Three-dimensional SU-8 structures by reversal UV imprint

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In this work, three-dimensional (3D) SU-8 micro- and nanostructures were fabricated using a reversal UV imprint process at low temperature and low pressure. The SU-8 polymer was coated on a patterned glass mold and then transferred onto various substrates by reversal UV imprint at a typical temperature of 50 °C, pressure of 1 MPa, and UV exposure of 1 s. The lower temperature and pressure used compared to conventional thermal imprint shorten the imprint time and alleviate pattern distortion. A combination of silanes was used to generate a medium surface energy on the imprint molds to enable polymer spin coating and mold release after imprint. In addition, an O_2 plasma was used for glass mold treatment to improve uniformity of silane coating and to increase substrate surface energy for better polymer adhesion. Using this technology, 100 nm–1 μm wide SU-8 gratings were fabricated on flat or patterned substrates with good fidelity. By repeating this process, multiple-level nanochannels, cavities, or air-bridging polymer structures with 400 nm–10 μm widths have been demonstrated. The surface energy of SU-8 was modified using an O_2 plasma to promote layer adhesion for 3D stacking. This reversal UV imprint technology offers versatility and flexibility to stack polymer layers and multiple-level sealed fluidic channels. *© 2006 American Vacuum Society.* [DOI: 10.1116/1.2335431]

I. INTRODUCTION

During the last decade, remarkable progress has been achieved in micro- and nanofluidic systems, lab-on-a-chip, photonic crystal, environment monitoring, biosensing, and tissue engineering. All these applications require continuous improvements in device performance, increased density, and reduced manufacturing cost. In addition, the ability to fabricate three-dimensional (3D) nanostructures offers unique advantages of high performance and integration of complex systems. For instance, 3D scaffolds that mimic natural cell microenvironment are essential for tissue engineering. 3D structures have obvious advantages over two-dimensional counterparts for cell growth,[1] 3D photonic crystals for optics,[2] 3D microelectromechanical systems (MEMSs) with integrated functions, and 3D circuits integration with compacted interconnects.[3] However, the traditional semiconductor fabrication processes that involve multiple deposition, planarization, lithography, and etch steps for 3D structures are rather complex, laborious, and expensive. The future development of nanotechnology would require simple, low-cost, and versatile methods to fabricate 3D micro- or nanosystems. For this endeavor, several techniques based on soft lithography,[1,4] nanoimprint lithography,[5–7] and bonding[8] have been developed to build 3D structures. Reversal thermal nanoimprint was developed to form 3D polymer stacks.[6] Nanotransfer printing was used to pattern 3D or multiple-layer metal structures.[4] Using adhesive bonding of SU-8 structures, a 3D microfluidic network was formed.[8]

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In this article, we report a reversal UV imprint technique to pattern SU-8 structures over topography to stack polymer layers and multiple-level sealed micro- and nanofluidic channels at lower temperature and pressure in comparison to conventional thermal nanoimprint or bonding process. The lower temperature and pressure used reduce the imprint time and alleviate pattern distortion. The effect of imprint temperature on polymer flow was studied. In addition, silane coatings and O_2 plasma were used to modify the surface energies of the glass molds, Si substrates, and SU-8 patterns for controllable 3D stacking. Two levels of sealed SU-8 channels were fabricated successfully by stacking two layers of polymer structures using this reversal UV imprint technology.

II. EXPERIMENT

In this reversal UV imprint technology, a glass mold with modified surface is spin coated with an UV curable polymer and then transferred onto a substrate during an UV imprint process. Figure 1 illustrates how this process works. First, UV curable polymer SU-8 is spin coated onto a glass mold, which is then brought into contact with a substrate. Moderate pressure is applied and temperature is set to about the glass transition temperature (T_g) of SU-8, 55 °C, to induce slight polymer wetting for adhesion to the substrate. After a couple of minutes, UV light radiation is supplied through the glass mold to the SU-8 layer. SU-8 monomers undergo cross-linking under the UV exposure to form hard epoxy material. Finally, the glass mold is removed and molded SU-8 structures are left on the substrate. The reversal UV imprint process is carried out in an Obducat 4 system, which allows flexible programming of the temperature, pressure, and UV exposure processes. Glass molds with structures as small as 400 nm were fabricated using an i-line stepper for pattern-

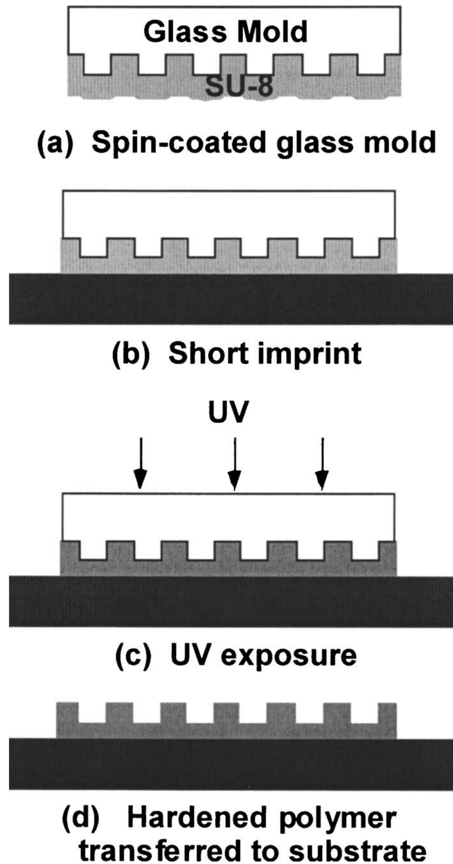


FIG. 1. Schematics of reversal UV imprint: (a) spin coating of UV curable polymer onto glass mold, (b) short thermal imprint at moderate temperature and pressure, (c) UV exposure through the mold to the polymer, and (d) UV curable polymer transferred to substrate after mold release.

ing, followed by dry etching of the glass using a CF_4 plasma. Molds with depth of 300–600 nm are formed with smooth surface.

For the complete transfer of SU-8 structures from the glass mold, the control of mold surface energy is important. Polymer coating requires the mold surface energy to be high enough to ensure good adhesion of the polymer to the mold surface. On the other hand, mold surface energy needs to be lower than that of the substrate so that the polymer will adhere more strongly to the substrate than to the mold. Therefore, we need to tailor mold surface energy to satisfy these requirements. Several different silanes, namely, perfluorodecyltrichlorosilane (FDTS), methacryloxypropyltrichlorosilane (MOPTS), phenethyltrichlorosilane (PETS), and their combinations, were used to alter mold surface energy. Glass molds were initially treated with an O_2 plasma at 250 mTorr and 200 W for 5 min to clean the surface and to generate a surface with uniform –OH bonds before silane deposition. Then the molds were soaked in 2% silane in *n*-heptane for 5 min, rinsed in acetone, and baked at 100 °C for 10 min. Contact angles of de-ionized water and acetone on the silane treated molds were measured and surface energies were calculated using the two-liquid method[9] as shown in Table I. The mold surface energy was 17.8 mJ/m^2 after treatment with FDTS, 19.7 mJ/m^2 with PETS:FDTS (2:1), 21.9 mJ/m^2 with PETS, and 50.0 mJ/m^2 with MOPTS. Spin coating of SU-8 in methylisobutylketone (MIBK) was successful on the glass molds treated with all the silanes and combination except FDTS alone. The surface energy of FDTS, treated molds was close to that of those treated with PETS:FDTS (2:1) and PETS. The failure of SU-8 spin coating on the FDTS-treated molds is due to its very low polar surface energy (0.01 mJ/m^2) compared to the dispersion surface energy (17.8 mJ/m^2). When one of the two materials in contact is nonpolar, only dispersion interactions are possible. Since SU-8 and its MIBK solvent are polar materials while FDTS-treated mold is nonpolar, the interaction energy is small and the adhesion is poor. On the other hand, a larger polar surface energy on the treated mold will help promote the adhesion of the polar SU-8. However, if the polar surface energy of the mold is too large, for example, 26.5 mJ/m^2 for MOPTS-treated molds, separation of the SU-8 from the mold after reversal imprint will be more difficult. The best mold treatments for polymer coating and subsequent polymer transfer to the substrate were found to be PETS:FDTS (2:1) and PETS.

In addition, O_2 plasma cleaning of the glass molds is critical for the silane coating to be effective. As shown in Table I, without O_2 plasma cleaning, the surface energy of PETS-treated glass mold was 50.1 mJ/m^2, which is much higher

TABLE I. Surface energies of glass molds and Si substrates treated with various silanes.

Mold	Silane	O_2 plasma exposure	Contact angle (deg)		Surface energy[a] ($\gamma = \gamma^d_+ \gamma^p$) ($mJ/m^2$)		
			H_2O	Acetone	γ^p	γ^d	γ
SiO_2	FDTS	Yes	117.0	43.0	0.01	17.8	17.8
SiO_2	PETS	Yes	96.0	38.0	3.0	18.9	21.9
SiO_2	PETS:FDTS (2:1)	Yes	101.0	42.5	1.8	17.9	19.7
SiO_2	PETS	No	50.7	0	26.4	23.7	50.1
SiO_2	MOPTS	Yes	50.8	7.8	26.5	23.5	50.0
Si		No	54.5	23.8	25.1	21.7	46.8
Si		Yes	4.6	0	48.9	23.7	72.6

[a]Surface energy γ is the sum of γ^d (dispersion component) and γ^p (polar component) (Ref. 8).

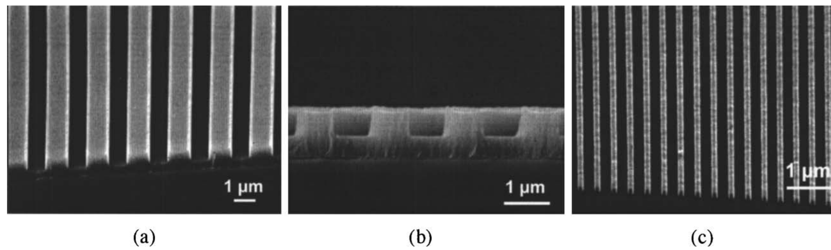


FIG. 2. SU-8 gratings transferred to Si substrate by reversal UV imprint. (a) Top view of 1 μm half-pitch SU-8 gratings with 650 nm depth, (b) cross-sectional view of 1 μm half-pitch gratings, and (c) 100 nm wide SU-8 gratings with 250 nm depth.

compared to those with an O_2 plasma cleaning (21.9 mJ/m^2). The reason is that trichlorosilane molecules interact with the hydroxyl groups, –OH, of the glass surface to form covalent bonds. However, unlike Si, the –OH density of the glass molds before cleaning is low and nonuniform.[10] O_2 plasma treatment can increase–OH density and therefore results in a more uniform and effective silane coating.[10] For a successful pattern transfer of polymer from the glass mold to the Si substrates, the surface energy of the substrate also plays a role. As shown in Table I, the Si substrate has a surface energy of 46.8 mJ/m^2, which can be increased to 72.6 mJ/m^2 by an O_2 plasma. Higher Si substrate surface energy is desirable since it results in better adhesion of SU-8 to the substrate.

After the glass mold was coated with appropriate silane, 900 nm thick SU-8 was spin coated on it. Then it was baked on a hot plate at 65 °C for 2 min and 95 °C for 5 min to drive out solvent. The SU-8 coated glass mold was placed in contact with a Si substrate. A 0.5 mm thick polycarbonate film was used to cover the mold and the substrate to form a pressure seal. The UV absorption by the polycarbonate film is negligible. Pressure was supplied by compressed N_2 above the polycarbonate film and heat was supplied from the bottom substrate holder. A typical reversal imprint process was carried out at a temperature of 40–85 °C and a pressure of 1–5 MPa. The imprint temperature needs to be in the range of T_g-15 °C to T_g+30 °C for effective adhesion of SU-8 to the Si substrate. After the temperature and pressure were applied for 2 min, UV light was turned on for 1–10 s depending on SU-8 thickness. UV intensity for 300–400 nm wavelength is $\sim$200 mW/cm^2 under pulse mode. 1 s exposure results in a dose of 200 mJ/m^2, which is sufficient to expose 900 nm thick SU-8. After the UV exposure, pressure and temperature were reduced and the glass mold and the substrate together with UV cured SU-8 were baked on a hot plate at 65 °C for 2 min and at 95 °C for 5 min for further cross-linking of SU-8 and to reduce its stress. Finally, the glass mold was separated from the substrate, and the SU-8 structures were transferred from the glass mold to the Si substrate.

III. RESULTS AND DISCUSSIONS

A. Imprint on flat substrates

Figure 2 shows cured SU-8 gratings with 1 μm half-pitch and 650 nm depth [Figs. 2(a) and 2(b)] and with 100 nm half-pitch and 250 nm depth [Fig. 2(c)] that were transferred onto the Si substrate using the reversal UV imprint. Typically, a 2 min thermal imprint at 85 °C and 5 MPa before UV exposure resulted in a good adhesion of SU-8 to the Si substrate. UV exposure for 10 s was followed at 50 °C and 5 MPa. After the imprint was completed, the glass mold was separated from the Si substrate and the SU-8 structures were transferred to the Si substrate with excellent uniformity. The depth and the width of the transferred SU-8 were identical to the dimensions of the molds. This reversal UV imprint shows good fidelity and high yield for both micro- and nanoscale devices.

B. Imprint on patterned substrates

One advantage of reversal UV imprint is its capability to pattern polymer structures directly onto devices with topography and create cavities underneath. We used the reversal UV imprint to transfer SU-8 gratings onto Si grating substrates and to control the flow of SU-8 during imprint. The SU-8 flow is determined by the amount of heating before UV exposure. Figure 3(a) shows SU-8 structures transferred onto the Si gratings with 1 μm depth. The reversal UV imprints were carried out at a pressure of 5 MPa, temperature of 40–70 °C, and 10 s UV exposures. Sealed cavities were formed for both the 1 and 10 μm wide gratings as shown in Fig. 3(b). Higher temperature used for the imprint caused more SU-8 flowing into the Si cavities. Figure 3(c) shows the amount of polymer flow into 2 μm wide Si gratings as a function of the imprint temperature. Reducing the temperature to be slightly below the glass transition temperature of SU-8 results in insignificant SU-8 flow and the SU-8 structures successfully bridge over the Si gratings to form cavities and sealed channels.

After the reversal UV imprint, the cured SU-8 structures become permanent parts of the devices. During the polymer transfer, there is a residual layer across the substrate, as shown in Fig. 2. Such a residual layer may need to be removed for some applications, such as in tissue engineering. The residue can be etched using an O_2 plasma with 5% CF_4. If only O_2 plasma is used to etch the SU-8 residue, the SU-8 surface becomes rough. The introduction of F in CF_4 significantly reduces the roughness of the etched surface and increases the etch rate.[11] Figure 4 shows the 1 μm wide and 600 nm deep SU-8 gratings over a Si grating substrate after 400 nm thick SU-8 residue was etched away. This structure can be used as scaffolds for tissue engineering or other applications requiring air-bridging structures.

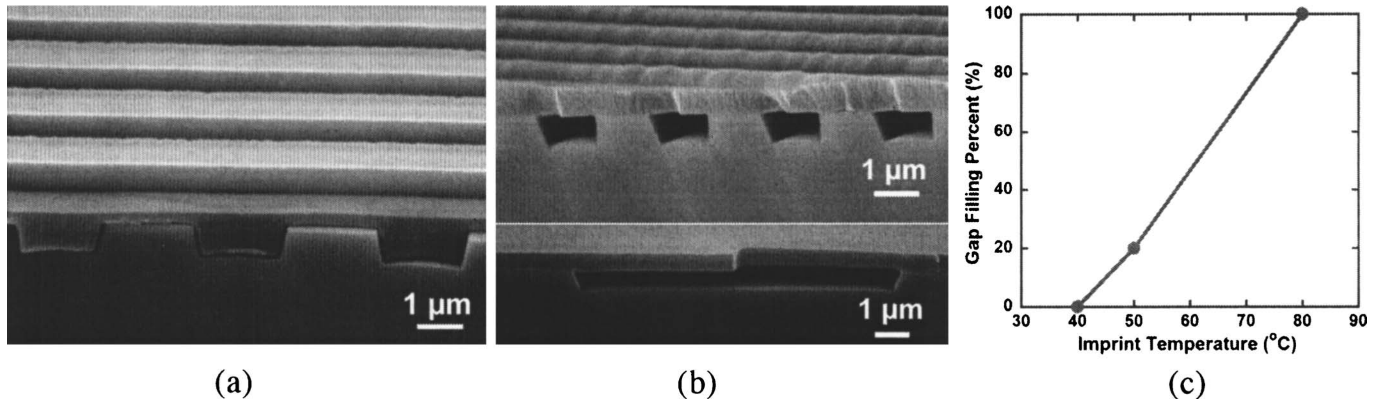


FIG. 3. SU-8 structures transferred onto a Si grating substrate with 1 μm depth: (a) SU-8 heated at 70 °C during imprint for 3 min, (b) SU-8 heated at 40 °C for 3 min, and (c) amount of SU-8 flow into the Si grating as a function of temperature.

C. Multiple-level SU-8 structures

By repeating the reversal UV imprint to stack SU-8 structures, multiple-layer 3D structures can be fabricated. However, SU-8 structures formed by the imprint have the surface energy of 28.0 mJ/m^2 which could cause low SU-8 transfer yield when using a second reversal UV imprint to transfer another SU-8 layer on top. For successful stacking, the SU-8 substrate was treated with an O_2 plasma to increase surface energy. Figure 5 shows the surface energy of SU-8 substrate as a function of the O_2 plasma exposure time. The SU-8 was exposed to an O_2 plasma at a pressure of 100 mTorr, a power of 80 W, and with no dc bias. We found that a 30 s treatment is sufficient to change the SU-8 surface energy from 28.0 to 72.0 mJ/m^2. Longer treatment had little effect on the surface energy but resulted in more removal of SU-8.

With the O_2 plasma treatment of SU-8, we could successfully transfer SU-8 structures on top of previously imprinted SU-8 layer to form 3D structures. Figure 6 shows two-level SU-8 gratings to form completely sealed channels. In Fig. 6(a), the bottom SU-8 layer was UV exposed for 10 s, while the top layer was exposed for 1 s. The different exposure times for the top and bottom layers caused different degrees of SU-8 cross-linking and resulted in different mechanical hardnesses. Therefore, the interface between the two layers can be clearly observed. The reversal UV imprint conditions were SU-8 heated at 40 °C for 3 min, then imprinted at 50 °C and 1 MPa, and followed by a 1–10 s UV exposure. Figure 6(b) shows 400 nm wide sealed SU-8 nanochannels by reversal UV imprint at 55 °C using the same mold as in Fig. 6(a). The SU-8 was heated at 45 °C for 3 min before imprint, and it caused more SU-8 flow into the bottom SU-8 structures. Sealed channels became narrower, reducing their width from 1 μm to 350 nm. For the channels shown in Fig. 6(b), both SU-8 layers were UV exposed for 1 s at 55 °C and 1 MPa. Since their mechanical hardness and degree of cross-linking were similar, the two layers merged together smoothly and the interface could not be distinguished.

Figure 7 shows two levels of SU-8 sealed channels formed by three layers of SU-8 gratings transferred from the same glass mold using the reversal UV imprint. The bottom layer SU-8 grating was UV exposed for 10 s while the middle layer and the top layers were exposed for 1 s. The top two layers merged together similar to what is shown in Fig. 6(b). Fabrication of each level of sealed channels needs only a single process, where no sacrificial layer or etching is involved. Imprint temperature, imprint time, and UV exposure dose are the key factors to control the channel dimensions.

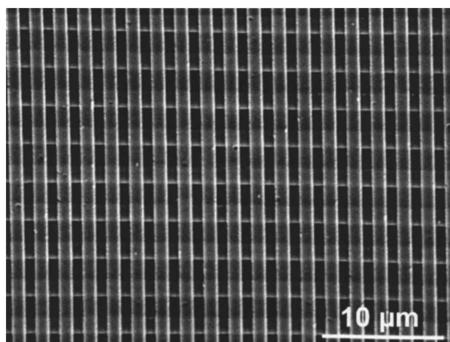


FIG. 4. 1 μm wide, 600 nm deep SU-8 gratings bridging on Si grating substrate after SU-8 residue removal.

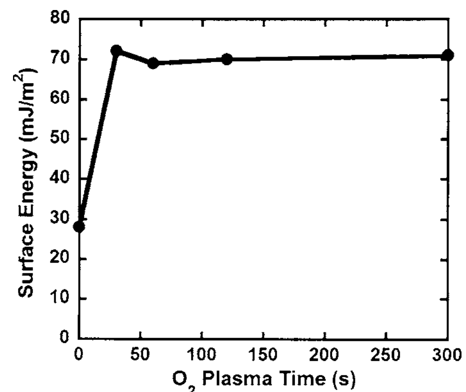
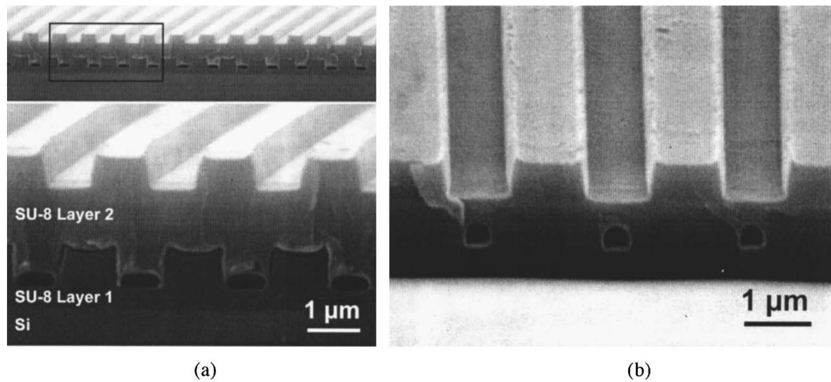


FIG. 5. Surface energy of cured SU-8 as a function of O_2 plasma exposure time.



(a) (b)

FIG. 6. Sealed SU-8 channels formed by reversal UV imprint of two layers of SU-8 grating structures: (a) imprinted at 50 °C, 1 MPa, top layer UV exposed for 1 s, bottom layer UV exposed for 10 s and (b) imprinted at 55 °C, 1 MPa, both layers UV exposed for 1 s.

For our imprinter, since the temperature variation across a 4 in. wafer is only 0.1 °C, the channel dimension can be precisely controlled with good uniformity.

IV. SUMMARY

We have developed a reversal UV imprint technique for the fabrication of 3D SU-8 micro- and nanoscale structures. The key word "reversal" refers to the SU-8 spin coating on the glass mold instead of on the substrate. For this purpose, the mold surface was modified with the combination of PETS and FDTS to obtain the suitable surface energy to enable uniform SU-8 spin coating and also successful mold release after imprint. Besides the silane coatings, exposure to an O_2 plasma is also effective in changing the surface energies of the glass molds, Si substrates, and SU-8 patterns. Using reversal UV imprint, multiple-level SU-8 nanochannels, grating structures, and cavities were fabricated with good controllability. These 3D SU-8 structures can serve as excellent permanent parts for devices with good mechanical strength. Since SU-8 is transparent, these sealed channels allow optical observation of particles or molecules flowing inside the channels. The reversal UV imprint offers a flexible and versatile technique for the fabrication of 3D micro- and nanoscale polymer structures at low temperature and low pressure, which can be used for a variety of applications in biosensing and analysis, photonic devices, and tissue engineering.

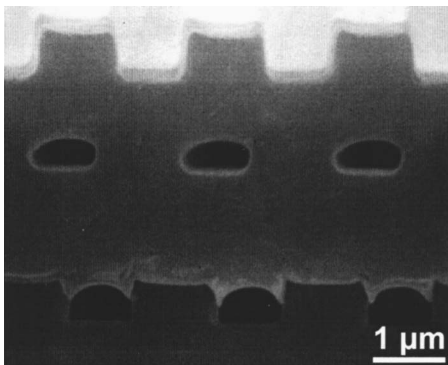


FIG. 7. Two layers of SU-8 grating structures forming two levels of sealed SU-8 channels. Imprints were carried out at 50 °C and 1 MPa. The top and middle layers were UV exposed for 1 s and the bottom layer was UV exposed for 10 s.

ACKNOWLEDGMENTS

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