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Research paper

Fabrication of polymeric dual-scale nanoimprint molds using a polymer stencil membrane

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ABSTRACT

We report on a simple and effective process that allows fabricating polymeric dual-scale nanoimprinting molds. The key for the process is the use of a thin flexible SU-8 stencil membrane, which was fabricated by either photolithography or thermal nanoimprint lithography (NIL). The stencil membrane with microscale pores was assembled into a nanopatterned substrate, producing a dual-scale structure. The assembled structure was used as a template to produce polymeric imprinting molds via UV-NIL. With this method, we demonstrated dual-scale nanoimprint molds having nano-pillars of 251 nm diameter and 146 nm high on top of microscale square protrusions of 5 µm wide and 3.6 µm high. The resin mold with the dual-scale structure was successfully used to produce a freestanding membrane with dual-scale perforated pores via UV-NIL. After metal coating and integrated into microfluidic devices, this freestanding membrane can potentially be used as a substrate for surface plasmon resonance sensors.

1. Introduction

The ability to produce multi-scale or dual-scale hierarchical structures is important for the development of applications such as surfaces with directional wetting and spreading, dry adhesive motivated by gecko, and lab-on-chips [1]. One interesting hierarchical structure for the lab-on-chip application is a freestanding polymer membrane with nanoscale through-holes (nanopores) integrated with microscale fluidic compartments. Such a structure can be used as a platform for molecular separation and sorting [2], cell trapping and enrichment [3], and organs-on-chips [4]. After a metal coating, such a membrane structure can also be used as a substrate for surface plasmon resonance (SPR)-based biosensing [5,6].

Nanoimprint lithography (NIL) is a promising method for the fabrication of the nanopore membrane with microscale compartments, due to its ability to produce large area structures at low cost and with high throughput [7]. Thermal-NIL has demonstrated sub-10 nm structures imprinted in a thin resist layer [8]. However, production of a nanopore membrane by thermal- and UV-NIL is more challenging than producing imprinted surface patterns, mostly because high aspect ratio NIL is usually required to achieve a membrane thickness for sufficient mechanical strength and easy handling of the membrane. Our group has demonstrated fabrication of a freestanding polymer membrane with nanopores with diameters less than 100 nm via thermal-NIL and used the membrane for detection of DNA molecules via transient current measurements across the nanopore membrane [9]. A Si mold with microneedles formed by wet chemical etching of Si was used, which makes it difficult to produce the membrane structure with high density nanopore arrays with microscale fluidic compartments. Such structures have never been realized by NIL, attributed mostly to the difficulty in fabricating a large area mold with the hierarchical structures.

There are mainly two top-down fabrication methods for the dual-scale hierarchical structures: one is a two-step photolithography and the other is a sequential thermal-NIL [1]. However, the two-step photolithography technique is limited to the fabrication of microscale patterns set by the wavelength of the light source used. For the sequential thermal-NIL, it is difficult to adjust the imprinting temperature to prevent collapse or reflow of predefined structures during the second imprinting. Two-step UV-assisted capillary molding technique was recently developed [10]. A partially UV cured microstructure was further molded to produce nanostructures on its top, making a monolithic hierarchical structure. However, controlling the degree of UV curing is exceedingly difficult. Here, we present a simple method to produce dual-scale nanoimprint molds using a polymer stencil membrane and single-step UV-assisted molding technique, which allow excluding two-step UV-assisted molding process.

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2. Materials and methods

The first step of fabricating polymeric dual-scale nanoimprint molds is to fabricate a polymer stencil membrane with microscale through-holes via photolithography or thermal-NIL. For photolithography, a 1 µm thick lift-off resist (LOR) layer (LOR-7B, MicroChem) was first spin-coated on a silicon (Si) substrate as a sacrificial layer and baked. On the LOR layer, a thick SU-8 layer of different thickness (SU-8 2005, SU-8 2010, SU-8 2020, and SU-8 2035, MicroChem) was spin-coated and baked. The spin-coating speed, soft and post exposure baking temperatures, and UV exposing energy were selected according to the company protocol provided. After exposure and development of the uncured SU-8, the membrane was released via dissolving the sacrificial layer with a developer (Microposit MF-319, MicroChem). For thermal-NIL, in the same combination of sacrificial and SU-8 layers the microscale pores were imprinted using a Si mold. Details of the process to form the polymer stencil mask via thermal-NIL have been published in [7].

The second step involves producing a nanostructured substrate, which has been done via thermal-NIL into poly(methyl methacrylate) (PMMA) substrate (750 µm thick sheet, Goodfellow). The thermal-NIL was carried out at the imprinting temperature and pressure of 130 °C and 3.5 MPa, respectively, for 5 min by using a commercial nanoimprinter (Eitre6, Obducat). Demolding was performed at 70 °C manually. The nanostructured substrate has nanoscale cavity patterns with the pitch, diameter and depth of 450 ± 30 nm, 240 ± 30 nm, and 200 ± 40 nm, respectively.

The released membrane was dipped into isopropyl alcohol (IPA) (isopropyl alcohol, Sigma-Aldrich) and then laid on a nanostructured substrate, which allowed achieving a good conformal contact between the membrane and the nanostructured substrate (Fig. 1a, Step 1). It should be noted that neither glue nor any sort of adhesive was used between the released membrane and the nanostructured substrate. The assembled substrate with dual-level pores, i.e. the microporous membrane on the nanostructured substrate, was treated with a thin polydimethylsiloxane (PDMS) layer in the liquid phase [11] or a fluorinated silane ((heptadecafluoro-1,1,2,2-tetrahydrodecyl)trichlorosilane, Gelest) in the vapor phase to reduce adhesion during the subsequent UV-NIL process.

A UV-curable resin (PUA511RM, Minuta Technology) was used to transfer patterns on the assembled substrate into UV-cured polymers via UV-NIL (Fig. 1a, Steps 2–4). Drops of the UV-curable resin were dispensed against the assembled substrate. Then, a flexible polycarbonate (PC) film (~250 µm thick polycarbonate sheet, ePlastics) was slightly pressed against the liquid drop and used as a supporting backplane. In the curing process, the sample was exposed to flash-type UV light (250–400 nm) for 10 s, at an intensity of ~1.8 W/cm^2 by using the nanoimprinter. Lastly, the UV-cured sample was peeled off from the assembled substrate using a sharp tweezer. The UV-cured sample was used as a master mold and replicated twice by UV-NIL to make a replica with the same polarity. This step is not necessary when the UV-cured sample from the assembled substrate with dual-level pores is directly used as the mold for the final UV-NIL process to produce a membrane with perforated dual-level pores.

In order to produce a membrane with perforated dual-level pores, the resin mold and polycarbonate (PC) substrate was first coated with a thin PDMS layer to avoid an adhesion problem between substrates (e.g. the resin mold and PC substrate) and a membrane to be created (Fig. 1b, Step 1). Then, a drop of the UV-curable resin (PUA511RM, Minuta technology) was dispensed between the resin mold and the PC substrates (Fig. 1b, Step 2) and UV-NIL was performed in the same NIL system with the UV exposure time of 10 s under 1 MPa (Fig. 1b, Step 3).

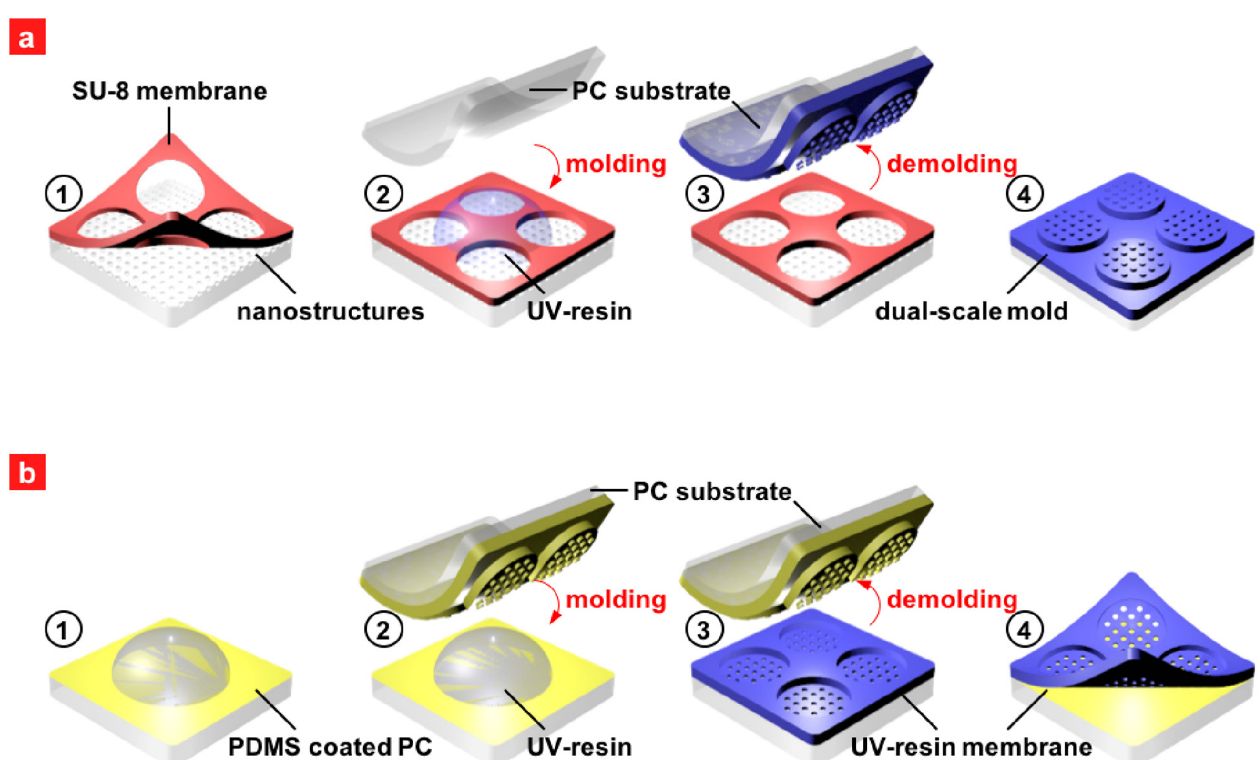

Fig. 1. (a) Schematics of fabricating dual-scale nanoimprint molds: place a piece of SU-8 membranes over a nanostructured substrate. Then, treat with a thin PDMS layer or a fluorinated silane (1); dispense drops of UV-curable resin and slightly press with a flexible PC substrate. Then, expose to flash-type UV-light (2); peel off the UV-cured sample (3); and coat a thin PDMS layer on the UV-cured sample for self-replicating (4). (b) Schematics of fabricating UV-resin freestanding membranes having dual-scale perforated structures: dispense drops of UV-curable resin on a PC substrate coated with the thin PDMS layer (1); slightly press with the UV-resin mold self-replicated from the UV-resin master and coated with the thin PDMS layer. Then, expose to flash-type UV-light (2); peel off the UV-resin mold (3); and gently peel off the UV-cured membrane from the PC substrate (4).