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Fabrication of Freestanding 1-D PDMS Microstructures Using Capillary Micromolding

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***Abstract*—A method to create freestanding, biocompatible polydimethylsiloxane (PDMS) microstructures is presented. First, capillary flow through micro channels on silicon (Si) substrates is used to create high fidelity PDMS structures that are a few μm wide and deep but several mm long (length to width/depth $\approx$ 500:1). Next, an improvised procedure is employed to remove the cured PDMS microstructures from the Si substrate without damaging them. The method is used to create extremely sensitive cantilever beams with stiffness less than 0.1 pN/μm, and micro platforms for cell biology studies. The PDMS microstructures created using this method have applications in cell mechanobiology as force and mass sensors. [2013-0106]**

***Index Terms*—BioMEMS, force sensors, cell mechanics.**

I. Introduction

Polydimethylsiloxane (PDMS) based micro devices and systems have been extensively used to study biological systems [1], [2]. Most initial applications involved using microfluidic circuits to perform biochemical assays and micro patterning to study the biochemistry of fundamental cellular processes. However, PDMS based micro systems are being increasingly used [3], [4] to study the mechanics of cells and tissues and the effects of mechanical microenvironment on their behavior, a field commonly referred to as mechanobiology.

The increasing adoption of PDMS, and other polymer-based, devices in mechanobiology is driven by several factors. From a biological perspective, the mechanical properties of polymers are closer to the *in vivo* environment of cells. Their surface chemistry can also be altered to better mimic the natural microenvironment of the cells. Furthermore, polymers like PDMS and polyacrylamide are optically transparent and therefore force measurement and immunofluoresence imaging of specific biological markers can be made simultaneously under light microscopes. This enables correlation of cell force generation/sensing with molecular processes and biochemical pathways. Some of the notable techniques in mechanobiology based on polymer-based micro systems include traction force microscopy [5], [6], microfabricated post array detectors [7] and micro cantilevers to measure contractile forces of cells [8], [9]. These techniques are focused on determining the origin of cell mechanosensitivity, and measuring the mechanical response of cells to mechanical and biochemical stimuli.

A more recent development has been the use of flexible polymeric devices in cell based bio-hybrid actuators [10]–[12]. In bio-hybrid actuators, the physical forces generated by cells are used to power the composite cell-polymer structure. But since cells can sense their mechanical microenvironment [13], [14] their migration, proliferation and force generation are dependent on the mechanical properties of the polymeric device. Thus, there is a strong coupling between the structure and physical properties of the bio-hybrid actuator and the biological processes that power it, which can be used to design more efficient biomachines and power generators [15], [16].

In this letter we outline a method to fabricate long, freestanding PDMS microstructures for use in mechanobiology studies and as platforms for bio-hybrid devices. The method uses capillary driven flow through micro channels to create PDMS structures that have width and depth of a few μm but are several mm long (length to width/depth $\approx$ 500:1). The technique requires only a single photolithography step for the creation of the silicon (Si) master mold, which can then be used repeatedly. While capillary forces have previously been used for lithography [17], [18], in those studies PDMS was primarily used as a mold for creating patterns of other materials on various substrates. Here, we use a similar principle to create ultra sensitive, freestanding PDMS cantilever beams (stiffness $<$ 0.1 pN/μm) as well as devices with more complex patterns for bio-hybrid systems.

II. Fabrication Process

First, a Si master mold is created using conventional photolithography techniques. A Si wafer is spin coated with photoresist (PR) and the pattern on the photomask is transferred to the PR layer. The PR layer is then hard baked and the wafer is transferred to a deep Si etching chamber (STS ICP-DRIE) to etch micro channels in the Si wafer to the required depth. The depth of the PDMS structures to be created will be the same as the depth of the micro channels. After the Si etch, a thin layer (about 100 nm) of polytetrafluoroethylene (PTFE) is deposited on the entire wafer, including the side walls and bottom surface of the micro channels. The deposition of the PTFE layer assists in peeling off the cured PDMS structures and is the final step in the fabrication of the Si master mold.

In the first step of the fabrication of PDMS structures, liquid PDMS is injected into reservoirs shown in Fig. 1. As the PDMS spreads in the reservoir it comes into contact with the inlet of the micro channels and is drawn into the channel due

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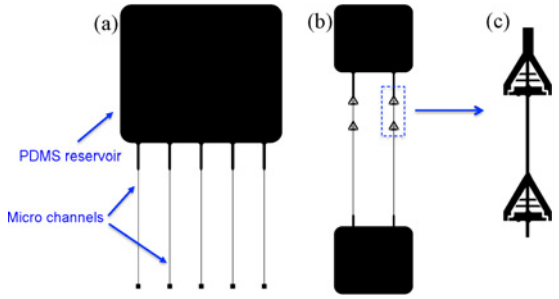

Fig. 1. (a) Top view of a pattern etched into the Si wafer. The micro channels are connected to a reservoir from which PDMS flows into them through capillary action. (b) Micro channel with a intricate pattern that is connected to two reservoirs. (c) Magnified view of the intricate pattern.

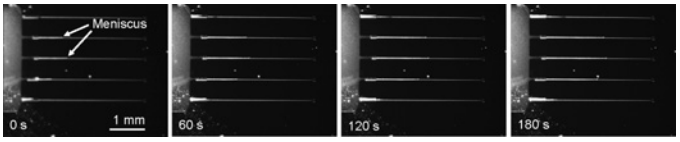

Fig. 2. PDMS flow in the micro channels. As time progresses, the rate of fluid flow reduces because while drag increases (proportional to the volume of fluid in the channel) the driving force resulting from the reduction in interfacial energy remains constant.

to capillary forces. The rate at which the channels get filled up depends on the viscosity of the liquid PDMS, the dimensions of the channel and the interface energy between PDMS and the PTFE layer. Typically, PDMS with a higher cross-linker to base ratio fills up faster since the cross-linker viscosity is lower compared to the base. Based on our experiments, micro channels that are about 3 mm long, 15 μm wide and 30 μm deep fill up in a period of 10 minutes when the base to cross-linker ratio is 4:1 (Dow Corning's Sylgard 184 elastomer kit was used in all our experiments). To fill channels that are longer or have more intricate features (Fig. 1b, 1c), more than one reservoir can be connected to the micro channels and PDMS can be injected simultaneously into multiple reservoirs. A series of snapshots of liquid PDMS filling up micro channels is shown in Fig. 2.

Once PDMS has completely filled the micro channels, it is cured by baking at 60 ^{o}C for 24 hours. Note that the PDMS structure is an inverse pattern of the Si mold and hence the micro channels in the mold become micro beams in the PDMS structure. After the PDMS is cured, the Si mold along with the PDMS is immersed in ethanol for 1–2 hours to ensure easy release of the PDMS structure. While it would be very difficult to release each individual PDMS micro beam, the task is greatly simplified in this case because the micro beams are still attached to the reservoirs. In the first step of the peeling process, the reservoirs, which have much larger area, are gently released from the Si mold. Then a small tweezer is used to grasp the reservoir and slowly peel off the micro beams. Since the tweezer does not contact the micro beams during their release, damage is considerably reduced. In effect, the reservoir in the Si mold serves two purposes: a) filling micro channels with PDMS, and b) avoiding damage to PDMS micro beams during release. A schematic of the entire fabrication process is shown in Fig. 3.

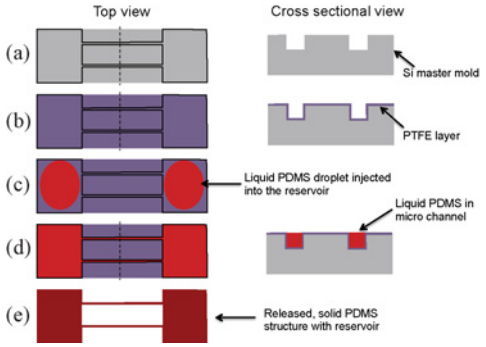

Fig. 3. Process flow for the fabrication of high aspect ratio PDMS structures.

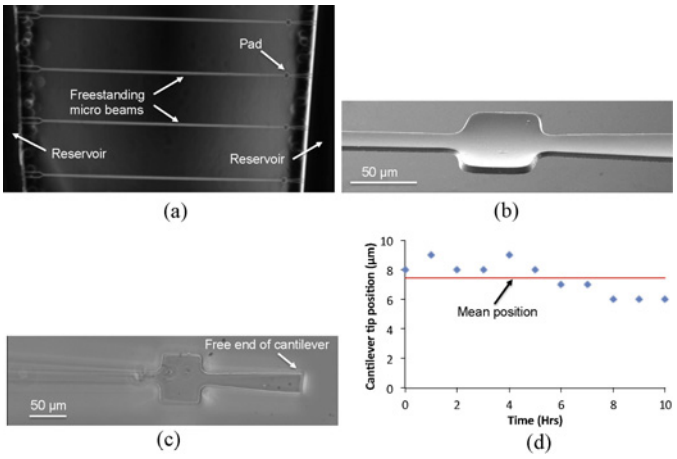

Fig. 4. (a) Freestanding PDMS beams after being released from silicon mold. (b) Scanning electron micrograph of the pad area showing a smooth, defect free topography. (c) Optical image of a free cantilever ($k < 0.1$ pN/μm) in water. The cantilever was obtained by severing one end of a micro beam from the reservoir near the pad area. The entire cantilever is not in focus because of the deflection due to buoyancy. (d) Variation in tip position of a free cantilever over a period of 10 h. The accuracy of the measurement is $\pm 0.5 \mu$m.

III. Results and Discussions

The process described above was used to fabricate different types of PDMS microstructures. Figure 4a shows a set of freestanding PDMS micro beams (with pads) that are attached to the reservoirs at both ends. The magnified image of the pad area shown in Fig. 4b reveals a smooth topography and a nearly perfect reproduction of the pattern on the silicon mold. Figure 4c shows a free cantilever beam, obtained by severing one end of a micro beam from the reservoir, in water. The length (L), depth (t) and width (w) of the cantilever beam are 3 mm, 8 μm and 20 μm, respectively. Thus, the stiffness, $k = Ewt^3/4L^3$, is 0.095 pN/μm (assuming a Young's modulus (E) of 1 MPa), which is more than three orders of magnitude smaller than the stiffness of the most compliant Si or PDMS based micro force sensors [19], [20]. In fact, the stiffness is low enough to directly measure mass change of single cells (about 1.5 ng [21]) during a cell cycle. Because the relative density of the cell (ρ_{cell}/ρ_{water}) is around 1.1, the apparent change in mass during the cell cycle is around 0.15 ng. But this extremely small mass change will still cause the PDMS

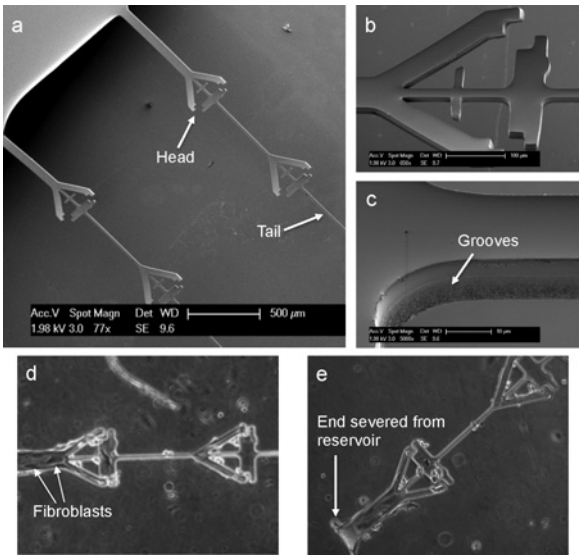

Fig. 5. (a) Intricate, freestanding PDMS structure with two triangular heads and a long, slender tail. (b) Magnified image of the head area. (c) A high-resolution image of the surface topography of the device revealing sub μm grooves that replicate the pattern on the Si mold. (d) Fibroblasts cultured on a PDMS device. (e) PDMS device with fibroblasts after being released from the reservoir.

cantilever beam to deflect by 15 μm, which can be easily measured in an optical microscope.

The PDMS cantilevers, because of their extremely low stiffness, would collapse under self-weight in air. However, because the mass density of PDMS ($\rho_{pdms} \approx 0.97 g/cm^3$) is similar to water ($\rho_{water} = 1 g/cm^3$), the effective density of PDMS ($\rho_{eff} = \rho_{pdms} - \rho_{water}$) in an aqueous environment is very small and negative. As a result, a cantilever ($L = 2.5mm$, $w = 10\mu m$ and $t = 10\mu m$) with $k = 0.16\ pN/\mu m$ would experience only a moderate upward deflection (δ_{buoy}) of $172\mu m$ due to buoyancy. It is worth noting that $\delta_{buoy}(1.5\rho_{eff}L^4/Et^2)$ scales differently than k ($Ewt^3/4L^3$). Therefore, by reducing t and L proportionately, we can keep k low while significantly reducing δ_{buoy}. Similarly, decreasing w would reduce k without increasing δ_{buoy}. Alternately, ρ_{pdms} can be altered to match the liquid density by adding a small amount of denser micro/nano particles to PDMS. In that case, $\rho_{eff} = 0$ and there would be zero deflection.

Apart from low stiffness, the cantilevers should also have minimal fluctuation/drift to ensure reliable force/mass measurements. To verify their stability, we monitored the tip deflection of a free cantilever over a period of 10 hours. During this time, the cantilever showed a maximum fluctuation of only ($\pm 1.5\mu m$) from its mean position (Fig. 4d).

In addition to simple beam-like structures, the technique can also be used to fabricate devices with more intricate geometry (Fig. 5). The depth of the PDMS devices shown in Fig. 5 (30–50 μm) is considerably larger than the cantilever beams (Fig. 4), demonstrating that deep structures can be released without damage. As can be seen from Fig. 5c, even the sub μm grooves on the silicon master mold (introduced during the Si etching) are faithfully replicated in the PDMS structure, attesting to the fidelity of the process. These PDMS structures were functionalized using fibronectin (50 μg/ml) and fibroblasts (Fig. 5d, 5e) were cultured on them. As the structures are extremely compliant, the forces generated by fibroblasts cause deflections as large as 1 mm of the tail-like structure when they are released from the reservoir.

In summary, we have developed a method to create long, 1D, freestanding PDMS microstructures. These PDMS structures can be used as ultra high resolution force/mass sensors in cell mechanobiology studies as well as substrates for bio-hybrid devices.

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