

Optimization of an optical disk manufacturing process for polymer microfluidic substrates by using the design of experiment methodology

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1 Introduction

Recently, polymer-based technologies have been widely used to fabricate biomedical devices that have lower costs, are more compatible to biocharacteristics, have more flexible process flows, or increased capability for mass production.^{1–4} For instance, polydimethylsiloxane (PDMS) is used for speeding up implementation of the prototypes for microfluidic systems.^{5,6} However, the cycle time for manufacturing a microfluidic substrate is too long for mass production. Moreover, a microfluidic structure on a dummy polymethylmethacrylate (PMMA) sheet is generated by a CO₂-laser machine⁷ without any photolithography. Unfortunately, the application of this method is only limited to the prototype due to possible damage that results from surfaces of the substrate of the microfluidic channel. Also, the long cycle time is a drawback for mass production. A lithography electroforming micromolding (LIGA) technique⁸ is conducted to produce a microfluidic structure on high-density polyethylene (HDPE) substrates with an aspect ra-

Abstract. We present an improved method for manufacturing microfluidic structures on a polymer-based substrate, and the design of experiment (DOE) is used to extract the optimum injection parameters. The long cycle time of the injection molding causes high costs in manufacturing, and this prevents conventional techniques from being widely used for mass production. Therefore, this study adopts a new optical disk process to reduce the cycle time. The cycle time of the new method can be reduced by more than ten-fold compared with that of traditional ones. Also, this new method can prevent damage on the mirror plate of the mold. The mold system is composed of a mold insert (stamper) holder and a vacuum system to join the mold insert with the mold. In this way, the time needed to change the stamper is drastically decreased. Our proposed method has the ability to reduce the time required to insert the mold from several hours to a few minutes, to prevent damage on the mirror plate of the mold, and to decrease the cycle time of molding from several minutes to 4 sec. The DOE is applied to study the effects of molding parameters on replication rate of depth, width deviation, birefringence, tilt and surface roughness of the microfluidic substrates. The experimental results show that the proposed method is suitable for mass production. © 2010 Society of Photo-Optical Instrumentation Engineers. [DOI: 10.1117/1.3491363]

Subject terms: injection molding; lithography electroforming micromolding; mold insert; microfluidic; optical disk.

Paper 09136PSSRR received Sep. 10, 2009; revised manuscript received Aug. 19, 2010; accepted for publication Aug. 19, 2010; published online Sep. 30, 2010.

tio of 10 by means of the injection molding method, which is expensive and requires a long cycle time as well. On the other hand, a LIGA-like technique is proposed to produce PMMA/polycarbonate (PC)/cyclic-olefin copolymer (COC) substrates by using hot embossing^{9–14} or the injection molding^{15–20} method. Although the LIGA-like technique can reduce the cost through changing the exposure light source from x-ray to ultraviolet (UV) light or e-beam, the method not only requires a long cycle time but also needs to take the molds off the injection machine to change the mold inserts. Due to the fact that the requirements of cost and cycle time has not been met in previous work, this work proposes a new method to manufacture microfluidic structures on polymer-base substrates. The major advantage is the low cost achieved by adopting the optical disk process. For example, by being made from PC or PMMA, the manufacturing cost of a compact disk (CD) or digital versatile disk (DVD) that contains microscale features will be less than \$0.25 USD.²¹ And unlike other conventional methods that take hours to change the mold inserts, this research is focused on saving time.

In addition, when the conventional optical disk technique is used for fabricating the microfluidic substrate, the mold insert induces an opposite electroform pattern on the backside, which may further induce damage on the mold. Therefore, the mold insert must be polished before being integrated with the mold. However, this backside polishing must be properly controlled to prevent overstress, or the overstress will warp the mold insert and cause loss of vacuum.

Therefore this work uses a nickel (Ni) plate to prevent the generation of backside patterns on the mold insert so that it will not damage the mirror plate of the mold. In addition, the proposed molding system not only includes a new spiral cooling system to reduce the cycle time, but also uses a mold insert holder and a vacuum system to integrate the mold insert with the mold. As a result, the time to change the mold insert is obviously reduced. Also, the design of experiment (DOE) method and parametric analysis are applied to investigate the effects of molding parameters on the microfluidic substrate quality. The molding parameters include inner/outer deviation, birefringence, tilt, surface roughness and cylinder temperature, mold temperature, clamping force, and injection speed. The interaction between those molding parameters is demonstrated in the following section. All in all, the proposed method has low cost, is highly efficient, and suitable for mass production to fabricate microfluidic structures on polymer-based substrates.

This work is organized as follows. Section 2 provides a description of the molding system. The new optical disk process is introduced in Sec. 3. We demonstrate our experiments in Sec. 4, and finally discuss the experimental results and draw our conclusions in Sec. 5.

2 Mold System

In this study, the Seikoh Giken F-type optical disk mold is adopted, as shown in Fig. 1. The molds are commonly used in the optical disk field. So far, these optical disk molds have sold more than 2000 sets.²² There are two steps to integrate the mold insert with the mold. First, the mold insert holder is installed in the mold [Fig. 1(a)]. Second, the vacuum system of the mold is used to keep the mold insert in place [Fig. 1(b)]. In general, to prevent an unwanted pattern of the mirror plate occurring in the substrate, the mirror plate of the mold should be coated with diamond-like carbon (DLC). The backside pattern of the mold insert has to be polished before injection due to the hard and fragile characteristics of DLC, which require no sharp pattern on the backside. However, when polished, the mold insert can warp due to possible overstressing, and further cause the mold insert to be unable to create a proper vacuum. This work proposes a new optical disk process to deal with all of these problems. Also, in order to reduce the cycle time, some spiral cooling water routes have been attached to the back of mirror plate. With the cooling water routes, the cooling effect of the polymer in the mold cavity will be greatly improved, and thus will shorten the injection molding cycle time. In this study a spiral cooling system with 5 turns was adopted, as shown in Fig. 1(c).

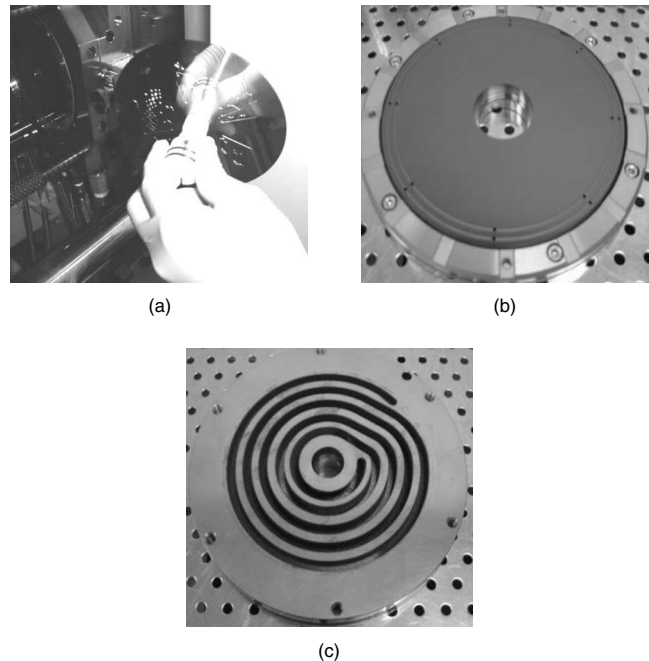


Fig. 1 Photographic images of (a) microfluidic mold insert hung on the fixture, (b) mirror plate of the mold, and (c) the spiral cooling system behind the mirror plate.

3 New Optical Disk Process

In Fig. 2, the new optical disk process is compared with the conventional optical disk process.²³ A Ni plate is used in the proposed process to prevent a backside pattern from being formed on the mold insert,^{4,13,14,16} so that the mold insert processed by the proposed method will not damage the mirror plate of the mold. The details of the conventional optical disk process are as follows.^{21,24} First, one piece of glass is coated with a layer of photoresist (PR) 20 to 250 nm thick. The PR on the surface of the glass is exposed by a gas laser of UV wavelength. When it is adopted by a one-time speed of exposure, it takes about 80 min, but when it is run by four times the speed, it will take 20 min. The power of exposure is 78 mJ/cm². Then, the PR layer is developed by the 2.38% tetra-methyl-ammonium hydroxide (TMAH), followed by sputtering of the seed layer, which will be later electroformed to a thickness of 300 μm , onto the PR. After being polished and punched, the mold insert is integrated with the mold insert holder and the mold. With the appropriate injection parameter adjusted, the plastic optical disk substrates can be fabricated as shown in Fig 2(a).

Figure 2(b) shows the new process flow. First, a Ni plate 8-in. in diameter and 300 μm thick is electroformed and then coated with PR. Next, the PR on the Ni is exposed and developed. In this work, the transparent photomask and emulsion glass mask are used for investigating the influence of performance. Finally, the Ni plate is electroformed again, and the mold insert, without any backside pattern, is produced.

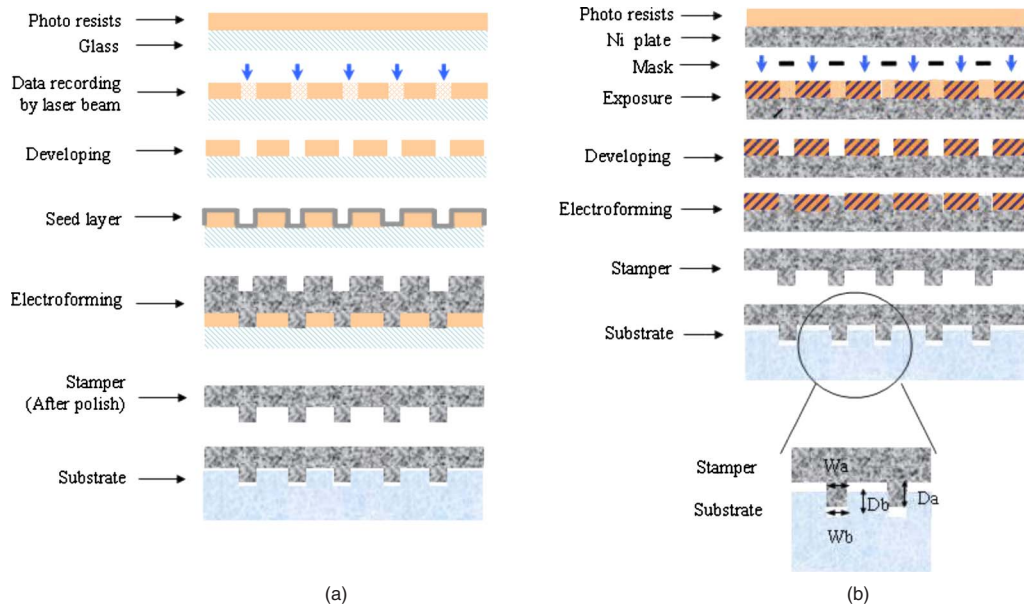


Fig. 2 Process of (a) the optical disk substrates and (b) the microfluidic substrates.

4 Experiments

4.1 Mold Insert

The mask is made by using a computer-aided design (CAD) file of the microfluidic pattern. The design of microchannel width is shown in Fig. 3(a). The width of location (inner) of the microchannel is $55\ \mu\text{m}$, and the width of location (outer) of the microchannel is $200\ \mu\text{m}$. In this research, a $300\text{-}\mu\text{m}$ -thick Ni plate is coated with SU-8 2100 negative PR by means of a spin coater. For soft baking, the Ni plate is first placed on a leveled hot plate at $80\ ^\circ\text{C}$ for 10 min and then at $100\ ^\circ\text{C}$ for 50 min. After soft baking, the PR is

cooled down gradually to room temperature prior to exposure, the power of which is $650\ \text{mJ}/\text{cm}^2$. The post exposure bake (PEB) is carried out at $65\ ^\circ\text{C}$ for 30 min and then at $80\ ^\circ\text{C}$ for 30 min. The PR cools down and is then developed in propylene glycol methyl ether acetate (PGMEA). The SU-8 mold is then placed in a 5 to 10% hydrochloric acid (HCl) solution for 5 min to remove the oxidative layer of Ni plate from the SU-8 mold. Then the SU-8 mold is rinsed in deionized (DI) water and quickly placed in a commercial boric-acid-based nickel sulfamate solution (Nickel Sulfamate 13409, Macdermid, Denver, Colorado). Boric

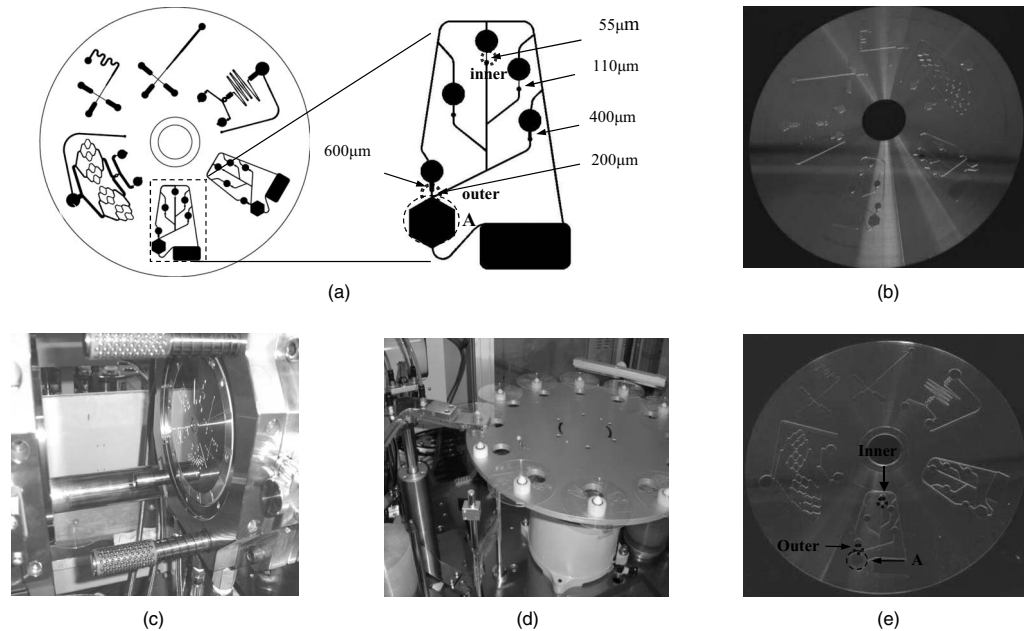


Fig. 3 (a) Diagram of microfluidic design, (b) microfluidic mold insert, (c) mold insert hung on the mold, (d) cooling and stacking after injection of the microfluidic substrates, and (e) microfluidic plastic substrate.

Table 1 Two levels of effective factors for injection molding of microfluidic substrates.

Factor	Level	
	1	2
Cylinder temperature, A (°C)	350	380
Mold temperature, B (°C)	118	128
Second clamping force, C (KN)	280	350
Injection speed, D (mm/sec)	120	150

acid acts as buffer to prevent the pH rising in the interfacial layer between the plated part and the electrolyte solution during the plating reaction. The buffering effect of the boric acid prevents the local formation of nickel oxides/hydroxides, especially in the microcavities of the structure. A wetting agent is added to the electrolyte and is used to counteract pitting.²⁴ Electroforming is conducted at $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ with a pH of 4 ± 0.2 , and at a low current density of 0.2 to 1 ASD (amp/decimeter²) minimize the internal stress and obtain a more uniform thickness.^{25,26} After being electroformed, polished, and punched, the mold insert (stamper) is completed.

4.2 Microfluidic Substrate

The injection molding experiments for fabricating the microfluidic substrate are conducted on a high-speed injection machine [Sumitomo (Tokyo, Japan) SD-35E], and the mold is a Seikoh Giken (Chiba, Japan) F type. The injection material in this experiment is optical-grade PC (Makrolon CD2005, Bayer Corporation, Germany). The glass transition temperature of PC is approximately $145\text{ }^{\circ}\text{C}$.²⁷ The clamping mode adopts the injection compression molding method. In addition, the initial mold open distance (platen position) is 0.55 mm. The mold will be fully closed within 0.1 sec after the injection, and then the mold clamping force is established. The third, fourth, and fifth mold clamping forces are set at 85, 65, and 70 kN, respectively. The

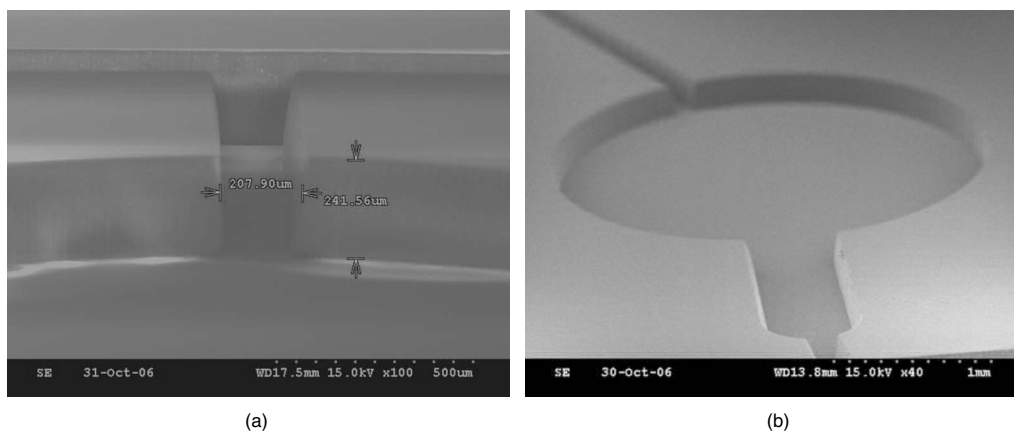
holding pressures of the injection are 180 and 200 bar. Furthermore, the holding times are both 0.1 sec under the two different holding pressures. The thickness of the substrate is 0.6 mm. Figure 3 shows the flow chart for making the microfluidic substrates. In this study, the DOE method is used to observe the interaction and main effects of molding factors on replication rate of depth, width deviation, birefringence, vertical run-out, and surface roughness. Cylinder temperature, mold temperature, clamping force, and injection speed are selected factors for injection molding. Each factor is designed with two levels, as shown in Table 1.

5 Results and Discussion

Figure 4 shows the results of the SEM image when using this method to make microfluidic substrates. It is obvious that the microfluidic structure is exposed by emulsion glass mask, as shown in Figs. 4(a) and 4(b).

Birefringence is formed due to the molecular orientation, the internal stress in plastic substrates, and the profile deformation resulting from the differences in cooling speeds of polymer solidification inside the mold.²⁸⁻³⁰ Figure 5 shows the birefringence patterns of different plastic microfluidic substrates measured by polarize plate and evaluation equipment. As a result, test samples 1 and 16 in DOE are similar under polarized plate measurement, as shown in Figs. 5(a) and 5(b). However, there is an obvious difference between test sample 1 and 16 measured by optical and mechanical evaluation equipment (PRO meteus MT 200 Blue, Dr. Schenk GmbH, Germany) as shown in Figs. 5(c) and 5(d).³¹ Accordingly, the birefringence distribution indicates the residual stress when forming the microfluidic structure. The value of the birefringence of test sample 1 is larger than that of test sample 16, because the setting condition of injection parameters of test 1 is at a lower cylinder temperature, a lower mold temperature, and a lower injection speed compared to that of test 16. The lower the flow rates are, the higher the residual stress is, because the flow of the polymer is stretched and solidified, which results in flow-induced stress during polymer flowing on the microfluidic structure of the mold insert.

The experimental results of injection molding by using

**Fig. 4** SEM images of microfluidic substrate fabricated by emulsion glass mask (a) channel and (b) valve and reservoir.

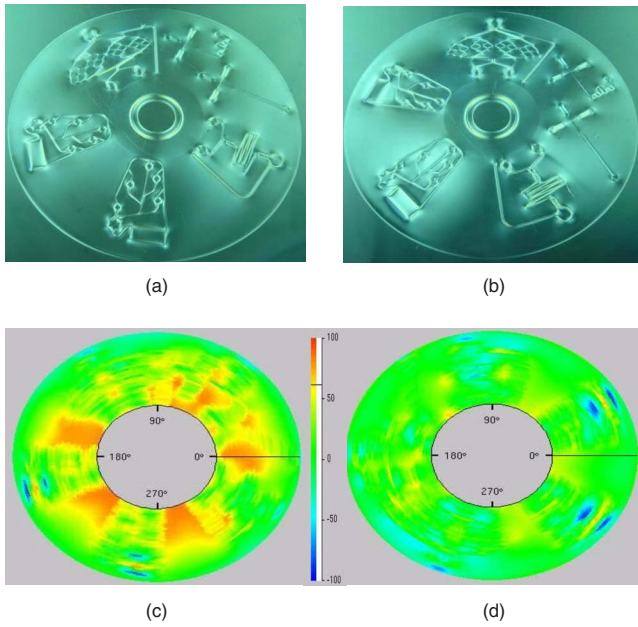


Fig. 5 Birefringence pattern of plastic microfluidic substrates of (a) pattern of test 1, (b) pattern of test 16 by polarization, (c) distribution of test 1, and (d) distribution of test 16.

the DOE method are shown in Table 2, where the parameter of replication rate of depth and width deviation are derived by

$$\text{replication rate of depth} = Db/Da \times 100\%, \tag{1}$$

where Da is the height of the microfluidic structure of the stamper, and Db is the depth of the microfluidic structure of the substrate, as shown in Fig. 2(b).

$$\text{width deviation} = (Wb - Wa)/Wa \times 100\%, \tag{2}$$

where Wa is the width of the microfluidic structure of the stamper, and Wb is the width of the microfluidic structure of the substrate, as shown in Fig. 2(b).

The depths and widths of the microfluidic structure in the substrates and the heights and widths of the microfluidic structure of the stamper are measured by a white light interferometer (Fogale Nanotech, France). The locations (inner) and (outer) of the substrate are shown in Fig. 3(e). The height and width of locations (inner) of the stamper are 204.5 and 57.5 μm . For location (outer), the height and width are 203.4 and 208.5 μm , respectively. That is, the aspect ratios are 3.56 for location (inner) and 0.98 for location (outer). The replication rate of depth reaches more

Table 2 Results of replication rate and effects of depth, width deviation, birefringence, vertical run-out, and surface roughness of microfluidic substrates.

Experimental run	Factor				Average of replication rate of depth (inner/outer) (%)	Average of width deviation (inner/outer) (%)	Average of birefringence (nm)	Average of vertical run-out (μm)	Average of roughness (Ra, mm)
	A	B	C	D					
Test 1	1	1	1	1	97.85/97.70	24.23/9.66	24.3	71.7	38.59
Test 2	2	1	1	1	97.88/98.11	24.23/7.09	11	74.0	39.48
Test 3	1	2	1	1	98.21/98.35	19.83/6.23	16.7	166.7	42.94
Test 4	2	2	1	1	98.36/98.80	18.17/4.1	4.7	205.0	43.04
Test 5	1	1	2	1	98.06/98.17	21.2/7.09	28.0	90.3	42.17
Test 6	2	1	2	1	99.19/98.59	3.01/5.55	18.0	95.0	43.69
Test 7	1	2	2	1	98.48/98.43	18.17/5.59	24.7	213.3	41.35
Test 8	2	2	2	1	99.34/98.91	3.01/3.94	11.0	194.3	44.21
Test 9	1	1	1	2	98.16/98.38	19.83/5.71	9.3	40.3	39.48
Test 10	2	1	1	2	98.53/98.68	18.17/5.52	1.0	98.3	43.99
Test 11	1	2	1	2	99.03/99.10	5.52/3.26	2.3	167.3	44.45
Test 12	2	2	1	2	99.54/99.18	3.01/2.97	-4.0	189.3	45.26
Test 13	1	1	2	2	99.62/99.28	1.13/2.28	17.0	62.7	46.51
Test 14	2	1	2	2	99.63/99.30	0.89/2.22	7.7	103.0	46.78
Test 15	1	2	2	2	99.63/99.33	0.83/1.97	9.3	190.7	48.55
Test 16	2	2	2	2	99.79/99.64	0.02/1.79	0.0	199.3	50.22

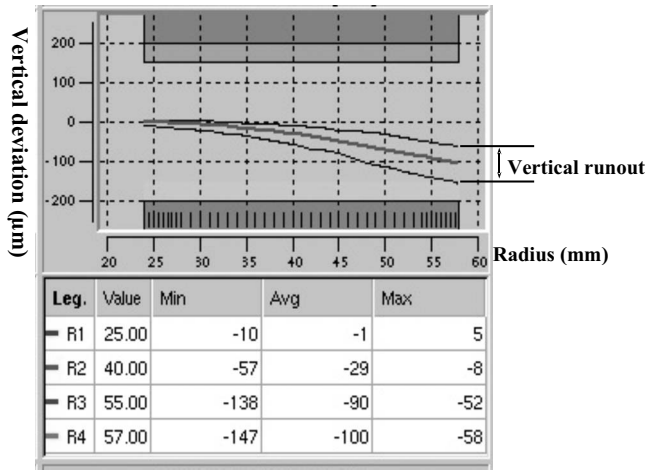


Fig. 6 Vertical deviation distribution of polymer microfluidic substrates.

than 97.5%. The width deviations of locations (inner) and (outer) are less than 24.23 and 9.66%, respectively. Furthermore, the width deviation of the inner location is larger than the outer location. The birefringence and tilt (vertical run-out) values are measured by optical and mechanical evaluation equipment, developed by the Dr. Schenk Company, as shown in Table 2. The vertical run-out is derived from the maximum and minimum correlated vertical deviations, as shown in Fig 6. The maximum vertical run-out value is located at radius 57 mm. The surface roughness of microfluidic structures and substrates are measured by a white light interferometer. The location of (A) in substrates is shown in Fig. 3(e). In this work, the roughness value is represented by the roughness average (*Ra*). The results of replication rate of depth, width deviation, birefringence, tilt, and surface roughness are calculated by averaging three samples for every test run. The experimental results are analyzed by means of Minitab (State Colleges Pennsylvania) Release 14 software. The Minitab is a kind of software for statistical analysis. In this work, the factorial function of DOE is adopted to generate statistics and create graphs.³² The results indicate that there are no interactions among

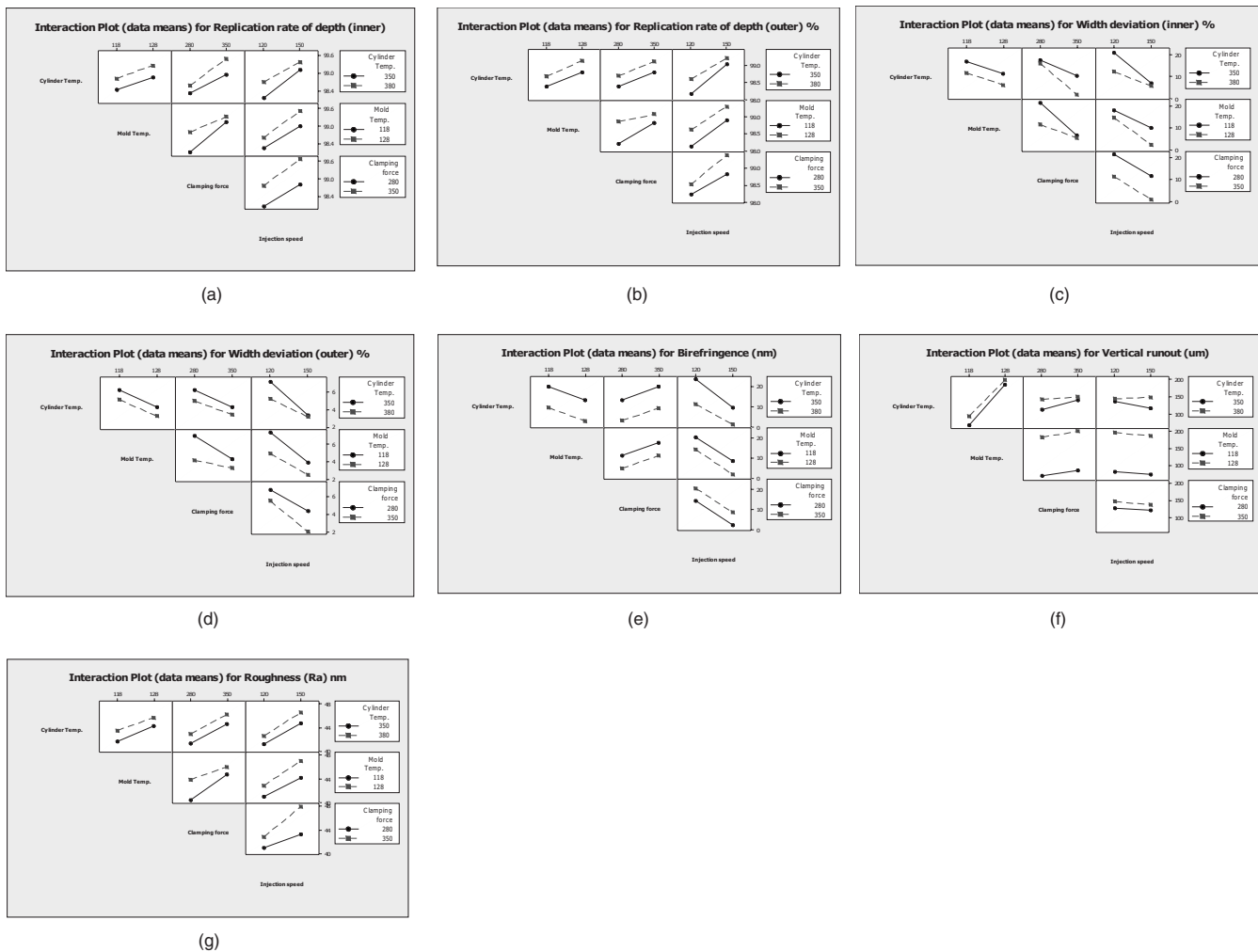


Fig. 7 Interaction relations of (a) replication rate of depth of location (inner), (b) replication rate of depth of location (outer), (c) width deviation of location (inner), (d) width deviation of location (outer), (e) birefringence, (f) vertical run-out, and (g) surface roughness with effective factors of injection molding.

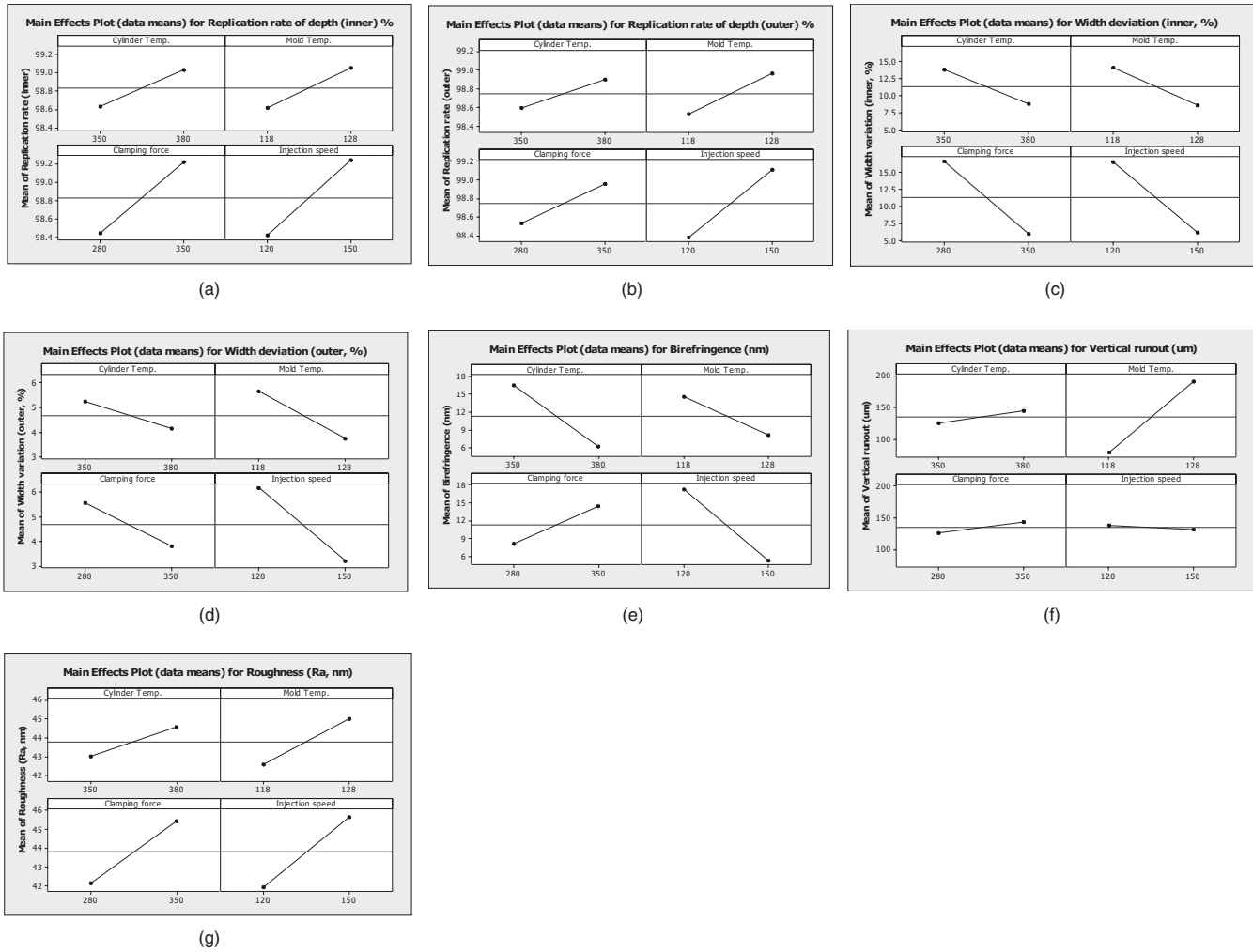


Fig. 8 Main effects of (a) replication rate of depth of location (inner), (b) replication rate of depth of location (outer), (c) width deviation of location (inner), (d) width deviation of location (outer), (e) birefringence, (f) vertical run-out, and (g) surface roughness with effective factors of injection molding.

replication rate of depth of location (inner)/(outer), width deviation of location (inner)/(outer), birefringence, tilt, surface roughness and cylinder temperature, mold temperature, clamping force, or injection speed, as shown in Fig. 7. Therefore, these results also indicate that each factor is independent of each other in the set value of the two levels of effective factors for injection molding of microfluidic substrates, as shown in Table 1. That is to say, even the parameter of each factor is adjusted; it will not influence the results of the experiment. Figure 8 shows the effects that cylinder temperature, mold temperature, clamping force, and injection speed have on the quality of manufacturing microfluidic substrates. However, Fig. 8(a) shows that clamping force and injection speed have greater effect on the replication rate of depth of location (inner). It is clearly seen that the replication rate of depth of location (inner) increases with increasing clamping force and injection speed. Figure 8(b) shows injection speed has a greater effect on replication rate of depth of location (outer). The replication rate of depth of location (outer) increases with the increasing injection speed. Greater effects of clamping force and injection speed have greater effect on width deviation of location (inner).

The effect injection speed has on width deviation of location (outer) is shown in Figs. 8(c) and 8(d). The width deviation of location (inner) decreases with the increasing clamping force and injection speed. The width deviation of location (outer) decreases with increasing injection speed. Figure 8(e) shows that injection speed and cylinder temperature have greater effects on birefringence. Figure 8(f) shows that mold temperature has a greater effect on vertical run-out, and Fig. 8(g) shows injection speed has a greater effect on the surface roughness of location (A). Birefringence decreases with the increasing cylinder temperature and injection speed. Vertical run-out decreases with the decreasing mold temperature, and surface roughness increases with increasing injection speed. These results can assist us to obtain suitable microfluidic substrates. The average cycle time is 4 sec. These cycle times are shorter than any of those in the literatures.⁸⁻²⁰

When using 300- μm -thick mold inserts to make injection microfluidic substrates, if one needs to change to another mold insert, taking the mold off the injection machine is not required, since the system uses a mold insert holder

and vacuum to hold the mold insert in place. In a traditional mold, to change to a new microfluidic design of mold insert, the mold has to be taken off the injection machine, taken apart, changed into another design, and then reassembled again. As a result, it takes 3 to 8 h to change a mold insert. However, in our proposed improved method, it takes only 5 to 10 min. As a result, our proposed method is extremely fast, and can be effectively applied to mass production.

6 Conclusions

This work proposes and demonstrates a new method to fabricate microfluidic mold inserts and substrates. The new mold insert design means that the mold does not need to be taken off the injection machine when making a change to a different microfluidic design. In addition, the time required to change a mold is reduced from 3 to 8 h to 5 to 10 min. Even if the mold inserts are injected more than 10,000 times, they show no sign of damage. The injection cycle time is 4 sec, which is much faster than any previous works reported. In addition, the replication rate of depths of all these experimental runs reaches more than 97.5%. The DOE method and parametric analysis are applied to study the effects of molding parameters on the microfluidic substrates quality. The results indicate that there are no interactions between replication rate of depth, width deviation, birefringence, tilt, surface roughness and cylinder temperature, mold temperature, clamping force, and injection speed. However, clamping force and injection speed have a greater effect on replication rate of depth and width deviation of location (inner), while the injection speed has a greater effect on replication rate of depth and width deviation of location (outer) as well. The injection speed and cylinder temperature have a greater effect on birefringence, the mold temperature has a greater effect on vertical runout, and the injection speed has a greater effect on location A of surface roughness. These results lead to the conclusion that the proposed method is low cost, highly efficient, and suitable for mass production to fabricate microfluidic structures on polymer-based substrates.

Acknowledgment

The authors would like to thank the RITEK Corporation, Hsinchu, Taiwan, for access to their equipment and for their technical support.

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