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Polymer shrinkage of hot embossed microstructures for higher aspect ratio and smaller size

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ABSTRACT

In this paper, a facile polymer fabrication approach by combination of hot embossing and polymer shrinking is presented to reduce the feature size and dramatically increase the aspect ratio of imprinted microstructures. Pre-pattern is hot embossed on a heat shrinkable polymer at low temperature to reserve a good shrinkage and recovery. The projected structures are removed by a polishing process. Finally, new microstructures derive from the pre-pattern at the absence of removed materials after baking process. Through this way, both two- and three-dimensional hot embossed structures were successfully shrunk into a smaller scale. The width along two lateral directions reduced to two-fifths, and the height along vertical direction increased by 6 times. Detailed features at different layers exhibit clearly three-dimensional shrunk microstructures. This polymer-shrinking process brings a new way to extend the fabrication capability of hot embossing process.

1. Introduction

In last decades, hot embossing [1–5] has been a well-established fabrication process for polymer microstructures in Micro-Electro-Mechanical Systems (MEMS), such as actuators [6], microfluidics [7–10], micro-optics [11–13], and other microdevices [14]. Although various microstructures can be hot embossed by careful design on the fabrication process [2,3,6], there are still many challenges to enable some features. For example, reducing the feature size and increasing the aspect ratio need much improvement on the mold insert fabrication [15,16], molding [17], and demolding processes [16,18]. However, high-aspect-ratio microstructures tend to be damaged by demolding force [18].

Polymer shrinking becomes another approach to generate structures by reforming pre-patterned structures [19–25]. In the meantime, some new features can be obtained. For example, the conservation of total volume requires anisotropic dimension change for a biaxial planar blank shrinkable polymer. In this case, the width of polymer sheet decreases by a factor of $\boldsymbol{n}$, while the height increases by a factor of $\boldsymbol{n}^2$. Therefore, the aspect ratio of height to width dramatically increases by a factor of $\boldsymbol{n}^3$. The $\boldsymbol{n}$ for commercial shrinkable polymers varies from 2 to 5, and the increased high aspect ratio can reach 100 [21,22]. This characteristic promises the reduced feature size and enhanced high aspect ratio by material itself, instead of improvement on the machining process. It was verified on different pre-patterning processes such as reactive iron etching (RIE) [21,22], mechanical engraving [20], screen-printing [23], laser ablation [25], etc. Zhao et al. [21,22] demonstrated a 100-fold increase in the aspect ratio of microstructures by RIE on polystyrene (PS) film. The width along the two lateral directions reduces to one fourth and one fifth. Minimal shrunk width of 1 μm was obtained after shrinking process. Chen et al. [20] presented a simple approach on obtaining structures at a microscale by hand for microfluidic chips. Manually scribed channels shrunk well into microscale. These pre-patterning methods were adopted well by shrinking process. However, we still need seek a low cost, mass productive and high quality pre-pattern method.

Hot embossing is expected to meet the above demand since it has been a well-established fabrication process for polymer microstructures. However, directly shrinking of hot embossed microstructures fails to generate new features as expected. Yokoo et al. [24] attempted to shrink hot embossed nanostructures. The pitch of a hole array reduced from 100 to 60 nm. But the depth of initial nanostructure tends to shallow instead of dramatically increase. We also find that the hot embossed pre-pattern at a microscale is a temporary deformation, and finally vanishes after baking process. We are curious why the shrinking of hot embossed initial pattern is different from that in other processes such as RIE [21,22], and more interested in how to activate hot embossed pattern to reduce feature sizes and increase the aspect ratio dramatically through shrinking process.

In this paper, we present a facile strategy to activate hot embossed microstructures for the shrinking process. This

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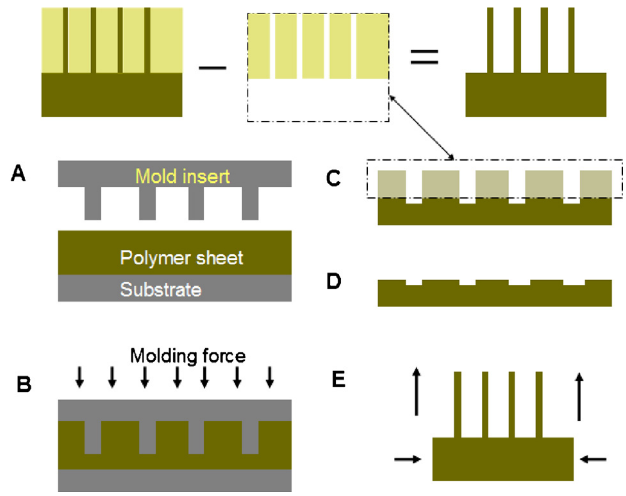


Fig. 1. Sketch on combination of hot embossing and heat shrinking processes. The final shape of shrunk microstructures is defined by the absence of removed materials, (a) Shrinkable polymer PS sheet is sandwiched by a mold insert and a hot plate, and heated to molding temperature of 120 °C, (b) The PS sheet is patterned by the mold insert with a molding pressure of 5 MPa, (c) and (d) Polymer in specific regions of hot embossed structures is removed by polishing process with sandpaper, (e) The shape of shrunk microstructures is homothetic to the mold insert with anisotropic dimension changes.

developed fabrication approach brings new vitality for conventional hot embossing process.

2. Experiment method

The shrinking performance of embossed pre-patterns is affected by the material properties. Heat shrinkable polymer is mechanically pre-stressed and oriented plastic with molecular chains stretched out [19,20]. They tend to recover to their randomly oriented and tangled state upon exposure to an external baking stimulus. Different from directly removing materials on local regions by RIE process, the hot embossing forms microstructures by re-distributing oriented materials. Embossed pre-pattern will almost vanish due to material reflow. For example, an original embossed step of 250 μm may turn into a small step no more than 2 μm.

Although the direct shrinking of embossed pre-patterns was blocked by the shape memory effect, there is still possibility to solve this problem. In fact, the re-distribution of materials in shrinking makes it possible to define final structure by removing specific regions. The sketch of a new shrinking process is shown in Fig. 1. The pre-pattern was pressed down into polymer materials during hot embossing process, which is expected to shrink into final microstructures. Because of the shape memory effect, it tends to rise and return to its original relative position during the shrinking process, and no shrunk microstructures can be seen directly. In fact, the shrunk pattern just submerged in non-pressed-down materials. Thus a feasible way of exposing the shrunk microstructures is to remove the non-press-down part. It was done by removal of the non-press-down materials by polishing before shrinking process. This strategy enables the pressed-down pre-patterns to shrink alone and expose without being submerged by other part. Microstructures are hot embossed above the glass transition temperature (T_g), as in Fig. 1(a) and (b). Projected regions on the surface layer are removed by a polishing process, and act as recessed parts after shrinking process, as shown in Fig. 1(c) and (d). The final shape is defined by the absence of removed materials before shrinking, as shown in Fig. 1(e). Through this method, hot embossed structures are activated and shrink into new patterns with a reduced size along lateral directions and an increased size in vertical direction. In the meantime, the aspect ratio was dramatically increased.

2.1. Materials

Various commercial available heat shrink films can be used for this fabrication process. Here shrinkable Polystyrene (PS) film (Grafix Inc., Maple Heights, OH) 250 μm thick is selected for a demonstration because regular PS has been routinely used in hot embossing. The suggested fast baking process starts at 150 °C. After shrinking process, the blank PS film retracts to two fifths of its original size, resulting in six times increase in thickness. Waterproof Silicon Carbide (SiC) sandpapers (3 M) with grit from P800 to P4000 are used to remove projected regions on hot embossed sample.

2.2. Mold inserts

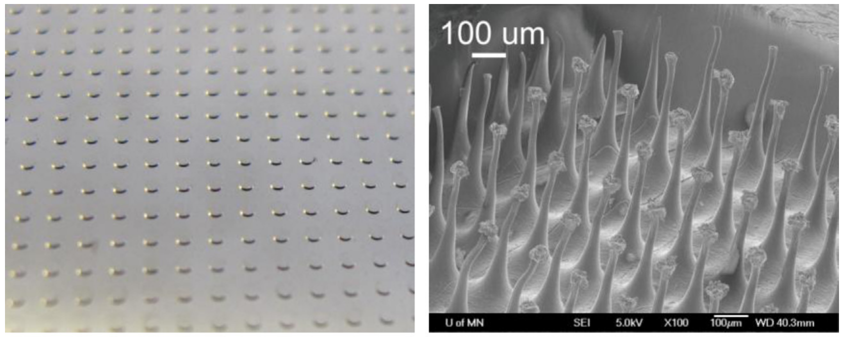
Two types of mold inserts including two- and three-dimensional structures were used for hot embossing process. The two-dimensional microstructures, including posts and needles, were designed and fabricated on a nickel mold insert through UV lithography and electroforming processes that are standard UV-LIGA [26,27]. The mold insert of around post with height and diameter of 250 and 50 μm respectively, is shown in Fig. 2(a). The mold insert of microneedle array with inner and outer diameter of 200 and 400 μm, respectively, is shown in Fig. 3(a). The height is also 50 μm. The mold insert of square post with height and side length of 250 μm, respectively, is shown in Fig. 4(a). SU8 3050 photoresist (MicroChem, USA) was spin-coated on a 4-in. silicon wafer with Cr/Au (50/100 nm) seed layers, soft baked at 95 °C for 15–30 min, and exposed by UV light with a dose of 250–300 mJ/cm^2 in Karl Suss MA6 aligner (Karl Suss, Germany) using printed mask (CAD Art Service, USA). Sequentially, it was post baked at 95 °C for 5 min, and developed in propylene glycol monomethyl ether acetate (Sigma–Aldrich, USA). The patterned SU8 was used as a mold for electroplating. Nickel 3 mm thick was deposited at a direct current of 2.0 A in plating station SE101 (Digital Matrix, USA). Nickel 3 mm thick was deposited on an around area with diameter of 91.6 mm at a direct current of 2.0 A in plating station SE101 (Digital Matrix, USA), ending in an average current density of 30.4 mA/cm^2. The electroformed Nickel mold insert was machined to adapt the work holder in hot embossing machine. Finally, the SU8 was removed by SU8 remover (MicroChem, USA).

United States one-cent coins (commonly known as a penny) were also used as mold inserts to demonstrate the shrinking ability on three-dimensional structures, as shown in Fig. 2.

2.3. Hot embossing process

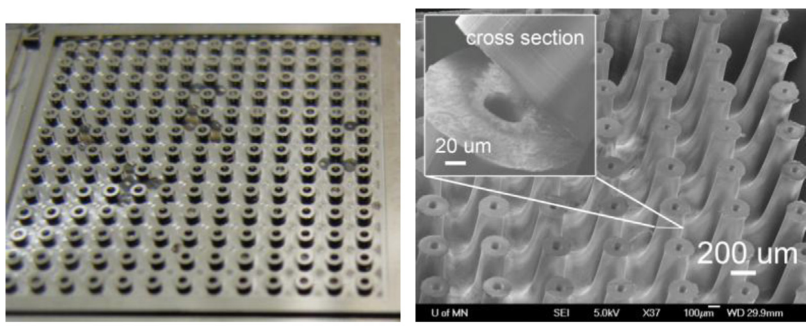
The hot embossing process is performed with a HEX01 (JENOPTIK, Germany) machine. There are two hot plates in this machine: a bottom stationary hot plate and a top movable hot plate. The maximum force and temperature are 50 kN and 320 °C, respectively. A rotary pump is connected to an embossing chamber to provide a vacuum of lower than 0.1 mbar. The polymer was patterned through a conventional hot embossing process with different process parameters such as molding force, temperature and time. The polymer and mold insert were heated up after holding together. Once the temperature of polymer reaches the setting value, the molding force was rapidly raised to target value in a linear manner.

The objective of hot embossing process is to reserve good shrinking ability. For shrinkable PS, the slow baking temperature of 110 °C is close to the glass transition temperature (T_g) of around 95 °C [21,22]. Thus the shrinkable polymer may be baked during molding process. Our solution is to decrease the molding temperature and reduce the heating cycle. In this case, the molding temperature is



(a) (b)

Fig. 2. Nickel mold insert of microposts (a), SEM view of shrunk polymer posts (b).



(a) (b)

Fig. 3. Nickel mold insert of microneedle (a), SEM view of the shrunk polymer needles with cross section (b).

Table 1
Hot embossing parameters.

Process parameter	Value
Molding temperature	120 °C
Molding force	5 MPa
Molding time	2 min
Demolding temperature	85 °C

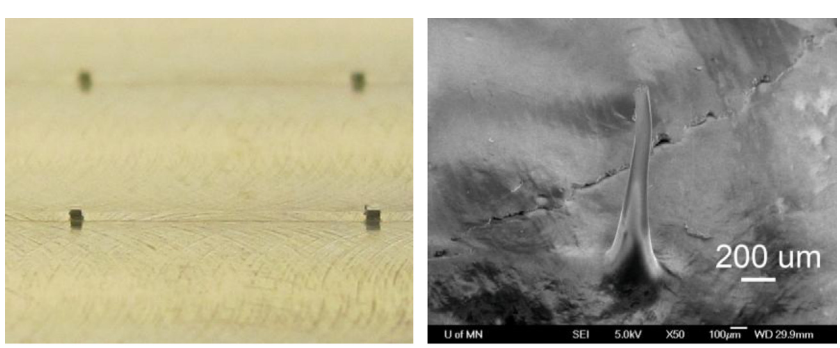
120 °C, slightly higher than the T_g. But the total duration at temperatures higher than T_g is less than 6 min. The overall hot embossing parameters are shown in Table 1.

2.4. Surface polishing with sandpaper

Projected regions of hot embossed polymer sample were manually removed by sandpaper. The embossed sample was attached to a flat plate by double side tape. Large grit sandpaper is used for fast polishing. Small grit sandpaper is used to get smooth surface. In this case, the hot embossed samples were polished by a combination of different grits sandpapers. For example, 50 μm thick polymer materials were removed by consecutive grits, P800, P1200, P2400 and P4000. During the polishing, a drop of water was dispensed on the sandpaper to smooth the polishing process.

2.5. Shrinking process

The suggested baking temperature varies from 110 °C to 160 °C. Lower baking temperature will slow down the shrinking process, and reduce the distortion. Literature reports that baking at 110 °C results in a long shrinking time up to 1 h [21,22]. High baking temperature will get the samples curled firstly and flat consequently. In this case, the polished samples were not sensitive to a quick shrinking process. Thus they were baked at 160 °C in an oven for 5 min. A



(a) (b)

Fig. 4. Micromachined posts with height of 250 mm on brass mold insert (a) and the shrunk polymer post with height over 1.0 mm (b).

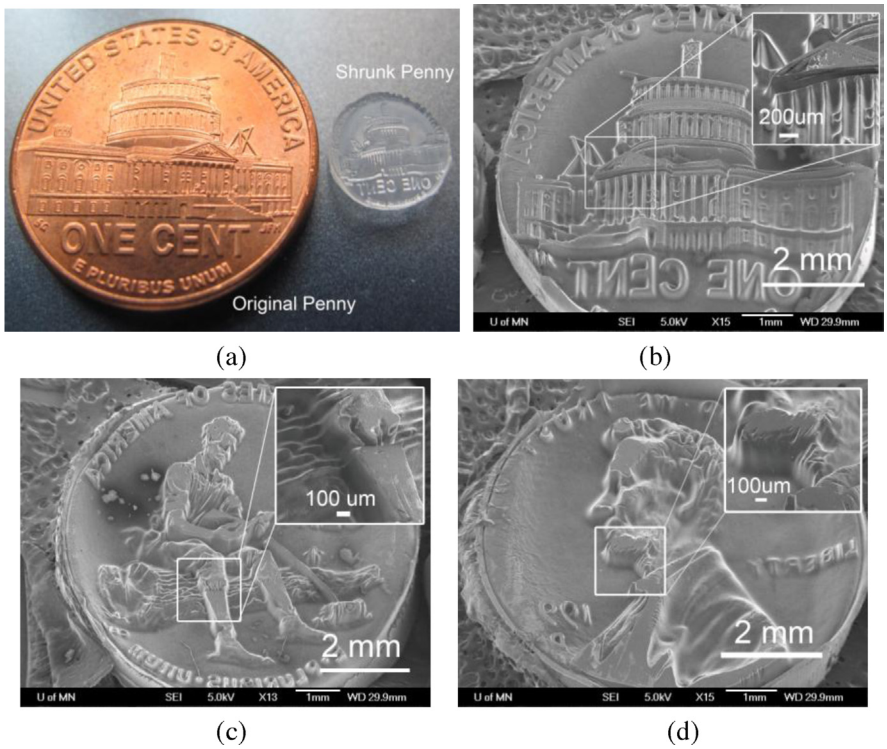


Fig. 5. United States penny and the shrunk polymer penny. United States penny of "Presidency in DC" and the shrunk polymer coin (a), SEM view of the shrunk polymer coin with details (b). SEM view of Lincoln cent of "formative Years in Indiana" with details (c). SEM view of shrunk polymer Lincoln cent with details (d).

medium clipboard was used to minimize sticking of PS samples to the oven.

3. Results and discussion

3.1. Results

Figs. 2–4 summarize the results of two-dimensional shrunk patterns from microneedles and posts with a vertical sidewall. The shrunk height for micro around post and needle array increases by 6 times and reaches 300 μm. This is consistent with the ratio for blank shrinkable PS film. Different from a homothetic shrinking of samples by RIE [21,22], our shrunk structures are not in an exactly homothetic shape of original hot embossed patterns. For example, the cross sectional diameter of posts and needles varies with depth, instead of a uniform diameter before the shrinking process. The root part is thicker than the top, resulting in inclined sidewalls. The top part is more uniform, compared to the root. This non-uniform transformation indicates some re-flow of polymer in shrinking process. One possible reason is that the polymer flow during hot embossing still remembers its original shape. While in Zhao's work, RIE process directly removed materials in designed regions with no polymer re-distribution, and enabled extremely uniform shrinking process.

Geometric transformation of shrinking process is also affected by the aspect ratio of pre-pattern. A low aspect ratio needs a small deformation during hot embossing process. The molecular chains are capable of keeping uniform, resulting in almost vertical sidewalls, as shown in Fig. 3(b). A high aspect ratio needs large deformation of polymer in local regions, causing molecular chains redistributed both in plane and at vertical direction. In this case, the cross section tends to be more non-uniform, and the sidewall becomes more inclined, as shown in Fig. 2(b). Thus a relative uniform posts can be obtained by reducing polished depth. In this case, the root part will be embedded inside polymer materials, and only the top part is disclosed.

The shrinking ability along vertical and lateral directions was also tested. Although the inclined side limits the shrinking in vertical direction, the maximum shrunk height reaches 1.1 mm, as shown in Fig. 4(b). This indicates that molecular chains can remember their original state even at large deformation along the vertical direction. Horizontal shrinkage is very critical for dense microstructures. We found that both single structure and array can be trimly shrunk, as shown in Figs. 2–4. The needles shrink well, and the inner microchannel can be clearly seen, as shown in Fig. 3(b).

Shrinking of three-dimensional microstructures is different from the two dimensional one. The polishing depth is fixed to all these imprinted regions, while the imprinted depth varies at different locations. Microstructures with a small imprinted depth may be totally removed during the polishing process. Will these structures vanish after heat shrinking process? Fig. 5 summarizes the results of shrunk US penny containing three-dimensional features. We can see many detailed patterns at different layers. For example, wrinkles can be clearly seen on the bark of trunk located under Lincoln's legs. This can be explained that geometry feature of a shallow structures can be transferred to deeper range in the vertical direction. This feature transfer effect enables the hot embossed three-dimensional microstructures using a shrinking process with surface polishing.

3.2. Discussion

3.2.1. Recovery ability of hot embossed microstructures

The recovery ability of embossed pre-pattern plays a key role in shrinking process. As described in process sketch (Fig. 1), the pressed materials are expected to be able to recover to its original relative position. This process may fail due to improper molding process. For example, high molding temperature tends to release

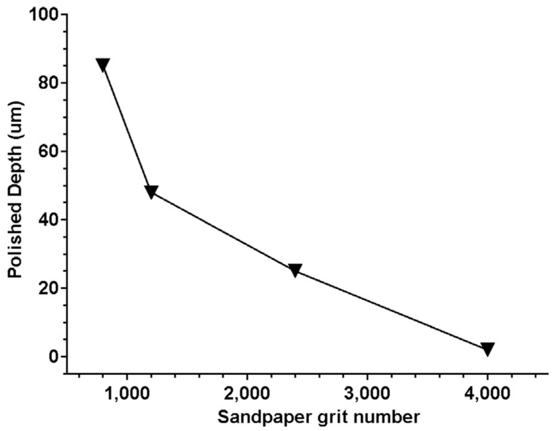


Fig. 6. Polished depth dependent on grits for polishing time of 5 min.

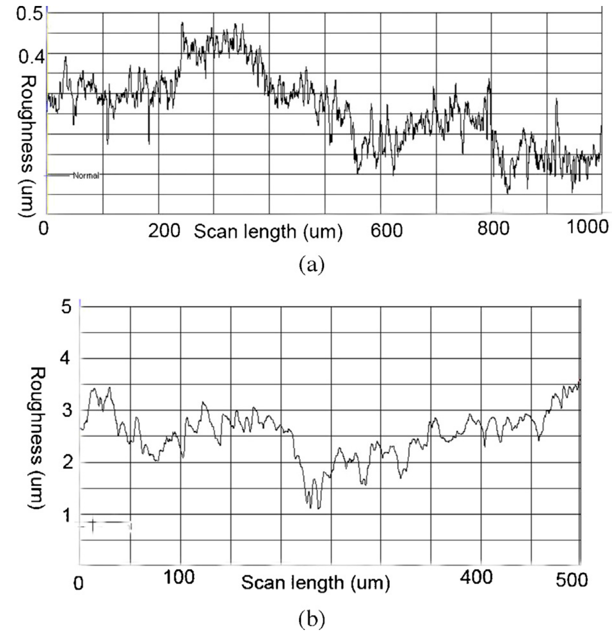


Fig. 7. Surface profile of polished polymer with grit 2400 before (a) and after (b) shrinking process.

these stretched polymer molecular chains into randomly oriented and tangled state, in result of losing recovery ability.

As for this concern, we examined the recovery ability of embossed step that is dependent on molding temperature. The height of the embossed step is 225 μm, which can promise a final shrunk height of over 1.0 mm. The molding temperature varies from 110 °C to 140 °C. The molding pressure and time are 10 MPa and 120 s, respectively. The line width of embossed height reduces from 1000 μm to 400 μm after shrinking process, and is consistent with that of blank PS film. This indicates that these embossed samples have a good shrinking ability along lateral direction. For molding temperature from 110 °C to 130 °C, the top surface of embossed step becomes almost equal with other un-pressed part. The residual height is below 3 μm. When molding temperature increases to 140 °C, the residual height quickly rises up to 5 μm, and the edge becomes sinking. One possible reason is that the molecular chains re-distribute and tends to stay there. The results suggest that molding temperature below 140 °C will promise good recovery ability on embossed microstructures.

3.2.2. Removal of polymer surface layer by sandpaper polishing

In this work, the hot embossed pre-patterns were activated by sandpaper polishing for heat shrinking process. Projected regions on the surface layer are removed by a polishing process. The final shrunk height of microstructures was determined by the polished depth. Thus a well-controlled polishing depth is desired. The typical polished depth in our work varies from several microns to two hundred microns. To meet this demand, a combination of consecutive grits sandpaper is employed to minimize the polishing time. Measured polishing depth by various grits is shown in Fig. 6. It rises rapidly for grit from P4000 to grit P800, corresponding to abrading particle sizes. Grit P4000 can remove a thin polymer layer with thickness of only 2 μm. For a given polishing depth, larger grit sandpaper was firstly used to polish the majority depth, and then smaller grit was used to polish the residual depth and precisely control the final polishing depth.

The roughness of polished area is also important for the shrinking process. It measured by profilometer, and the results show that it will be “shrunk” with microstructures, too. For example, the roughness of polished surface by grit 2400 sandpaper increased from 0.43 μm to 2.49 μm after shrinking process, as shown in Fig. 7. While the roughness of polished surface by grit 1200 sandpaper will increase from 2 μm to 7.64 μm. This means that very shallow shrunk microstructures may be submerged by the “shrunk” roughness. The final shrunk height is expected to be over the shrunk roughness, otherwise the shrunk pattern submerged in the rough surface. Considering that the typical shrunk roughness on polished surface by grit P2400 is below 2.5 μm, thus most shrunk microstructures still can be clearly observed. For shallower shrunk microstructures, an extremely fine polishing is needed. This demand can be met by sandpaper polishing with abrading particles size down to 50 nm or chemical mechanical polishing (CMP) apparatus [28].

Compared to other more established methods such as Zhao’s [21,22] etching method, our shrunk microstructures have warped walls due to the shape memory effect and polymer reflow. Thus this shrinking method does not get an extremely uniformity. But it promises several other advantages: (1) Due to the excellent replication abilities of hot embossing, this process can shrink most exiting microstructures in a facile way, suitable for extremely high shrunk microstructures; (2) The experiments results show this process has a good three dimensional shrinking abilities, it is very useful for some complex microstructures; (3) This process needs no mask on polymers, it will simplify the fabrication process; (4) This process has less requirements on experimental setup, and more time efficiency. Even complex microstructures may be shrunk by hand.

4. Conclusions

A facile polymer fabrication approach by hot embossing and shrinking is presented to reduce the feature size and dramatically increase the aspect ratio of imprinted microstructures. The shrinking ability of hot embossed microstructures is reserved by molding at low temperatures with less cycle time. Hot embossed pre-pattern was activated for shrinking by removing projected structures. The final shape is defined with the absence of removed materials before shrinking. Both two- and three-dimensional hot embossed structures were successfully shrunk into smaller scale. Detailed features at different layers exhibit clearly in three-dimensional shrunk microstructures. This polymer-shrinking process brings a brand new way to extend the fabrication capability of hot embossing process

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Biographies

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