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Using imprinting technology to fabricate three-dimensional devices from moulds of thermosetting polymer patterns

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Abstract
The fabrication of moulds for imprinting can be simplified significantly by using specialized cross-linking polymers to define the pattern on a silicon wafer. Thermosetting polymers (SU-8) can be used to pattern silicon moulds for imprinting technologies because (1) silicon oxide moulds bearing a thermosetting polymer pattern can be obtained using conventional semiconductor technologies and (2) thermosetting polymers have no obvious glass transition temperature (T_g) because of their cross-linked structure, but the hardness decreases significantly when the temperature is above the T_g. In this study, we used Su-8 resist as the thermosetting polymer pattern to obtain moulds on a silicon wafer. We have tested the thermal properties of thermosetting (SU-8) and thermoplastic polymers (22A4) for use as imprinting patterns and imprinted resists. We fabricated a hill-like structure by applying an electron beam strategy and used this thick film to increase the adhesion between the pattern and the silicon wafer. We used scanning electron microscopy to investigate the resolution of the thermoplastic polymer resist (22A4) pattern that we imprinted using the thermosetting polymer (SU-8) pattern. To define the feature size after imprinting, we determined the feature size shrink factor after separation of the thermosetting polymer pattern (SU-8) from the thermoplastic polymer (22A4) resist. In addition, we have fabricated a microlens of polydimethylsiloxane (PDMS) through replication using the thermoplastic polymer resist (22A4) obtained after imprinting the mould with the microlens structure of the thermosetting polymer (SU-8).

1. Introduction

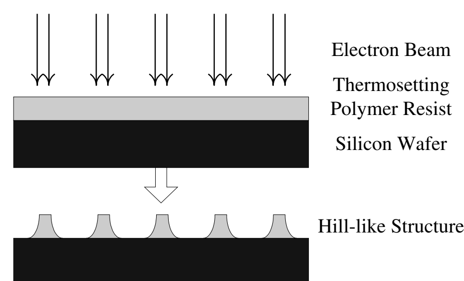
The application of nanofabrication to printing processes has the potential to develop into a competitive parallel process that may serve the future technological demands of nanoelectronics and related areas. Nanoimprint lithography (NIL) is a very useful technique for fabricating various nanostructure devices, such as quantized magnetic discs [1–4]. The resist pattern is fabricated by deforming the physical shape of the resist. The technique has excellent resolution: pattern possessing feature sizes >10 nm within a large area can be realized in high throughput and at a low cost. A conventional mould is usually delineated using electron beam lithography and dry etching, which usually results in a two-dimensional pattern.

Thermal imprint lithography is a promising method for fabricating integrated fine patterns using a variety of materials [5]. Two most important properties of polymeric materials are the melt transition temperature (T_m) of crystalline polymers and the glass transition temperature (T_g) of amorphous phase. The temperature at which a hard glassy polymer becomes a rubbery (softening) material is called the glass transition temperature (T_g). The T_m is the melting temperature of the crystalline domains of a polymer sample. In this process, a thermoplastic polymer is heated above its glass transition temperature (T_g) and then a fine mould is pressed on the polymer. After cooling to below its T_g, the mould is released and the fine pattern on the mould is transferred to the polymer. The fabrication of high-aspect-ratio [6] and curved cross-sectional patterns [7] and the transfer of fine patterns onto novel plastic plates [8] have been reported using thermal imprint processes. The residual resist layer in the compressed areas is finally removed from the substrate by oxygen plasma etching.

The moulds used in NIL are usually fabricated by a quartz or silicon wafer through a multi-step process. A double resist layer is spin-coated on a wafer and patterned by e-beam lithography. Chromium deposition, lift-off and reactive ion etching of the chromium mask are performed to define the stamp pattern [9]. The surface of quartz or silicon usually forms a layer of silicon oxide due to the presence of oxygen and water in the air. In order to improve mould quality and smooth the mould separation process for imprint, certain low surface energy monolayer materials have been used to coat and react with the silicon oxide on the surface. Trichlorosilane (R-Si-Cl_3) is used to react with the silicon oxide on the silicon surface. The alkyl fluoride functional groups of the trichlorosilane are able to self-assemble on the surface to form a thin layer of lower surface energy coating of the silicon oxide. The mould with lower surface energy can reduce sticky problem and thus enhances the quality of patterns after the mould separation process in nanoimprint.

Cross-linking plastics, so-called thermosetting polymers, are being used increasingly as engineering materials because of their excellent stability towards elevated temperatures and physical stresses. They are dimensionally stable under a wide variety of conditions because of their rigid network structures. Such polymers will not flow when heated above their T_g, unlike thermoplastic polymers, which tend to soften and flow when heated. Therefore, thermosetting polymers are suitable moulds for imprinting. In this case, the imprinting must be performed on the thermoplastic polymer surface by using the thermosetting polymer as the pattern.

In this paper, we describe the fabrication of hill-like structures using e-beam lithography to increase the adhesiveness between the thermosetting polymer pattern and the silicon wafer. Such a hill-like structure prevents lift-off of the thermosetting polymer pattern from the silicon wafer when it is separated from the thermoplastic polymer resist. In addition, we demonstrate the fabrication of working moulds using the thermosetting polymer pattern and their applications for NIL, such as the preparation of a microlens of PDMS.



Scheme 1. The hill-like structure fabricated using electron beam strategy for a thick film.

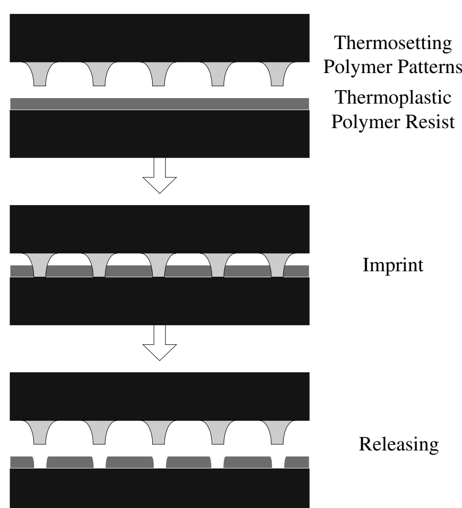
2. Experimental details

2.1. Thermosetting polymer pattern (SU-8)

SU-8 resist obtained from the Microchem Company (Massachusetts) was used to fabricate the thermosetting polymer pattern (SU-8) with the aid of an electron beam. The ingredients of the resist, provided from the vendor, are 50–70% epoxy resin, 25–50% γ-butyrolactone, and 1–5% propylene carbonate and triarylsulfonium hexafluoroantimonate salts. Electron beam exposure was performed using a Leica Weprint Model-200 stepper (Jena, Germany). The shaped electron beam energy was 40 kV, with a beam size of 20 nm and a beam current of 40 A cm^{-2}. The developer for the SU-8 resist was a 98–100% 1-methoxy-2-propyl acetate solution. The SU-8 resist was spun onto a 6 inch silicon wafer, with resist thickness of 3.5 μm, at a spin rate of 6000 rpm for 0.5 min, followed by soft-baking for 10 min at 65 °C and then for 2 min at 90 °C. The glass transition temperature was measured using a differential scanning calorimeter (Seiko SSC-5000) and the stress variation of the thin film coated onto the substrate during the heating procedure of the imprinting process was obtained from wafer curvature measurement (Tencor FLX-2320). The feature size was evaluated using cross-sectional SEM (Hitachi S-4000).

2.2. Mould with thermosetting polymer (SU-8) pattern fabrication

SU-8 resist, a thermosetting polymer resist, was exposed to an electron beam at a dosage of 15 μC cm^{-2}, followed by developing and hard-baking processes for 10 min at 105 °C. In the case of thick film exposure, multi-layer structures can be fabricated through e-beam exposure, which results in hill-like structures due to the scattering effect illustrated in scheme 1. When the photo-resist layer is exposed to the e-beam, the thickness scale is greater than the focus of the e-beam. The resist around the pattern is exposed by such a defocused e-beam and results in a hill-like structure around the pattern. The resist around the pattern is exposed by the insufficient dosage of the defocused e-beam, and thus results in incomplete thickness around the pattern after development. Therefore, the pattern exposed by e-beam for thick resists tends to form the hill-like structure (multi-layer structure) [10]. Trichloro(1*H*,1*H*,2*H*,2*H*- perfluorooctyl)silane (FOTS; Aldrich, used as received) was used as the precursor for obtaining the self-assembled monolayers (SAM) on the mould,



Scheme 2. The imprint process by using a mould with a hill-like structure of thermosetting polymer.

with the thermosetting polymer pattern as the mould-releasing and anti-sticking layer for imprinting.

2.3. Imprint process

22A4 resist (Sumitomo Chemical Company, Japan) was used for imprinting. The success of using combinations of the resist and mould surface modifications can be demonstrated using an imprint tool developed by Nanonex (nx-1000, US). For multiple imprint investigations, an experimental series of 50 imprints is printed continuously using one mould on two silicon wafers. Each of these two wafers is printed with 25 imprints at a spacing of 1 mm. The mould featuring the thermosetting polymer pattern has a size of 1×1 cm^2 and contains the test structures at a depth of 2 μm. These feature structures consist of lines and dots. The contact between the resist and mould had preprint pressure of 200 psi at a preprint temperature of 110 °C, and then an imprint force of 380 psi was used to press the mould into a 700 nm thick resist for a duration of 5 min at 130 °C. Scheme 2 illustrates the imprinting processes.

2.4. Surface energy calculations

Surface energy is directly related to the adhesiveness between the thermosetting polymer pattern and the thermoplastic polymer resist; it is evaluated using the Lifshitz–van der Waals acid/base approach (three-liquid acid/base method) previously proposed by van Oss *et al* [11, 12]. This methodology introduces a new concept of 'apolar' (Lifshitz–van der Waals, γ^{LW}) and 'polar' (Lewis acid/base, γ^{AB}) surface; the latter cannot be represented by a single parameter such as γ^{p}. Briefly, the theoretical approach follows the additive concept suggested by Fowkes [13],

$$\gamma = \gamma^{d} + \gamma^{AB} \tag{1}$$

where γ^{d} represents the dispersive term of the surface tension. The superscript AB refers to acid/base interaction. By

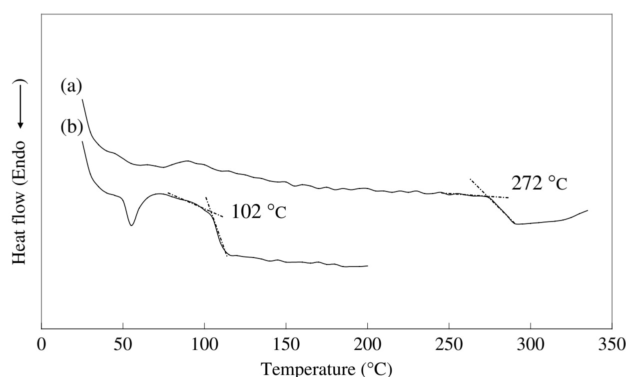


Figure 1. DSC curves of the chemically amplified resists. (*a*) Thermosetting polymer (SU-8); (*b*) thermoplastic polymer (22A4).

regrouping the various components in equation (1), the surface energy can be expressed as

$$\gamma = \gamma^{LW} + \gamma^{AB}. \tag{2}$$

In addition, two parameters are created to describe the strength of the Lewis acid and base interactions:

- γ_s^+ = Lewis acid parameter of surface free energy,
- γ_s^- = Lewis base parameter of surface free energy,

$$\gamma^{AB} = 2\left(\gamma_s^+\gamma_s^-\right)^{1/2}. \tag{3}$$

Van Oss, Good and their coworkers [11, 12] developed a 'three-liquid procedure' (equation (4)) to determine γ_s by using the contact angle technique:

$$\gamma_L(1+\cos\theta) = 2\left[\left(\gamma_s^{LW}\gamma^{LW}{}_L\right)^{1/2} + \left(\gamma_s^+\gamma_L^-\right)^{1/2} + \left(\gamma_s^-\gamma_L^+\right)^{1/2}\right]. \tag{4}$$

To determine the components of γ_s of a polymer solid, three liquids are needed: two of them are polar and the third is apolar. Polar pairs of water and ethylene glycol or water and formamide are recommended to provide the best results. The apolar liquid is either diiodomethane or r-bromonaphthalene because the Lewis acid and Lewis base parameters of these liquids are readily available. The values of the LW, Lewis acid and Lewis base parameters of γ_s can then be determined by solving these three equations simultaneously. By measuring contact angles for these three well-characterized (in terms of γ_s^{LW}, γ_L^+ and γ_L^- [14, 15]) liquids, three equations with three unknowns are generated. Water, diiodomethane and ethylene glycol were the three liquids that we employed in this study.

3. Results and discussion

3.1. Glass transition temperature measurement

The imprint technique is based on the diversity of glass transition temperatures (T_g) between the thermosetting polymer pattern (SU-8) and the thermoplastic polymer resist (22A4). Therefore, it is important to measure the phase transition temperature of the resist material. In this study, we used the conventional DSC method to determine the glass transition temperatures of the thermosetting polymer pattern (SU-8) and the thermoplastic polymer resist (22A4) used for imprinting applications. Figure 1 displays the DSC curves of

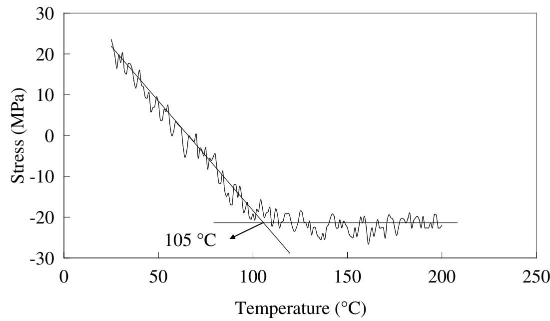


Figure 2. Plot of stress versus temperature for the thermoplastic polymer resist (22A4).

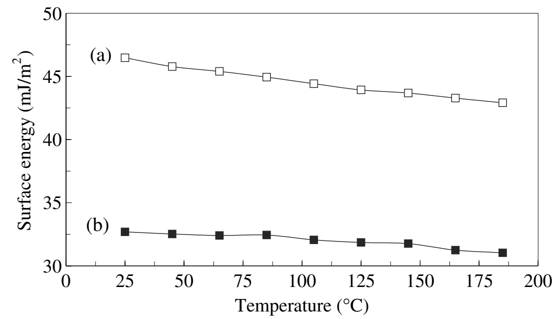


Figure 3. Plot of surface energy versus temperature of (*a*) the thermoplastic polymer resist (22A4) and (*b*) the thermosetting polymer (SU-8). Surface energies were calculated from the contact angles of diiodomethane, ethylene glycol and water as a function of the annealing temperature.

the thermosetting polymer (SU-8) and the thermoplastic resist (22A4); the endothermic peak at about 50 °C is probably due to the endothermic effect of the resist additive. The endothermic peak at about 102 °C is caused by the glass transition of the 22A4. The glass transition temperature of the SU-8 is not clearly defined from the DSC curve because of the nature of the cross-linked structure. The glass transition temperature of the thermosetting polymer is close to 272 °C, which is significantly higher than the value of T_g of the 22A4. Therefore, the SU-8 pattern remains a high-modulus solid at 150 °C, the imprint temperature.

Another method for determining phase transition temperatures is wafer curvature measurement (WCM), which is usually performed using a stress measurement instrument to obtain the radius of curvature and stress variation of a thin film coated on the substrate during a predetermined heating procedure [16]. In our experiment, we performed stress measurement using the SU-8 and 22A4 resist coated on a silicon substrate. The heating rate was maintained at 10 °C min^{-1} and the ambient gas was air. As presented in figure 2, the stress for the thermoplastic polymer resist varied from −30 to 30 MPa. Figure 2 indicates that the phase transition temperature of the thermoplastic polymer resist is about 105 °C. The value of T_g measured by this WCM method is only slightly different from that obtained from DSC analysis for the 22A4 resist. As mentioned above, the glass transition temperature for the SU-8 cannot be detected when using this WCM method because of its cross-linked structure. This thermosetting polymer exhibits a stable stress when the temperature was increased >100 °C. The DSC technique measures the calorimetric transition; in contrast, the WCM technique detects the mechanical relaxation transition.

3.2. Contact angle and surface free energy

Adhesiveness is a feature of the separation process during nanoimprinting (NIL). Strong adhesion may arise from capillary, electrostatic and van der Waals forces and, in some cases, from hydrogen bonding interactions. During imprinting, the transfer of the pattern from the mould to the resist on the substrate requires a completely anti-wetting surface of the mould to minimize any defects induced by adhesiveness. The value of the surface energy is commonly used as an indicator of the adhesive characteristics of materials [17, 18]. Table 1 displays the values of γ_L^{LW}, γ_L^{+} and γ_L^{-} that we calculated using the contact angles of water,

Table 1. Surface tension parameters calculated from the advancing contact angles of water, diiodomethane (DIM) and ethylene glycol (EG) on a thermosetting polymer (SU-8) and a thermoplastic polymer resist as a function of temperature.

	Contact angles for testing liquid (°)				Surface energy (mN m^{-1})			
Temperature (°C)	Category	DIM	Water	EG	γ_s^{LW}	γ_s^{-}	γ_s^{+}	γ_s
25	22A4	59.1	81.1	49.1	40.21	4.80	2.04	46.47
	SU-8	38.8	71.6	51.0	29.09	0.46	7.08	32.68
45	22A4	59.3	81.4	49.7	39.56	4.63	2.09	45.78
	SU-8	40.1	72.2	51.6	28.98	0.45	6.95	32.52
65	22A4	59.7	81.5	50.2	39.36	4.43	2.06	45.40
	SU-8	40.5	72.8	52.2	28.75	0.49	6.86	32.40
85	22A4	60.0	81.6	51.1	38.95	4.35	2.06	44.94
	SU-8	41.3	73.2	52.7	28.58	0.57	6.58	32.44
105	22A4	60.2	82.0	51.2	38.69	4.00	2.05	44.42
	SU-8	41.8	74.1	53.4	28.46	0.48	6.67	32.06
125	22A4	60.6	82.1	51.4	38.28	3.86	2.07	43.93
	SU-8	42.6	74.6	53.9	28.23	0.49	6.69	31.85
145	22A4	60.7	82.3	51.8	38.17	3.80	1.99	43.68
	SU-8	42.8	75.0	54.5	28.17	0.49	6.60	31.76
165	22A4	61.0	82.8	51.9	37.86	3.76	1.96	43.28
	SU-8	43.4	75.4	55.1	28.00	0.39	6.73	31.24
185	22A4	61.3	83.0	52.2	37.60	3.74	1.89	42.91
	SU-8	43.9	75.8	55.8	27.83	0.38	6.71	31.03

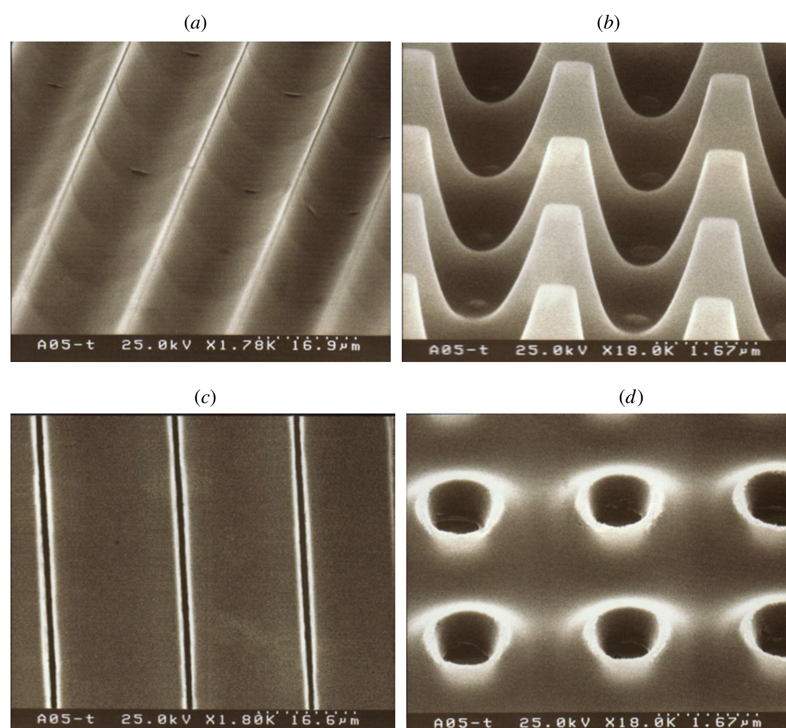


Figure 4. SEM images (top views) of the surface mould of the thermosetting pattern (SU-8) displaying (*a*) isolines and (*b*) dense cylinders, and those of the thermoplastic polymer resist (22A4; 650 nm thickness) imprinted using the mould of the thermosetting pattern (SU-8) displaying (*c*) isotrenches and (*d*) dense contact holes.

diiodomethane and ethylene, which we then used to calculate the respective surface energies. Figure 3 displays plots of the critical surface energy as a function of temperature. In general, the surface energy of a polymer decreases only slightly upon increasing the temperature [16, 17]. The surface energy of the thermoplastic polymer resist decreased upon increasing the temperature; its surface energy was about 43.5 mJ m^{-2} at 150 °C, i.e., the imprint temperature. This result indicates that the SU-8 is suitable for imprinting applications.

3.3. Mould with thermosetting polymer (SU-8) pattern and the imprinted pattern (22A4)

Figure 4 displays SEM micrographs of the mould presenting the SU-8 pattern. The SU-8 pattern of dense lines possesses hill-like structures for fabricating trench patterns during the imprinting process, as demonstrated in figure 4(*a*). The formation of such hill-like structures in the SU-8 is caused by the scattering effect of the electron beam [18]. The hill-like structures of the thermosetting polymer increase the contact area between the pattern and the silicon wafer surface, which results in increased adhesiveness that prevents lift-off during the separation step of the imprinting process. The feature size at the top of the hill-like structure of these dense lines is about 835 nm. Figure 4(*b*) displays the hill-like structures of the thermosetting polymer; the dense cylinders fabricate contact holes during the imprint process. The heights of the hill-like patterns for preparing isolines and cylinders were about 3.5 μm.

Figures 4(*c*) and (*d*) display SEM micrographs of the 22A4 resist (650 nm thickness) after separation from the mould of the SU-8 pattern of hill-like isolines and dense cylinders

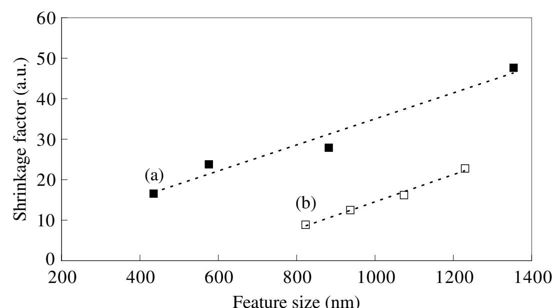


Figure 5. The shrinkage factor as a function of the feature size of the thermosetting polymer pattern on a mould for (*a*) isotrenches and (*b*) dense contact holes.

prepared under a typical process temperature of about 150 °C and a typical pressure of about 100 bar. The SU-8 pattern for the negative tone transfers the feature to the 22A4 resist for the positive tone, which results in the pattern of isotrenches and dense contact holes. The feature sizes of the isotrenches and dense contact holes on the 22A4 resist surface are smaller than those of the isolines and dense cylinders on the mould of the SU-8 pattern. During hot embossing lithography, the feature size of the 22A4 resist shrinks slightly after cooling below its value of T_g, while the feature size of the pattern on the mould remains close to constant because the thermal extension coefficient of the silicon wafer mould is significantly lower than that of the resist. In this study, we observed that both the SU-8 and 22A4 shrank upon cooling, which resulted in tight contact between them. The adhesion between the SU-8 and 22A4 drives the thermoplastic polymer to shrink

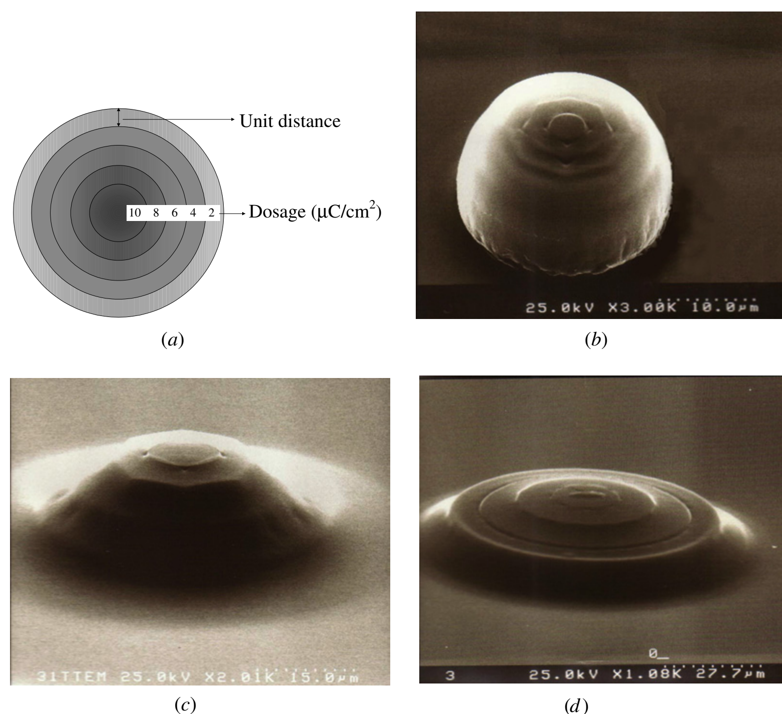
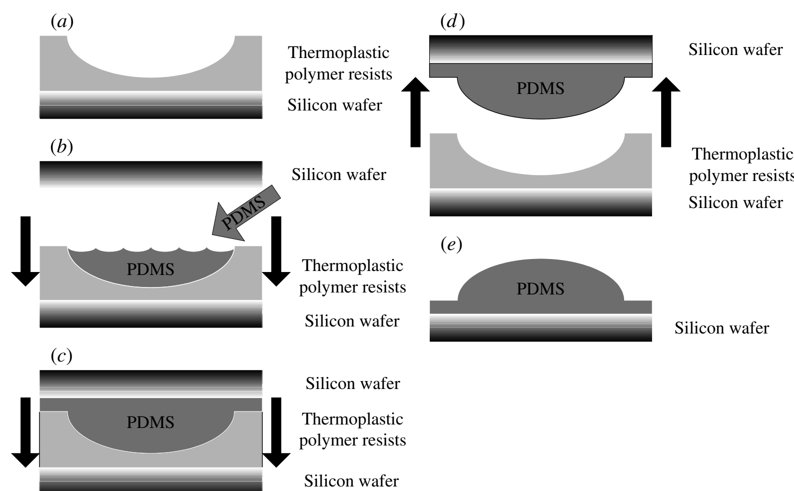


Figure 6. (*a*) The strategy of exposure to an electron beam to fabricate microlenses of the thermosetting polymer. (*b*) SEM images (45° tilt) of the thermosetting polymer microlenses for the mould obtained before imprinting when the unit distance was (*b*) 3 μm, (*c*) 5 μm and (*d*) 7 μm.

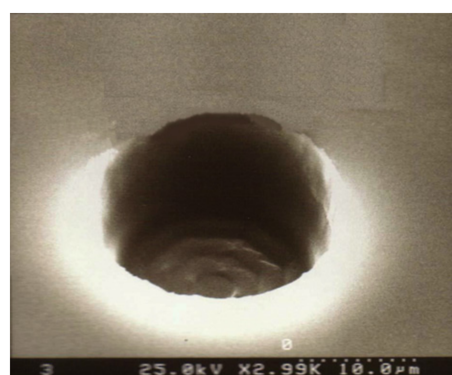


Scheme 3. PDMS microlens fabrication. (*a*) Thermoplastic polymer resist imprinted using a mould of the thermosetting microlens. (*b*) PDMS cast onto the thermoplastic polymer resist imprinted using the mould of the thermosetting microlens. (*c*) Pressing and baking PDMS using an NIL apparatus. (*d*) Removal of the Si wafer from 22A4. (*e*) PDMS microlens.

its feature sizes when the SU-8 pattern separates from it. A useful relative measure of the shrink factor can be calculated from the relationship $f = 1 - (d_t/d_l)$, where d_t is the feature size of an isotrench on the 22A4 surface and d_l is the feature size of an isoline for the SU-8. Figure 5 demonstrates that the shrink factor (f) increases upon increasing the feature size of the isolines of the SU-8. This observation suggests that the adhesion between the SU-8 and 22A4 increases upon increasing the contact area; a larger-feature-size pattern possesses a larger contact area and results in a larger value of its shrink factor. The total contact area between the SU-8 and 22A4 for imprinting a trench is larger than that required to imprint a single hole; thus, an imprinted trench possesses a larger value of shrink factor.

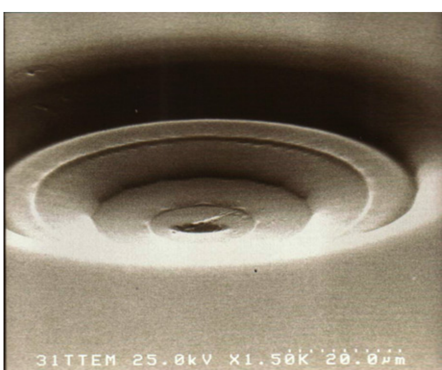
3.4. Fabrication of a microlens

Three-dimensional (3D) structures are required in various optical devices (e.g., microlenses and photonic crystals). Electron beam lithography is very useful for fabricating 3D structures, as we demonstrated previously with the preparation of hill-like structures [10]. Figure 6(*a*) presents a scanning



(*a*)

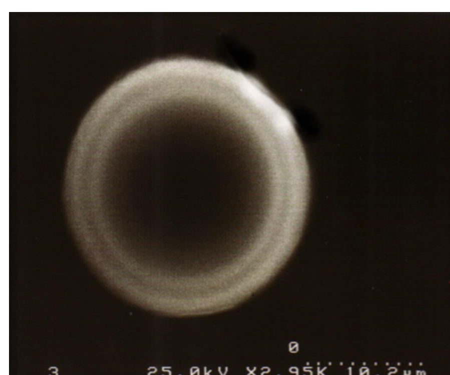
(*b*)



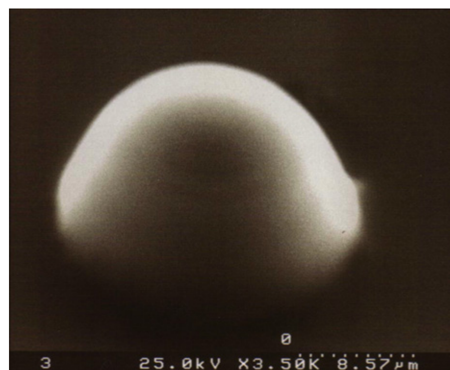
(*c*)

Figure 7. The thermoplastic polymer resists (22A4) having 3.3 μm thickness imprinted using the thermosetting microlens moulds. SEM images (40° tilt) of the thermoplastic polymer resist imprint of the thermosetting microlens at unit distances of (*a*) 3 μm, (*c*) 5 μm and (*d*) 7 μm.

electron microscopy (SEM) image of microlenses of the SU-8 that were fabricated using a strategy of electron beam exposure on a silicon wafer. Figures 6(*b*)–(*d*) display various SU-8 microlenses fabricated by dividing a circular pattern into five sections, each possessing the same centre of a circle, and then exposing the five sections to electron beams of 10, 8, 6, 4 and 2 μC cm^{-2} dosage, respectively. The unit distances of the microlenses presented in figures 6(*b*)–(*d*) are 3, 5 and 7 μm, which results in their possessing radii of 12.5, 17.6 and 33.8 μm, respectively. The curvature radius of the microlenses increases upon increasing the unit distance. Figures 7(*a*)–(*c*) display SEM images of the 22A4 resists imprinted with the



(*a*)



(*b*)

Figure 8. (*a*) Top-view and (*b*) 40° tilt SEM images of a PDMS microlens fabricated using the reversal mould of the thermoplastic polymer resist at a unit distance of 3 μm.

microlens patterns of various curvature radii that are presented in figures 6(*b*)–(*d*), respectively.

Polydimethylsiloxane (PDMS) is used in soft lithography techniques as a replication material [22, 23]. Soft lithography is a non-photolithographic fabrication process based on self-assembly and replicated moulding. It provides a convenient, effective and low-cost method for forming and manufacturing micro- and nanostructures having feature sizes ranging from 50 nm to 100 mm. In this study, we obtained a 22A4 resist containing a reverse microlens pattern, as indicated in figure 7(*a*), by imprinting onto PDMS coated on a silicon wafer by using an NIL apparatus. The 22A4 surface was precoated with a thin layer of silane-coupling agent to reduce the critical surface tension.

Scheme 3 displays a schematic of the fabrication process of the PDMS microlens using the NIL apparatus. We prepared the reverse microlens mould of the 22A4 resist by using the imprinting process described in section 3.3. The PDMS was cast onto the reverse microlens mould, which was then pressed onto a bare silicon wafer using the NIL apparatus. The PDMS film thickness was controlled by the imprinting pressure. The PDMS was baked at 75 °C for 15 min during pressing. After pressing, the PDMS, which became stuck to the upper bare silicon wafer, was removed from the 22A4 resist reverse microlens mould to fabricate the PDMS microlens. Figures 8(*a*) and (*b*) display top- and side-view 3D SEM images, respectively, of the PDMS microlens. The radius and height were 10.2 and 3.2 μm, respectively; these values

are nearly identical to those of the reverse microlens mould of the 22A4 resist on the silicon wafer.

4. Conclusions

We fabricated the mould pattern of a thermosetting polymer (SU-8) using an imprint process to simplify the processes of etching and resist removal during mould fabrication. Hill-like structures fabricated in a thick film by an electron beam prevented the thermosetting polymer (SU-8) pattern from lifting off from the silicon wafer surface when it was separated from the thermoplastic polymer resist (22A4). We fabricated two- and three-dimensional patterns by using thermoplastic polymer resists (22A4) of 650 nm and 3.3 μm dimensions for imprinting the mould with the thermosetting polymer (SU-8) pattern. In the 2D imprinting process, we observed a shrinking effect after the SU-8 pattern separated from the 22A4 resist. We have demonstrated that three-dimensional microlens mould may be fabricated using an electron beam exposure strategy. We delineated various 3D microlens moulds by increasing the unit distance for exposure to the electron bean; the mould was then imprinted onto the thick thermoplastic polymer resist (22A4) using an NIL apparatus. The 3D mould obtained after the NIL process maintained its original shape; we then successfully imprinted 3D structures onto the 22A4 resist. These results reveal that the 3D mould fabricated by electron beam lithography can be applied to the NIL. Furthermore, we fabricated a PDMS microlens by using a 22A4 resist reversal microlens mould. In this study, we have demonstrated that a mould containing a thermosetting polymer (SU-8) pattern may be very useful for fabricating 2D patterns or 3D optical devices such as microlenses.

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