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Chapter 1

Fabrication of Three-Dimensional Concave or Convex Shell Structures with Shell Elements at Micrometer Resolution in SU-8

Louis WY Liu, Qingfeng Zhang and Yifan Chen

Additional information is available at the end of the chapter

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Abstract

This chapter presents a photo-lithographically-based technology for mass production of three-dimensional (3D) micro-structures with shell elements. In this technology, shell elements are photo-lithographically fabricated at micron or sub-micron resolution by illuminating with ultraviolet light radiating an ultraviolet light beam onto UV-opaque SU-8 monomer. The technology does not require any steps involving micro-injection molding or micro-stereolithography. Several prototypes have been fabricated to demonstrate the feasibility of this technology.

Keywords: SU-8, Micro-fabrication, Injection molding, Micro-stereolithography, Graytone lithography

1. Introduction

Micrometer features with hollow parts are increasingly common in many applications [1]. Microneedles, drug delivery systems, and vacuum micro-electronics operating at millimeter-wave frequencies all have hollow micro-structures with dimensions that extend from half millimeter to 1 μm scale. However, mass producing hollow objects at micron resolution is a known challenge in the field of micro-fabrication [2]. Preliminary information about the enabling technologies for realization of suspended or hollow micro-structures is discussed in the sections which follow.
2. Micro-injection molding

Micro-injection molding is currently the technology most widely used for mass-fabrication of three-dimensional (3D) hollow micro-structures [3,4,11–13]. In micro-injection molding, thermoplastic granules are first melted in the plastifying unit of a micro-injection machine. Then, the molten plastic is injected at high pressure into the hollow space of an injection molding tool. After a cooling process, the injection tool is disassembled and the molded object removed. Although micro-injection molding is an established technology that supports mass production of hollow objects, the process is not without limitations. To date, the sizes of hollow objects made in conventional micro-injection molding technology are typically in millimeter range, not in micron range. Injection molding of an object at millimeter resolution is not possible without a special injection machine and auxiliary equipment. The mold to be used for formation of object at millimeter resolution has to be equipped with inlets and outlets in order to allow high-speed injection, gas evacuation, and the expulsion. More importantly, the process of micro-injection molding involves many energy intensive steps which are eco-unfriendly.

3. Micro-stereolithography

Another most widely used 3D micro-fabrication technology is micro-stereolithography [1,2,8]. It works by scanning an UV laser on a liquid monomer, curing the monomer into solid polymeric slices layer by layer, and stacking together all these polymeric slices with various contours. This UV-induced photo-polymerization repeats in a layer-by-layer fashion until the desired 3D object is fully formed. This technology has made it possible to fabricate any form of 3D micro-structures. The surface profile of a fabricated micro-structure can be as complicated as a human face. However, micro-stereolithography is a time-intensive process. The typical scanning speed of a micro-stereolithography machine is about 200–300 layers per hour [8], depending on the geometry and the resolution of the 2D slice to be formed on each layer. Fabricating a simple 3D object of 1 mm in height can take more than 30 minutes. Fabricating a small array of micro-needles can take anywhere between 30 minutes and several hours. Micro-stereolithography technology is currently being pushed developed aggressively focusing on for improvements in both resolutions, speed and flexibility in choice of photo-curable materials. However, due to the use of a laser and a scanner system, the initial investment costs of a micro-stereolithography-based process are unavoidably high.

4. Graytone lithography

Graytone lithography [9,10] is another inexpensive 3D micro-fabrication technology. This technology has been evolved to one step mask-less fabrication using SU-89. For the applications not requiring or not suited with no access to using an expensive micro-stereolithography machine, gray tone lithography is an interesting alternative to micro-stereolithography. Graytone lithography is a modification of conventional 2D optical lithography. It works by exposing a positive photoresist to a UV light through a grayscale mask which defines the
patterns of 3D micro-structures to be formed. The UV light through the grayscale mask produces local intensity modulation. Following a UV exposure, the 3D profile on the surface of the positive photoresist can be formed on the substrate by stripping off the UV-exposed photoresist. While this technology is well known for its potential for mass production, the technology itself is not without limitation. One such limitation is that grayscale lithography based on a positive photoresist does not support fabrication of hollow or suspended micro-structures without change in the technology.

5. Comparison between different 3D micro-fabrication technologies

To the authors’ knowledge, to date, there has not been any cost-effective approach dedicated to mass production of hollow micro-structures at micron or sub-micron resolution. In this paper, the proposed fabrication process presented is a fundamentally different approach based on an improved version of another process published in references [5,6]. The proposed process can be carried out photo-lithographically with conventional photo-lithographic equipment. It provides a convenient alternative for researchers without access to a micro-stereolithography machine or other expensive fabrication facilities. It is not intended as a replacement of other already established and accessible 3D micro-fabrication technologies. Instead, we believe that the proposed process should be used in conjunction with other 3D micro-fabrication technologies to optimizing maximize the advantages in speed, precision, repeatability, and costs of manufacture. Unlike micro-stereolithography, which is only suitable for fabricating small objects in the micron range, the proposed technology can be used to fabricate larger objects with dimension in excess of 1 mm with no sacrifice of speed. The fabricated structures or micro-structures are optically smooth. Table 1 summarizes the advantages and disadvantages in comparison with other competing technologies.

<table>
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<tr>
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<tbody>
<tr>
<td>Initial investment</td>
<td>High</td>
<td>High</td>
<td>Low</td>
<td>Low</td>
</tr>
<tr>
<td>Materials</td>
<td>A negative photocurable material</td>
<td>A thermoplastic</td>
<td>A positive photo-curable material</td>
<td>A UV-opaque SU-8</td>
</tr>
<tr>
<td>Processing speed</td>
<td>Depends on the thickness of the structure</td>
<td>Fast</td>
<td>Fast</td>
<td>Fast</td>
</tr>
<tr>
<td>Feasibility for fabricating shell or hollow components</td>
<td>Yes</td>
<td>Yes</td>
<td>Not easy</td>
<td>Yes</td>
</tr>
</tbody>
</table>
6. SU-8 combined with a UV-blocking impurity

Standard SU-8 fabrication processes normally require a UV-transparent SU-8 [14–17] to proceed. To start fabricating a micro-structure with shells or suspended layers, however, we need a custom-made SU-8 monomer. This SU-8/impurity composite essentially contains a mixture of an SU-8 monomer and a UV-opaque impurity. The UV-opaque impurity is an organic chemical compound satisfying the following criteria: i) it must be opaque to UV lights, ii) it must be highly soluble in SU-8, and iii) it must be substantially non-adhesive. Our experimental results suggest store-bought plasticines or synthetic rubber Blu-Tack™ can be used as an example of the UV-opaque impurities in the proposed process. In this study, Blu-Tack plasticine is the UV-opaque impurity added to the SU-8 to increase its UV opacity. The
ratio of SU-8 to Blu-Tack is 5:3 by volume. The mixture is then baked at 110°C for 4 hours to evaporate off the majority of the solvent (i.e. GBL).

Before exposure to any UV light, it is essential to ensure that the SU-8/impurity composite can be reshaped upon heating and retain its chemical properties after cooling down. However, when this SU-8/impurity composite is exposed to a UV light, as shown in Figure 1a, only the surface exposed on which the ultraviolet light has shone to can be polymerized into a thin layer. The thickness of this layer can be changed by changing the duration of the UV exposure. In general, the longer the UV exposure, the thicker this layer will be.

Since the UV light cannot reach the SU-8/impurity composite underneath the UV exposed surface, the SU-8/impurity composite underneath the UV-exposed surface will remain uncured. The heat then applied to this experimental setup will melt this uncured SU-8/impurity composite into a liquid, as illustrated in Figure 1b. Since the removed SU-8/impurity composite removed remains photo-curable, it can be reused to fabricate other hollow parts.

7. Fabrication of an embossing stamp

Another key element for mass production of micro-structures is an embossing stamp, as shown in Figure 2e. The embossing stamp serves as a patterned template for casting the desired 3D patterns on the surface of the SU-8/impurity composite. A variety of methods can be used to fabricate this embossing stamp. If the 3D features to be formed have many complicated contours, then gray tone lithography or micro-stereolithography [5–8] should be used. If the 3D patterns to be formed are just a combination of several arbitrarily profiled sidewalls, then the method illustrated in [5,7,18] may be adopted.

8. Casting of three-dimensional micro-structures and formation of suspended or hollow parts

Once the embossing stamp and the SU-8/impurity are available, we can proceed to replicate the desired 3D micro-structures and fabricate suspended and hollow parts. This process is illustrated in Figure 2a–h.

In step 1, as shown in Figure 2a, several height-defining blocks of equal height are fabricated onto the corners of the wafer. These height-defining blocks are used to ensure the SU-8 monomer to be deposited on the wafer becomes even and accurate in thickness.

In step 2, as shown in Figure 2b, the SU-8/impurity composite is deposited onto the surface of the substrate until its thickness slightly exceeds the height of the height-defining blocks. The wafer is then heated so that its temperature is slightly above about 70–80°C. The SU-8/impurity composite is heated at this temperature for a prolonged period until the SU-8/impurity composite is void of any solvent.
In step 3, as shown in Figure 2c, the top surface of the SU-8/impurity is pressurized and ironed flat with a glass slice. Then, the heat source is removed.

In step 4, as shown in Figure 2d, patterns defining the hollow or suspended regions are metalized. This metallization process can be carried out by painting with metal ink followed by etching. The purpose of this step is to create an embedded mask which allows selective UV exposure in the later step. After the UV exposure step, the embedded mask created in this step will become a sacrificial layer to be removed in the final step.

In step 5, as shown in Figure 2e, the embossing stamp fabricated in the previous stage is manually aligned with a mask-aligner and pressed downwards slowly. This step not only casts the conical pattern on the right of Figure 2e, but it also forms hemispherical solids by pressing the metal layers downwards. Since the top surface of the semi-molten SU-8/impurity composite has its own surface tension, this step will ensure that a smooth surface with a spherical profile is formed on the top of the hemispherical package.

In step 6, as shown in Figure 2f, the embossing stamp is removed from the wafer. The wafer is then cooled down for about 3 hours until the 3D patterns on the SU-8/impurity composite become fully solidified. This step further eliminates the surface adhesion on the top of the wafer.

In step 7, as shown in Figure 2g, areas requiring hollow or suspended parts are selectively exposed to a UV light. This step can be realized by two methods. We can expose the wafer to an ordinary UV light through a photomask that defines the patterns of hollow or suspended parts. Since the SU-8/impurity composite has been mixed with a UV-opaque impurity, the UV-exposed surface of the SU-8/impurity composite can be polymerized into a polymeric layer. This polymeric layer can be easily thickened by increasing the duration of the UV exposure. This polymeric layer can be as thin as a membrane or as thick as the application demands. The SU-8/impurity underneath the UV-exposed surface will remain uncured and become removable by melting.

In step 8, as shown in Figure 2h, the wafer is baked at 110°C. This heat temperature not only hardens the polymerized surface from step 7 but also melts the uncured SU-8/impurity mixture underneath the UV-exposed surface, into a liquid. Traces of the SU-8/impurity not removable by melting can be further removed by developing in an appropriate SU-8 developer.

The fabricated component on the top left corner of Figure 2h is a hemispherical package used to protect RF-MEMS devices against humidity. This hemispherical package has been glued onto a metallic washer which serves as a radiofrequency ground for a radiofrequency-printed circuit board. The package is intended to be capped on the substrate of a printed circuit board on which the RF-MEMS devices are mounted. The interface between the hemispherical package and the substrate can be further sealed with PDMS.

The fabricated component on the top middle Figure 2h is a spherical hollow object with a diameter equal to approximately 500 μm. It is realized by gluing together two hemispherical shell elements fabricated using the same process as illustrated in Figure 2a–h. Gluing the two hemispherical shell elements together involves manual alignment under a microscope.
The component in the top right corner of Figure 2h is a conical funnel with base diameter equal to approximately 50 μm.

Figure 2. (a–h) Cross-sectional view illustrating the process flow of the technology for fabricating hollow or suspended micro-structures.
9. Fabricating devices for drug delivery applications

Over the past decades, there has been no shortage of interest in nano- or micro-fabrication in the field of drug delivery. Among all drug delivery devices, micro-needles appear to be the most popular apparatus in terms of the number of papers that have been published over the past three decades. With the above fabrication technique, fabricating a micro-needle or similar devices would be as easy as brewing coffee. Formation of hollow objects at micro-scale is undoubtedly one of known challenges in the field of micro-fabrication, especially polymer micro-fabrication. The following sections demonstrate how the previously developed techniques [14–19] can be employed for realization of conical micro-funnels for drug delivery applications. As explained in the previous sections, our fabrication methodology is based purely on photo-lithography.

In step 1, as shown in Figure 3a, we need to prepare a pre-cured SU-8 which is UV opaque. A standard SU-8 monomer resin is first mixed with a UV-opaque impurity at an elevated temperature until the final mixture becomes almost opaque in the UV spectrum. This UV-opaque impurity can be an appropriate plasticine that is opaque to UV lights and does not form any complex in SU-8. In addition, this UV-opaque SU-8 resin will have to undergo a prolonged dehydration bake to increase its viscosity and to decrease its surface adhesion. The wafer of this pre-cured SU-8 will be maintained at an elevated temperature (preferably slightly above the glass transition temperature) so that this UV-opaque SU-8 resin becomes partially molten and its upper surface becomes non-adhesive. In so doing, this UV-opaque SU-8 resin should be able to be reshaped upon heating without excessive change in its physical and chemical properties.

In step 2, as shown in Figure 3b, we need to fabricate an embossing stamp which is basically a master mold having an array of cylindrical rods. This embossing stamp will be used as a patterned template for casting of the inverted conical patterns on the surface of the UV-opaque SU-8 from step 1. A variety of methods can be employed to fabricate this embossing stamp. In the present study, an embossing stamp with high aspect ratio micro-rods was fabricated using a high-quality standard UV-lithographic process but high aspect ratio is not really a prerequisite in the present application.

In step 3, as shown in Figure 3c, the upper surface of the UV-opaque SU-8 resin from step 1 is physically deformed by a mechanical impact produced by the embossing stamp moving downwards at high speed. At the same time, the wafer temperature is tuned down. As a result of this impact, an array of micro-conical wells will be 3D cast on the upper surface of the UV-opaque SU-8. It is important to understand that the stroke speed of stamping will determine the sharpness of the tip of each conical well and the surface profile of the inner wall. In general, if the stroke speed is higher, the sharp tip of each conical well will accordingly become sharper.

In step 5, as shown in Figure 3d, the embossing stamp is removed from the wafer while the wafer is being cooled down. Following this cooling step, the conical micro-wells will become highly solidified.
Figure 3. Cross-sectional illustration of the procedure for fabrication of a micro-funnel. (a) A wafer containing a bath of UV-opaque highly dehydrated SU-8 resin. (b) Cross-sectional view that illustrates the embossing stamp with micro-rods. The diameter of each micro-rod is 40 μm. The length of each micro-rod is 200 μm. (c) Casting of micro-wells by surface deformation using the embossing stamp. (d) Removal of the embossing stamp from the wafer. (e) UV exposure. (f) Removal of uncured SU-8 resin by melting at an elevated temperature.

In step 4, as shown in Figure 3e, the shell of each micro-funnel is formed and thickened by increasing the dosage of UV exposure. The micro-well patterns on the wafer can be photolithographically defined, patterned, and exposed to a UV light using a photomask. Since the SU-8 resin in the wafer contains a UV-opaque impurity, the micro-wells become partially transparent at ultraviolet spectrum. In the presence of the UV-opaque impurity, the interior
surface of each micro-well is the only region fully exposed to the UV lights. The SU-8 resin attached to the opposite side of the UV-exposed surface will be partially cured. As a result, the interior surface of each micro-well will be polymerized into a hard and thick layer. The thickness of this polymeric layer can be easily increased by increasing the duration of the UV exposure. In general, this polymeric layer can be as thin as a membrane or as thick as the application demands, depending on the duration of UV exposure.

In step 5, the uncured UV-opaque SU-8 resin attached to each of the micro-well is removed from the micro-well by melting at an elevated temperature. The SU-8 resin attached to the opposite side of the UV-exposed surface will remain uncured and melted into a liquid when the wafer is subjected to a strong heat. As a result of this heat, the UV-exposed surface will be significantly hardened. Traces of the uncured photosensitive resin which remain attached to the micro-well array can be stripped off by developing in 1-methoxy-2-propanol acetate.

In step 6, a hole is formed on the tip of each conical micro-well by dry etching. This step is intended to turn each conical micro-well into a micro-funnel. A hole can be formed on the tip of each conical micro-well by dry-etching the wafer in oxygen plasma for 100 seconds using a Trion RIE/PECVD tool. The oxygen plasma also sharpens the tip of each micro funnel during the dry etching process. In the present study, the process parameters were 90% O2, 10% CF4, an RF power of 100 w, and a chamber pressure of 1.6 Torr.

10. Results and discussions

Figure 4 shows one of the micro-funnels which have been fabricated using the abovementioned method. The micro-funnels were designed to have a sharp tip and wide top, that is a low aspect ratio geometry. This design allows a larger amount of drug to be encapsulated per micro-funnel. The conical geometry used in this study had a volume in excess of 24.4 nl. In addition, the micro-funnels were found to have sufficient mechanical strength for inserting into the

Figure 4. Photo illustrating a fabricated micro-funnel.
human skin. The smallest tip diameter which has been achieved in this study is within 50 μm, which is sharp enough to pierce the human skin.

The proposed fabrication methodology can be further improved and extended to realize other more complicated systems involving 3D micro-structures.

11. Conclusion

For the first time, this article has presented a method that enables concave or convex micro-structures with shells elements or hollow space to be photo-lithographically fabricated. The method comprises four basic stages. The first stage involves preparation of a composite containing a mixture of SU-8 monomer and a UV-opaque impurity. The second stage involves fabrication of an embossing stamp for casting the desired 3D patterns. In the third stage, the desired 3D hollow or suspended micro-structures are cast using the embossing stamp. In the final stage, features requiring with suspended or hollow parts are selectively polymerized by UV exposure. The concepts of this technology have been demonstrated by fabrication of prototypes.

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