

# Fabrication of size controllable SU-8 nanochannels using nanoimprint lithography and low-pressure thermal bonding methods

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A new and simple method is demonstrated to achieve size controllable nanochannels by using nanoimprint lithography and low-pressure thermal bonding methods. The flexible free-standing SU-8 allows the bonding of the nanochannels' pattern features at such a low pressure primarily because of 'sequential' bonding made possible by the bonding layer flexibility and the conformal contact made between the bonding layer and the structure layer in a large area. The simple geometrical argument shows that the height of the enclosed nanochannel can be determined by the depth of the cross-linked SU-8 trenches as well as by the initial thickness of the thin unexposed SU-8 layer. The height of the nanochannels can also be controlled by adjusting the ratio of the ridge width to the trench width on the SU-8 trenches. These simple relationships can serve as a guideline in choosing the parameters for SU-8 nanochannel fabrication.

**1. Introduction:** In the newly emerging field of bionanotechnology, further downsizing of the fluidic channels to a nanometre scale is attractive for both fundamental studies, such as fluid transport and molecular behaviour at extremely small dimensions, and in technical applications such as manipulation and high sensitivity detection of single molecules for biosensing, chemical analysis and medical diagnostics. Since the dimensions of the microchannels go down to the nanometre scale, not only a higher integration level and a faster analytical speed can be achieved but also some extraordinary properties such as reduced electro-osmotic flow, increased viscosity, overlapping electric double layer, ion enrichment as well as depletion emerge is also increased because the surface effects become comparable or even dominant to the bulk effects inside the nanochannels. There have been many efforts to scale down and control the width of the nanochannels.

In creating low-cost nanofluidic channels for single biomolecule analysis, the fundamental challenges are to reduce and control the size of the nanoscale trenches and seal these trenches to complete the functional nanofluidic devices [1]. Although the traditional electron beam lithography and focused ion beam milling techniques have a high resolution in generating nanoscale structures, both technologies have the disadvantages of a low throughput and costly processes with limited yields. One widely used scheme to reduce and control the dimensions of the nanochannels is silicon grating oxidation. Several novel and unconventional methods such as size reduction lithography [2], edge lithography [3] and nanopattern oxidation [4] utilise various forms of this technique to reduce the dimensions during or after the pattern transfer step. However, these techniques suffer from limitations such as high voltage, high temperature, an absolutely defect free and flat surface which results in a low throughput and a high cost and oxidation non-uniformities resulting in a high line edge roughness. Moreover, the oxidation-based techniques are not applicable to materials other than silicon, or organic materials. In contrast, nanoimprint lithography (NIL) is a parallel high-resolution technique that makes it possible to create nanometre-scale features over large substrate surface areas at a high throughput and low cost. Among the polymeric materials that have been used for NIL, the SU-8 photoresist is a highly

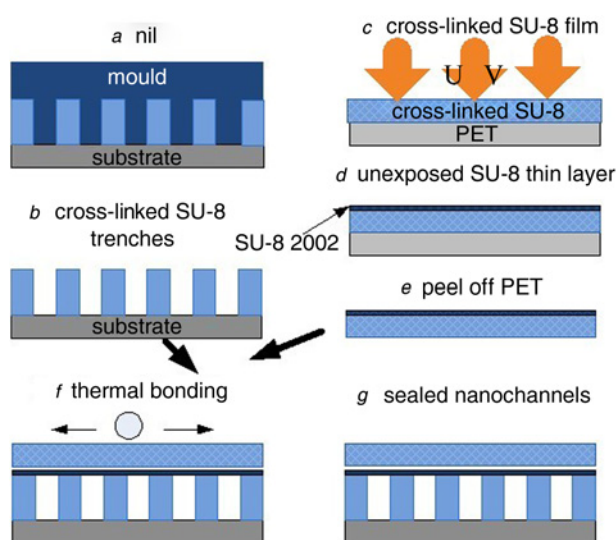
desirable polymer for making micro/nanofluidic channels because of its good chemical resistance, optical transparency and excellent capability for micro/nanofabrication. UV cured nanoimprint of SU-8 has been well developed to fabricate nanostructures with a high resolution and low cost. To date, direct thermal bonding [5] approaches are especially desirable as they allowed the formation of enclosed SU-8 micro/nanochannels over entire surfaces composed of the same polymeric material and introduced no foreign material. In the process, bonding temperatures are kept just over glass transition temperature, and a relatively high bonding pressure is applied to provide a close contact between the structure layer and the bonding layer. However, such a method makes it difficult to obtain a large area uniform micro/nanochannel, because a high bonding pressure may induce global and localised geometric deformation of the substrates or leave an interfacial layer with a significant thickness variation. Furthermore, the nanochannels may be torn apart, clogged or even destroyed by the high pressure because of the uneven pressure distribution over a large area.

The aim of the work reported in this Letter was to develop a novel approach to achieve nanochannels with well-controlled dimensions in a large area by thermal bonding at low pressure. The nanochannels can be prepared by simply imprinting fully cross-linked SU-8 trenches into a thin unexposed SU-8 film cast on a cross-linked SU-8 film in a single step. The high bonding temperature makes the melted thin unexposed SU-8 fill into the SU-8 trenches inevitably and achieves the chain entanglement of the polymer over the boundary by the capillary force. Furthermore, it is easy to control the nanochannel dimensions by a simple relationship involving the initially thin unexposed SU-8 layer thickness and the cross-linked SU-8 trenches' pattern configuration. Besides, our approach is capable of generating an ultra-small line pattern, which goes beyond the limitation of the original height of the template.

**2. Experimental details:** In our fabrication process, high-density arrays of nanofluidic trenches were first fabricated by using NIL. The NIL mould patterns were generated by a laser holographic lithography on photoresist coated silica substrates. By using the patterned resist as an etching mask, reactive ion beam etching is performed by a self-made ion milling machine to form lines/

spaces templates in silica. The etching plasma is a mixture of Ar and  $\text{CHF}_3$  gases in the optimised ratios to harden the photoresist masking layer as well as to passivate the sidewall, as described in our previous paper [6].

Fig. 1 presents the schematic process steps for the fabrication of the size controllable SU-8 nanochannels by nanoimprinting and thermal bonding methods. As per our previous paper and the work of others [7–9], SU-8 is used both as a structural material for the channels and the adhesive bonding material. Firstly, an SU-8 resist with a thickness of about 20  $\mu\text{m}$  was spin-coated on a glass substrate and soft baked at 90°C for 20 min. Then, silica templates were pretreated with Dow Corning 20, made up of polydimethylsiloxane and a high molecular weight silicone resin, to reduce the surface energy for easy separation from SU-8 during the following demoulding process. The imprint of the SU-8 was conducted at 90°C for 15 min using a pressure of 2 MPa (shown in Fig. 1a). In this process, a home-made mechanical press with a screw clamp was used to perform the imprint and the pressure adjusted using a torque wrench. After imprinting and before separating the mould-substrate assembly, the SU-8 resist was exposed to UV light (365 nm wavelength) through the transparent glass substrate to cure the imprinted SU-8 trenches (shown in Fig. 1b). Figs. 1c–e present the schematic process flow for a fabricated free-standing SU-8 bonding layer. In the fabrication process, a flexible polyethylene terephthalate (PET) film was employed as a sacrificial substrate for the bonding layer. Two flat SU-8 layers with thicknesses of about 20 and 50 nm, respectively, were spin-coated consecutively on a PET substrate to serve as the cover layer, where a thin and uncured layer of SU-8 2002 was applied to act as an adhesive layer which also controls the height of the nanochannels and a thick SU-8 2025 layer was fully cured as a supporting layer (shown in Figs. 1c and d). Then, the PET was peeled off from the SU-8 cover layer before bonding to form a flexible free-standing SU-8 bonding layer, as shown in Fig. 1e. The PET film can be reused many times, only needing nitrogen blowing each time it is reused to remove the particles attached from the laboratory environment.



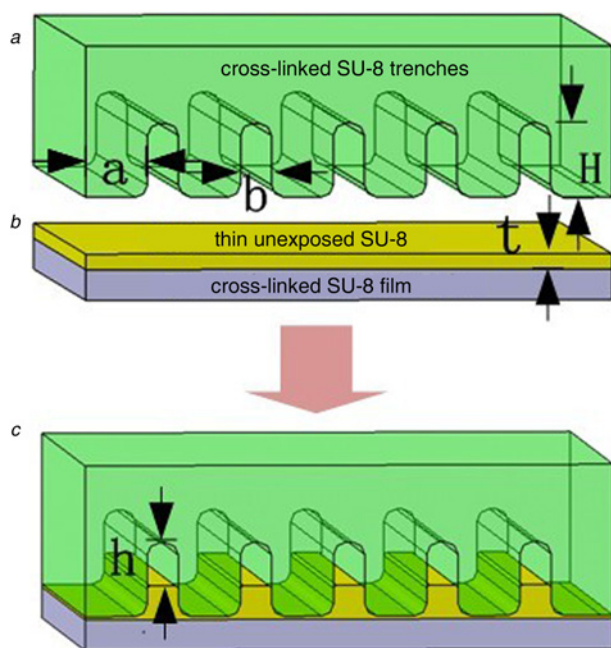
**Figure 1** Schematic process flow for fabrication of size controllable nano-fluidic channels

- a Nil
- b Fabrication of SU-8 trenches
- c Fabrication of SU-8 film
- d Spin-coated unexposed SU-8 thin layer
- e Peel off PET
- f Thermal bonding
- g Sealed nanochannels

For a successful bonding, the SU-8 trenches should be treated with oxygen plasma to increase the surface energy after the demoulding process. Then, the oxygen treated SU-8 trenches and the SU-8 cover layer are aligned and thermally bonded together on a hotplate, as shown in Fig. 1f. After the stage temperature reached the set point (about 80°C), a rounded bottom roller that was made of polydimethylsiloxane (PDMS) was used to uniformly contact the bonding layer with the SU-8 trenches. The rolling contact should be initiated at the centre region and then rolled outward for an efficient removal of the entrapped air. Then, we simply put a stainless steel block weighing 0.25 kg on top of a flat PDMS block, which was inserted between the weighting block and the SU-8 bonding layer. After maintaining a high temperature (above  $T_g$  of the SU-8 (55°C)), typically 80°C, for 10–15 min, followed by UV flood exposure for 1 min to cross-link the thin SU-8 adhesive layer. Therefore, the freestanding SU-8 cover layer is transferred onto an SU-8 fluidic channels substrate, forming sealed fluidic channels, as shown in Fig. 1g.

**3. Results and discussion:** Traditionally, bonding temperatures were kept just over glass transition temperature, and a relatively high bonding pressure was applied to provide a close contact between the structure layer and the bonding layer. However, for a high aspect ratio structure after NIL, the SU-8 trenches may be easily torn apart, deformed, blocked up or even destroyed by high pressure. Besides, such a method makes it difficult to obtain a large area uniform micro/nanochannel, because a high bonding pressure may induce global and localised geometric deformation of the substrates or leave an interfacial layer with a significant thickness variation. Therefore a low-pressure thermal bonding would be possible if a conformal contact can be made between the bonding layer and the substrate and if a sequential, parallel, imprinting of the pattern features on the substrate surface is ensured by some means. One way to achieve the conformal contact and the sequential bonding is to use a flexible bonding layer, as demonstrated in this Letter. The conformal contact and the sequential bonding made possible by the use of a flexible freestanding SU-8 bonding layer are the key to this low-pressure thermal bonding method. A large conformal contact can be easily achieved because of the freestanding SU-8 layer's good flexibility. The use of a rounded roller is another key step during bonding because the rolling process can increase the contact intensity and the uniformity. Owing to the conformal contact and the rolling process between the SU-8 substrate and the bonding layer, the dimensions of the nanochannels can be controlled by an initially thin unexposed SU-8 layer and a cross-linked SU-8 trenches' configuration.

Fig. 2 shows the schematic of the SU-8 nanochannels dimensions controlled by varying the initially thin unexposed SU-8 layer thickness and the cross-linked SU-8 trenches' configuration [10]. As we know, the glass transition temperature ( $T_g$ ) of the unexposed SU-8 is 55°C, whereas the glass transition temperature of the crosslinked SU-8 is more than 200°C. Therefore a bonding temperature of 80°C will accelerate the fluidity of the melting unexposed SU-8 polymer chain filling into the SU-8 trenches and have little effect on the cross-linked SU-8 trenches, as shown in Fig. 2. Fig. 2a shows the key dimensional parameters for the cross-linked SU-8 trenches: 'a' is the SU-8 ridge width; 'b' is the SU-8 trenches' width; 'H' is the trenches' depth; 't' is the initial thickness of the thin unexposed SU-8; and 'h' is the nanochannel height after the bonding process. If a very thin unexposed SU-8 layer 't' is used during thermal bonding, the displaced polymer will not be able to completely fill the cross-linked SU-8 trenches 'H', thereby creating enclosed nanochannel features, as shown in Fig. 2a. Considering that the deformation of the cross-linked SU-8 trenches and the cross-linked SU-8 bonding layer under a bonding temperature of 80°C is very small, and assuming that the thin unexposed SU-8 is incompressible, the nanochannels are obtained by the thin

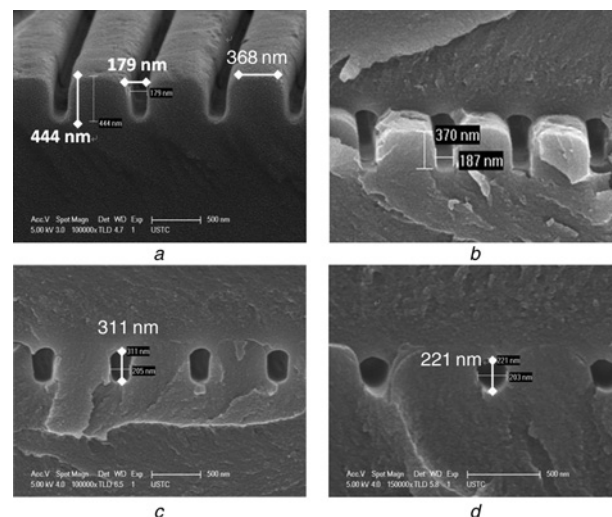


**Figure 2** Thin unexposed SU-8 displacement into SU-8 trenches during bonding process (Fig. 2a); height of enclosed nanochannel can be determined by depth of cross-linked SU-8 trenches as well as by initial thickness of thin unexposed SU-8 layer (Fig. 2b); height of nanochannels can also be controlled by adjusting ratio of ridge width to trench width on SU-8 trenches (Fig. 2c)

unexposed SU-8 displacement during the bonding process. The relationship between them can simply written as  $h = H - t \cdot a/b$ . The simple geometrical argument shows that the height of the enclosed nanochannel can be determined by the depth of the cross-linked SU-8 trenches as well as by the initial thickness of the thin unexposed SU-8 layer, as can be seen in Fig. 2b. Considering the SU-8 trenches' fabrication using NIL, the imprint depth 'H' is equal to the height of the template pattern, which can be easily controlled by adjusting the etching duration during the template fabrication. Fig. 2c shows that the height of the nanochannels can also be controlled by adjusting the ratio of the ridge width to the trench width on the SU-8 trenches. These simple relationships can serve as a guideline in choosing the parameters for the SU-8 nanochannel fabrication.

To demonstrate the easy control of the channel dimensions, we fabricated the NIL mould and the size controllable nanochannels by using the nanoimprinting and the thermal bonding methods, as shown in Fig. 3. Fig. 3a shows the scanning electron microscope (SEM) photograph of the replicated trenches on the SU-8 resist which is in accordance with the size of the NIL mould. The depth of the trenches is approximately 444 nm, the width of the ridge width is 368 nm and the width of the trenches is about 179 nm, respectively. For such high aspect ratio structures, the imprinted SU-8 itself can be directly used as fluidic channels for the applications of biological and chemical analyses. However, following the imprinting, the SU-8 bonding layer must be laminated to the structure substrate to complete the fluidic conduits using the direct thermal bonding approach.

In our bonding process, the SU-8 bonding layer could be peeled off because of the low surface energy of the PET before bonding. The SU-8 foil is free-standing and flexible, and conformal contact with the structure layer is easy to achieve with a rounded-bottom roller to expel the entrapped air. This step was found to be critical to expel the entrapped air and thus achieve conformal contact between the SU-8 trenches and the SU-8 bonding layer. When



**Figure 3** SEM of size controllable nanochannels using different initially thin unexposed SU-8 layer

a Original imprinted SU-8 trenches

b Height of nanochannels is 370 nm and initial unexposed SU-8 is 25 nm

c Height is 311 nm and initial unexposed SU-8 is 50 nm

d Height is 221 nm and initial unexposed SU-8 is 75 nm

the bonding process was performed without this rolling contact, there remained unbonded regions on the polymer surface because of the entrapped air bubbles. The rolling contact should be initiated at the centre region and then rolled outward for efficient removal of the entrapped air. Then, we simply put a steel block weighing 0.25 kg on top of the SU-8 bonding layer at 80°C for 15 min, followed by a UV flood exposure for 1 min to cross-link the thin SU-8 adhesive layer. Since the glass transition temperature of the cross-linked SU-8 is more than 200°C, the bonding process will have no effect on the cross-linked SU-8 trenches and the cross-linked SU-8 bonding layer, the bonding temperature of 80°C makes the thin unexposed SU-8 layer melt and fill into the trenches inevitably and achieve the chain entanglement of the polymer over the boundary by the rolling process. Size controllable nanochannels with a uniform channel width in a large area as multiple continuous sheets can be achieved by using this bonding process. The different channels with a depth of 370, 311 and 221 nm were fabricated by a particular thin unexposed SU-8 layer with thicknesses of 25 (Fig. 3b), 50 (Fig. 3c) and 75 nm (Fig. 3d), respectively. These data clearly demonstrate that the new approach indeed offers a good resolution for patterning multiple fluidic channels with a great width and height uniformity over a centimetre-scale ( $1.5 \times 1.5$  cm) channel length. No air bubble and clogging were found using the low pressure thermal bonding.

**4. Conclusion:** We have successfully fabricated arrays of sealed nanochannels with well-controlled dimensions in a large area by using a combined NIL and the thermal bonding methods. The advantage of this process is that the dimensions of the nanochannels can be adjustable simply by the depth of the cross-linked SU-8 trenches as well as by the initial thickness of the thin unexposed SU-8 layer. On considering the SU-8 trenches' fabrication using NIL, the imprint depth is equal to the height of the template pattern, which can be easily controlled by adjusting the etching duration during the template fabrication. The height of the nanochannels can also be controlled by adjusting the ratio of the ridge width to the trench width on the SU-8 trenches. This method is preferable if a large area needs to be sealed uniformly in a single step. This method also could be used to control the channels or the other structures for the other materials.

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## 6 References

- [1] Jay Guo L., Cheng X., Chou C.-F.: 'Fabrication of size-controllable nanofluidic channels by nanoimprinting and its application for DNA stretching', *Nano Lett.*, 2004, **4** (1), pp. 69–73
- [2] Yan X.-M., Kwon S., Contreras A.M., Bokor J., Somorjai G.A.: 'Fabrication of large number density platinum nanowire arrays by size reduction lithography and nanoimprint lithography', *Nano Lett.*, 2005, **5** (4), pp. 745–748
- [3] Bai J.G., Chang C.-L., Chung J.-H., Lee K.-H.: 'Shadow edge lithography for nanoscale patterning and manufacturing', *Nano Technol.*, 2007, **18**, p. 405307
- [4] Zhao Y., Berenschot E., de Bore M., *ET AL.*: 'Fabrication of a silicon oxide stamp by edge lithography reinforced with silicon nitride for nanoimprint lithography', *J. Micromech. Microeng.*, 2008, **18**, p. 064013
- [5] Abgrall P., Low L.-N., Nguyen N.-T., *ET AL.*: 'Fabrication of planar nanofluidic channels in a thermoplastic by hot-embossing and thermal bonding', *Lab Chip*, 2007, **7**, pp. 520–522
- [6] Li X., Wang X., Jin J., *ET AL.*: 'Fabrication of micro/nano fluidic system combining hybrid mask-mould lithography with thermal bonding', *Microelectron. Eng.*, 2010, **87**, pp. 722–725
- [7] Wang X., Chen Y., Banu S., *ET AL.*: 'High density patterns fabricated in SU-8 by UV curing nanoimprint', *Microeng. Eng.*, 2007, **84**, p. 872–876
- [8] Patel J.N., Kaminska B., Gray B.L., Gates B.D.: 'PDMS as a sacrificial substrate for SU-8-based biomedical and microfluidic applications', *J. Micromech. Microeng.*, 2008, **18**, p. 095028
- [9] Abgrall P., Charlot S., Fulcrand R., *ET AL.*: 'Low-stress fabrication of 3D polymer free standing structures using lamination of photosensitive films', *Microsyst. Technol.*, 2008, **14**, pp. 1205–1214
- [10] Hoff J.D., Cheng L.J., Meyhöfer E., Guo L.J., Hunt A.J.: 'Nanoscale protein patterning by imprint lithography', *Nano Lett.*, 2004, **4**, (5), pp. 853–857