



Design and Fabrication of CD-Like Microfluidic Platforms for Diagnostics: Polymer-Based Microfabrication

*L. James Lee,¹ Marc J. Madou,² Kurt W. Koelling,¹
Sylvia Daunert,³ Siyi Lai,¹ Chee Guan Koh,¹
Yi-Je Juang,¹ Yumin Lu,² and Liyong Yu¹*

¹Department of Chemical Engineering, The Ohio State University,
Columbus, Ohio 43210

²Department of Materials Science and Engineering, The Ohio State
University, Columbus, Ohio 43210

³Department of Chemistry, University of Kentucky

Abstract. Several microfabrication methods for polymer-based CD microfluidic platforms are presented in this paper. For prototyping, both traditional CNC-machining and photolithography techniques were used. For mass production, mold inserts were made by CNC-machining of tool steel and LIGA-like processes such as UV photolithography, photolithography/electroplating, and photolithography/deep reactive ion etching (DRIE). Several molding methods were tried, including liquid resin casting, thin wall injection molding, and hot embossing. Advantages and disadvantages of each method are explained. Plastic bonding for microfluidic platforms is also briefly discussed.

Key Words. microfabrication, CNC-machining, photolithography, electroplating, deep reactive ion etching, liquid resin casting, thin wall injection molding, hot embossing

Introduction

In the past, micro-electro-mechanical-systems (MEMS) including microfluidic devices have been fabricated almost exclusively in silicon or glass because similar technologies are available in the microelectronics industry. For many applications, particularly in the biochemistry and biomedical field, these materials and the associated production methods are too expensive, or the material properties often induce problems like lack of optical clarity, low impact strength, and poor biocompatibility. Polymeric materials, on the other hand, offer a wide range of physical and chemical properties. They also have the advantages of low cost, good processibility for mass production, and are biocompatible and recyclable. Polymer microfabrication techniques, however, are still not well developed. In this paper, we present a series of fabrication techniques capable of prototyping or mass producing polymer-based microfluidic devices such as CD platforms.

Prototyping Fabrication

For testing the design concept of microfluidic devices, low-cost prototyping techniques are highly desirable. If the feature size is larger than 50 μm or different feature depths are required, computerized numerically controlled (CNC) milling can easily be used to mechanically manufacture the device. Figure 1 shows a CNC-fabricated plastic CD platform with four sets of 2-point calibration structures. CNC machining does not provide either a smooth surface finish (surface roughness around several μm), a high aspect ratio (height vs. width ratio is limited to less than 2), or good dimension control (often 10% large than design). It does, however, have no material limitations, and various metals, glass, and plastics can all be used. For feature sizes smaller than 50 μm (but larger than several micrometers), micro-milling or laser ablation can either be used alone or in conjunction with CNC machining for prototyping fabrication. Yet all of these prototyping fabrication processes are labor intensive and slow, and it typically takes several hours to make a single CD platform.

Thick photoresist-based lithography has been widely used in micromachining [1–3]. It has several advantages. For example, high aspect ratio features are attainable [3] and very small feature sizes ($\sim 1 \mu\text{m}$) can be achieved. In our study, a thick layer of negative photoresist (SU-8 100 from MicroChem) was applied. The substrate could be a silicon wafer, a glass plate, or a plastic plate. Smoothness of the wall surface depends strongly on the quality of photomask used. A high-resolution transparency containing the design of the features, created in a CAD program, can be used as the mask in photolithography [4,5]. Figure 2 compares two CD platforms made by UV-photolithography of SU-8. The one developed from a chrome-coated photomask shows much better feature shape and surface smoothness compared to that developed from a transparency photomask (3386 dpi). The latter, however, is much less expensive (a few



Fig. 1. A CNC-machined CD microfluidic platform.

dollars vs. several hundred dollars). For feature sizes larger than $20\text{ }\mu\text{m}$, the transparency photomask is a very low-cost way for initial design and testing of microfluidic devices.

Mold (Master) Making

In order to make CD platforms economically viable for a broad range of medical diagnostic applications, they must be low-cost and disposable. To do so, one needs to succeed in replicating microstructures successively, in a so-called infinite loop, without going back to CNC-machining or lithography to remake the primary structures. A mold insert (or master) has to be generated, that can be used over and over in the mass production process.

The mold inserts (or masters) can be fabricated by a variety of techniques [1,2]. For large features ($> 50\text{ }\mu\text{m}$) with tolerances and repeatability in the range of about $10\text{ }\mu\text{m}$, traditional CNC-machining of materials like tool steel and stainless steel are often accurate enough. The advantage of this technique is that materials used are the same as those in conventional polymer molding, so their design, strength, and service life are well established. Complicated 3-dimensional structures can also be easily machined. The main drawbacks are that it is difficult to make sharp corners or right angles, and the surface quality is usually poor. Diamond-based micromilling or microdrilling [6], and

excimer or femtosecond laser-based [7] direct removal processes can reduce the surface roughness to $1\text{ }\mu\text{m}$ or less [8]. While diamond-based methods can also make features smaller than $10\text{ }\mu\text{m}$, they are only applicable to “soft” metals such as nickel, aluminum, and copper. For smaller feature sizes (down to sub-micron), LIGA (German acronym for X-ray lithographie, galvanoformung, and abformtechnik) [1,2] and LIGA-like methods (i.e., surface machining) have to be employed [1]. In this approach, different mold inserts (plastics, glass, silicon, and metals) for plastic molding (liquid resin molding, injection molding, or hot embossing) can be created by surface and bulk micromachining as illustrated in Figure 3.

The procedure of making a photoresist mold by UV-photolithography is the same as in making the platform, except for the use of a negative photomask. A “mother-daughter” mold making approach can also be used to fabricate various plastic molds by casting. In this method, a “positive” mother mold is used to produce a “negative” polydimethyl siloxane (PDMS) mold. The mother mold can be manufactured using LIGA-like techniques such as photolithography, deep reactive ion etching (DRIE), etc. to pattern structures with high resolution and accuracy. The mother mold can be preserved and reused, which leads to faster turnaround time and cost-effectiveness. The “negative” PDMS mold is then used for casting a liquid resin (e.g., epoxy, and polyurethane) to produce a “positive” plastic mold (daughter mold). Figure 4 shows photos of a photoresist

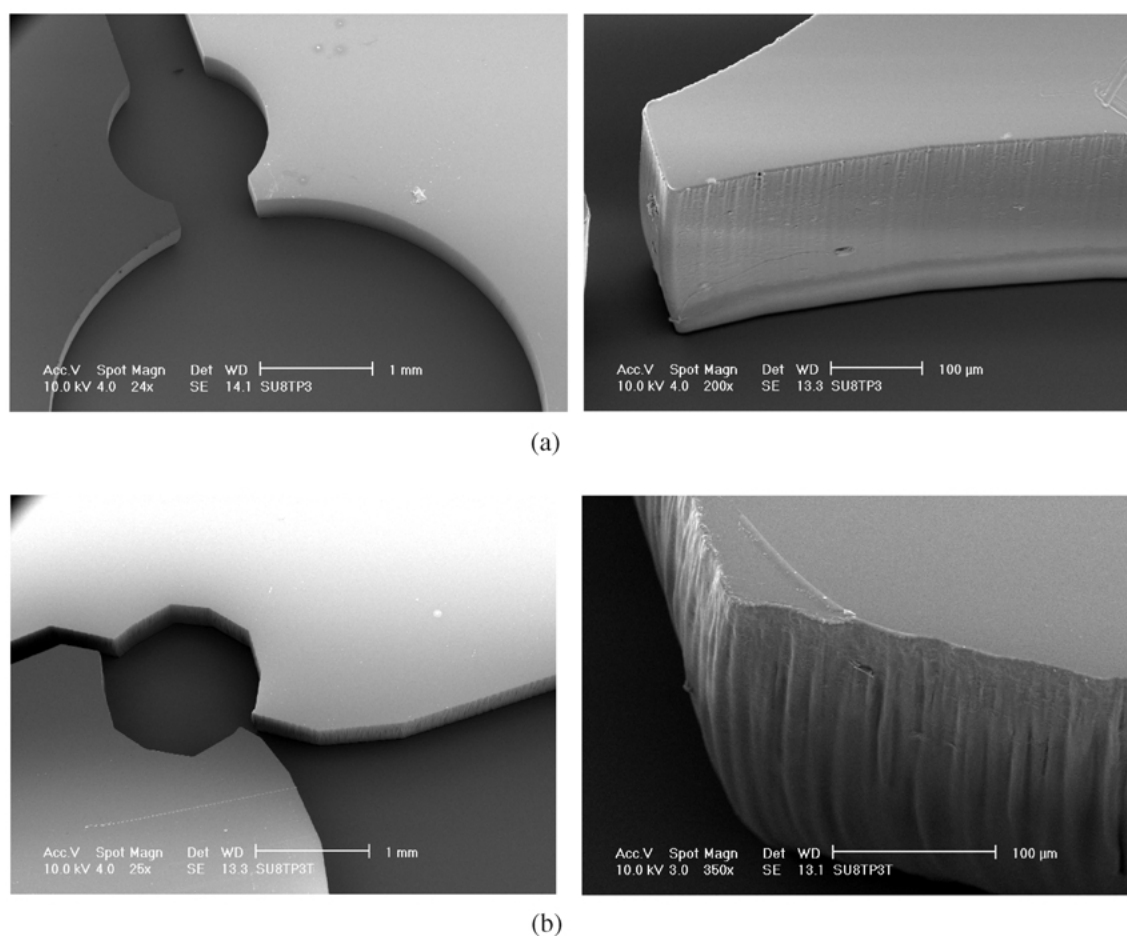


Fig. 2. SEM photos of SU-8 based structural features developed from (a) a chrome-coated photomask, (b) a transparency photomask.

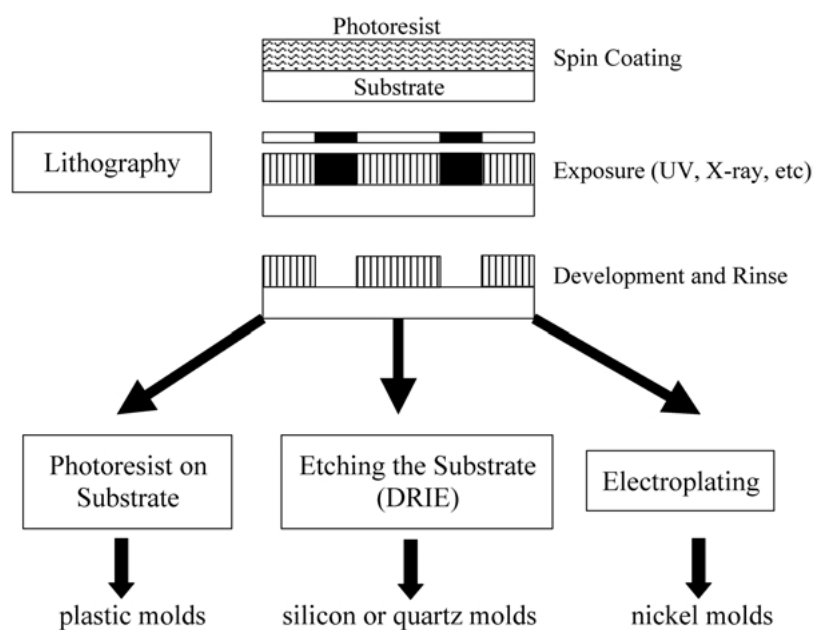


Fig. 3. Surface machining of mold inserts (master).

mold (SU-8) and an epoxy mold (3M, PR-500 epoxy resin). An exact replica of the mother mold with good surface quality can be made through this technique. Because of their low cost and simplicity, the plastic molds are suitable for small volume production in low-pressure molding processes, such as liquid resin casting. However, plastic molds are difficult to use in high-pressure molding processes, such as hot embossing or injection molding, due to their low thermal and mechanical strength and a tendency to adhere to the molded polymer.

For small quantity production where the lifetime of mold inserts is not crucial, a silicon wafer or quartz etched by DRIE can be utilized directly as a mold insert. In our study, a thin layer (10 μm) of a positive photoresist AZ 4620 (Clariant Inc.) was patterned on a silicon wafer as the DRIE masking. The DRIE process was carried out in a PlasmaTherm SLR-770 ICP Bosch Etcher for 140 minutes to produce features of 200 μm in depth. The patented Bosch process uses a series of alternating depositions and etches to maintain very low undercut and vertical sidewalls (anisotropic etch). The remaining photoresist was stripped off and the silicon wafer was ready for plastic molding. A silicon mold is often too brittle to endure very high molding pressure; therefore, one improvement is to anodically bond the silicon wafer to a thick glass support.

Metal is the ideal mold insert material for plastic molding due to its high mechanical and thermal strength. To make small feature sizes with a high aspect ratio, LIGA and LIGA-like processes can be used to produce mold inserts made of “soft” metals like nickel. The fabrication of a nickel mold insert consists of photolithography and electrodeposition. Issues such as surface roughness and sidewall draft angle of the mold insert need to be considered, in order to ensure that the molded plastic can be easily demolded after molding. It has been reported that a positive draft angle of greater than $1/4^\circ$ is essential for demolding in plastic injection molding [9]. In our study, we used a high resolution, chrome-coated photomask to achieve a low surface roughness. The draft angle of the mold insert depends strongly on the photolithography parameters. The formed photoresist mold may have overcut, vertical, and undercut (see Figure 5a) sidewalls after photolithography. According to Lorenz et al. [10], the change in sidewall profile is caused by the volume change of SU-8 during polymerization, chemical diffusion of crosslinking agents, and various illumination parameters (beam divergence, reflectivity of the substrate, UV-absorption in SU-8, reflections at the SU-8 surface). It has been shown that the exposure dose greatly affects the dimensional change during pattern transfer. Under-dose or over-dose may cause a negative

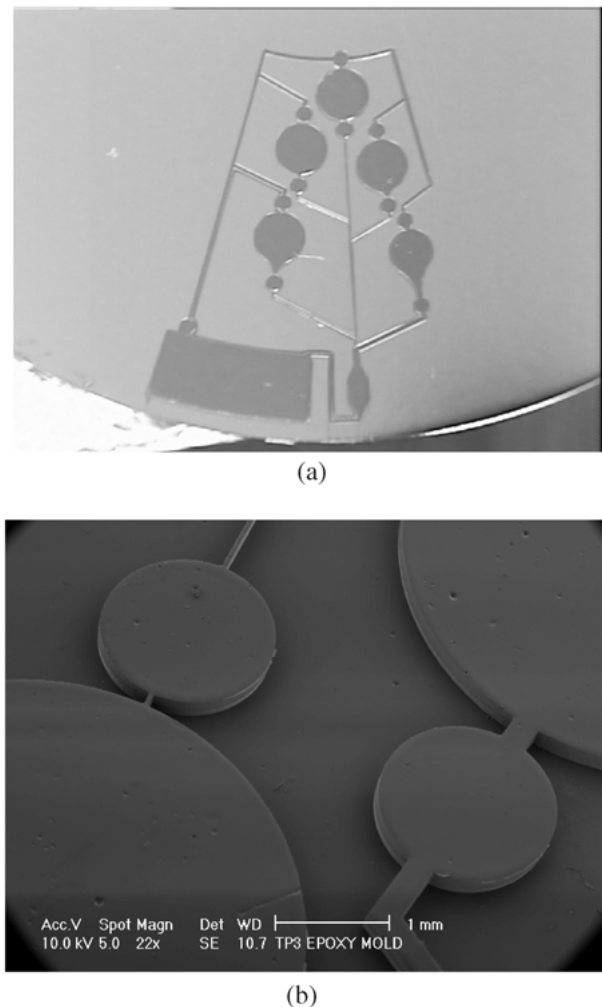


Fig. 4. (a) An SU-8 mold insert (380 μm deep) made by UV-photolithography and (b) SEM photo of an epoxy mold insert (200 μm deep).

or positive dimensional change in the transferred structures, respectively [11].

Two methods were used to fabricate the nickel mold inserts in our study. The first method used a silicon wafer or glass plate as a substrate [12,13], while the second method used a nickel plate as the substrate [14]. The schematic of the first method is shown in Figure 5b. A thin sacrificial layer of titanium (50 nm) and a seed layer of gold (100 nm) were thermally evaporated on a silicon wafer. This was done to improve electrical conductivity and surface adhesion between the photoresist and the substrate. These metals would be etched during lift-off. Photolithography was performed as described previously. The formed negative SU-8 mold was placed in a commercial nickel sulfamate solution (Technics, Inc.). Electroplating was conducted at 50°C with a pH of 3.5 to 4, and at a low current density of 1 to 3 A/dm^2 (Amps/

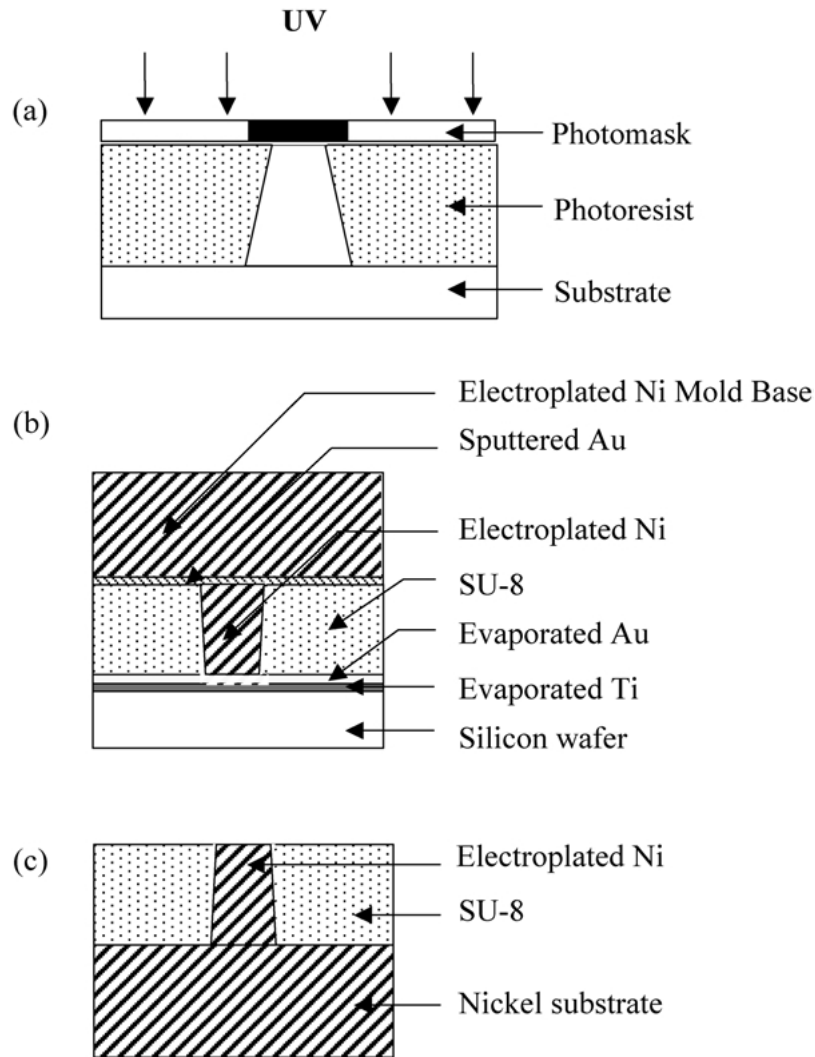


Fig. 5. (a) Photolithography showing an undercut sidewall profile, (b) Fabrication of nickel mold inserts by photolithography and electroplating using a silicon wafer as substrate and (c) a nickel plate as substrate.

decimeter²) in order to minimize internal stress in the nickel mold. The electrolyte was filtered regularly to prevent hydrogen pitting and impurity buildup. The electroplating itself was separated into two stages, where the first stage involved nickel-plating to fill the developed areas of the photoresist. Since the photoresist is non-conductive, a thin layer of gold was then sputtered on the top surface of the photoresist. This ensured that the plating would take place on top of the photoresist. The second stage was carried out to form the mold base. After electroplating, the nickel mold insert was released by etching the metal sacrificial layer in hydrofluoric acid and stripping the photoresist in hot NMP (1-methyl-2-pyrrolidion). The schematic for the second method is shown in Figure 5c. Photolithography was performed on a nickel plate instead of a silicon wafer. Electroplating

was conducted using the same conditions described above until nickel completely filled all of the micro-features. Any protruding nickel was polished and the photoresist was stripped off.

To produce a positive draft angle on the mold insert, the photoresist mold should have an overcut sidewall profile in the first method, and an undercut profile in the second method. Since the negative photoresist (SU-8) exhibited a predominantly undercutting profile in all our experiments (which is similar to results obtained by others [10,11]), therefore, the second method was chosen. This method also requires less processing steps. Figure 6a shows a nickel mold insert with one 2-point calibration structure, while Figure 6b shows a positive draft angle in an enlarged view (top view).

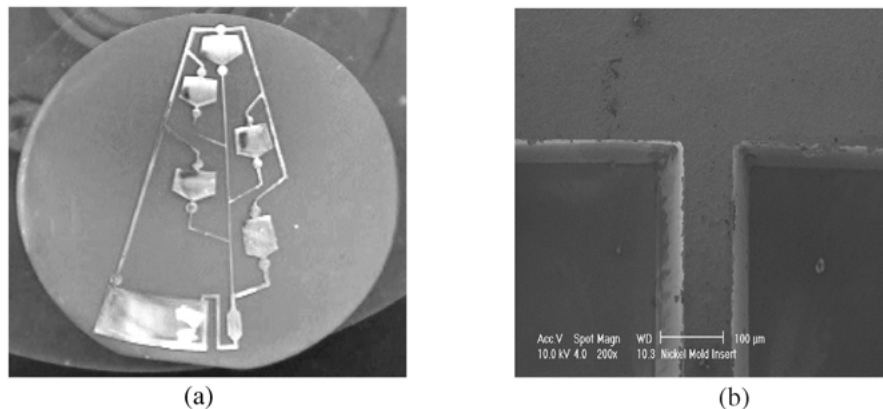


Fig. 6. (a) Photo of a nickel mold insert, (b) SEM photo showing positive draft angle (top view).

Replication Techniques

Liquid resin molding

Micromolding based on low viscosity liquid resins (instead of high viscosity polymer melts) is a very attractive approach, since mold inserts made by photolithography techniques are limited to soft metals (e.g., nickel), silicon, quartz, and plastics. During liquid resin molding, the low viscosity reactive polymer components are mixed shortly before injection into the mold cavity, and polymerization takes place during the molding process. Both reaction injection molding (RIM) and transfer molding, two techniques widely used in conventional processing of thermoset resins, are options for mass production [15]. For new design of microfluidic devices, casting is an attractive method for rapid prototyping. Whitesides and his group at Harvard University [4,5] combined a photolithography technique with PDMS molding for microfabrication. The PDMS resin was cast onto a photoresist mold produced by photolithography on a silicon wafer and cured at elevated temperatures. The polymer replica of the master containing a negative relief of features could be easily peeled away from the silicon wafer and either used as the microdevice directly [16,17], or as a master for micro-contact printing [16], micromolding in capillaries [5], or micro-transfer molding [18]. This method, called soft lithography, has also been used by other researchers [19,20] because of its simplicity. The long cycle time (several hours) and limitation to only PDMS rubber, however, make it difficult to use for mass production in most large scale BioMEMS applications.

Thin wall injection molding

Injection molding is based on heating a thermoplastic material until it melts, thermostating the parts of the mold, injecting the melt with a controlled injection

pressure into the mold cavity, and cooling the molded polymer. Injection molding is probably the most widely used technique in macroscopic production of polymer parts.

Injection molding of parts with small features and low-aspect ratios (like CDs) has been widely applied. Currently, the main challenge is to extend this technique to the fabrication of components with a smaller feature size but larger aspect ratio, needed in many medical and bio-chemical applications. In recent years, some research work has been initiated in Europe. Ehrfeld and his co-workers at the IMM (Institut für Mikrotechnik in Mainz), Germany [21,22], used precision injection molding machines, similar to those commonly used for the fabrication of CDs, to mold MEMS-components based on mold inserts made by LIGA. Another group at the Institut für Materialforschung in Karlsruhe, Germany [23–27], used CNC-machined and laser ablated metal molds in microinjection molding. Wimberger-Friedl in the Netherlands [28] fabricated sub-μm grating optical elements by injection molding. The mold inserts were made by E-beam lithography together with nickel electroplating, and by RIE in SiO₂ (fused quartz). In the United States, Edwards et al. [29] used SU-8 molds and Kelly [14] used LIGA-produced nickel molds for injection molding to make devices such as micro heat exchangers. In general, these studies showed that the molds need to fill rapidly in order to prevent early freezing. A mold temperature above the “no-flow” temperature can guarantee a complete filling. Shape deviation and damage of the fragile mold walls occur quite easily, possibly due to shrinkage of the polymer or defective filling and release. Since the mold cavity is filled at a mold temperature that exceeds the melting point or the glass transition temperature T_g of the polymer, the mold needs to be cooled down to obtain a sufficient strength before part ejection. In addition, conventional venting of the cavity is not feasible due to

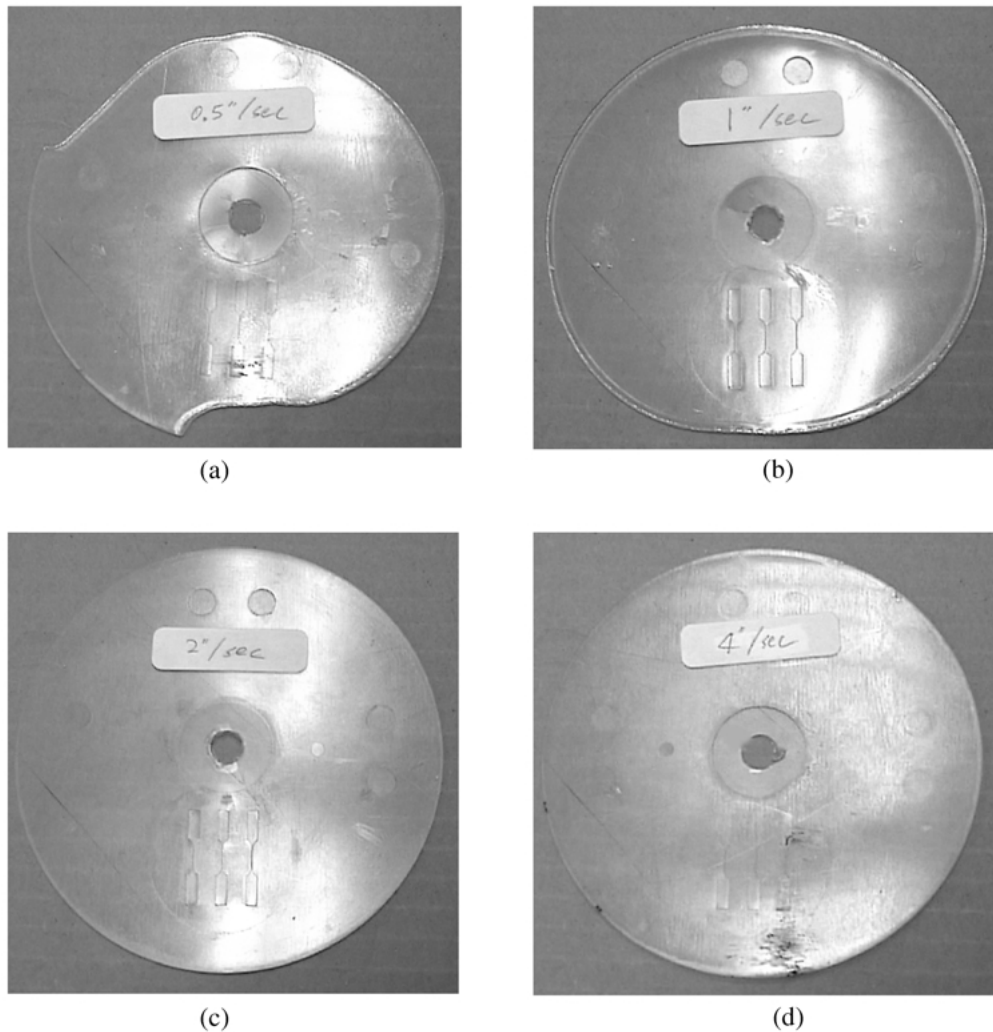


Fig. 7. CD platforms made by injection molding at different injection speeds (a) 12.7 mm/sec., (b) 25 mm/sec., (c) 51 mm/sec., and (d) 102 mm/sec.

the presence of microfeatures in the mold inserts. Therefore, prior evacuation of the mold cavity is needed. As a result, the cycle time is five minutes or longer, including the time needed for evacuation, heating and cooling of the mold. Molding of microfeatures with large aspect ratios or the use of materials with a higher viscosity leads to even longer cycle times.

Thin wall injection molding is a high-speed injection molding process [15,30]. Speeds can be ten times faster than in conventional injection molding, reducing the thickness of the polymer skin on the mold. The polymer melt can quickly be injected into the mold to fill all the detail structure of the cavity before solidification occurs. It has a great potential for mass production in microfabrication, because the mold temperature does not need to be raised to a high level during molding.

In this study, the injection molding experiments were conducted on a Sumitomo 200 ton high-pressure and high-speed machine. An optical quality polycarbonate

(OQPC, Lexan from GE plastic) and a polymethyl methacrylate (PMMA, PL 25 from Plaskolite) were selected as the molding materials, which are common polymer substrates in BioMEMS applications. The glass transition temperatures of PMMA and OQPC are approximately 100°C and 135°C, respectively. The melt temperature was controlled at 290°C for OQPC and at 232°C for PMMA. The mold temperature was held at 30°C and 80°C. The holding pressure was 2.1 to 6.9 MPa (300 to 1000 psi) and the holding time was 5 to 10 seconds. A mold release agent (55-NC, Dexter Polymer Systems) was used for easy demolding.

Figure 7 shows the injection molding results on a CD platform (about 1 mm thick). The quality of replication becomes accurate only at injection speeds of 50 mm per second or higher. A birefringence technique was used to qualitatively examine the stress in the polymer parts (Figure 8). Samples showing a sharp color contrast have large molded-in stresses. At lower flow rates the residual

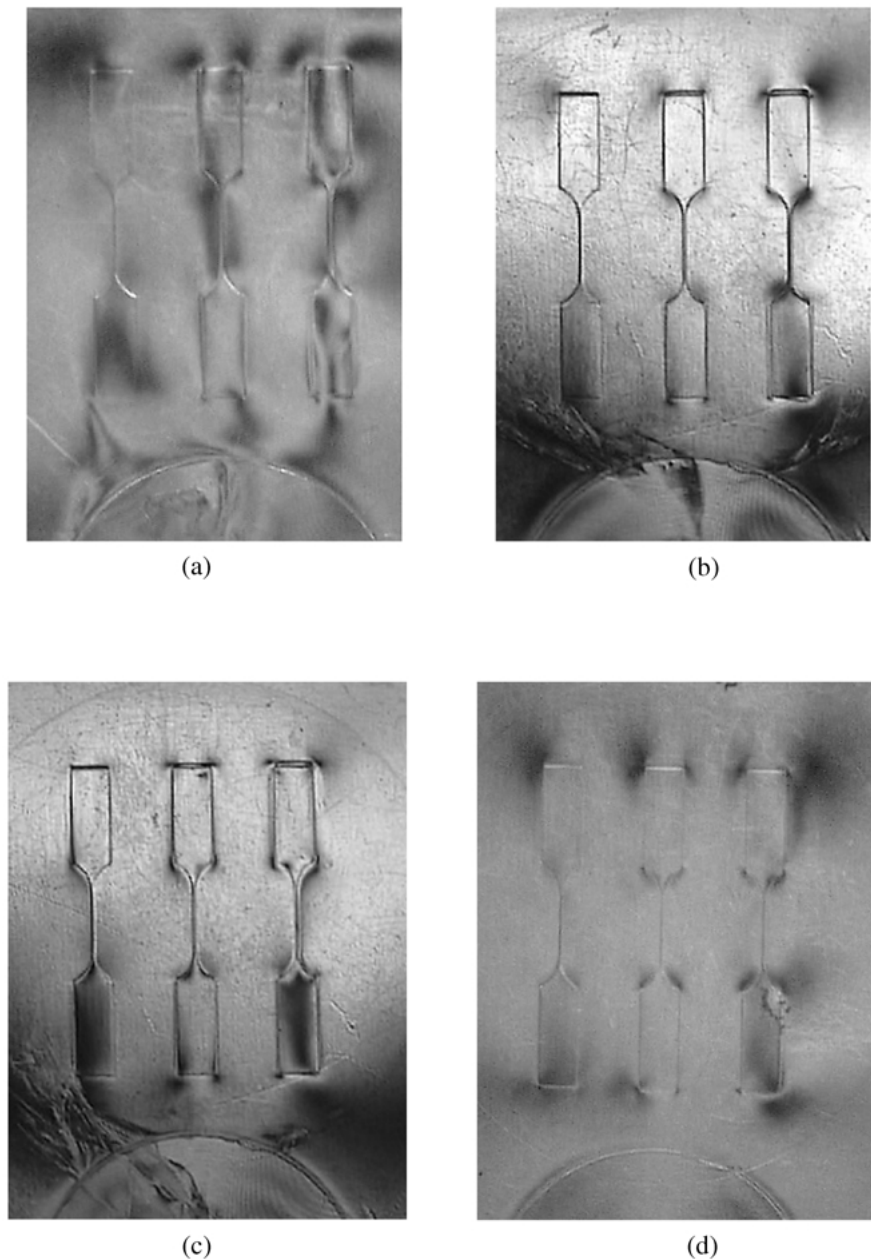


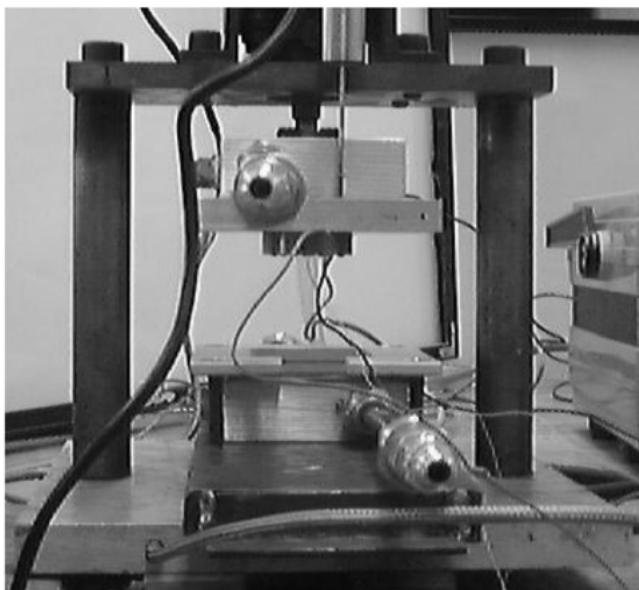
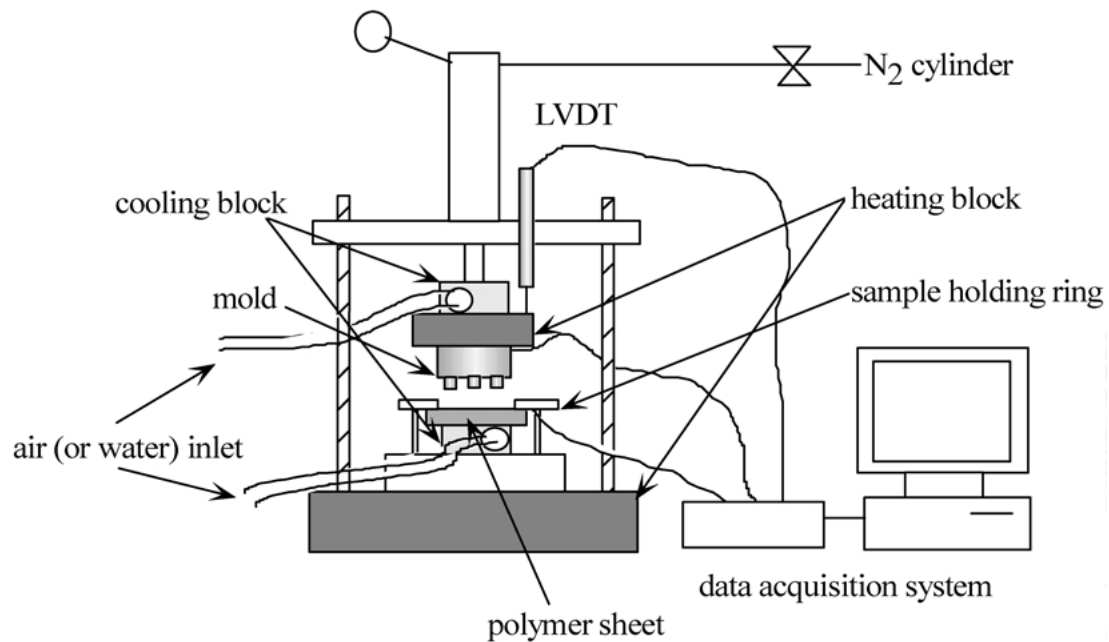
Fig. 8. Birefringence of CD platforms made by injection molding at different injection speeds (a) 12.7 mm/sec., (b) 25 mm/sec., (c) 51 mm/sec., and (d) 102 mm/sec.

stress is higher, because during the flow the polymer has been stretched and solidified, inducing significant flow-induced stresses. Increasing the flow-rate decreases the amount of residual stress, because the polymer can quickly fill in the mold and relax before solidification occurs. Nevertheless, some stresses remain that may cause warpage or a lowered chemical resistance.

Hot embossing

Hot embossing (or relief imprinting) [31,32] provides several advantages compared to injection molding, such

as relatively low costs for embossing tools, a simple process, and a high replication accuracy for small features. The basic principle of embossing is that the polymer substrate is first heated above its glass transition temperature, T_g (or softening temperature). A mold (or master) is then pressed against the substrate, fully transferring the pattern onto it (embossing). After a certain time of contact between the mold and the substrate, the system is cooled down below T_g (or softening temperature), followed by separating the mold and the substrate (de-embossing). Replication of micro-



Steel Mold Insert



Fig. 9. Schematic and photo of hot embossing setup.

and nano-size structures has been successfully achieved [32–38]. Adding an anti-adhesive film to reduce the interaction between the mold and the replica during embossing has also been studied [39,40]. Instead of the conventional nickel molds, the possibility of using silicon molds has been demonstrated due to their excellent surface quality and easy mold release [41,42]. Also, the use of a plastic mold in the embossing process was recently illustrated [43]. This can be achieved in either a cyclic or continuous process [15]. In a cyclic process, a metal master is placed in a hydraulic press. A

heated polymer sheet is stamped by applying the appropriate force, thus replicating the structure from the master to the polymer. This constitutes a low-cost method for making prototypes. For mass production, a continuous process is preferred. A polymer sheet stretches through a temperature chamber and several masters, mounted on a conveyor belt to continuously produce parts. The process also may incorporate a lamination station to enclose certain features.

Processing parameters include thermal cycle, compression force and compression speed. The temperature

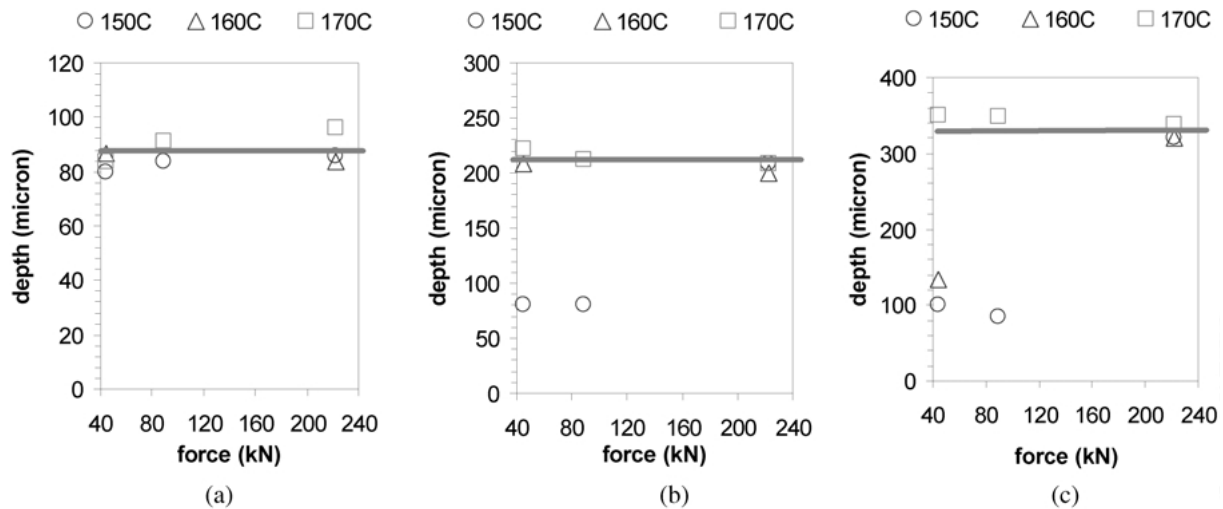


Fig. 10. Depths of replicated channels in PC vs. Applied force (5 minutes compression time at mold temperature and 2 hours cooling) (a) small channel (depth/width = 92/57 μm), (b) medium channel (depth/width = 220/120 μm), and (c) large channel (depth/width = 340/170 μm).

difference between embossing and de-embossing determines the thermal cycle time, typically from 25°C to 40°C. In principle one could, after hot embossing, cool down the whole device to room temperature before de-embossing or, at the other extreme, one could de-emboss just below or at the glass-transition temperature. A compromise is needed: the quality of the replication may not be good if one tries to remove the master when the polymer is still soft, while cooling all the way down to room-temperature takes too long. A narrower small temperature cycle leads to smaller induced thermal stresses. Such a narrower temperature cycle also reduces replication errors due to different thermal expansion coefficients of the tool and substrate. By actively heating and cooling the upper and lower bosses, a cycle time of about 5 minutes is achieved.

In our study, we used a cyclic process in a 0.3 MPa (50 psi) pneumatic press to fabricate a compact disc-based fluidic platform (see Figure 9). The materials used are OQPC (GE Plastics, Lexan, $T_g = 135^\circ\text{C}$) and regular PC (GE Plastics, Lexan, $T_g = 145^\circ\text{C}$). The variables explored involve a compression force (from 17.8 kN to 222 kN (2 to 25 tons)) and embossing and de-embossing temperatures. Figure 10 plots the depths of replicated channels in PC vs. the applied force. The smallest channels can be replicated at a relatively low force and temperature. For the deeper channels one has to use a higher temperature and a greater force. This means a longer cycle time and a larger residual stress. That is why embossing is good for small structures and low aspect ratios and difficult for large structures and features. For 3-dimensional structures (e.g., channels with different depths), the embossing pressure is often very high. Correspondingly, the molded-in stresses are also very large. Figure 11 shows the displacement profile during

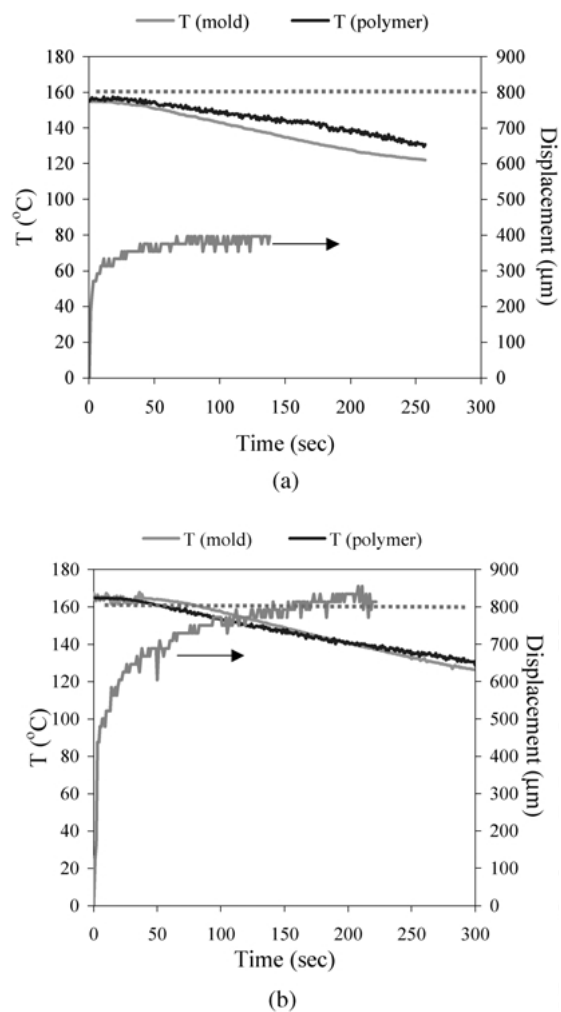


Fig. 11. Typical displacement and temperature profiles during embossing (single-depth mold, PC, 0.16 MPa); embossing temperature at (a) 155°C and (b) 165°C.

embossing of PC. At 155°C embossing temperature and under 0.16 MPa (23 psi) compression pressure, the final displacement was around 400 μm , which is about half the depth of the mold insert. However, as the embossing temperature increased to 165°C, the final displacement reached 800 μm and the feature was completely transferred to the polymer substrate. The birefringence patterns under different processing conditions were also investigated. A strong birefringence pattern is observed at lower embossing temperatures and higher compression pressures, indicating larger molded-in stresses. In contrast, a higher embossing temperature and a lower compression pressure provide lower molded-in stresses. Note that although less molded-in stresses are obtained by increasing the embossing temperature and reducing the compression pressure, the cycle time becomes longer.

By comparison, there is little flow in casting, so the molded part is relatively stress-free.

Plastic bonding

A major concern of polymer-based microfluidic devices is how to bond parts together. Bonding together of plastic microparts or applying plastic lids to plastic platforms can be achieved using adhesives or tapes, plastic welding (e.g., hot-plate and ultrasonic welding) and selected organic solvents (i.e., partially dissolving the bonding surfaces and evaporate the solvent [44]). In our study, several plastic bonding techniques were tried, including vacuum-assisted heat bonding, bonding with double-sided adhesive tape, and water-soluble polymer-assisted bonding. Figure 12 shows cross-sections of bonded microchannels by these three methods. For large features

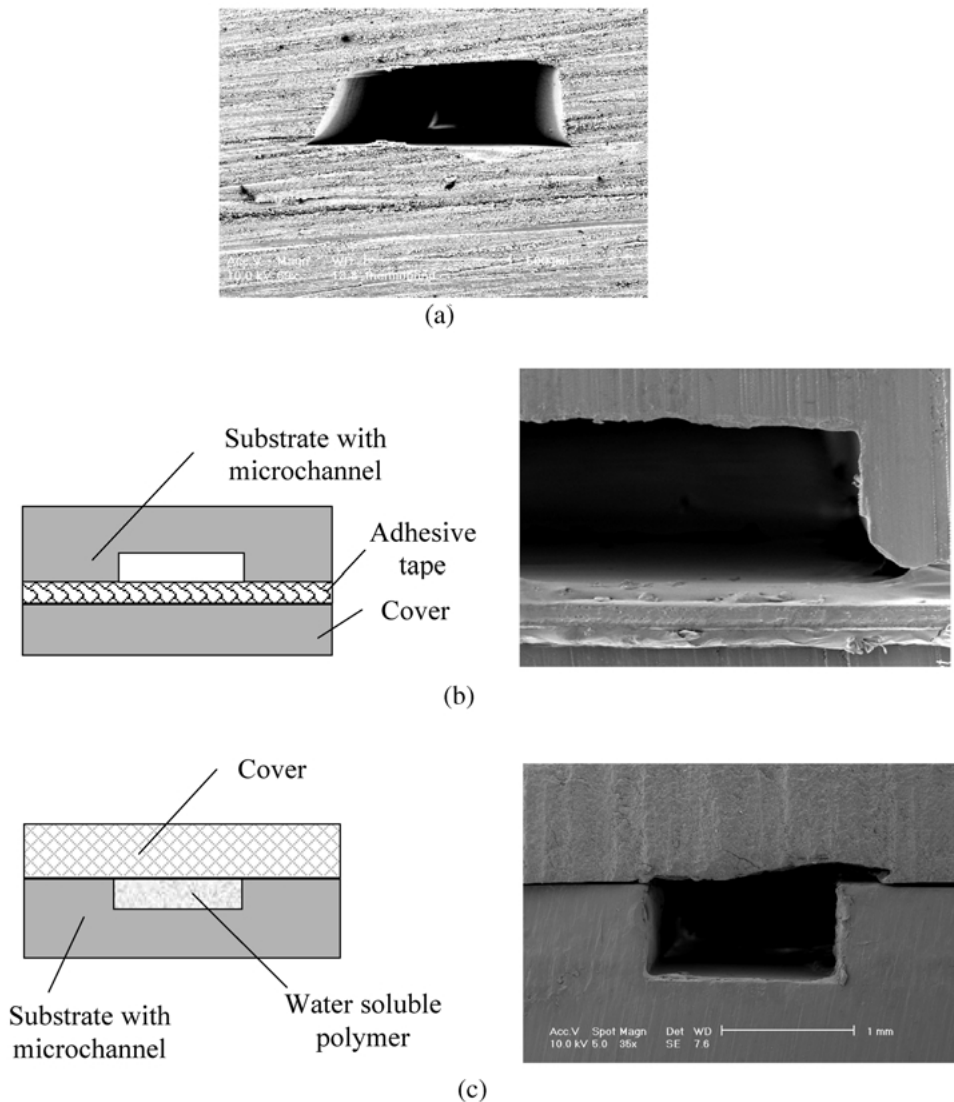


Fig. 12. SEM photos of bonded microchannels (a) vacuum-assisted thermal bonding, (b) adhesive tape bonding, and (c) water-soluble polymer-assisted bonding.

(several hundred micrometers to millimeters), conventional plastic welding techniques such as hot-plate, ultrasonic, and IR welding can be used. Applying a vacuum before welding may reduce voids at the welded interface; consequently, lower pressure and temperature can be used in welding. This will minimize the molten polymer flowing into the bonded channels. Ideally, small overflow traps should be designed along the edge of the channels to prevent them from being completely blocked by the molten polymer during welding. For smaller channel sizes (less than 100 μm), the design and fabrication of overflow traps become difficult. Double-sided adhesive tape can be a useful way for microbonding as shown in Figure 12b. The main disadvantage is that the presence of an adhesive tape tends to change the channel size and channel wall properties. For very small channels (say, less than 1 μm), pre-filling the channels with a water soluble polymer (serving as a sacrificial material), drying the polymer, sealing the plate with channels to a cover lid by either welding or adhesive bonding, and then washing out the water soluble polymer is a viable way to prevent channel blockage during bonding (see Figure 12c). This method, however, is more tedious and may not be applicable in some applications. The development of low-cost, high speed, and reliable bonding techniques for microfluidic devices remains an important and challenging issue in BioMEMS fabrication.

Conclusion

A number of prototyping and mass production techniques for mold making and polymer fabrication are presented. They can be used to produce CD microfluidic platforms for BioMEMS applications. The effects of mold inserts, molding methods, and feature size on replication accuracy and “molded-in” stresses are briefly discussed. Table 1 compares the three molding methods. Proper bonding without affecting the shape and size of micro-sized channels is still an unsolved issue in fabricating polymer based microfluidic devices.

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Table 1. Comparison of micro-molding methods

	Liquid resin molding	Injection molding	Hot embossing
Mold inserts	Any molds	Metal molds (Silicon molds suitable for prototyping)	Metal and silicon molds (Plastic molds suitable for prototyping)
Feature size	No limit	<ul style="list-style-type: none"> • Good for small features with low aspect ratio, or large features with high aspect ratio • Good for 3-dimensional features 	<ul style="list-style-type: none"> • Good for small features • Difficult for high aspect ratios • Difficult for multiple depth • Planar features only
Material	Liquid resins (thermosets provide high chemical resistance)	Mainly low molecular weight thermoplastics	Low and high molecular weight thermoplastics
Processing	<ul style="list-style-type: none"> • Simple (except for RIM) • Easy mold filling • Closed mold process • Long cycle time (hr) • Mold release problem for some resins 	<ul style="list-style-type: none"> • Short cycle time (sec ~ min.) • Closed mold process • High automation 	<ul style="list-style-type: none"> • Simple • Medium cycle time (min) • Potential for continuous production • Open mold process
Replication accuracy	<ul style="list-style-type: none"> • Less dimensional control (polymerization shrinkage) 	<ul style="list-style-type: none"> • Excellent dimensional control 	<ul style="list-style-type: none"> • Less dimensional control
Part quality	<ul style="list-style-type: none"> • Low molded-in stresses • Contamination (resin residue) 	<ul style="list-style-type: none"> • High stress on mold insert • High molded-in stresses 	<ul style="list-style-type: none"> • High molded-in stresses
Cost	<ul style="list-style-type: none"> • Low tooling cost (except for RIM) • For prototyping and low volume production 	<ul style="list-style-type: none"> • High tooling cost • For large volume production 	<ul style="list-style-type: none"> • Low tooling cost • For low and medium volume production

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