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Two-stage polymer embossing of co-planar microfluidic features for microfluidic devices

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Abstract

A two-stage embossing technique for fabricating microchannels for microfluidic devices is presented. A micromachined aluminum mold is used to emboss a polyetherimide (PEI) substrate with a relatively high glass transition temperature (T_g). The embossed PEI is then used as a mold for embossing an amorphous polyethylene terephthalate (APET) substrate with a lower T_g. The resulting APET substrate has the same features as those of the aluminum mold. Successful transfer of features from the aluminum mold to the APET substrate was verified by profilometry, and an application of this method in production of a microfluidic device is presented.

Keywords: Polyetherimide; PEI; Amorphous polyethylene terephthalate; APET; Glass transition temperature; Hot embossing; Microfluidic

1. Introduction

Microfabrication technology is a rapidly growing area with new methods reported each year from many research groups. Microfluidic devices have become increasingly popular due to their ability to handle minute quantities of samples, and due to their high throughput sampling capability. Advantages of device miniaturization include decreased consumption of reagents and samples, reduced analysis time, more portable instrumentation, and in some instances lower limits of detection. Miniaturized devices can be coupled to one another to produce micrototal analysis systems (μTAS), which can incorporate sample pre-concentration, separation, and detection steps in a single device. In addition, μTAS devices can reduce the chance of human error, such as mislabeling and contamination, by decreasing the number of times a sample is handled [1–3].

Most microfluidic systems were initially fabricated with glass substrates partly because of well-established pre-existing fabrication techniques such as photolithography and chemical etching processes used to produce these devices in the microelectronics industry [4,5]. The surface properties of glass were also well suited for use in μTAS. However, there are drawbacks associated with the laborious fabrication procedures and the tools required for their production. These disadvantages have made polymers attractive alternative materials for μTAS fabrication. They are preferred over silicon or silica due to their low-cost and ease of fabrication. Moreover, plastic devices are attracting interest as alternative biocompatible materials for microfluidic applications. Fabrication methods have been demonstrated using mass replication technologies such as hot embossing, injection molding and casting of polymers including polymethylmethacrylate (PMMA) [6–8], polycarbonate (PC), polyethylene terephthalate (PETE) and polydimethylsiloxane (PDMS) [9,10]. The number of cost effective and relatively rapid fabrication procedures demonstrated with polymer materials for microfluidic device production has increased as the need for single use, disposable microchips for chemical and bioanalytical analyses have grown.

Micromachining has provided a way to produce individual miniaturized, three-dimensional structures on metal substrates. CNC machining makes use of standard G-code instructions that drive the machine tool to fabricate a device by selective removal of the metal. However, CNC machining places limits on the tool size, and thereby limits the sizes of features to be developed, and is a slow process for replication of large numbers of objects. CNC milling is more appropriate if only a single device to be used as a mold or master is needed. The machining process used in this work involved milling positive features into an

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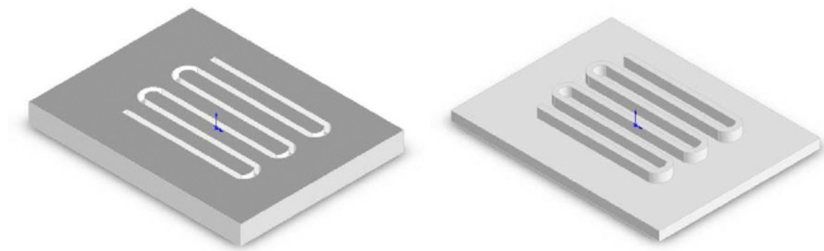

Fig. 1. Master designs produced by micromachining. Recessed features (left) created by cutting into the metal stock, and raised features (right) created by removing the excess metal around the desired feature areas, result in molds to be used for feature replication.

aluminum substrate. The fabrication of a device with recessed features as were needed in our device is considerably easier and faster than creating negative features in a master mold, as illustrated in Fig. 1. The CNC programming needed to create the design shown in Fig. 1(a) is much simpler, the amount of material to be removed is significantly reduced, and therefore, the fabrication process is much more rapid than is the case for production of the design in Fig. 1(b).

The motivation for this two-stage method is the need for co-planarity of bonding surfaces at the interface between substrates in a microfluidic chip. If these surfaces are not co-planar, full contact at the interface will not be achieved and leakage will likely occur either at the chip boundary or between microfluidic features. This goal of co-planarity is difficult to achieve in micromachining because a variety of cutting tools are used for specific purposes. Large tools ($D > 1$ mm) are used for large features such as initial substrate planarization and step features with large widths such as those typically found along the chip perimeter. Small tools ($D < 1$ mm) are used for small features such as ridges between microfluidic channels. During the machining process, tools must be interchanged leading to tool-offset errors. An illustration of the impact of tool-offset errors on co-planarity is shown in Fig. 2: (a) an idealized scenario of no tool-offset error in which co-planarity is achieved; (b and c) features that stand proud of or lie below the substrate plane as a result of tool-offset error; and (d) the scenario used in two-stage embossing which is independent of tool-offset errors and co-planarity is always preserved.

Hot embossing traditionally employs a single processing step in which a mold with *negative* features is used to emboss *positive* features into a thermoplastic substrate. The term "positive" refers to the desired geometry of the features, whereas the term "negative" refers to the inverse of the desired features. In the two-stage embossing method, two processing steps are needed (see Fig. 3 for the schematic diagram of the embossing process). In the first step, a primary mold with *positive* features is used to emboss *negative* features into a thermoplastic substrate to form a secondary mold. In the second step, the secondary mold is used to emboss *positive* features into a second thermoplastic substrate to form the final product. It is critical that the secondary mold have a significantly higher T_g than the final product in order to ensure no deformation of the secondary mold during the second embossing step. A similar conclusion was reached by Belligundu and Shiakolas [11] using a silicon to polymer to polymer embossing technique. The second mold used was polycarbonate with a T_g of 145 °C; PMMA with a T_g of 106 °C was used as the final substrate. In this study, we introduce the use of a polymer substrate, polyetherimide (PEI) which possesses a T_g of 210 °C. PEI is a transparent polymer with high rigidity, excellent heat resistance and comparatively great dimensional

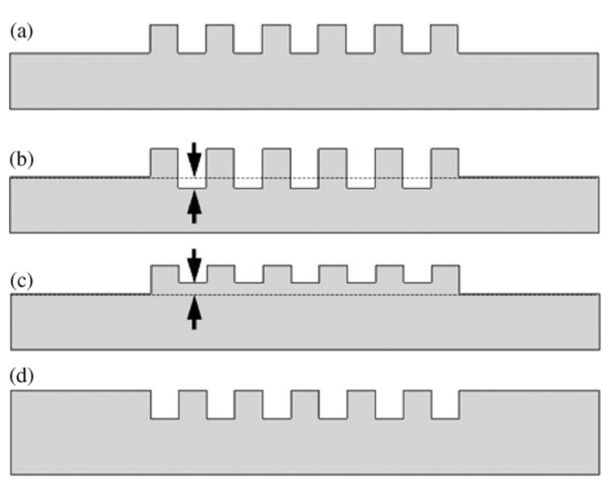

Fig. 2. An illustration of the impact of micromachining tool-offset error (black arrows) on the co-planarity of macroscale and microscale features; (a) co-planar with no tool-offset error; (b) excessive tool-offset; (c) insufficient tool-offset and (d) tool-offset independent.

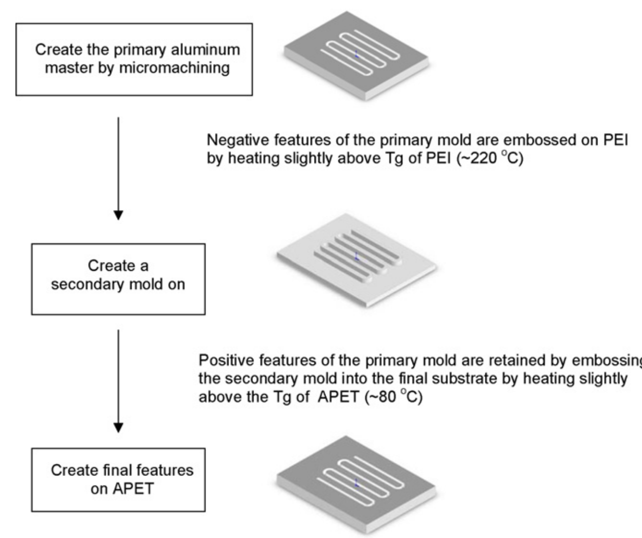

Fig. 3. Schematic diagram of the two-stage embossing procedure.

Table 1
Nanoimprinting conditions used for embossing PEI substrate (top) and PETE (bottom)

Temperatures (°C)	Pressures (bar)	Time (s)
Nanoimprinting program for PEI		
80	0	60
160	0	60
220	0	150
220	65	150
Nanoimprinting program for APET		
50	0	60
65	0	60
80	0	150
80	65	150

Constant pressure at 65 bar was applied at the end of the program until substrate reaches demolding temperature of 50 °C.

stability. The use of this substrate as a secondary mold allows us to work with variety of substrates of lower T_g, such as PC, PMMA, or PETE. APET (amorphous polyethylene terephthalate) was selected to demonstrate the feasibility of this technique for microfluidic device fabrication.

2. Experimental

The primary mold was milled from aluminum stock on a Tool Crafter mill (CMS CNC, Laguna Hills, CA, USA). In the first embossing process (Table 1, top), the positive features of the primary mold were imprinted as negative features into a PEI substrate (McMaster-Carr, Santa Fe Springs, CA) ($T_g \sim 210$ °C) to produce the secondary mold. In the second embossing process (Table 1, bottom), the negative features of the secondary mold were imprinted as positive features into an APET substrate (ALRO Plastics, Jackson, MI) ($T_g \sim 75$ °C) to produce the final product.

The microchannels were made by conformally sealing a PDMS slab on top of the embossed APET. PDMS (Sylgard 184, Dow Corning, NC, USA) was mixed in a 10:1 ratio of monomer to the curing agent, poured onto a clean unpatterned glass slide, and thermally cured for 3 h at 60 °C. The PDMS layer was then peeled off, cleaned with ethanol and thoroughly dried. Holes ($D = 1.5$ mm) were punched through the PDMS slab to form the reservoirs for the fluid inlet and outlet. A 10^{-3}M solution of Rhodamine B dye (Lambda Physik, Acton, MA, USA) in methanol was injected at the inlet of the channels at a very low pressure and the flow was observed under a microscope (Zeiss Axiotron, Carl Zeiss SMT, Germany). The solution was injected into the channel through the inlet reservoir hole using a Microport Interface (Cascade Microtech, Inc., Beaverton, OR, USA).

3. Results and discussion

The two-stage embossing procedure was carried out on an APET substrate using aluminum and PEI devices as the primary and secondary molds, respectively. A single channel design with a dimension of 215 μm width, 85 μm depth, and 80 mm length

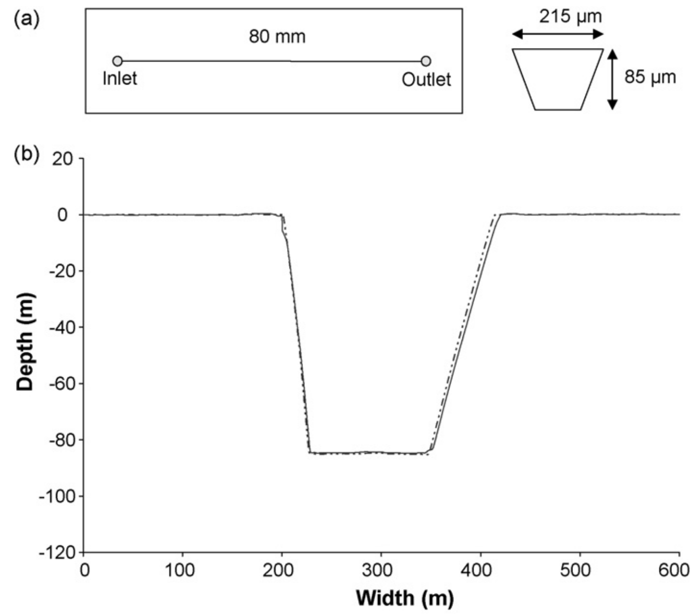

Fig. 4. (a) Schematic of the microchip layout showing the channel lengths and the approximate cross-sectional dimensions. (b) Measurements of the microchannel features obtained by surface profilometry. Depths and widths of the channel of the primary aluminum mold (- - - line) vs. APET replica #8 (solid line) are compared.

was used; a schematic of the microchip layout is provided in Fig. 4a.

The CNC machining operation used to fabricate the aluminum master followed a set of G-code instructions by selectively removing parts of the metal to create the desired final design. In this work, the master was fabricated by milling the design into the aluminum substrate resulting in a mold with recessed features. This technique makes the machining process significantly simpler and more rapid than the conventional method, in which a large area of metal material is removed to produce a master with raised features.

The embossing cycle was repeated eight times on the APET substrate to confirm the reproducibility of the technique. Microfluidic chips produced with this technique were shown to have smooth and reproducible features. The channel features were measured using a profilometer. The channels were characterized under the surface profilometer on the basis of the depths and widths of the channels. Fig. 4b shows the overlay profiles for the aluminum primary mold and the embossed APET final device surface. The results clearly show that the profile of the channel on APET corresponds well to that of the primary mold. The co-planarity of the surface, which is important for bonding purposes, can also be preserved during the aluminum master fabrication and throughout the APET embossing process as can be seen from the figure. The figure shows the comparison between the channel features of the primary aluminum mold and the final features of the APET substrate after 8 repeated cycles on successive blank APET substrates. The results show that there is a slight decrease in the depth of <1 μm (<1%) and an increase in width of 7 μm (∼3%) in the APET substrate for replica #8 compared to the primary mold.

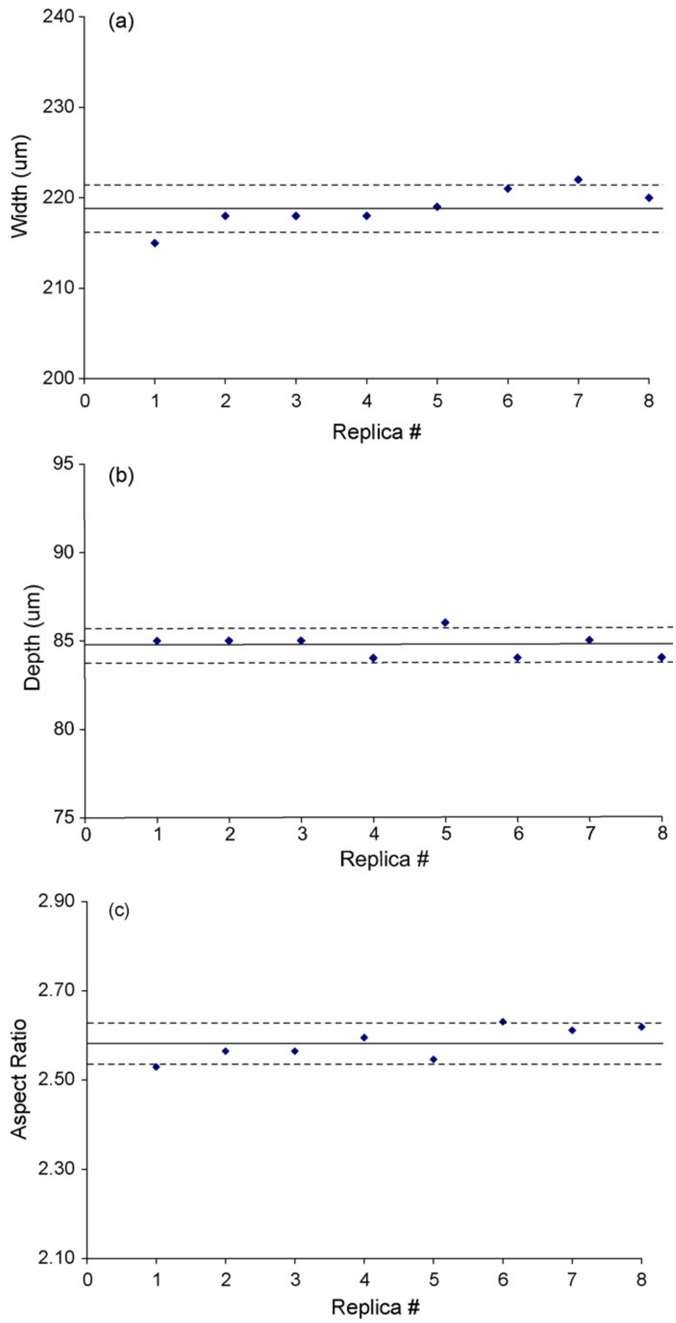

Fig. 5. Comparison of the channel width (a), depth (b), and the aspect ratio (c) of the 8 APET embossed replica. The solid line represents the mean, and the dotted lines represent ±1 standard deviation of the mean.

Variability in widths (*W*) and depths (*D*) were also assessed for the APET replicas (Fig. 5a and b). The differences in channel widths and depths relative to the aluminum mold were ∼2 and ∼1%, respectively. The *W*/*D* aspect ratio of the 8 replicas ranges between 2.53 and 2.63 as compared to *W*/*D* aspect ratio of aluminum mold of 2.53 (Fig. 5c). These results demonstrate success in transferring features from the primary mold to the final substrate.

Another experiment was also conducted using a more complex design master in order to validate the viability of the embossing process. A photograph of the machined aluminum mold is shown in Fig. 6a. The fabrication process from the primary mold to the final APET substrate was carried out using the procedure described above. The final result on APET was characterized by profilometry as shown in Fig. 6(b and c). The microchannels on the primary mold have widths ranging from

Fig. 6. Photograph of the aluminum primary mold (a), surface profile of primary mold (b) and APET substrate (c) measured with the profilometer.

Fig. 7. The channel was formed by conformally sealing the APET chip with a PDMS slab, and filled with 10^{-3}M Rhodamine B dye in methanol under a very low pressure.

310 to 320 μm and depths from 40 to 48 μm. The resulting APET channels have widths of 308–318 μm and depths of 40–46 μm. This embossing procedure was performed five times, and the results indicate good repeatability of the imprinting process.

An embossed APET chip fabricated using the two-stage embossing procedure was then used to create a proof-of-concept microfluidic device. Rhodamine B solutions were introduced into the APET microchannel to demonstrate the device integrity and utility. Fig. 7 shows photomicrographs of the Rhodamine B solution flowing through the APET channel. For this purpose, a PDMS sheet was used to conformally seal the channel. Rhodamine B was injected into the channel from the inlet reservoir using a microport connected to an LC pump. No leaks were detected around the channel when Rhodamine B solution was introduced through the microchannels. By preserving the co-planarity of the master mold during machining, the final embossed APET chip had good surface homogeneity that allowed for effective bonding on a second polymeric substrate, showing the potential of two-staged embossed chips in microfluidic applications.

4. Conclusions

A novel metal–polymer–polymer two-stage embossing technique was demonstrated to be an effective, robust, low-cost fabrication method for replication of features on polymer substrates. The method is simple, reliable, and reproducible. The PEI secondary mold can be used repeatedly as a tool for microfluidic chip fabrication on lower T_g polymer substrates. This technique is especially well suited for fabrication of a primary mold that requires the removal of a large amount of metal to create the design. With this technique, tedious procedures of fabricating the primary mold can be avoided, thus decreasing the time needed for mold fabrication. Preliminary results show the potential of chips fabricated with this method in microfluidic applications. Future work will include the fabrication of low-cost, disposable microfluidic devices using this technique for chemical and biomedical analyses. Future work will also verify this method with other substrates such as PMMA ($T_g \sim 105\,^{\circ}$C) and PC ($T_g \sim 160\,^{\circ}$C).

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Biographies

Myra T. Koesdjojo received her BS in chemistry from the State University of New York at Plattsburgh in 2003. At the time this work was submitted she had completed her third year of study as a PhD candidate in the chemistry program at Oregon State University. She is the author or coauthor on 4 publications.

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