced. The first method is deep-UV patterning of PMMA using printed wax shadow mask. Instead of using laser mask writer, sodaline glass and UV source in the cleanroom, this research used wax printer, wax and self-made deep-UV source as the mask fabrication method, mask materials and light source, this significantly lowered the cost of the fabrication. The minimum width of the microchannel fabricated using this method is as low as 50 μm. A low-cost CO₂ laser processing method for polymer-based microfluidic systems will also be introduced in this chapter. In this research, the CO₂ laser was used either directly ablate microchannels on the surface of polymers or inducing the bumps on the surface of polystyrene. A deep-UV LIGA process will also be introduced in this chapter, this low-cost method can easily achieved an aspect ratio of 5:1 for metal structures.

2.1 Deep-UV Patterning for Low-cost Microchannel Fabrication

As introduced in the previous chapter, PMMA is one of the most widely used polymers for microfluidic devices. PMMA is a cheap commercial product sold with different brand names with the commercial name “acrylic”. PMMA was first used in the
microfabrication process from the introduction of the LIGA process in 1980s (Becker & Miinchmeyer, 1986). At the early stages of using PMMA in microfabrication, PMMA was used as a photoresist in photolithography (Kawamura & Toyoda, 1982), the PMMA dissolved in solvent was spin coated on a substrate (silicon, glass or quartz) and patterned by photolithography. Nowadays, besides using the PMMA as a photoresist, microstructures can also be directly patterned on PMMA films or sheets with various fabrication methods.

Different types of radiation sources have been used for the patterning of PMMA, such as x-ray (H & R, 1990; P et al., 2002), e-beam (W et al., 2003; M & C, 2005) and deep-UV (Burke et al., 2008; Haiducu et al., 2008; Sameoto & Menon, 2010; Fan et al., 2012). When patterning the PMMA as a photoresist, a radiation source with shorter wavelength is preferred, generally these kinds of radiation source (i.e. x-ray, e-beam) are relatively expensive. However, PMMA can also be exposed and patterned by deep-UV radiation with very low cost. In our approach, we used a deep-UV radiation source with the wavelength of 254 nm. While this wavelength is slightly longer than those deep-UV radiation sources usually used (wavelength lower than 250 nm), it is still within the upper limit of the effectiveness (Johnstone et al., 2008). The reason for using this 254 nm deep-UV to pattern PMMA is the low-cost of the light source. To generate deep-UV radiation, mercury-vapor lamps with the strongest peak at 254 nm were used in our experiment as the radiation source for the patterning of microchannels on a PMMA substrate. The custom-made deep-UV exposure system is shown in figure 2.1, this low-cost deep-UV exposure box was made of aluminum and mounted with 8 mercury-vapor lamps inside the box. A rotational stage inside the box was designed to help the PMMA substrate uniformly absorb radiation from the light source. A UV sensor was also mounted inside the box to monitor the dose from the radiation source.
During the deep-UV exposure process of PMMA, the chemical chain of the PMMA molecules will scission due to the absorption of high energy radiation, the main chain scissions will lower the molecular weight at the exposed area on the bulk PMMA substrate, thus causing an increase of solubility in those exposed areas. The exposed areas can then be developed to remove the soluble material. Besides the solubility, the deep-UV exposure will also change some of the PMMA’s physical properties such as absorption spectrum, refractive index and glass transition temperature.

The exposed PMMA can be developed using several different methods. The most frequently used solvents are a mixture of MIBK (methyl isobutyl ketone) and isopropanol (IPA) (Yasin & D.G. Hasko, 2002; Olzierski, 2004) or a mixture of water and IPA (Yasin et al., 2001; Uchiya et al., 2007).

For all the development process of PMMA in this research, the deep-UV exposed PMMA was developed using the mixture of IPA/water. The advantages of this method are the low-cost of the solvent, and the mixture of water/IPA has higher sensitivity and contrast compared with the mixture of MIBK/IPA (Yasin et al., 2002). The method of using the mixture of IPA and water to develop the exposed PMMA was found in 1996 (Brambley & Bennett, 1996). It is interesting that, both IPA and water are not solvents for PMMA at the room temperature, but the mixture of IPA can work as a co-solvent system as a developer for PMMA.

In order to conduct a successful development, the chemical components, developing temperature and developing time need to be carefully controlled to ensure the full dissolution of the exposed area, which has the lower molecular weight, without influencing the unexposed area. Improper control of these parameters can lead to the
swelling and dissolving of the unexposed area, which would damage the polymer-based microfluidic chips.

2.1.1 Printed Wax Mask for the Deep-UV Patterning of PMMA

In this section, a novel low-cost method of fabricating PMMA based microfluidic devices with deep-UV patterned microchannels will be introduced. A commercial solid ink printer (Xerox Phaser 8560DN) was used to print the wax patterns on a thin layer of polymer film and then this wax layer was transferred to a PMMA substrate. After that, the PMMA substrate underwent a deep-UV exposure followed by the development of the exposed area. During the exposure process, the transferred wax layer worked as a photomask. Finally, after bonding another polymer layer to seal the microchannel, the device was ready for testing.

Wax printers have previously been reported for fabrication of paper-based open-channel microfluidics, in which the wax layer works as a hydrophobic barrier in the hydrophilic paper (Lu et al., 2009a; Lu et al., 2009b). In this research, we used the printed wax layer as the shadow mask for deep-UV patterning in a custom-made exposure box, which significantly lowers the cost of mask fabrication and deep-UV exposure. Further, the whole fabrication process for this microfluidics device can be performed without the cleanroom environment.

2.1.1.1 Fabrication Process

PMMA (Goodfellow Cambridge Limited, England, 1.1 mm in thickness) was chosen as the substrate for the patterning of the microchannels. The deep-UV radiation source is shown in figure 2.1. The schematic of the fabrication procedures is illustrated in figure 2.2.

The first step was wax printing quality test, the designed patterns were printed on various thin films using Xerox Phaser 8560DN solid ink printer. The graphics software AutoCAD 2010 was used to sketch the patterns, including a micromixer and microchannels, with varied widths for the testing of minimum channel width (shown in figure 2.3). Different polymer films (sourced from Goodfellow Co., Ltd, England) were
tested for the transfer quality of the wax patterns onto the PMMA substrate (Table 2.1). Result shows the high density polyethylene (HDPE) film (10 μm thick) has the best transfer quality of wax patterns. To perform the wax printing on HDPE film, the HDPE film was attached at the center of a standard A4 paper and sent through the wax printer for the printing (figure 2.2a).

Figure 2.2 Schematic of the fabrication process for PMMA based microfluidics (Fan et al., 2012). (a): Wax layer was printed on the surface of HDPE film. (b): Transfer the wax layer onto the PMMA substrate using thermal laminator. (c): Peel off the HDPE film after cooling down. (d): Deep-UV exposure. (e): Development process. (f): Remove the residue wax fragment. (g): Bonding another layer of PMMA to enclose the microchannel.

Figure 2.3 Layout of the designed microfluidic structures (Fan et al., 2012). (a): Designed patterns for testing. (b): Wax layer transferred on the PMMA substrate.
After wax printing, the HDPE film was carefully detached from the A4 paper and ready for the wax layer transfer process. A commercial thermal laminator (Heatseal H600 Pro, GBC Co., Ltd, USA) was used for transfer of the wax layer (shown in figure 2.2b). During the wax layer transfer process, the wax pattern printed on the HDPE film was in direct contact with the PMMA substrate and then went through the thermal laminator, the temperature of the roller in the thermal laminator was set at 93.3 °C with the speed of 3 mm/s. To reduce the stress (may cause pattern skewing) on the thin HDPE film during this thermal transfer process, the HDPE film was attached on a piece of paper of the same size during the pattern transfer in the thermal laminator.

<table>
<thead>
<tr>
<th>Polymer Films For Transfer</th>
<th>Film Thickness</th>
<th>Transfer Ratings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyethylene terephalate (PET/PETP) Film</td>
<td>0.1mm</td>
<td>+*</td>
</tr>
<tr>
<td>Poly carbonate (PC) Film</td>
<td>0.25mm</td>
<td>+</td>
</tr>
<tr>
<td>Polystyrene(PS) Film</td>
<td>0.125mm</td>
<td>++</td>
</tr>
<tr>
<td>Polypropylene (PP) Film</td>
<td>0.03mm</td>
<td>+++</td>
</tr>
<tr>
<td>Poly(methyl methacrylate) (PMMA) Film</td>
<td>0.05mm</td>
<td>+++</td>
</tr>
<tr>
<td>Polytetrafluoroethylene (PTFE) Film</td>
<td>0.1mm</td>
<td>++++</td>
</tr>
<tr>
<td>High-density Polyethylene (HDPE) Film</td>
<td>0.01mm</td>
<td>+++++</td>
</tr>
</tbody>
</table>

*+ represents the poorest transfer while 5 +’s represents the finest among those polymer films

After the wax pattern transfer procedure, it is essential to wait for the wax to cool down to the room temperature (23 °C) and solidify before peeling off the HDPE film (figure 2.2c) from the PMMA substrate. Figure 2.3b shows the PMMA substrate covered with a wax layer (~10 μm thick) that thermally transferred from a HDPE film. The reflow of wax layer to lower the roughness at the edge of wax pattern was also tested by baking the polymer substrate on a hot plate, due the hydrophobic contact between liquid wax and PMMA substrate, this reflow test is not successful.

The next step was deep-UV exposure, the PMMA substrate covered with the wax mask was placed on a rotating stage and exposed to deep-UV radiation for 15 hours (with a dose of 4.3 KJ/cm², figure 2.2d). For the development process (figure 2.2e) after deep-UV exposure, the solution of 7:3 IPA:Water (Johnstone et al., 2008) was used at 28 °C in a water bath for 30 minutes. The etching duration in this procedure is 30 minutes, which
this etching time is enough for developing the microchannel with depth of 100 µm, for
the microchannel with depth less than 100 µm, more etching time (several extra minutes)
is not going to have significant change of the depth of the microchannels, because the
channel depth is decided by the dose from deep-UV exposure. Most of the wax patterns
on the PMMA substrate could be removed during the development process, with the
residual wax fragments were cleaned up using ultrasonic cleaning in DI water (figure
2.2f). An SEM image of a microchannel cross-section is shown in figure 2.4, taken by a
Quanta 600 FEG, FEI Co., Ltd, USA. The depth of the microchannel depends on the
material properties of the PMMA as well as the exposure dose. In our experimental
process, we found that, microchannels with the depth of over 100 µm were easily
achievable. After development, the microchannels were sealed by thermocompression
bonding with another layer of PMMA sheet (figure 2.2g). It is also worth to mentions that,
the relations between channel depths and the exposure time using 254 nm deep-UV
patternning PMMA has been well studied in (Haiducu et al., 2008).

![Figure 2.4 Cross-section SEM image of the microchannel after development (Fan et al., 2012).](image)

For the testing of the fabricated microfluidics device, two syringe pumps (Fusion 200,
Chemyx Co., Ltd, USA) were used to drive the fluid into the device. Figure 2.5 shows the
laminar flows in the microchannels on this PMMA-based microfluidics device. In figure
2.5, three water flows propagate in parallel in the main channel without any obvious
mixing, this laminar flow behaviour is because of the low Reynolds number (Re) of the
fluid flow inside the microchannel. The upper stream and bottom stream were water dyed
with cresol red and the centre stream was water dyed with blue (Parker fountain pen ink).
Instead of using direct deep-UV exposure for the fabrication of microchannels on PMMA surface, by inverting the desired patterns prior to printing, a positive relief of the microstructure can be used as the mold for fabricating microfluidic devices. For demonstration, PDMS (Sylgard 184, Dow Corning, USA) mixed with a ratio of 10:1 of curing agent, and poured on the PMMA mold, a 1 mm thick PDMS layer was then formed on the PMMA mold. The PDMS layer with mold was then sent for curing at 90 °C for 1 hour in the drying oven (Thermo Scientific, model 6947). After curing, the PDMS layer was carefully peeled off from the mold and sent to a 1 hour deep-UV exposure (254 nm) where ozone was generated in the exposure box to activate the surface of the PDMS. Finally, the PDMS layer was attached to a glass substrate and baked in the oven at 90 °C for 2.5 hours to achieve bonding. The optical image of the PDMS-based microfluidic device is shown in figure 2.6b.

In this section, a low-cost method for deep-UV patterning of PMMA was introduced. The wax pattern was printed on a thin HDPE film and then transferred onto the surface of
PMMA substrate using a thermal laminator. The PMMA substrate covered with wax mask was then sent for deep-UV exposure, and then a mix of IPA/water was used to develop the microchannels.

The minimum width of the microchannel this method can achieve on a PMMA substrate is 50 μm, the depth of the microchannel can be controlled by the exposure time (dose). The limitation of the minimum width this method can achieve comes from this low-cost commercial wax printer. Since the deep-UV light source in our method is not collimated because of the usage of low-cost mercury lamps, the etch rate on PMMA substrate is also none linear, the detailed etch rate information and selection of etch recipes can be found in (Haiducu et al., 2008; Johnstone et al., 2008). However, the accuracy of this fabrication method for PMMA-based microfluidics device is suitable for most of the microfluidics applications in the chemical and biological fields. This proposed microchannel fabrication method is simple and low-cost, without the requirement of any sophisticated instruments and the cleanroom environment.

2.1.2 Deep-UV LIGA Process

In this section, a Deep-UV LIGA process using PMMA as the photoresist and 254 nm deep-UV radiation sources is introduced for the fabrication of high aspect ratio metal structures, these metal structures can be used as the mold in soft lithography for the fabrication of microfluidics device.

LIGA is the German acronym for lithography, electroplating, and molding, which describes a fabrication technology employed to create high aspect ratio microstructures. LIGA technology is traditionally used to produce microstructures with high aspect ratio geometry using X-ray lithography. Since a synchrotron radiation source for X-rays is extremely expensive, the traditional LIGA process is costly and time consuming. As a result, researchers began to use a UV LIGA method, which uses SU-8 and a radiation source at 365 nm to fabricate high aspect ratio SU-8 microstructures (Che-Hsin et al., 2002; Ronald, 2005; Shew et al., 2005), microstructures with thickness exceeding 1000 μm and aspect ratio of 30:1 could be achieved using this method (Campo & Greiner, 2007). However, SU-8 has high initial stress and is hard to remove after exposure and
development (Chang & Kim, 2000), which limits the aspect ratio and the finest features this method can achieve.

In this study, instead of using the costly X-ray or 365 nm radiation source, we used a low-cost 254 nm deep-UV source to perform the Deep-UV LIGA process, which lowers the cost and simplifies the fabrication process for the fabrication of high aspect ratio microstructures. Since the SU-8 has a strong adhesion to the silicon wafer (Liming et al., 2006), which can hardly be removed after electroplating. In this research, the thick PMMA layer was used as the structure material for the electroplating process, after electroplating, the thick PMMA layer could easily be stripped using toluene.

2.1.2.1 Fabrication Process

The Deep-UV LIGA fabrication process is shown in figure 2.7. The process started with sputtering Cr/Au (50nm/100nm) layer on a silicon wafer (figure 2.7a) which the Cr/Au layers works as the seed layer for the electroplating process at the last step. After metal sputtering, a layer of 30 µm thick PMMA was deposited on the seed layer by casting (figure 2.7b), the casting was conducted by baking the solution of PMMA in anisole on a hot plate at 120 °C for 6 hours. After that, a SU-8 layer with the thickness of 8 µm was sprayed on top of the PMMA layer using SU-8 MicroSpray (figure 2.7c), and soft baked at 95 °C for 10 minutes. Then, the SU-8 layer is exposed in the mask aligner at 365 nm with an exposure dose of 250 mJ/cm² (figure 2.7d), post exposure baked at 95 °C for 10 minutes, and developed in SU-8 developer for 5 minutes. The patterned SU-8 has high absorption of radiation at the wavelength of 254 nm, thus could be used as an opaque mask for the PMMA exposure (figure 2.7e). After the deep-UV exposure of the PMMA, the sample was developed in PMMA developer to obtain the structure for electroplating (figure 2.7f). The electroplating process was conducted in an electroless Ni plating solution (Transene, Co., Ltd, USA) at 95 °C for 60 minutes (figure 2.7g). Finally, the sample after electroplating was cleaned with DI water and the residual PMMA was stripped using toluene (figure 2.7h).
Figure 2.7 Fabrication high aspect ratio metal structure using deep-UV LIGA method (Huawei et al., 2012b). (a): Sputtering Cr/Au as seed layer. (b): Casting a layer of 30 µm thick PMMA. (c): Spray a layer of SU-8 on the top of PMMA layer. (d): Patterning of the SU-8 layer. (e): Deep-UV patterning of PMMA layer using SU-8 layer as mask. (f): Development process. (g): Electroplating process. (h): Removal of the PMMA and SU-8 layer.

Figure 2.8 (a): Metal structure on the seed layer after the removal of photoresist (Huawei et al., 2012b). (b): Enlarged view of the smallest part (“Y” junction) of the structure on the seed layer.

The images of the metal structures after electroplating and removal of PMMA are shown in figure 2.8. The height of the nickel mold was 50 µm and the minimum line width achieved was 10 µm, the aspect ratio we achieved here is 5:1. The smallest feature
this method can achieved was restricted by the resolution of the photolithographic mask, which depends on the resolution of the Laser mask writer (Heidelberg μPG 101, Germany). To further increase the aspect ratio, either casting a thicker PMMA layer or using multiple castings could easily further increase the maximum aspect ratio of this method.

In this section, a low-cost deep-UV LIGA process for the fabrication of high aspect ratio metal structures was demonstrated. This metal layer can be used as the replica mold for PDMS based microfluidic devices where high aspect ratio microchannels are needed.

### 2.2 Laser Processing for Low-cost Microchannel Fabrication

As introduced in chapter 1, laser processing has been extensively used for the fabrication of microchannels on various substrates such as polymers, glass and silicon. Various laser systems with wavelength ranges from UV to infrared have been used for the fabrication of microchannels, among these laser systems, the CO₂ laser (10.6 μm in wavelength) is the cheapest option with the appropriate precision for microchannel fabrication on polymer materials. Various researchers have been using CO₂ laser to ablate the microchannels on PMMA (Klank et al., 2002a; Davim et al., 2008; Nimai et al., 2008).

In the first part of this section, polystyrene (PS) instead of PMMA, was used as the material for the microchannel fabrication by CO₂ laser ablation. PS can provide features such as biocompatibility, inert, resistivity against alcohols and polar solvents, and transparency of lights in visual range. Since PS has been widely used as the material for cell petri dish and microplate (Chen et al., 2008), and there is much background research on making PS biocompatible. Further, the surface quality of PS can easily be changed with various chemical or physical methods (Kaur et al., 2008). This demonstrated CO₂ laser ablated PS-based microfluidic fabrication process could have wide applications in the biological field.

The second part of this section will introduce an interesting finding of laser induced bump formation process on PS. During our experiment using laser processing on PS surface, we observed that, if we lowered the energy of CO₂ laser below a certain threshold, a bump instead of a ablation trench was formed on the surface of the PS. The
height of the bump will increase if multiple laser scans were applied. Microchannel molds with multiple depths can be fabricated using this interesting phenomenon.

Besides fabricating PS-base microfluidic devices, the third part of this section will introduce a low-cost flexible plastic paper based microfluidics device with CO$_2$ laser ablated microchannels.

2.2.1 Laser Ablated Microfluidic Devices Based on Polystyrene Substrate

In this research, the CO$_2$ laser was used for the fabrication of polystyrene based microfluidic devices. As introduced in chapter 1, the ablation process for the CO$_2$ laser is purely thermal, the polymer materials will melt, decompose and vaporize during the laser ablation process. The profile of the laser ablated microchannel on polymers depends on the intensity distribution, laser power, cutting speed and the properties of the material. Usually, a Gaussian like profile of the microchannel will be formed after laser ablation. A simple schematic diagram of the laser ablation process on polymer substrate is shown in figure 2.9.

2.2.1.1 Materials and Instruments

In this research, the polystyrene substrate was 2 mm thick with a molecular weight of 267.8 kDa from Goodfellow Cambridge Limited, England. In the experimental process,
the band of mineral oil is Sigma-Aldrich (light oil, produce code: M5904). DI water was dyed with cresol red (1%) for the easy observation of the flow inside the microchannel.

The CO$_2$ laser system used in this research and all the research involved using the CO$_2$ laser in this chapter was Universal PLS6.75 (Universal Laser System Inc., AZ, USA). The laser source has a wavelength of 10.6 µm which is in the infrared region. The laser beam was focused with a 60 mm focal length and the spot size is 0.127 mm in diameter. The effective working area of this laser system was 813 cm by 457 cm, with the maximum scanning speed of 300 mm/s. The laser source equipped with an air-cooled free-space gas slab laser, which had a maximum power output of 75 W, working in pulse mode with a highest resolution of 1000 PPI (pulses per inch). Based on the previous mentioned parameters of the laser beam, the calculated depth of field is 2.58 mm, and the highest power intensity at 75W is 5920 W/mm$^2$. The motion of laser head is controlled by proprietary software that allows the user to modify the output power, speed, points per inch (PPI) and height of the work area. With the help of control software (Universal Control Panel), the power output, scanning speed and resolution can be adjusted from 0% to 100%. The motion of laser head was controlled by three step motors, and the scanning path could be generated by AutoCAD, CorelDraw or other graphic packages. An SEM (Quanta 600 FEG) and profilometer (DEKTAK 150) were also used in this research for the imaging and profile measurement of the fabricated microchannels.

### 2.2.1.2 Fabrication Process

The effect of different laser powers and scan speeds for the ablation of polystyrene microchannels was studied in detail. In the fabrication process, multiple microchannels were laser ablated with power settings ranges from 3.75 W to 33.75 W and scanning speed settings from 10 mm/s to 52.5 mm/s. After the laser ablation process, the width and depth of the microchannels were measured using profilometer. The whole measurement process was repeated 10 times to ensure the reproducibility, and the average value was taken as the measurement result.

A simple microfluidic device (droplet generator) was fabricated for the demonstration of this low-cost fabrication technology. The microchannel was laser ablated with the laser
power of 10 W and scanning speed of 50 mm/s on a 2 mm thick polystyrene substrate. After laser ablation, the microchannel was closed by bonding another 2 mm polystyrene sheet using thermocompression bonding method (102 °C for 120 minutes at 700 N). The cross-section SEM images of laser ablated microchannel before and after bonding are shown in figure 2.10. Figure 2.10b shows the sealed microchannel fabricated by laser ablation, the bottom of the microchannel appears to have a bump, which is because of the thermal reflow during the bonding process and the bumpy surface left after laser ablation on the surface of polystyrene.

![Figure 2.10 (a): SEM images for the cross-section of the laser ablated microchannel before bonding. (b): Cross-section of the laser ablated microchannel after bonding (Li et al., 2011a).](image)

2.2.1.3 Result and Discussion

The laser ablated microchannels with various laser power and cutting speed settings were measured by profilometer. The result is shown in figure 2.11, 2.12 and 2.13, for each measuring point, the data of the width and depth of the microchannel was based on the average of 10 measurement results (standard deviation is less than 3 %). The measurement result of depths, widths and calculated aspect ratio at different laser energy and scanning speed are shown in figure 2.11, figure 2.12 and figure 2.13, respectively. From these figures, we can conclude that, the depth and width are increasing with the rising of laser power and lowering of scanning speed.

In figure 2.11, the depth of the laser ablated microchannel seems to reach a plateau and rate of depth increasing is slower, which is the result of two reasons. The first reason
is when the microchannel goes deeper, more energy at the bottom of the channel is being conducted to the bulk material, this will increase energy lost and slowing down the channel depth growing. The second reason is the laser beam is focused on the surface of the polymer material, as microchannel goes deeper focus points no longer as affective, which caused less energy being absorbed by polymer material. The width of the microchannel is also increasing with higher laser energy and slower laser scanning speed (shown in figure 2.12), because more residue time of the laser beam at certain spot will cause more material melt down (larger molten pool) and more material will evaporate, thus, the microchannel width is increased with slower laser scan speed.

Figure 2.11 The depths of the microchannels laser ablated under 20 different laser powers and 6 laser scanning speeds (Li et al., 2011a).
Figure 2.12 The widths of the microchannels ablated under 20 different laser powers and 6 laser scanning speeds (Li et al., 2011a).

Figure 2.13 The aspect ratio of the microchannels ablated with different laser powers and cutting speed (Li et al., 2011a).

The aspect ratio (shown in figure 2.13) of the microchannel is also growing with the increasing of the laser power, but decreasing with increasing scan speed. The aspect ratio appears to achieve saturation when laser power is higher than 30W, the underlay physics of this interesting phenomenon is still not clear at this point, an educated assumption is: the laser power higher than 30W could provide sufficient energy for the polymer melt down, decomposition and evaporation at bottom of the microchannel. With careful
selection of laser energy and scan speed, the profile of microchannels can be chosen to match the application.

For a demonstration of this technology, a Y-shaped droplet generator was fabricated (shown in figure 2.14). The microchannel was fabricated with laser energy of 11.25 W and scanning speed of 25 mm/s. The width of the microchannel is 308 μm, and the depth of channel is 324 μm. The DI water dyed with cresol red was used as the dispersed phase in the continuous phase of mineral oil. The flow rate of the water and mineral oil was controlled by syringe pump (Fusion 200, Chemyx Inc. TX, USA) at 0.005 ml/ min and 0.01 ml/ min, respectively. It can be seen clearly that the water droplet was generated in the laser ablated microchannels on PS.

With the enlarged image (image with yellow frame) in figure 2.14, bulges besides the microchannel can be observed; these bulges were generated during the laser ablation process (solidified polymer materials ejected from the molten pool during laser ablation). Although researchers have proposed some methods for eliminating this bulge, such as coating a PDMS layer before laser ablation (Chung et al., 2005), we found these bulges are actually helpful for sealing the microchannel in the thermocompression bonding process which will be introduced in details in the next chapter.

Figure 2.14 Polystyrene based droplet generator with laser ablated microchannels (Li et al., 2011a).
In this work, a low-cost microchannel fabrication technique for PS-based microfluidic devices was introduced. A commercial CO₂ laser system was used to thermally ablate microchannels on a PS substrate. The channel depths, channel widths and aspect ratio of the laser ablated microchannels under various laser powers and cutting speeds were carefully characterized. A microfluidic device for demonstration was also fabricated. This new method for fabricating microchannels on polystyrene is simple, low-cost and rapid, without the requirement of cleanroom facilities. In addition, this laser ablation method is also applicable for other polymer materials in addition to polystyrene. The minimum channel width this method can achieve is around 120 μm, the limitation comes from this commercial CO₂ laser system, which originally built for the laser engraving.

2.2.2 Laser Induced Bump Formation on Polystyrene and Applications in Microfluidic Devices

During the previous research in using laser ablation to fabricate microchannels on polystyrene substrate, we observed that if the power of the laser is low enough (below the vaporization threshold), a bump instead of ablation trench will formed on the surface of the polystyrene substrate. In addition to that, we found the height of the bump could be controlled by laser power and repeat of scans. In this section, the bump formation process on polystyrene will be carefully studied. For the demonstration of this bump’s application in microfluidics, the laser induced bumps were used as the mold for the fabrication of microchannels with multiple depths.

2.2.2.1 Bump Formation Process Using a Low Power CO₂ Laser on Polystyrene

In this research, the laser bumps were formed on a 1.2 mm thick polystyrene sheet (Goodfellow, molecular weight of 267.8 kDa, product code: ST313120) using a CO₂ laser system (Universal PLS6.75). Detailed information for this low-cost commercial CO₂ laser system could be found in the previous section in this chapter. For characterization of laser induced bump, the profile of the bump was measured using DEKTAK 150 profilometer, and SEM images were obtained using Quanta 600.
As introduced in the previous section, the CO₂ laser ablation process is purely a thermal process. When the laser is focused on the surface of the polystyrene, the temperature of the irradiated spot will increasing sharply, when the temperature exceeds a certain vaporization threshold, the decomposition and evaporation of the materials will occur, leaving a trench (microchannel) with a Gaussian-like profile. In this study, during the laser scanning on the surface of PS substrate, the energy of the laser was carefully controlled at a value below the vaporization threshold to prevent material decomposition and vaporization. By lowering the laser energy, a bump rather than a trench was formed on the PS surface (illustrated in figure 2.15).

![Figure 2.15 Illustration of the bump formation on polystyrene using CO₂ laser (Huawei et al., 2012a).](image)

The mechanism for the bump formation is shown in figure 2.16, and this complicated bump formation mechanism can be simply described as the “fast cooling effect” (Shiu et al., 1999; Pantani, 2003). During the laser irradiation, a volume increase of the material due to thermal expansion causes the bump formation on the surface of polystyrene substrate.
As shown in figure 2.16, when the laser irradiation starts, the temperature of the irradiated spot on polystyrene will quickly raise from solid state (point A) to rubbery state (point B), because of the low heat capacity and low heat conductance of polystyrene. When the laser irradiation stopped, cooling will start and the spot leaves point B. Since the cooling process after laser ablation is much faster than the cooling of the bulk polystyrene during the production process, the polymer chains do not have time to pack themselves as densely as they have after the bulk fabrication. The difference of the cooling rate will lead to a change of glass transition temperature from $T_g$ to $T_g'$ at the laser ablated area. It can be seen clearly from this graph that, when the whole process stopped at point C, there will be an increasing of the volume, which causes the bump formation in the laser ablated area. In other words, the melt and thermally expand polymer cools and solidifies faster than it can mechanically contract, locking in the expanded dimensions, which finally causing the bump formation. SEM image of the bump formed on the polystyrene substrate is shown in figure 2.17. The power of the CO$_2$ laser was set at 0.6 W with a scanning speed of 300 mm/s, which is the maximum scanning speed of this laser system.
The relation of the bump height and laser power was also carefully studied. The result (shown in figure 2.18) indicates that, the height of the bump will increase with increasing laser power. An SEM image with the bumps fabricated with different laser powers is also shown in figure 2.19, the laser power was set at 5 W, 4 W and 3 W, with the scanning speed at 300 mm/s. However, the experimental results also indicate that, the power of the laser should be kept below 5.3 W to avoid breaking the vaporization threshold. A depression on the summit of the bump will appear when the threshold has been broken at laser powers higher than 5.3 W (the profile is shown in figure 2.20, laser power is 5.5 W with the scanning speed of 300 m/s, repeated 5 times, the bump heights’ standard deviation is less than 5 %). The depression on the top of the bump in figure 2.20 indicating the break of the threshold, the PS starts to decompose and evaporate.
Figure 2.18 Relation between the bump height and laser power (Huawei et al., 2012a).

Figure 2.19 Bumps fabricated on polystyrene with laser power at 5 W, 4 W and 3 W (left to right) (Huawei et al., 2012a).
Another noteworthy finding shows the height of the bump can be increased by multiple scans of the same area by laser beam. Figure 2.21 shows the height of the laser induced bump rose from 4 µm to 33 µm by multiple scans (from 1 scan to 23 scans). In this multiple scan process, the power of laser was kept at 3.75 W with the scanning speed at 300 mm/s. The waiting time between each scan is 1 minute to ensure the polymer material has enough time to cooling down. In this figure, each point indicates the height measurement data from an average value of 5 measurements, with the standard deviation less than 5 %. There is a jump of the height after 20 scans as shown in figure 2.21, the underlay physics is still not clear at this point.
2.2.2.2 Laser Induced Bump Used as the Mold for the Fabrication of Multi-depth Microchannels

For the demonstration of this bump’s application in microfluidics, a PDMS-based droplet generator with microchannel of multiple depths was fabricated using the laser induced bumps on polystyrene as the mold. The fabrication process is shown in figure 2.22, the bumps with different height were fabricated with the multiple scan of CO$_2$ laser at 3.75 W with a scan speed of 300 mm/s. As depicted in figure 2.22, the bump in green was laser scanned 15 times and has a bump height of 15 µm. The bump in yellow was laser scanned 22 times and results in a bump height of 33 µm. Then, the PDMS (Dow Corning Sylgard 184) mixed with curing agent was poured on the polystyrene mold with bumps and cured at 80 °C for 1 hour. Finally, the patterned PDMS sheet was bonded to a glass substrate after the oxygen plasma surface treatment.
Figure 2.22 Fabrication process of the PDMS microfluidic devices with multiple microchannel depths using polystyrene mold (Huawei et al., 2012a). (a): Multiple laser scanning on the surface of polystyrene. (b): Replica molding of PDMS using polystyrene mold.

The schematic diagram of the fabricated PDMS based droplet generator is shown in figure 2.23. The mineral oil and water (DI water dye in red) were pumped into the microchannel from two inlet ports with the same flow rate of 0.05 µL/min. The water droplet was generated at the T-junction and keeps proceeding in the main channel, when entering the area with deeper microchannel (shown in figure 2.24) the droplet will reshape because of the surface tension of the water droplet.

Figure 2.23 PDMS based droplet generator with multiple depth channel (Huawei et al., 2012a).

Figure 2.24 Droplet reshaped when entering the deeper microchannel (Huawei et al., 2012a).
2.2.2.3 Surface Roughness comparison

In the previous section, the PDMS molding was used for the fabrication of microchannel with multi-depth. The surface roughness of the microchannel fabricated on PDMS, the surface roughness of the laser induced PS bump and the surface roughness of the laser ablated microchannel on polystyrene will be compared in this section.

The laser induced bump was fabricated with laser energy of 3.75 W and scanning speed of 300 mm/s, the height of the bump is 18 μm after 17 scans. The PDMS based microchannel was the replica molding of this laser induced bump on PS. The laser ablated microchannel on PS was fabricated using laser energy of 13.5 W, with a scanning speed of 300 mm/s, the depth of the microchannel is 18.2 μm.

The surface roughness was measured using a profilometer (Dektak 150, Veeco Inc, USA). The surface roughness (Ra) of these three samples was measured along the direction of the bump or channel’s axes with a length of 4 mm. The measurement results are the average of 5 measurements, with the standard deviation less than 8 %. The laser induced bump on PS has a Ra of 138nm, the microchannel on PDMS after molding is 46 nm, and the laser ablated microchannel on PS has a Ra of 6.9 μm.

We can conclude from these measurement results that the laser induced bump has a much smaller Ra than the laser ablated microchannel on PS, and the PDMS replica molding can even reduce the Ra during the replica molding. For the applications where a smooth microchannel is needed, this low-cost PDMS molding method using laser induced bump on PS could be an ideal choice.

In this section 2.2.2, a laser induced bump formation phenomenon and bump growth on PS substrate was studied. For demonstration, a PDMS-based droplet generator was also fabricated using multi-height bumps on a PS substrate as the mold. The technology proposed in this research gives a low-cost and rapid method for the fabrication of microchannels with multiple depths. Multi-depth microchannels fabricated using this method could have various applications in the biological field, such as trapping cells during single cell analysis (Yue & Xue-Feng, 2006). The limitation of the minimum feature this method can achieve comes from the CO₂ laser system, as a commercial
engraving system, the accuracy of the laser power/scan speed control and the spot size of the laser beam are the bottleneck for the improving of the feature size quality.

2.2.3 Plastic Paper Based Microfluidic Devices with Laser Ablated Microchannels
In the third part of the low-cost laser ablated microchannels research, this section will present a novel rapid prototyping method for plastic paper based microfluidic devices. Instead of using the thick and rigid polymer substrates that have been used in the previous sections, the plastic paper (also known as permanent paper) was used as the substrate for microfluidic devices with laser ablated microchannels. The thickness of this plastic paper is 135 \( \mu \text{m} \). As a commercial product, the detailed composition of this product is trade secret, but the main composition is polyester. The band is Model PP5WASC (Southwest Binding Company, Maryland Heights, Missouri, USA). Besides the ultra-low-cost of the material (~$20 for 1 pack of 100 sheets), the flexibility of the plastic paper also enables the potential of fabricating bendable 3D microfluidic devices.

2.2.3.1 Fabrication Process for Paper Based Microfluidic Devices Using Laser Ablation
A microfluidic device (droplet generator) was fabricated for the demonstration of this new technique. The fabrication process is illustrated in figure 2.25. The process starts with laser ablation of microchannels on plastic paper (135 \( \mu \text{m} \) thick) with the CO\(_2\) laser system we have introduced previously. Microchannels with the desired dimensions were fabricated using laser ablation (figure 2.25a) at a controlled laser power of 7 W and scan speed of 220 mm/s. Then, the CO\(_2\) laser system was used again to cut the inlet and outlet ports on a thin piece of PMMA film (Goodfellow Cambridge Limited, England, 50 \( \mu \text{m} \) thick), which will be later used as a cover sheet to seal the microchannels on the plastic paper.
Figure 2.25 Fabrication process of the plastic paper based microfluidics device. (a): Laser ablate microchannels on plastic paper. (b): Laser cut through holes on PMMA film. (c): Bonding between PMMA film and the plastic paper with laser ablated microchannel. (d): Connecting pipes. (e): Schematic view of the thermocompression bonding system (Yiqiang et al., 2013).

To enclose the microchannels, the patterned plastic paper and a thin transparent PMMA film with through holes for inlet and outlet ports were bonded using the thermocompression bonding method, which the bonding process will be introduced in details in the next chapter. The bonding was carried out using an Instron Dual Column Testing System (illustrated in figure 2.25e) fitted with an Instron 3119 environmental chamber for the temperature control. In order to securely bond the patterned plastic paper to the thin PMMA film, the bonding temperature was set at 150 °C with a bonding pressure of 0.4 MPa for 70 minutes. After bonding, “world-to-chip” connectors were attached to the inlet/outlet ports and the device was ready for testing after piping. The “world-to-chip” connector is a laser fabricated cuff to increase the contact surface between microfluidic chips and pipes. Figure 2.26 shows the low-cost and flexible microfluidic device after fabrication.
2.2.3.2 Result and Discussion for Paper-based Microfluidic Devices Using Laser Ablation

An optical image of the laser ablated microchannels on plastic paper is presented in figure 2.27a, the power of the laser was set at 7.5 W with the scanning speed of 220 mm/s. Figure 2.27b shows the droplet generated (water droplet in oil) in this plastic paper-based microfluidic device, the red stream was DI water and the transparent stream was mineral oil. The profile of the laser ablated microchannels on plastic paper was measured using a profilometer (Ambios XP-200). Figure 2.28a shows the profile measurement of a laser ablated microchannel, with the laser power setting 7.5 W and scanning speed of 220 mm/s. The channel has a width of 110 μm and a depth of 50 μm. The dimension of the microchannels could be controlled by the power and scanning speed of the laser. Since the plastic paper used in this study is only 135 μm in thickness, the power and speed of the laser needs to be carefully controlled to avoid cutting through the whole thickness. Figure 2.28b shows the relationship between the depth of the microchannel and the laser power at four different laser scanning speeds, each data point was the average value of four measures. From figure 2.28b, we can conclude that within the usable range of laser power (from 0 to 20 W) the depth of the channel will increase with the increasing laser power. The depth of the microchannel will also increase with a slower laser scan speed. The power of the laser must not exceed 20 W with the minimum scan speed higher than
180 mm/s, unless through holes are desired, as the high laser power will cut through the entire thickness of the plastic paper.

The cross-sectional SEM images (shown in figure 2.29) of the bonded microfluidic device were obtained using a Quanta 600 FEG. The upper layer was a transparent 50 μm thick PMMA film and the lower layer was a 135 μm thick plastic paper with the laser ablated channels. From the SEM images, we can observe that the thermocompression bonding could effectively enclose the microchannels on plastic paper with the thin PMMA film.

Figure 2.27 (a): Optical image of the microchannels after the laser ablation on plastic paper. (b): Droplet generated in the microfluidic device (Yiqiang et al., 2013).
Figure 2.28 (a): Profile of the laser-ablated microchannel on plastic paper. The channel has a width of 110 µm and a depth of 50 µm. (b): The measurement of the channel width on plastic paper with various settings of laser power (ranges from 3 W to 20 W) at four different scan speeds (ranges from 180 mm/s to 300 mm/s) (Yiqiang et al., 2013).
This work reported a novel technique for the low-cost prototyping microfluidic devices using plastic paper. A droplet generator was fabricated to demonstrate the effectiveness of this process. In this low-cost process, microchannels were laser ablated on a piece of plastic permanent paper with width as low as 100 μm. The patterned plastic paper was then bonded to a transparent PMMA film to enclose the channels. The device fabricated was thin (total thickness is 185 μm) and flexible, making it suitable for the 3D applications. Since the microfluidic devices fabricated with this technique are highly flexible, users can easily bend or fold it without damaging the device. The advantages of low-cost and rapid fabrication, without the requirement of any cleanroom facilities makes this technique a useful tool for researchers in biological and chemical analysis fields where low-cost, flexible and disposable microfluidic devices are needed. The limitation of this method comes from both the laser system and the material, with the commercial CO₂ laser system used in this research, the minimum width of microchannel is around 120 μm. Since the plastic paper is very thin (135 μm), the laser ablated microchannel deeper than 100 μm can hardly be fabricated, as the laser can easily ablated the whole thickness of the plastic paper.
Chapter 3 Low-cost Polymer-based Microchannel Sealing (bonding) Technologies

The previous chapter introduced various low-cost microchannel fabrication methods. After the fabrication of microchannels on a polymer substrate, enclosing the microchannel by bonding a cover plate (cap layer) is the next key step in the fabrication process of microfluidic systems.

As introduced in the chapter 1, unlike the bonding process for silicon and glass materials, there are a wide variety of bonding methods for polymer materials, especially for the thermoplastics. Compared with other polymers such as elastomers and thermoset plastics, thermoplastics can be softened and melted by heating, and solidified again when the temperature drops below the glass transition temperature without adversely affecting the material properties (Tsao & DeVoe, 2008).

In this chapter, a low-cost thermocompression bonding (also known as “thermal fusion bonding”) method will be introduced. This direct bonding method is low-cost and simple, without the need for any chemical to promote the adhesion, and adaptable to most of the thermoplastics. Besides bonding polymers of the same kind, which provides identical surface properties, the method introduced in this chapter can also be used for the bonding of different polymer materials which have similar glass transition temperatures. During the thermocompression bonding process, the temperate rises above the glass transition temperature for both of the polymer substrates, and pressure is applied between the two substrates. The high temperature and pressure significantly enhances the entanglement of the loosened polymer chains, resulting in a secure bond.

The contents in this chapter will be introduced in the following order: first, the thermocompression bonding between a PMMA substrate to a PS substrate will be introduced as a demonstration of this technology. With the help of this method, microfluidic devices with a combination of PMMA and PS layers can be bonded, which allows the combined advantages of easy fabrication of PMMA and easy surface functionality, highly transparency of visible light of PS. Then, the bonding strength will be tested, the bonding strength was measured by two different methods and the results
were compared. Finally, to prevent the microchannel deformation during the thermocompression bonding process, a low-temperature bonding method was also reported. A 254 nm deep-UV surface treatment was applied to the bonding surface before the thermocompression bonding process, which can significantly increase the surface wettability, thus lowering the temperature and compression force required during the bonding process.

3.1 Thermocompression Bonding Process

3.1.1 Fabrication of Microchannels

For the demonstration of the thermocompression bonding process, the microchannels were fabricated on PMMA substrate prior to bonding. The microchannels were laser ablated on a 1 mm thick PMMA sheet using the CO₂ laser system (Universal PLS 6.75, AZ, USA). The laser beam generated by the system has a wavelength of 10.6 µm, the power of the laser was set at 37.5 W, with the scanning speed of 150 mm/s. Figure 3.1 shows the illustration of the laser ablation process for the microchannel fabrication on PMMA substrate.

![Figure 3.1 Illustration of the laser ablation of the microchannel on PMMA substrate (Fan et al., 2013b).](image)

3.1.2 Thermocompression Bonding of PS-PMMA

The PS sheet (1.2 mm thick, Goodfellow Cambridge Limited, England, molecular weight of 267.8 kDa) was used as the cover plate to seal the microchannels fabricated on PMMA substrate using thermocompression bonding process. The Young's modulus (measured by
INSTRON 5960 Dual Column Tabletop Universal Testing Systems) of PS and PMMA substrate was 2640 MPa and 2870 MPa, respectively. The dimensions of the PS and PMMA sheets are identical, at 25.4 mm by 76.2 mm (1 inch by 3 inch).

The bonding process for this microfluidic device is shown in figure 3.2. After laser patterning the PMMA substrate, a blank PS cover plate was attached and together they went through the thermocompression bonding process. As shown in figure 3.2c, in order to protect the surface of the polymers, the PS and PMMA substrates were sandwiched between two glass coverslips. This stack of chips was then sent into the bonding system (INSTRON Dual Column Testing Systems) presented in figure 3.3. The bonding system consists of the lower and upper studs (arms), and the compression force between these two studs can be preprogrammed and controlled during the bonding process. The whole bonding system was setup in an environmental chamber (INSTRON 3119 Environmental Chamber), which can control the temperature of the entire stack during the bonding process.

![Figure 3.2 Fabrication and bonding process. (a): Blank 1 mm thick PMMA substrate. (b): Laser ablate microchannel on PMMA substrate. (c): PS cover plate and PMMA substrate were attached and sandwiched between two glass coverslips, then went through the thermocompression bonding process. (d): Remove the glass coverslip after bonding (Fan et al., 2013b).](image)

The temperature of the environmental chamber was set above the glass transition temperature of PMMA (higher than 110 °C), and this temperature is also higher than the $T_g$ of PS. The compression force was supplied by the two studs in the bonding system. As
previously mentioned, both the high temperature and compression force will significantly enhance the entangling of the mobile polymer chains at the adjoin surface of the PS and PMMA. In order to compare the bonding strength at different bonding temperatures and compression forces, temperatures were set in the ranges from 110 °C to 125 °C and the compression forces were ranged from 700 N to 1000 N (0.36 Mpa to 5.17 Mpa).

Figure 3.3 Illustration of the thermocompression bonding system setup. The chip set was placed between the lower and upper studs. Bonding force was controlled by two studs while the temperature was controlled by an environmental chamber (Fan et al., 2013b).

Table 3.1 shows the temperature and compression force settings during the thermocompression bonding of PS and PMMA. For each condition of different temperature and compression force, three chip sets were bonded and the bond strengths were tested. The detailed testing result will be discussed in the next section. During the thermocompression bonding process, the ramping rates of the temperature and compression force were set at 15 °C/min and 200 N/min, respectively. After reaching the desired temperature and the compression force, the two studs were held for 50 minutes and then released the bonding specimen with a ramp of 300N/min. After natural cooling (temperature inside the environmental chamber is below 50 °C), the chip set could be removed from the environmental chamber. The glass coverslips will automatically
separate from the polymers during the cooling period because the polymers’ thermal expansion coefficient is almost 10 times higher than glass’.

Table 3.1 Temperature and compression force settings for bonding testing process (Fan et al., 2013b).

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature (°C)</th>
<th>Compression Force (N)</th>
<th>Pressure (kPa)</th>
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</tr>
</tbody>
</table>

3.2 Bonding Strength Testing and Result

3.2.1 Introduction of Two Methods for the Bonding Strength Testing

The bonding strength was tested using two methods. The first method is the double cantilever beam (DCB) method, which is also known as crack opening method (Vallin et al., 2005). The DCB method is simple and rapid, and has been widely utilized for the testing of bonding strength after either adhesive bonding or direct bonding on various materials, such as wood (Ebewele et al., 1979) and silicon wafer (Martini et al., 1997).

With the desire to simplify the experimental process and maintain a low cost, we used the DCB method to test the bonding strength between two bonded polymer substrates. As illustrated in figure 3.4, the DCB bonding test was performed by inserting a razor blade (wedge) between the two bonded layers, and forcing the two bonded layers to separate thus opening a crack. Based on the length of the crack and the material properties, the bond strength can be calculated. In our bonding experiment, both of the polymer
materials (PMMA and PS) are transparent in the visible range, thus the length of the crack (L) can be directly measured after the crack stabilizes. It is worth mentioning that, for substrates which are opaque at visible ranges, but transparent in infrared range (i.e. silicon wafer), the crack opening can be measured using an infrared camera (Maszara et al., 1988).

![Double cantilever beam method](image)

**Figure 3.4 Double cantilever beam method for the bond strength testing of polymer materials (Fan et al., 2013b).**

The bonding strength with the double cantilever beam method could be calculated using the equation (1), which is modified from (Gillis & Gilman, 1964). In equation (1), $\gamma$ (mJ/m²) is the bonding strength, $d_1$ and $E_1$, $d_2$ and $E_2$ are the thickness and Young's modulus of the PMMA and PS, respectively. The thickness of the blade was represented by $t_b$. Then, using equation (1), the bonding strength could be calculated with the measurement of crack length.

\[
\gamma = \frac{3000}{16} \cdot \frac{t_b^2}{\left(\frac{1}{E_1d_1^4} + \frac{1}{E_2d_2^4}\right)^{1.4}} \tag{1} \text{(Fan et al., 2013b)}
\]

The second method used for the bonding strength testing was the tensile test (also known as pull-test). In this bonding strength test, the PMMA and PS sheet were bonded with an overlap area of 25.4 mm by 25.4 mm, and then the bonded polymer sheets were attached to self-built pull studs (illustrated in figure 3.5). Then, these pull studs were mounted on a tensile test machine (INSTRON testing system), the system setup is shown in figure 3.6. The advantage of this pull studs setup is without the need to glue the samples onto the pull studs as previously reported (Jiao et al., 1995; Kim et al., 1995).
The two bonded PMMA and PS substrates can be simply insert between two pull studs, have the screws tightened and begin the testing. During the testing, the machine automatically recorded the maximum force ($F_{\text{max}}$) at which PMMA and PS separate. The force $F_{\text{max}}$ can then be divided by the bonded area ($A$) to have the bonding strength as shown in equation 2.

$$\delta = \frac{F_{\text{max}}}{A} \quad (\text{Fan et al., 2013b})$$

Figure 3.5 Schematic of self-built pull studs setup for the tensile strength testing.

Figure 3.6 Self-built pull studs setup was mounted to the tensile strain testing system (Fan et al., 2013b).
3.2.2 Bonding Strength Testing Result and Discussion

The bonding process and bonding strength test were repeated 3 times for each of the bonding temperature and compression force settings in table 3.1, using both bond strength testing methods introduced in the previous section.

The bonding strength results of the bonded PMMA and PS substrates measured using DCB method at different temperatures are shown in box plot in figure 3.7. Since the glass transition temperatures of PMMA and PS are around 105 °C and 100 °C, respectively (varying slightly based on molecular weight). The bonding temperature was set above 110 °C to ensure the polymer backbone of PMMA and PS can freely moving at long-range and entangle with each other to achieve bonding. On the other hand, the bonding temperature was controlled below 125 °C to avoid the large scale deformation of polymer chips or even the collapse of the laser ablated microchannels on PMMA substrate. Bonding measurement data from figure 3.7 clearly shows the bonding strength (surface energy) was increasing with the increase of bonding temperature. It is worth mentioning that, the choice of bonding temperature can be very difficult when bonding polymer substrates with dissimilar glass transition temperatures. In our case for the PMMA-PS bonding, the similar T_g made bonding possible using the thermocompression bonding method.

![Figure 3.7 Bonding strength obtained under different bonding temperatures at different bonding force using DCB method (Fan et al., 2013b).](image)
The influence from the compression force on bonding strength is also shown in figure 3.7. During the thermocompression process, the compression force was set between 700 N to 1000 N (0.36 MPa to 0.52 MPa). It’s apparent from the boxplot in figure 3.7 that the bonding strength was also increasing with the enhancement of the compression force. This can be interpreted as the compression force also contributed to the entanglement of PMMA and PS polymer backbones. Statistics analysis tests (ANOVA) have also been performed to verify the statistically significant difference between the bonding strengths at different bonding temperatures and difference bonding forces. Again, during the thermocompression bonding process, the compression force needs to be carefully controlled within a certain range to avoid collapsing the microchannels under the high pressure and temperature.

We can observe some fluctuations of the boxplot shown in figure 3.7, which were primarily due to random errors during the bonding and strength testing processes. Although having been used for several decades, the DCB method is still immature. The reproducibility is relatively low and for some strongly bonded materials (e.g. anodically bonded silicon wafers), the blade is hard to insert between the bonded substrates. To verify the bonding strength testing results obtained using the DCB method, the tensile test was also performed. The bonding strength measurement results using the DCB method (measured as surface energy) and the tensile test (measured as Pascal) were compared in figure 3.8. Each dot shown in this scatter plot represents a pair of PMMA and PS substrates bonded under the temperature and compression conditions enumerated in table 3.1. The x-axis and y-axis in figure 3.8 represent the bonding strength measurements from the DCB method and tensile test methods respectively for each set of bonding conditions. The linear regression line was calculated and overlapped in this scatter chart, the calculated correlation coefficient \( R^2 \) was 0.892, which indicated the two methods shows a close agreement statistically.

An SEM image taken by a Quanta 600 of the cross-section of bonded PMMA (lower) and PS (upper) substrates is shown in figure 3.9. In this SEM image, the PMMA substrate with laser ablated microchannel and PS cover plate were bonded at 120 °C with the compression force of 800 N.
3.3 Low Temperature Bonding with Deep-UV Surface Treatment

As introduced in chapter 1, the surface treatment has been widely used for polymer-based microfluidic devices for different purposes. In this section, a simple and low-cost surface treatment is introduced to lower the bonding temperature prior to thermocompression bonding.
Due to the high temperature and the compression force applied during thermocompression bonding, the channels in the micro/nano-scale can deform or collapse without the proper handling of the bonding temperature and compression force. In order to lower the bonding temperature while maintaining bond strength, surface treatment is needed. The general strategy is to increase the surface energy and wettability (Tsao & DeVoe, 2008) between the two contacting surfaces by breaking the long chains of polymers at the contacting surfaces using a surface treatment. Different energy resources can be used for breaking the chemical bonds, such as plasma (Brown et al., 2006; Abgrall et al., 2007) and UV/ozone (Tsao et al., 2007a; Liu et al., 2008). However, these surface treatment methods require sophisticated instruments (e.g. plasma source, vacuum chamber) and gas supply.

The method we proposed in this research, as part of the low-cost polymer bonding method, used the 254 nm deep-UV source (shown in figure 3.10) to break the chemical bonds on the surface of polymer materials. During the deep-UV exposure, the absorption of energy at the surface of the polymers will cause scission and oxidation of the long chains, resulting in photo-degradation (Emanuel & Buchachenko, 1987). The average molecular weight is decreased after deep-UV surface treatment, thus lowering the glass transition temperature on the surface of the bulk polymer material. With the lower glass transition temperature at the contact surfaces, two polymer substrates can be bonded at a lower temperature. Since the depth of the deep-UV affected regions can be controlled by the duration of exposure, the properties of the bulk polymer substrates will not be influenced by the surface treatment, the detailed etching rate of using 254 nm deep-UV exposure on PMMA can be found in previous work from other researchers’ in (Haiducu et al., 2008).
For demonstration of this low temperature bonding method, the surface of the PMMA and PS was exposed in a self-built deep-UV exposure box with a dose of 432 J/cm². Then, the PMMA and PS substrates were bonded using the thermocompression bonding method introduced in the previous section of this chapter. The bonding temperature was set at 115 °C with the compression force of 800 N. The bonding strength was measured using DCB method and repeated 3 times. The average of bonding strength was 2,436 mJ/m², comparing to 1805 mJ/m² for the PMMA-PS substrates bonded at the same temperature and compression force without the surface treatment.

It is worth mentioning that, there was a side-effect of using the deep-UV surface treatment on PMMA. Owing to the production of some photoproducts during the deep-UV exposure, a yellowing effect can be observed (Seybold & Wagenblast, 1989). An image of untreated and deep-UV surface treated specimens after bonding is shown in figure 3.11. The yellowing effect may reduce the optical transmission of the device and obscure the observation of the flow inside the microchannels.
This chapter introduced a low-cost thermocompression bonding method for enclosing the microchannels fabricated on polymer substrate by bonding another layer of polymer material. Using the bonding method introduced in this chapter, with the proper control of the bonding temperature and compression force, same kind of polymer material or different polymer materials with similar glass transition temperatures can be easily bonded. The bonding strength was also tested in this chapter for the microfluidic chips bonded under the different bonding temperatures and bonding forces. The concept of using deep-UV surface treatment to lowering the bonding temperature was also tested. However, there are also some drawbacks of this method. Due to deformation of the polymer-based chips during the thermocompression bonding, the microchannels could collapsed/clogged with improper setting of the bonding temperature and compression force. Besides that, this method is not suitable for bonding two different polymer materials with very dissimilar glass transition temperatures. This method is also not suitable for enclosing the microchannels fabricated on silicon/glass materials, as the thermocompression bonding temperatures required for silicon/glass are too high for the proposed low-cost thermocompression bonding system.
Chapter 4  Integration Technologies in Low-cost Microfluidic Devices

Chapter 2 and 3 introduced various low-cost microchannel fabrication and chip bonding technologies. In this chapter, several integration technologies will be presented. Different miniaturized components, such as metal electrodes and microlenses were integrated in the polymer-based microfluidic systems. Some low-cost back-end processing technologies, like wafer dicing and piping, will also be introduced in this chapter.

As introduced in the chapter 1, with the desire to integrate the components and functionality of a typical room-sized laboratory into a small chip, the concept of micro total analysis systems (μTAS), or “lab-on-a-chip (LoC)” was introduced in the later 90s. In addition to applications in traditional chemical analysis, integrated microfluidic technology has also been widely used in DNA analysis (Khandurina et al., 2000; Zhang et al., 2006), cell handling (Gu et al., 2004; Hung et al., 2005) and immunoassay analysis (Lim & Zhang, 2007; Ren et al., 2012). For these applications, in addition to microchannels, other components like electrodes and microlenses usually need to be integrated into the microfluidic chips for various purposes such as temperature control, signal collection and flow observation. This chapter provides some low-cost methods to integrate these components in polymer-based microfluidic systems.

4.1  Integration of Metal Electrodes on Microfluidic Systems

Metal electrodes have been extensively integrated in the microfluidic systems for different purposes. The first commonly used application of metal electrodes in microfluidic devices is heater (Liu et al., 2002; Sadler et al., 2003). The temperature control of fluid inside the microchannels is critical for applications like polymer chain reaction (PCR) used for DNA analysis (West et al., 2002). The temperature of fluid for PCR process should be controlled within an accuracy of 1 °C (Wittwer & Garling, 1991), in this case, sensors might also be integrated in the microfluidic devices for the closed-loop control of the temperature.

Metal electrodes integrated in microfluidic chips can also work as a sensing component. Various kinds of metal-based sensors have been integrated into microfluidic
devices, for instance, thermocouples for temperature sensing (Rodriguez et al., 2003),
giant magnetoresistive sensors for DNA detection (Liang et al., 2008), electrodes for
conductivity measurement in cell separation (Adams et al., 2008), electrodes for
capacitance sensing of droplets inside microchannels (Chen et al., 2004). It is also worth
mentioning that, as the sensing component in the microfluidic devices, the metal
electrodes may or may not have direct contact with the fluids, as a dielectric layer is often
used to separate the metal electrodes from the fluid flow inside the microchannel.

Besides working as the sensing components, metal electrodes can also be part of
actuators for flow or droplet manipulation inside the microchannels. In recent years,
many kinds of metal-made actuators based on different principles such as thermocapillary
actuation, (Chen et al., 2004), electrowetting (Pollack et al., 2000) and dielectrophoresis
(Ahn et al., 2006) have been integrated into microfluidic systems for droplet
manipulation.

However, most of the previously mentioned methods of integrating metal electrodes
in microfluidic devices usually use standard photolithographic methods for the patterning
and deposition of the metal layer. In these cases, highly sophisticated instruments for
mask fabrication and photolithography will be conducted in the cleanroom environment,
consuming costly quartz or soda lime mask blanks for each new design.

In this section, a low-cost method of fabricating sputter deposition masks for metal
electrodes and shadow masks for deep-UV patterning will be introduced. With the help of
this method, users can easily integrate metal electrodes in polymer-based microfluidics
with very low-cost materials using common lab instruments. The method of fabricating
metal electrodes introduced in this part is a useful and powerful supplement for the
previously introduced low-cost fabrication techniques for polymer-based microfluidic
systems.

### 4.1.1 Laser Microfabricated Wax-coated Plastic Permanent Paper as Sputter
Deposition Mask for Metal Electrodes on Polymer-based Microfluidic Devices

In this section, a low-cost method for fabricating the metal sputter deposition mask and
depth-UV shadow mask for polymer-based microfluidic systems using a wax-coated
plastic permanent paper will be introduced. For the demonstration of this technology, a T-shaped droplet generator with the metal electrodes for droplet counting was also fabricated.

4.1.1.1 Fabrication Process

The first step of the fabrication process was coating the plastic paper with a layer of wax. This plastic paper (135 μm in thickness) has been previously used in flexible paper-based microfluidics device introduced in chapter 2. For wax coating, a layer (~10 μm thick) of wax was printed on the plastic paper using Xerox Phaser 8560D multifunction solid ink printer, the solid ink is Genuine Xerox Solid Ink. After wax-coating, the CO₂ laser system (Universal PLS6.45) was used for the laser ablation of a metal sputtering mask (illustrated in figure 4.1a) and a deep-UV exposure mask (illustrated in figure 4.1d). For the metal sputtering mask, the laser ablated the desired patterns with the laser energy of 37.5 W and scanning speed of 150 mm/s, the pulse of the laser was set at 1000 PPI (plus per inch). For the deep-UV exposure mask, the detailed discussion of dimensions and spacing of the cutting lines (shown in figure 4.2a) under different laser powers and scan speeds will be provided in the next section. Comparing with the deep-UV patterning using printed wax mask method introduced in chapter 2, the mask fabrication process introduced in this section is more simplified with fewer steps.

After laser ablation, the two wax-coated plastic paper masks were thermally bonded to two separate PMMA substrates (1 mm thick, illustrated in figure 4.1b, 4.1e). The thermal bonding of the PMMA substrates and paper mask was conducted using a thermal laminator (GBC HeatSeal H600 Pro). The wax covered surface of the plastic mask was placed in contact with the PMMA substrate and then sent though the thermal laminator with a temperature setting of 76 °C with a feed rate set at 3 mm/s. In this process, the wax partially melts and works as an adhesive layer to achieve the low temperature bonding of plastic paper mask to the PMMA substrate.
Figure 4.1 Fabrication process for the T-shaped droplet generator integrated with metal electrodes for droplet counting. (a): Laser ablation for the pattering of wax-coated plastic paper as metal sputter mask. (b): Attach the mask on PMMA substrate and sputter a layer of 50 nm thick gold. (c): Remove the plastic paper mask from the PMMA substrate. (d): Laser ablated patterns on wax-coated plastic paper as the deep-UV exposure shallow mask. (e): Deep-UV patterning on the PMMA surface. (f): Remove the plastic paper mask after deep-UV exposure. (g): Two PMMA substrates were bonded to enclose the microchannel, piping and wiring (Fan et al., 2013a).

The PMMA substrate covered with the metal sputter mask was then placed into a low-cost desktop metal sputtering system (Q300T, Quorum Technologies), the sputtering current was set at 50 mA with the coating time of 1 minute at the high vacuum of $5 \times 10^{-5}$ Pa (illustrated in figure 4.1c). A gold layer of 50 nm was sputtered onto the PMMA substrate, figure 4.2b shows the gold layer on PMMA substrate after peeling off the plastic paper mask. The gold layer shows a strong adhesion to the surface of PMMA, however, the thickness of the gold layer should not less than 50 nm to prevent the cracking during the followed thermal compression bonding process.
Another PMMA substrate covered with the paper mask was sent through a deep-UV exposure for 4 hours in a self-built 254 nm deep-UV exposure system that has been used in the research introduced in chapters 2 and 3. Alternatively, other low-cost deep-UV radiation sources can also be used for the exposure of the PMMA substrate (e.g. Stratalinker UV Crosslinker). During the exposure, the plastic paper covered with wax layer will block the deep-UV light from etching the PMMA substrate. After UV exposure, the PMMA substrate was developed using a solution of 7:3 IPA:water, the development process was conducted at 28 °C for 30 minutes in a water bath. The fabricated T-junction microchannel is illustrated in figure 4.1f.

Thermocompression bonding (shown in figure 4.1g) was used for bonding the two PMMA substrates, which contain the metal electrodes and microchannels, respectively. The detailed bonding system setup and detailed introduction of this bonding method can be found in chapter 3. To achieve proper bonding of the PMMA substrates and protecting the sputtered metal layer, the bonding temperature was set at 140 °C with the compression pressure of 0.5 MPa for 50 minutes.

Some back-end processes were required to finish the fabrication. Three plastic pipes (Silastic tubing, Dow Corning Corporation, USA) were connected to the two inlet and one outlet ports in the chip. With the help of conductive glue (PELCO Conductive Silver
18, USA), wires for signal collection could be simply glued to the metal electrodes avoiding the need for wire-bonding. The microfluidic device after fabrication is shown in figure 4.3.

![Figure 4.3](image)

**Figure 4.3 Fabricated T-shaped droplet generator integrated with metal electrodes for droplet counting (Fan et al., 2013a).**

4.1.1.2 **Experimental Result and Discussion**

The schematic view of the fabricated T-shaped droplet generator integrated with gold electrodes for droplet counting is shown in figure 4.4a. The transparent liquid was the mineral oil and the red liquid was DI water. During the experimental process, when the oil stream enters the main channel, the oil droplet starts to grow into the downstream direction, with the pressure gradient and drag of the water flow, the oil flow will neck and an oil droplet will break free into the water flow and move downstream (Garstecki et al., 2006). An optical image of the oil droplet in the main channel is shown in figure 4.4b.

This droplet counter is based on the coulter counter operational principle. A voltage source was connected between the two gold electrodes, when an non-conductive oil droplet passes between the two electrodes, the sudden resistance change between two electrodes will cause a current drop, by monitoring this current change, we can calculate the number of droplets that have been generated in the main channel. This could be useful when accurate control of droplets is needed in chemical or biological, analysis or synthesis.
In this research, laser ablation was used to fabricate the masks for both the metal sputtering and the deep-UV exposure. The minimum spacing of laser ablated lines on the wax-coated plastic paper was tested; the result is shown in figure 4.5. Experimental results indicate that the minimum reliable spacing was about 0.5 mm, if the line spacing was smaller than 0.5 mm, the thermal deformation caused by the laser ablation will severely warp the plastic paper. For the line spacing larger than 0.5 mm, slight deformation could also be observed; however, some of the deformation is mended when this plastic paper mask goes through thermal lamination to the PMMA substrate.

Figure 4.4 (a): Illustration of the droplet generation in a T-junction droplet generator and (b): Optical image of oil droplet in water (dye in red) captured by microscope (Fan et al., 2013a).

Figure 4.5 Warping of the wax-coated plastic paper after laser ablation. The minimum spacing between laser ablated lines needs to be wider than 0.5 mm to avoid significant warping (Fan et al., 2013a).
An SEM image of the laser ablated lines at the T-junction on the wax-coated plastic paper mask is shown in figure 4.6a, the corresponding area on the patterned PMMA substrate after deep-UV exposure and development is shown in figure 4.6b. Generally, the minimum feature of a lithography method depends on the wavelength of the radiation source and the space between the mask and substrate. However, in this masking method the bottleneck is the minimum width of the laser ablated line on the laser cut deep-UV mask, because of the imperfect focus of the laser beam, the minimum feature this method could consistently achieve was around 200 μm.

Microchannels fabricated using deep-UV exposure and direct laser ablation are compared in figure 4.7 and 4.8, where it can be easily observed that the deep-UV patterned microchannel has a smoother inner wall compared with the laser ablated microchannel, which could make the optical observation of the fluid inside the deep-UV microchannel much easier. The surface difference was due to the two different fabrication methods, for deep-UV patterning, the energy is absorbed and the chain scission will happen at the surface of the PMMA, causing the increase of solubility and the subsequent development of the exposed region. Thus, the formation of the microchannel during development in the deep-UV patterning method is a chemical process, which generates a relatively smoother surface. The laser ablation process for microchannels on PMMA is purely thermal, the violent phase change of materials...
through melting and evaporation in a short time lead to the rough surface after laser ablation.

![Figure 4.7 Images of microchannel fabricated by (a): Deep-UV lithography and (b): Laser ablation (Fan et al., 2013a).](image)

![Figure 4.8 SEM images of microchannel fabricated using (a): Deep-UV lithography and (b): Laser ablation (Fan et al., 2013a).](image)

The low-cost self-built deep-UV exposure box was equipped with low-pressure mercury vapor lamps which the light was not collimated. The chips were placed on a rotational stage during UV exposure inside the exposure box to even out the exposure, the non-collimated deep-UV light slightly enlarged the features during the exposure process to transfer the pattern from mask to PMMA substrate, which was known as the “undercut effect” (Lin, 1975). Figure 4.9 compares the feature size on the plastic paper mask and the actual feature size after deep-UV exposure and development on the PMMA substrate. On average, an 80 μm undercut was observed when comparing the feature size on mask and the developed microchannel on the PMMA. This 80 μm can be easily
compensated for by reducing the feature size on the plastic paper mask in the chip design process. The profile measurement (Ambios XP-200 profilometer) of the deep-UV patterned microchannel on PMMA is shown in figure 4.10, this microchannel has a width of 410 μm and channel depth of 54 μm with a smooth inner wall.

Figure 4.9 The undercut effect of the microchannel during deep-UV exposure and development process (Fan et al., 2013a).

Figure 4.10 The profile measurement of the microchannel fabricated using deep-UV exposure on PMMA substrate (Fan et al., 2013a).
Moreover, because of the spacing of between the plastic permanent paper mask and the PMMA substrate underneath, the sputtered metal layer was also enlarged during the sputtering process. The reason for the enlarged metal layer is during the sputtering process, the atoms have a diffuse transport to the polymer substrate and the spacing between mask and PMMA substrate make a full shadow impossible. Figure 4.11 shows the comparison between the feature size on the paper mask and the actual feature size of the sputtered metal layer. Again, this expansion on feature size could be compensated for by shrinking the size on mask during the design process on CAD (Computer-aided design) packages.

![Figure 4.11 The comparison between the size of the feature on mask and actual sputtered metal features, with the 1:1 reference line (Fan et al., 2013a).](image)

Comparing with evaporation metal deposition method, the instrument cost of the metal sputter deposition is much lower. The sputtered metal layer typically has a better adhesion on the substrate than evaporated metal layer, because of the metal atoms impact energetically on the surface of the polymer substrates. Besides that, the metal sputter deposition instrument contains no hot parts and is compatible with reactive gases such as oxygen, which the evaporation instruments usually need to melt down the metal target. During experimentation we found that metal layers thinner than 50 nm were easily
damaged (cracking) during the thermocompression bonding process. Thus we adopted a 50 nm thick gold layer in order to maintain reliable electrodes after bonding, which we deposited using multiple sputtering steps.

The UV transmissions of the wax layer and the plastic paper with/without wax coating were also tested to validate the ability of using wax-coated plastic paper as the shadow mask during the deep-UV exposure. The result indicates that, the wax-coated plastic paper has a transmission of 0.8333 % at the wavelength of 254 nm. The plastic paper without wax coating has a transmission of 2.247 % at the wavelength of 254nm. The quartz (transparent to UV light) coated with wax layer has a transmission of 0.853 % at the wavelength of 254 nm. These results validates that the wax-coated plastic paper can effectively block the UV light with the wavelength of 254 nm during the exposure.

The width of the lines ablated on the wax-coated plastic paper under various laser powers and scan speeds is shown in figure 4.12. Similar to the result of laser ablated microchannels on a PS substrate, the line width will increase with increasing laser power and reduce with the higher scanning speeds.

![Diagram](image)

Figure 4.12 Width of the laser ablated lines on plastic paper under various laser scanning speed and laser power (Fan et al., 2013a).
The experimental setup for the droplet counting system is shown in figure 4.13. A voltage source supplies a constant voltage of 12 V between two electrodes; an oscilloscope was connected to a 1 Ω resistor in this series circuit to measure the voltage on this resistor. By monitoring the voltage on the resistor, the corresponding current in the circuit can be easily calculated. As previously mentioned, when an oil droplet goes through the electrodes, a transient current drop could be observed as a reduction in voltage. The signal measurement from oscilloscope is shown in figure 4.14, each trough in the signal represents an oil droplet passing between the two gold electrodes.

Figure 4.13 Schematic of the droplet counting system setup. When a non-conductive oil droplet goes through two electrodes, the sudden current drop was recorded (Fan et al., 2013a).

Figure 4.14 Signal measured by oscilloscope, each peak indicates an oil droplet goes through the electrodes (Fan et al., 2013a).
In this section, a low-cost and rapid technique for fabricating metal sputtering and deep-UV patterning masks using wax-coated permanent plastic paper was introduced. The research in this section provided a rapid and simple solution to fabricate deep-UV patterned microchannels integrated with metal electrodes. For demonstration of this technical innovation, a PMMA-based microfluidics device integrated with gold electrodes for droplet counting was also introduced. The whole process is simple, rapid and low-cost, without the requirement of a cleanroom; users can easily adapt this process using the lab facilities they already have. This technique could have wide applications in lab-on-a-chip technologies. The drawbacks and limitations of the method introduced in this section come from the instruments, the commercial laser engraving system used in this research limited the minimum dimension of microchannels/metal electrodes this method can achieve. Besides that, the non-collimated deep-UV light and spacing between paper masks and PMMA substrates caused the enlarging of the patterned structures.

4.2 Integration of Microlenses in Low-cost Polymer-based Microfluidics

Microlenses and microlens arrays (MLAs) have been widely used and developed in the areas in optical communications (Iga et al., 1982), light-emitting diodes (Li et al., 2011b), optical fibers (Cohen & Schneider, 1974), etc. Recently, with the development of microfluidics technologies, microlenses or MLAs have been integrated in microfluidic systems for various purposes, such as fluorescence detection (Roulet et al., 2001), size sorting of nanoparticles or cells (Sun et al., 2006) and point-of-care diagnostics (Myers & Lee, 2008).

Different fabrication technologies have been invented for polymer-based microlenses and MLAs, including: direct ink-jet-deposit of UV-curable resin (O’Neill et al., 2005), reflow of photoresist (O’Neill & Sheridan, 2002), hot embossing (Ong et al., 2002) and replica molding (Shih et al., 2006). These fabrication methods for polymer-based microlenses have the potential to be integrated into polymer microfluidic systems. Various polymer materials were used for the fabrication of microlenses, such as PMMA (Beinhorn et al., 1999), PDMS (Chronis et al., 2003) and SU-8 (Yang & Wang, 2004). However, there are some drawbacks to these methods for polymer-based microlenses, for ink-jet-deposition, the profile of the microlens is not well controlled; for the photoresist
reflow method, a cleanroom environment with expensive instruments is needed and photo resist lenses are absorbing in the UV range limiting their application to fluorescence based systems; replica molding is very costly unless large quantities are required.

In this section, a low-cost microlens and MLA fabrication technology based on PS will be introduced. PS is highly transparent in the visible light spectrum, and has been extensively used as a material for optical fibers (Rao et al., 1991) and waveguides (Singh et al., 1992). In this research, the microlenses and MLAs were directly fabricated on the surface of a PS substrate, which also acts as either the cover plate or substrate in the microfluidic system. In this way, the microlenses can be easily integrated into the polymer-based microfluidic system without adding another layer of material. The microlens fabrication method proposed in this section is low-cost and rapid, without the requirement of any costly processing tools or cleanroom. Microlenses with different dimensions and profiles can be fabricated using these two methods.

4.2.1 Low-cost Fabrication and Integration of Microlenses with Convex and Concave Profiles
In this section, a low-cost method for fabricating polymer-based microlenses with both convex and concave profiles will be introduced. This polymer-based microlens fabrication method can easily be integrated in the fabrication process of microfluidic devices for various applications. In this approach, the PS microlenses with both convex and concave profiles were fabricated using the thermocompression molding method. This thermocompression molding method originated from the thermocompression bonding method introduced in chapter 3, the same concept and system setup was adapted for this research. For the molding process, since the glass transition temperature of PMMA is higher than PS, PMMA was used as the mold for the patterning of PS. The PMMA mold was fabricated using low-cost and rapid CO₂ laser patterning.
4.2.1.1 Thermocompression Molding for Microlenses with both Concave and Convex Profiles

Thermocompression molding for fabricating the microlenses is illustrated in figure 4.15. The first step was fabricating the PMMA mold, multiple hemisphere shaped depressions were laser ablated on the surface of a PMMA sheet (2.5 mm thick, Thai Poly Acrylic), using an Epilog Legend CO₂ laser system (illustrate in figure 4.15a). During the laser ablation process, the energy of this laser system was set at 50 W with the frequency of 5kHz, the maximum resolution this setting could achieve was 600 dots per inch (ppi). As introduced in chapter 2, high-energy laser ablation on the surface of polymers will cause the materials to melt and evaporate. Based on this, a series of hemisphere depressions with the diameter of 800 μm were fabricated on the surface of the PMMA sheet, which will be used as the mold for the following thermocompression molding process.

Figure 4.15 Fabrication of microlens with convex and concave profiles using thermocompression molding method (Fan et al., 2014b). (a): Fabrication of PMMA mold using laser ablation. (b): Applying a drop of DI water for the fabrication of microlens with concave profile. (c): Thermocompression molding for the microlens. (d): Demolding after cooling down.
The second step was defining the convex and concave microlens on PMMA mold, 1 μL of DI water was dropped in the hemisphere depression using a pipette where concave microlenses were needed (illustrated in figure 4.15b). The formation principle of the convex and concave microlenses is shown in figure 4.16. During the thermocompression molding process, the PMMA mold was covered with a PS substrate (1.2 mm thick, Cambridge Limited), then the PMMA mold and PS substrate were sent through the thermocompression system that has been introduced in chapter 3 (shown in figure 4.15c) for the molding process. To perform the molding, pressure was applied between the PMMA and PS, and the temperature was controlled at 120 °C by the environmental chamber and held for 50 minutes.

During the thermocompression molding, as illustrated in figure 4.16, the water trapped in the hemisphere depression will rapidly vaporize and pressure will build up inside the chamber (pressure increased 1,700 times). The surface of PS was soft because of the temperature inside the environmental chamber was above the glass transition temperature of the PS, thus the high pressure inside the chamber will form a perfect concave profile in the PS. For the hemisphere depressions on the PMMA mold without the water the softened PS will be pushed into the mold to form a convex microlens. It is important to note that the convex surface was not in contact with the PMMA mold and thus the roughness of the laser cut depression is not transferred to the lens, because of the air trapped inside the chamber. During the thermal compression molding process, the profile of the convex microlens was formed by the surface tension of polystyrene, which was controlled by the temperature inside environmental chamber and compression force. After thermocompression molding, the PMMA mold and the PS substrate were detached and the PMMA mold can be reused.

For the demonstration of the easy integration of this microlens fabrication method into the fabrication process of a microfluidics device, a T-shaped droplet generator with the integrated microlenses was fabricated. The fabrication process is shown in figure 4.17, a PS substrate was laser patterned with the microchannels (figure 4.17a), and another PS substrate (cover plate) was laser patterned with the inlet and outlet ports. This cover plate then had microlenses fabricated on it using thermocompression molding (figure 4.17b). Finally, the PS substrate and the PS cover plate with microlenses were thermally bonded.
Figure 4.18 shows the microfluidics testing system setup and the fabricated convex microlens overlapping the microchannel.

Figure 4.16 Illustration of the formation of microlenses with both convex and concave profiles during the thermocompression molding (Fan et al., 2014b).

Figure 4.17 Fabrication of microchannels and integration of microlenses in the microfluidic device (Fan et al., 2014b). (a): Laser ablation for the microchannels on PS substrate. (b): Fabricated PS cover chips after thermocompression molding. (c): Bonding system setup.
4.2.1.2 Result and Discussion

SEM (Quanta 500FEG) images of the fabricated microlenses with both convex and concave profiles are shown in figure 4.19. The profile measurements of these two microlens are shown in figure 4.20 (Ambiox XP-200 profilometer). The measurement result shows the convex microlens to have a height of 38 $\mu$m with a width of 550 $\mu$m, and the concave microlens to have a depth of 75 $\mu$m with a width of 660 $\mu$m.

Some edge roughness could be observed for the convex microlens, which is mainly due to interaction of the PS with the surface of the PMMA mold during the thermocompression molding process and can be improved by making the mold deeper to avoid contact. However, the roughness at the edge will not have a significant influence on the optical qualities of the convex microlens. The surface of the concave microlens was smooth, which is due to the effect of the high pressure from the vaporized water on the soft surface of the PS past its glass transition temperature.

The temperature control of the thermocompression molding was critical. For the proposed method in this section PMMA was used as the mold material for the thermocompression molding of microlenses in PS. Thus, the temperature setting needs to be higher than the glass transition temperature of PS, but slightly lower than the glass transition temperature of the PMMA mold. Thus, the surface of PS will be soft enough
for the formation of microlenses and the PMMA mold will not have significant
deformation under the molding pressure. Overheating may cause the deformation of the
PMMA mold and bonding of the PMMA and PS as described in chapter 3.

Figure 4.19 Scanning electron microscope images. (a): Convex microlens and (b): Concave microlens
(Fan et al., 2014b).

Figure 4.20 The cross-section measurement of the fabricated microlens with convex and cave profiles
(Fan et al., 2014b). (a): Profile measurement of the convex microlens. (b): Profile measurement of the
concave microlens.

The diameter of the convex microlens fabricated in this research was around 500 μm. In this research, the convex microlens were used for giving the magnified view of the fluid inside the microchannel. Even though the microlenses could be made smaller with the thermocompression molding method demonstrated in this research, we were constrained by the required focal length. The convex microlenses were fabricated on the
PS substrate, which formed the plano-convex lens system (shown in figure 4.21). After calculation using the lens’s-maker’s equation, in the case of the PS material (refractive index at the center of visible light 587 nm is 1.5916), the focal length equals to 1.69 times the radius of the convex microlens. Thus, in order to keep the focal length long enough to magnify the view of the fluid inside the microchannel, the dimensions of the lens need to be large enough for the focal length to be greater than the thickness of the PS plate.

The two optical images shown in figure 4.22 were obtained from a microscope (Axio Observer, Zeiss, Germany) with the magnification setting at 10X. Figure 4.22a was taken with the convex microlens overlapping the microchannel, while the figure 4.22b was taken directly above the microchannel with no microlens. It can be seen that, with the help of the convex microlens fabricated using the method proposed in this section, the object was enlarged by about 1.61 times (by pixel count).

Figure 4.21 Schematic of the plano-convex lens system for the convex microlens fabricated on PS substrate.

Figure 4.22 The droplet in the microchannel observed with (a) and without (b) the microlens (Fan et al., 2014b).
In this section, a low-cost method for direct integrating microlenses on polystyrene based microfluidic devices was introduced. With the help of the thermal compression molding system and the laser fabricated PMMA mold, the microlens both concave and convex profiles can be fabricated. However, this research is still at the proof of concept stage, the detailed relations between the curvature/radius of the microlens and the molding temperature/pressures are not investigated in details. Besides that, the roughness of the microlens is not characterized under the different molding temperature and molding pressure.

4.3 Low-cost Silicon Wafer Dicing Using a Craft Cutter

A silicon wafer contains many repetitions of the desired device after fabrication; each of these repeated blocks is called a “die”. Each die needs to be separated from the wafer after fabrication, so that they can be separately packaged. This dividing up process of the wafer is called dicing. Various technologies have been developed for the dicing of silicon wafers, most of which are invented for the semiconductor industry. Mechanical dicing was the earliest dicing method and still widely used today, during mechanical dicing, a saw blade rotating at very high speed is used to directly cut the silicon wafer (Efrat, 1993). Due to vibration and stress during mechanical dicing, some damage such as chipping and cracking is unavoidable. The trend of using thinner wafers (thickness less than 100 μm) in the semiconductor industry makes mechanical dicing even harder, thus laser ablation (Lizotte, 2003; Krishnan et al., 2008) has also been used for the dicing of silicon wafers. However, the high temperature and deformation during laser ablation of the silicon wafer may cause damage to the structures fabricated on silicon wafer. To improve these shortcomings during laser ablation, the so called “stealth dicing” method was invented (Kumagai et al., 2007; Ohmura et al., 2008), where instead of direct laser ablation on the surface of the silicon wafer, the focus point of laser is adjusted to process inside of wafer. This leaves the wafer clean, and without any contamination due to chipping which other dicing method will generate during the processing.

However, for the low-cost microfluidic structures fabricated on a silicon wafer in a research context, all the above mentioned methods are too expensive: the mechanical dicing tools usually cost a minimum of fifty thousand dollars, laser dicing tools cost even
more, and all of these dicing methods are generally performed in a “dirty” area of the cleanroom. In this section, an ultra-low-cost and rapid silicon wafer dicing method will be introduced. This method was designed for the dicing of microfluidic devices fabricated on silicon wafers with a minimum die dimension larger than 3 mm. The dicing tool used in this method is a craft cutter, which is a relatively ultra-low-cost tool (cost lower than one thousand dollars) and easily accessible for most labs without need for a cleanroom environment.

### 4.3.1 Silicon Wafer Dicing Process

The ultra-low-cost silicon wafer dicing method demonstrated in this research requires only two simple instruments to perform the whole process. The system setup is shown in figure 4.23, which contains a craft cutter (Silver Bullet, That’s Scrap, Inc., USA) mounted with a diamond tip and a computer for dicing route design and device control. The dicing path, speed of the diamond tip on the silicon wafer and contact force between diamond tip and the wafer can be preprogramed and controlled.

![Figure 4.23 Ultra-low-cost silicon wafer dicing system setup (Fan et al., 2014a).](image)

The detailed alignment and dicing process is shown in figure 4.24. A standard 4 inch (100) orientated silicon wafer (500 μm thick) was used for the demonstration of the dicing process. Square-shaped dies of four different dimensions (3 mm, 5 mm, 7 mm and 10 mm) were fabricated on this silicon wafer. Each die contained a deposited metal structure, the metal structure was a combination of a layer of 100 nm thick gold on top of a 50 nm thick chrome which works as the adhesion promoter between the silicon wafer and gold layer.
After the patterning of metal structures on the silicon wafer, a series of alignments need to be performed before the dicing. A transparency film (product code: PP2500 3M, Inc, Italy) with printed alignment marks was aligned with an A4 size cutting mat which is covered with a layer of low-tack adhesive (shown in figure 4.24b). Then, the silicon wafer was carefully hand aligned on the cutting mat with the help of the alignment marks on the transparency film (figure 4.24c). The detailed alignment relations between transparency film, cutting mat and silicon wafer are shown in figure 4.25. The transparency film was removed after the wafer aligned on the cutting mat (figure 4.24d).

Figure 4.24 The wafer alignment process on cutting mat. (a): A 100-oriental silicon wafer with deposited metal patterns. (b): The alignment process between the cutting mat and transparency film. (c): Wafer attached on the cutting mat with the help of alignment marks on transparency film. (d): Transparency film was removed. (e): Scribing of silicon wafer in the craft cutter (Fan et al., 2014a).
After the three-step alignment process, the scribing can start. Since the cutting mat was coated with a layer of low-tack adhesive, the silicon wafer can safely be attached to the cutting mat during the scribing process and then easily removed after scribing. The scribing route of the diamond tip was designed using CorelDraw X4, the software for the craft cutter control was Sure cuts a lot 3. For the wafer scribing process (figure 4.24e), the cutting mat with attached silicon wafer was fed into the craft cutter, the origin point of the scribing route was set using the alignment mark on the cutting mat. Scribing speed of the diamond tip on silicon wafer was 40 mm/s with the force setting of 0.4 N between the tip of diamond dicing blade and silicon wafer. After scribing, the silicon wafer was carefully removed from the cutting mat and blown with nitrogen gas to clean the surface. The final step was cleaving, the silicon wafer was carefully cleaved along the scribing lines to separate each die on the silicon wafer using tweezers.

To avoid damage and contamination of the silicon wafer’s surface during the scribing and cleaving process, a method of backside wafer dicing will also be briefly introduced in this section. For the protection of the structural layer fabricated on the silicon wafer, a layer of 4 μm thick photoresist (AZ ECI 3027, AZ Electronic Materials, USA) was spin coated on the front surface (surface with deposited metal structures) of the silicon wafer.
Then, the wafer was aligned using the identical procedure introduced in figure 4.24 with the front surface contacting the adhesive layer on cutting mat. After that, the backside of the silicon wafer was scribed using the craft cutter, since the grooves during the scribing were on the backside of the silicon wafer, damage and contamination of the front surface of the wafer can be avoided. After scribing, the wafer was carefully detached from the cutting mat, cleaved to separate each die, followed by stripping of the photoresist layer on the front surface using acetone. Thus, using this back dicing method, the damage and contamination on the surface of the silicon wafer can be avoided during the scribing and cleaving process.

4.3.2 Dicing Results and Discussion

An optical image of the scribed lines on the surface of a silicon wafer is shown in figure 4.26a. Square-shaped dies with different dimensions (3 mm, 5 mm, 7 mm and 10 mm) separated from the silicon wafer are shown in figure 4.26b. It can be observed from these two figures that, the scribing line was slightly offset from the center of the scribing street by approximately 300 μm, which was primarily due to the unavoidable alignment error in the alignment process and mechanical limitations of this low-cost craft cutter.

The alignment process contains three steps to fix the two degrees of freedom between the silicon wafer and cutting mat, since the alignment process was purely manual in this demonstrated process, some alignment error was unavoidable. However, after careful evaluation of the common lab users without specific training on alignment, the square-shaped dies with the dimension of 3 mm could easily be achieved, which was suitable for most microfluidic devices fabricated on silicon wafers.
Figure 4.26 Scribing lines and dies after the dicing process. (a): Scribing lines on the silicon wafer. (b): Dies after wafer dicing process, with the dimensions of 3 mm by 3 mm and 5 mm by 5 mm (Fan et al., 2014a).

Figure 4.27 SEM images. (a): Scribing line on silicon wafer and (b): Edges of the die after dicing process (Fan et al., 2014a).

Figure 4.27 shows the scribing grooves on the silicon wafer and the edge of the die after cleaving. Some roughness can be observed for the scribing grooves and the edge of the die, which was due to the mechanical scribing of the diamond tip on the surface of the silicon wafer and the manual cleaving. The profile measurement of the scribing grooves is shown in figure 4.28, indicating the groove has a width of 52 μm and depth of 7.5 μm.
In this study, an ultra-low-cost silicon wafer dicing method was introduced. Compared with other silicon wafer dicing methods, this method only requires a commercial craft cutter and does not require a cleanroom environment. Square dies with a side length of 3 mm could be easily achieved using this method, which is suitable for most microfluidic applications. In addition, this method also provides a simple solution for the multi project wafers (MPW), in which dies of different dimensions could be fabricated on the same wafer and diced using the method provided in this section. However, there are also many limitations for using this ultra-low-cost silicon wafer dicing method proposed in this research, only silicon wafer (or material with crystalline structure) can be scribed and cleaved. The precision of this method (3 mm) is still much lower than conventional wafer dicing instruments in the cleanroom (lower than 0.01 mm), because of the usage of a low-cost commercial craft cutter to perform the scribing process.

4.4 Fabrication of Microfluidics Chip Holder and Connector Using 3D Printer
In order to simplify the piping process after the fabrication of low-cost polymer-based microfluidics chips, a simple and low-cost chip holder set will be introduced in this section. With the help of this chip holder, the user only needs to insert the polymer-based microfluidic chip into the holder after chip fabrication, tighten the holder set and start the experiment.
4.4.1 Design and Fabrication

This chip holder was designed for holding chip with a standard dimension of 25.4 mm by 76.2 mm (1 inch by 3 inch). The dimension of the chip holder set could easily be resized if needed to fit chips with nonstandard dimensions. This chip holder set contains 12 inlet/outlet ports on the cover plate in order to meet varied requirements for inbound and outbound flow. The design of the chip holder ensures the smooth insertion and secure position of the microfluidic chip.

The fabricated chip holder set is shown in figure 4.29, this chip holder set consists of a substrate plate (figure 4.29 left) and a cover plate (figure 4.29 right), both of which were fabricated using a 3D printer (Maker Gear M2, USA). The 3D printing system used in this study was based on the fused deposition modeling (FDM) of PLA (Poly(lactic acid)) filament. The temperature setting of the filament fusion at the injection head was 215 °C with a deposition bed temperature of 70 °C. The chip holder set was printed layer by layer, with the layer thickness of 0.2 mm, and the density of the solid model was set at 30 percent. The whole 3D printing process took around 2 hours.

![Fabricated chip holder set, substrate (left) and cover plate (right).](image.png)

Figure 4.29 Fabricated chip holder set, substrate (left) and cover plate (right).

After the 3D printing process, the microfluidic chip in standard size (1 inch by 3 inch) can be directly inserted into the holder substrate, then the cover plate was attached and four screws were tightened to ensure that the gaskets compress and a fluid tight seal is
formed (shown in figure 4.30). For demonstration, a microfluidic droplet generator chip was tested in this 3D printed chip holder set (shown in figure 4.31).

![Figure 4.30 Direct insertion of a microfluidic chip into chip holder (left), attached cover plate with four screws tightened to ensure sealing (right).](image)

![Figure 4.31 A droplet generator chip was inserted and tested in the 3D printed chip holder set.](image)

### 4.4.2 Result and Discussion

In this work, a simple and low-cost chip holder set was fabricated using 3D printing based on fused deposition modeling. With the help of this chip holder set, the piping process for the microfluidic system can be significantly simplified, the user only needs to insert the chip, attach the cover plate and start the experiment. However, there are also some drawbacks and limitations of this 3D printed chip holder set. Due to the low-cost of the 3D printer, the precision and resolution this 3D printer can achieve is relatively low, some swelling of the 3D printed structure can also be observed.
Chapter 5 Conclusion

5.1 Conclusion

The technologies demonstrated in this research provide a flexible choice of low-cost fabrication methods and polymer materials based on the users’ requirements for channel profile, surface quality, chemical resistance and light transmittance.

The low-cost microchannel fabrication methods introduced in this research can be classified into deep-UV patterning and laser processing. For deep-UV patterning, a self-made low-cost 254 nm deep-UV source was used for the patterning of microchannels on the surface of PMMA. A novel printed wax mask was introduced in this deep-UV patterning process. Based on this deep-UV patterning method, a deep-UV LIGA process was also introduced in this research for the fabrication of high aspect ratio metal structures. For laser processing of microchannels, a commercial low-cost 10.6 μm CO₂ laser was used for the thermal ablation of microchannels on the surface of various polymers. The profiles of the microchannels were carefully studied at various laser energies and scan speeds. An interesting phenomenon of bump formation on the surface of PS under low laser energy was also introduced, with the help of this method, the height of the bump can be controlled by laser energy and number of scans. A novel method of fabricating multi-depth microchannels via PDMS molding, using this laser induced bump was also presented in this research. The surface qualities of the deep-UV patterned and laser ablated microchannels were also compared and discussed.

A low-cost thermocompression bonding method was introduced in this research for the sealing of the microchannels after fabrication. Various polymer materials such as PMMA and PS were successfully bonded using this method. The bonding strength was measured using two methods and compared. In order to prevent channel deformation during the thermocompression bonding, a deep-UV surface treatment method was tested to lower the bonding temperature. This thermocompression bonding method is widely applicable to most of the thermal plastics.

Several low-cost integration technologies were introduced in this dissertation as a supplement for the low-cost fabrication methods in polymer-based microfluidics. Metal
electrodes were integrated in the polymer microfluidic devices using metal sputtering with the help of a laser ablated wax mask. This laser fabricated wax mask could be used both for the deep-UV patterning of PMMA and metal sputtering. For easier observation of the flow inside the microchannel, microlenses fabricated by thermal molding were also presented in this research. With the help of these fabrication technologies, microlenses can be directly integrated on the surface of polymer microfluidic devices. Other than these integration technologies, an ultra-low-cost silicon wafer dicing method using a craft cutter was also presented in this research, which significantly lowers the cost of silicon wafer dicing. A 3D printed microfluidic chip holder was introduced at the end of this dissertation to simplify the piping process after chip fabrication.

5.2 Published Contributions

5.2.1 Journal Publications


5.2.2 Conference Publications


5.3 Future Directions
The fabrication and integration methods for low-cost polymer-based microfluidic systems introduced in this research provided a simple and low-cost solution for multiple research purposes in biological, medical and chemical fields. The future directions of this research lie in, but are not limited to two directions. The first direction is providing more low-cost fabrication methods to lower the access of microfluidics technologies, the future work in this direction includes: low-cost digital microfluidics, modular microfluidics, disposable microfluidics and low-cost high-throughput microfluidics. The second direction is discovering the underlying physics of the interesting findings in this dissertation. For instance, the underlying physics of the laser induced bump formation on PS could be a very interesting topic, detailed bump formation process is still not clear at this point. Besides that, the detailed study of physical/chemical changes during the laser ablation on polymer materials could be very beneficial to other researchers who want to perform laser processing on polymer materials. In the followed section, the future direction in modular microfluidics will be briefly introduced.

5.3.1 Modular Microfluidics
In the previous chapters, various low-cost fabrication methods for polymer-based microfluidic systems have been introduced. As a future direction, a modular microfluidic system could be developed based on the technologies introduced in this research. This
modular microfluidics system could further lower the access of the microfluidics technologies for researchers in biological and chemical field. By directly combining pre-fabricated modules, users could easily build their own microfluidic system without any requirement of microfabrication experience and equipment.

This proposed modular microfluidic system falls into two parts, the supporting platform and functional microfluidic modules. The supporting platform would help to combine the modules, and provide some necessary supply (gas, signal collection, temperature control) for each of the microfluidic module if needed. Different microfluidic modules could be designed with different functions, each module will have its own function, such as mixer, droplet generator, flow sensor, valves and pump chips. With the customized combination of the modules using supporting platform, users without any experience in microfabrication can easily build their own multifunctional microfluidic systems. Another advantage of this system is a single module could easily be replaced if contaminated or damaged during the experiment, while other modules can be reused. The selection of materials for each module could be flexible based on their function, various kinds of polymers (PMMA, PS, PDMS), glass and silicon could be used. The low-cost fabrication methods introduced in previous chapters could be very beneficial in fabricating these modules with various materials and precision requirements.

This future direction of modular microfluidics also has a potential for commercialization. A custom-made microfluidics toolkit containing various microfluidic chips and a supporting platform could be a powerful tool for researchers. The commercialization for modular microfluidics in massive production could further lower the cost, which the price for each chip could falling as low as several dollars. This could be very beneficial for researchers who want to apply microfluidics technology to their researches.

5.4 Final Word

This dissertation provided a comprehensive solution for low-cost polymer-based microfluidics, covering all three stages in a microfluidic chip fabrication process: microchannel fabrication, microchannel sealing (bonding) and integration technologies. With the help of the technologies demonstrated in this work, researchers who wish to
apply microfluidics in their research can easily fabricate their own microfluidic chips using common lab instruments at a very low cost.
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