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Silicon based solvent immersion imprint lithography for rapid polystyrene microfluidic chip prototyping

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ABSTRACT

Polystyrene (PS) is preferred over polydimethylsiloxane (PDMS) in microfluidics for applications in cell biology. However, PS has not found widespread use in microfluidics due mainly to the lack of rapid prototyping techniques. Here we address this issue by developing a silicon based solvent immersion imprint lithography (Si-SIIL) technique. Silicon is rigid, mechanically robust, and highly compatible with standard microfabrication processes, and therefore, is a promising candidate for molds. Various PS microfluidic channels as small as 20 μm in width with the aspect ratio as high as 5 were demonstrated using Si-SIIL. Bubbles and bending generated in the fabrication process were analyzed and eliminated. The surface roughness was about 27 nm (rms). Compared to the untreated PS, the molded PS retained almost the same surface properties, as characterized by contact angle measurement and X-ray photoelectron spectroscopy. Cell culture was tested to demonstrate the utility of Si-SIIL in cell biology applications. The results show that PS, with the aid of Si-SIIL, can be an alternative material to PDMS in building microfluidic chips.

1. Introduction

In the past decade microfluidics in biological applications has experienced significant growth due to its advantages of small volume, low cost, short reaction time, and high throughput [1–5]. The materials for microfluidics usually include silicon, glass, and polymer [6]. While silicon and glass were commonly used in the earlier years of microfluidics development [7,8], polymer has become an increasingly attractive alternative [9–12]. Polymers encompass a large class of materials, including two major categories: elastomers and thermoplastics [13]. Since Whitesideset al. [14] fabricated complex microfluidic devices based on polydimethylsiloxane (PDMS), it has been widely employed in microfluidics due to its low cost, optical transparency, biocompatibility, and simple processing and prototyping [9,15,16]. However, despite all the afore mentioned beneficial properties, PDMS suffers from easy deformation, rapid liquid evaporation, absorption of molecules into the polymer, leaching of uncross-linked oligomers, and hydrophobic recovery [17,18], which significantly limit its adoption in microfluidics for biological research.

As an alternative microfluidic material, polystyrene (PS), one of the mostly used thermoplastics, has been studied and used for macroscopic cell culture and bioanalysis, thanks to its low cost, optical transparency, biocompatibility, chemical stability, and physical rigidity [19,20]. Furthermore, it can easily be transferred from hydrophobic to hydrophilic by plasma treatment and remains hydrophilic for 4 weeks, about 4 times longer than PDMS [21]. As such, PS is preferred over PDMS in microfluidics for cell biology applications. However, the fabrication of PS microfluidic chips is usually more difficult and expensive than PDMS. Therefore, it is crucial to develop simple and cost-effective processes with high resolution and repeatability for rapid PS microfluidic prototyping.

In the past, a number of PS microfabrication methods have been explored. Hot embossing relies on relatively high temperature (120 °C, 20 °C above the glass transition temperature of PS) and metal molds to create microfluidic devices [22,23]. However, metal molds are fabricated by a laser system, which process is time-consuming and of high cost, and therefore, may not be suitable for

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rapid lab prototyping. Laser cutting is another technology to fabricate microfluidic devices [24]. For example, Li et al. [25] used a CO_2 laser to create droplet microfluidic devices on a PS substrate. Laser cutting is a mask-free method, but its process is sequential and becomes very time-consuming and costly with a large number of devices. In contrast, injection molding is capable of fabricating multiple microfluidics simultaneously with low costs. However, it requires dedicated tools such as injection moldingmachines [26]. Johnson et al. [27] and Pentecost et al. [28] developed a method similar to the injection molding method but free of injection molding machines. In their method, PS powder was poured into an aluminum weighing dish and then heated to 250 °C for several hours. Unfortunately, this process does not allow for room temperature fabrication. To circumvent such an issue, Nargang et al. [29] used several toxic chemicals, e.g., toluene, isopropanol, and cyclohexanone, to dissolve PS before the use of a PDMS mold. A major drawback of this method is the distortion (such as swelling) in the PDMS mold in the presence of those chemical solutions [30]. Therefore, it is crucial to find a solvent that can dissolve PS, but does not distort the PDMS mold. Gamma-butyrolactone and delta-valerolactone were found to be appropriate solvents based on their ability to dissolve PS without swelling PDMS [30]. However, they need seven days to form a PS solution in a tube filled with PS solid and organic solvent [30]. Recently, solvent immersion imprint lithography (SIIL) has been developed that enables complete PS microfluidics prototyping in a single processing step [31,32]. In this method, the PS surface is first softened by acetone and then imprinted with a PDMS mold. SIIL is simple and rapid and does not require sophisticated tools or heating processes. However, PDMS has an elastic modulus of about 1–3 MPa, three orders of magnitude lower than that of thermoplastics like PS (~3 GPa), which makes PDMS easy to deform [17,31]. Consequently, it is difficult to transfer structures with high fidelity from a PDMS mode to PS, especially when a high aspect ratio is needed [31].

Here we developed a silicon based solvent immersion imprint lithography (Si-SIIL) method for rapid PS microfluidics prototyping. Silicon is much more rigid than PDMS and highly compatible with standard microfabrication processes, and therefore, is a promising candidate for molds. In this article, we present the details of the Si-SIIL and contrast it with PDMS-SIIL whenever possible. Various microfluidic channels as small as 20 μm in width with the aspect ratio as high as 5 were demonstrated. Characterization of the Si-SIIL molded PS chips using contact angle measurement, X-ray photoelectron spectroscopy (XPS), and cell cultivation is also discussed.

2. Material and methods

2.1. Fabrication of Si mold

The silicon mold with inverse structures was fabricated using standard microfabrication technology using the following steps. (1) A silicon wafer was cleaned using a mixture of H_2SO_4 and H_2O_2 solution. (2) A 300 nm SiO_2 layer was deposited on the wafer by plasma enhanced chemical vapor deposition (PECVD), which was used to form the mask layer for subsequent silicon etching. (3) The silicon wafer was spin-coated with a 5 μm AZ4620 photoresist layer, followed by baking at 95 °C for 120 s, exposure to UV light for 5.5 s, and development for 45 s. In order to increase the strength of the photoresist layer, the wafer was baked at 110 °C for 120 s. (4) The SiO_2 layer was etched by reactive ion etching [33] for about 40 min. (5) The Si wafer was etched for different amount of time using deep reactive ion etching [34] to obtain different depths. (6) Finally, the photoresist was removed using acetone.

2.2. Si-SIIL protocol

The basic procedures of Si-SIIL are summarized as follows. The PS surface was first softened using solvent, then the softened surface was imprinted with a Si mold, and finally the PS was bonded to a PS substrate. Acetone was used to soften PS according to the SIIL method [31]. During Si-SIIL, a 1.48 mm thick PS slab was immersed in acetone solvent for 0.5–3 min at room temperature. Acetone diffuses into the PS to form a surface "gel" layer (Fig. 1(a)). A drop of acetone was dropped on the surface of a Si mold until acetone spread completely over the mold (Fig. 1(b)). The immersed PS was removed from the acetone solution and subsequently placed on the Si mold. A weight (about 1 kg) was placed on the other side of the PDMS slab via a 2 mm thick PDMS slab to provide pressure for structure transfer (Fig. 1(c)). The PS slab and the mold were placed in a small vacuum chamber for 1.5 h to let acetone evaporate and subsequently release the PS from the mold. Note that without the vacuum chamber, acetone evaporation and the PS release take 10 h. In contrast, in PDMS based SIIL, the porous PDMS enables rapid solvent removal from the polymer and quick PS release [31]. Finally, another PS slab punched with inlets/outlets was immersed in acetone for about 5 s, and then bonded with the PS slab with structures (Fig. 1(d)).

2.3. Gel layer thickness and transmittance

The immersed PS slab after acetone evaporation was used for the ultraviolet-visible (UV-vis) measurement. Then the PS slab was broken into two pieces to measure the gel layer thickness.

2.4. Water contact angle measurement

To study the effect of immersion time in acetone on the PS surface properties, the static water contact angle was measured using droplets of water (about 1 μl) applied to the free surface of PS slabs that underwent acetone immersion, acetone immersion followed by O_2 plasma treatment, and no treatment.

2.5. Cell culture

The biocompatibility of the PS after Si-SIIL was assessed by 24 h cultivation of transduced ATDC5 cells, which are derived from mouse teratocarcinoma. The responsiveness and proliferation of the cells on the chip were determined by drug-induced luminescence, while cell morphology and death rate were visualized by

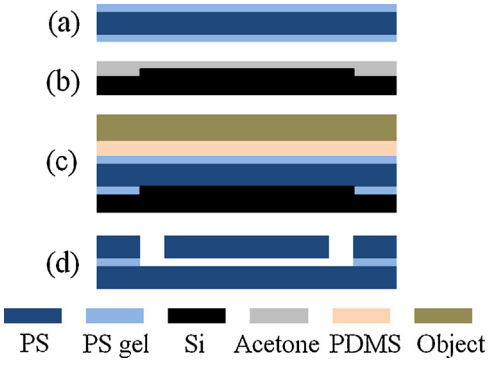

Fig. 1. Illustration of the Si-SIIL protocol. (a) A PS slab is immersed in acetone for 0.5–3 min under room temperature to form a surface "gel" layer on both sides. (b) A drop of acetone is dropped and uniformly distributed on the silicon mold surface. (c) The softened PS is placed on the silicon mold and then a weight is placed on the other side of the PS slab via a PDMS cushion (2 mm thick) to provide the pressure for structure transfer. (d) Another PS slab with inlets/outlet is immersed in acetone for about 5 s and then bonded with the first PS slab via manual pressing.