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TECHNICAL NOTE

Spun-cast micromolding for etchless micropatterning of electrically functional PDMS structures

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Abstract

Polydimethylsiloxane (PDMS) is widely used in bioMEMS applications; however, patterning of this material to form complex structures is often challenging. Chemical etches are typically ineffective due to the inertness of the material. Plasma processing of bulk material can be time intensive and presents concerns regarding the mechanical properties of the post-etched polymer due to etch-induced cross-linking of surrounding material. Presented in this paper, the etchless process of spun-cast micromolding (SCμM) is used to create an array of patterned, PDMS, electrical microcables. The microcables are arranged in a net-like array and incorporate electrical functionality. The geometries fabricated with these techniques include straight and sinusoidal microcables. In addition to the cables themselves, specific regions of the cables' top insulating layer can also be patterned using a hierarchical application of the SCμM process, creating exposed electrical access sites useful as electrical access points for electrophysiological applications. The SCμM process is a simple, relatively rapid technique that can be used to make highly compliant electronic structures with patternable geometries.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

This paper outlines a simple process flow for arrays of electrically functional, elastomer microcables. Elastomeric electronics is generally comprised of an elastomer that integrates an electrical conductor in some fashion, commonly by mixing conductive particulates into the bulk elastomer. Examples of conductive particulates include conductive polymers, carbon nanotubes and graphite [4, 5, 12]. Using bulk electrically conductive elastomers in micropatterned devices presents challenges in controlled, precise patterning of the conductive media and in incorporating the conductive component with selectively insulated and exposed regions. Thin-film gold metallization of silicone rubber addresses these considerations as gold is frequently patterned in microfabricated devices and silicone can tolerate some but not all standard process techniques [14]. Gold has relatively soft and non-brittle mechanical properties, which are advantageous for use in elastomer electronics. It also does not form a surface oxide, making it suitable for features such as the electrical access nodes described here. PDMS shape and topography can be controlled by several mechanisms, including photosensitive polymerization, laser ablation, reactive ion etching (RIE) and micromolding [1, 2, 10, 11]. While these techniques have been critical in developing silicone–rubber microstructures, they have limitations in applicability or convenience.

Photopatternable commercially available PDMS may have a higher modulus than desired [14]. Reactive ion etch micropatterning of PDMS for electrical devices is a

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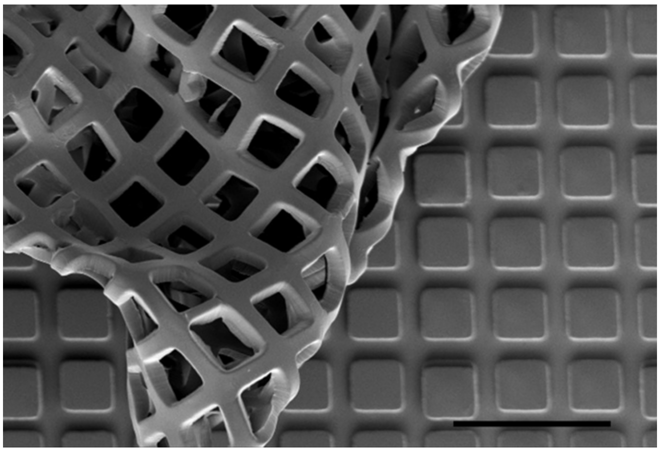

Figure 1. A PDMS membrane is shown as it is pulled back from the SU-8 on which it was spun-cast. The PDMS was spun-cast thin enough on the SU-8 mold so that through holes in the membrane are created. The membrane thickness and mold height are both 16 μm. The scale bar is 200 μm.

time intensive process [6]. Laser ablation is traditionally a serial process and may require registration in some instances. Micromolding when used alone does not create vias or other through-hole features. Although micromolding has been used extensively to pattern PDMS for non-electrical applications, molded microstructures with electrical functionality are less prevalent [11, 13]. The advantages of micromolding include the reusable mold and potential bench-top fabrication. Micromolding can be combined with spin-casting to create through-hole-containing PDMS membranes, suitable for electrically insulating structures [7, 8].

2. Approach

The through holes created by spun-cast micromolding (SCμM) can be used to create net-like membranes. The net-like geometry is useful in biological applications because it is more conformal than a planar sheet and does not isolate the target structure from gas and fluid exchange. A simple micromold and its released elastomer membrane, demonstrating the SCμM concept, are shown in figure 1. The released membrane is a net-like structure with through holes created by the mold posts. SCμM has been used here in a hierarchical process to create an electrically active net-like array, the strands of which are referred to here as microcables.

When designing a mold for a SCμM structure, the spacing of the mold features and the resulting effect on material retention must be taken into account. PDMS is retained between the posts during spin casting in a space and geometry-dependent manner; if the mold is an array of posts with the same size and spacing, the membrane will have a consistent thickness. If different features (e.g. posts) have different shapes and spacing, the parameters of one feature set will constrain the spacing and shape of the surrounding features; otherwise, the thickness of the membrane will be inconsistent (figure 2), and the thinner areas can make the membrane fragile and difficult to handle. The amount of PDMS retained for a given spin speed and duration depends on the post spacing, with narrower post spacings retaining more PDMS. This

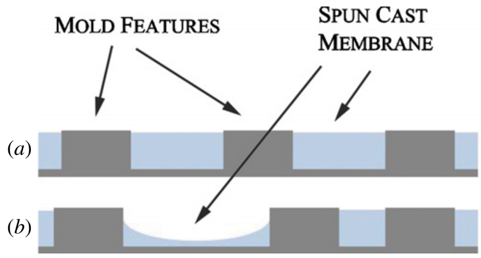

Figure 2. The feature spacing determines the thickness of a spun-cast film. (*a*) Evenly spaced mold features (within a finite spacing limitation) produce membranes with an even thickness. (*b*) Variations in the feature spacing create differences in the film thickness.

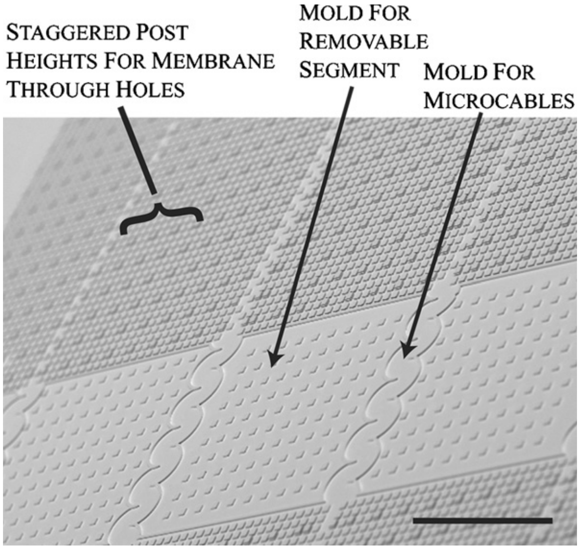

Figure 3. The SU-8 mold for the net-like array includes sinusoidal lines and multi-height posts to create an array of microcables and through holes in the surrounding membrane. The spacing of the posts between the microcable features is farther apart than the spacing in the rest of the mold because the wider post spacing allows for a thinner film, which is easily removed during the release of the microcable array from the mold. The scale bar is 1 mm.

feature can be exploited intentionally if variations in thickness are desirable; it is used here to facilitate the separation of the microcables by creating thin, easily removed segments that become open spaces after the membrane is released from the mold. The mold design for the surrounding membrane was constrained by the dimensions and geometry of the microcables. The sinusoidal walls define the cables while the surrounding posts retain PDMS to create a membrane that frames the microcable array (figure 3). If the mold consisted solely of the microcable features, the surrounding area required for handling and packaging would be too thin and too fragile to handle without breaking. The posts were selectively removed from metallized areas to prevent an electrical discontinuity due to post placement. The membrane framing the microcables maintains a semi-open geometry with through holes created by the subset of taller posts. The post spacing, 20 μm, edge to edge, is smaller than the 200 μm wall spacing for the microcables because it has been observed experimentally that the length of the mold feature, orthogonal to the spin-casting centrifugal force is positively correlated with the amount of

PDMS retained; the 2 mm long microcable-defining walls retain approximately as much PDMS as the 60 μm square posts.

The chemicals traditionally used for feature patterning in microelectronics processing are not generally suitable for use on silicone rubber because it absorbs many organics and is vulnerable to degradation from some highly acidic and alkaline aqueous solutions. The problems this generates are twofold. First, microfabrication processing frequently relies on organic or strongly alkaline/acidic etchants or developers. If the etchant or developer partitions into the substrate, its residue can interfere with subsequent steps. Additionally, we have observed that if the PDMS is patterned with metal, the swelling from absorption of organics causes the metal to fracture, destroying the electrical continuity.

The processing here uses aqueous materials. When resists and developers are used, the parameters were optimized to minimize exposure to the substrate. The release layers to remove the microcable array from the SU-8 mold are dextran, a water-soluble starch molecule, and agarose, a water-soluble polysaccharide molecule that deters the dextran dissolution during processing, particularly during the metallization lift-off. The dextran dissolves easily in water and has been used previously as a release layer for microfabrication [3]. The aqueous-based processing dissolves enough of the dextran (when used alone) that it partially releases the microcables off the SU-8 mold. This is problematic because the top, insulating PDMS layer is patterned using an aligned photomask, requiring the features to be in their original orientation on the mold. The agarose layer acts as a slowly dissolving seal over the quickly dissolving dextran. The agarose was not simply used in place of the dextran because it gels at room temperature at very low concentrations (<1% w/v solution with water), making its application as a bulk release layer difficult. The dextran release layer (20% w/v solution in water; liquid at room temperature), by contrast, was applied over two applications and supplies enough bulk dissolvable material to facilitate release of the cables. When the microcables are ready for release, the mold is placed in a water bath at room temperature for several hours to allow the agarose and dextran to dissolve. After the release layers are solubilized, the microcables are easily pulled from the SU-8 mold with a pair of tweezers. The microcable array leads are contained within a larger elastomer substrate for ease of handling (figure 4). The electrical leads transition into wider gold tracks on the substrate that terminate in square pads that can be easily attached to hook-up wires for testing.

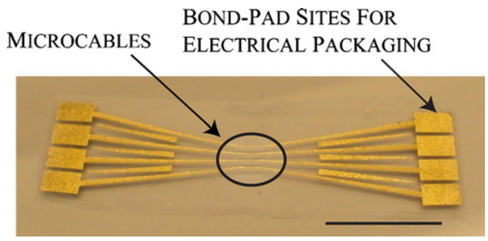

Figure 4. The microcable array is centered within a larger membrane, shown above, that has a footprint approximately 1.5 × 4 cm. The rectangular gold pads on each end are to facilitate packaging for electrical testing. The gold, triangle-shaped features between the leads were created to ease resist removal during metallization lift-off by reducing the contiguous area to be attacked by the resist stripper. As these features are artifacts of processing they are not electrically functional components of the array. The scale bar is 1 cm.

3. Methods

The mold for the array was photolithographically patterned with SU-8 photoepoxy. The photomasks were emulsion-printed mylar sheets (Fine Line Imaging; Colorado Springs, CO). The mold was made with multiple layers of SU-8. All SU-8 processing was done according to manufacturer instructions. A test-grade silicon wafer was coated with a 2 μm layer of SU-8 2002, flood exposed and hard baked. A second 16 μm thick layer of SU-8 2015 was spun-cast, patterned and developed as the mold substrate. A third layer, 26 μm thick, of SU-8 2015 was deposited on the substrate to increase the height of a subset of the mesh posts (26 μm versus 16 μm) to create mesh through holes throughout the membrane. This subset of posts is visible in the micrograph of the mold shown in figure 3. The heights of the molds were confirmed with an optical micrometer (Quadra-Chek 200, Metronics; Bedford, NH). All posts are 360 μm^2 in area, spaced 20 μm edge to edge. The walls creating the microcables are 30 μm wide.

The release layer, 20% v/w dextran (Sigma; St. Louis MO) in water was spun on the mold after a 30 s plasma discharge (EMS 100 glow discharge unit, Electron Microscopy Sciences; Hatfield, PA). The dextran was applied twice, followed by a 1% w/v agarose (Sigma) in water spin coat. The dextran and agarose were spun 40 s at 800 rpm, and each layer was dried (hotplate, 100 °C, 30 s) before the next application. Applying the agarose required heating it until the gel melted and then spin-casting the melted solution. Sylgard 184 PDMS was then spun onto the mold at 4000 rpm for 4 min. The thickness of the spun-cast silicone was 16 μm. Figures 5(*a*1) and (*a*2) shows the first layer of PDMS on the SU-8 mold. The micrographs of this step, figures 5(*a*3) and (*a*4), show the mold before and after the PDMS layer.

The gold leads were patterned using lift-off metallization and thermal deposition (figure 5(*b*1) and (*b*2)). Before the lift-off resist was applied, the silicone substrate was treated with a 30 s negative plasma discharge to improve photoresist dispersion on the PDMS. Negative resist, NR9-8000 (Futurrex; Franklin, NJ) was deposited 9 μm thick, pre- and post-exposure baked on a hotplate at 95 °C, for 60 s and developed with RD-6 developer (Futurrex). The exposure dose was 125 mJ, which is lower than the dose recommended by the manufacturer; however, the resulting undercut in the resist improved its release during lift-off. PVD 75 filament evaporator (Kurt J Lesker; Clairton, PA) was used for the thermal deposition of a 5 nm chrome adhesion layer and a 50 nm gold layer. The deposition was done at 10^{-5} Torr. After deposition, the lift-off resist was removed with aqueous RR3 resist stripper (Futurrex; concentrated tetramethyl ammonium hydroxide), rinsed with deionized water and dried.

The photoresist posts used to pattern a sacrificial mold are shown in figure 5(*c*). The photoresist mold preserves

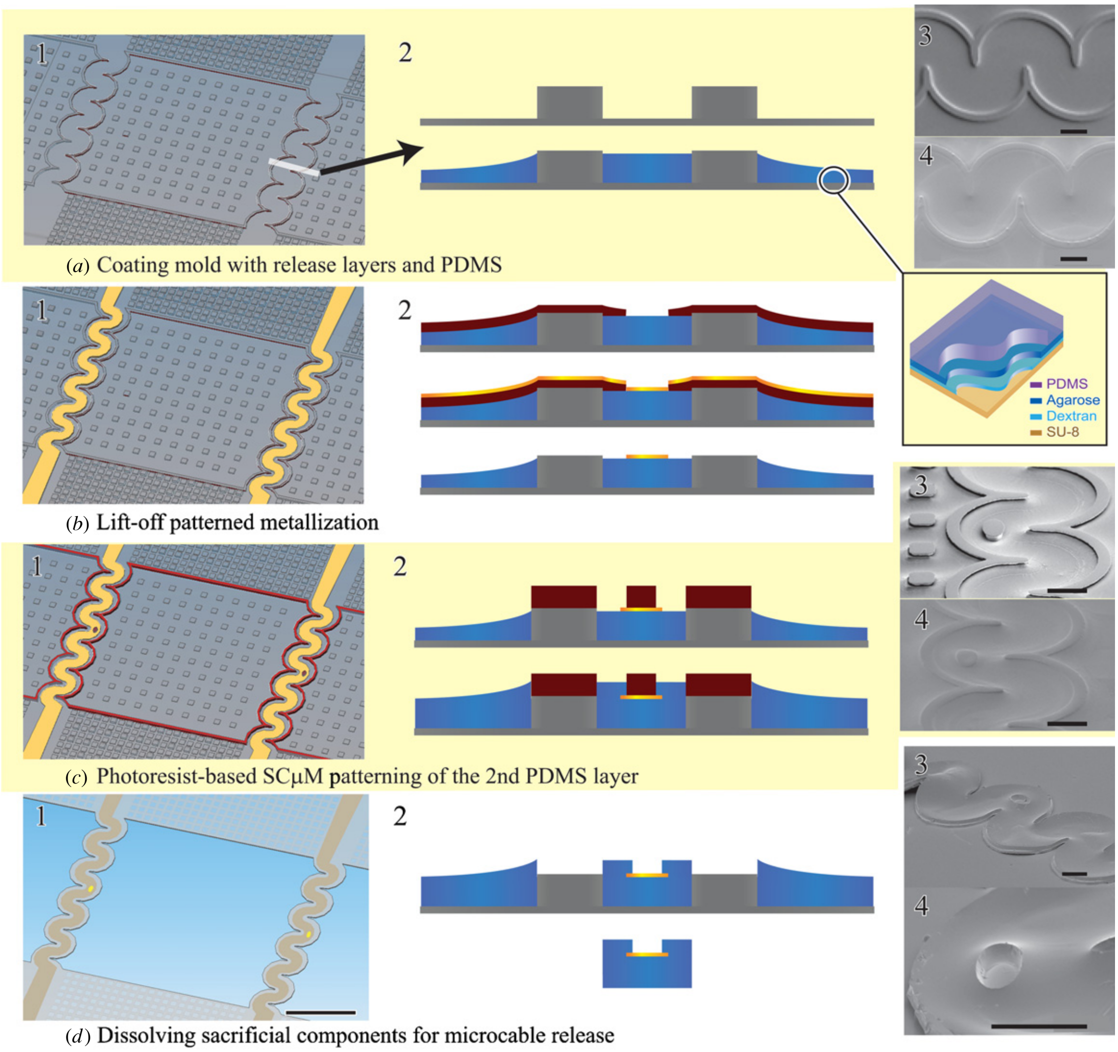

Figure 5. The main steps in the process flow are shown in graphical sequence. The three-dimensional representation (left) highlights specific steps in the process, while the corresponding process flow is shown in its entirety in the middle column. Relevant scanning electron micrographs of the microcables during processing are shown in the right column. (*a*1) The mold is sequentially covered with dextran and agarose release layers and PDMS. (*a*2) A cross-section of the mold and spun-cast layers is shown in the middle column. The thickness of the PDMS across the membrane varies depending on the spacing and geometry of the mold features. (*a*3) and (*a*4) The mold is shown with and without the PDMS layer. (*b*1) The electrical lead patterning is done using traditional metallization lift-off. (*b*2) A negative image of the leads is patterned with photoresist and metal is deposited over the sample. Dissolving the photoresist leaves the patterned metal trace on the PDMS substrate. (*c*1) The top insulating layer is defined using a sacrificial photoresist mold. (*c*2) The sidewalls of the microcable and the access node are defined with photoresist. (*c*3) The microcable array is shown in micrographs with the photoresist mold and (*c*4) after the top layer of PDMS has been spun cast. (*d*1) and (*d*2) After the photoresist is dissolved, the access node is exposed and the device is ready to be released. (*d*3) and (*d*4) A water bath is used to dissolve the agarose and dextran and the microcable array is separated from the mold. The scale bars are 500 μm for the drawing (left) and 100 μm for the micrographs (right).

the microcable structure and creates electrical access nodes along the cable length. The NR9-8000 photoresist is spun 30 μm thick, pre- and post-exposure baked in an oven at 85 °C, for five minutes, patterned with an exposure dose of 850 mJ, and developed with RD-6 developer. In figure 5(*c*), the cable and mold are shown before and after another layer of PDMS is spun-cast. (4000 rpm, 8 min; cured at 100 °C for 10 min.) After curing, the photoresist is removed with RR3, leaving the metal exposed at a selectively uninsulated access node (figure 5(*d*)). Note that in the process, the photoresist shields the gold surface from contact with the PDMS. After the PDMS is cured, dissolving the photoresist in a bath of TMAH creates an uninsulated site. The array is released in a water bath to dissolve the agarose and dextran. A single released microcable is shown in figure 5(*d*3). The access node is shown in a magnified view in figure 5(*d*4). After release, the array is placed on a microscope slide for mechanical support (figure 4). For electrical tests, the pads at the ends of the leads are connected to hook-up wire with electrically conductive silicone (Silicone Solutions; Twinsburg, OH)

4. Fabrication results

The SU-8 micromold and thick photoresist have been used to specify the geometry of an elastomer-based microcable array. The walls and posts respectively define microcables

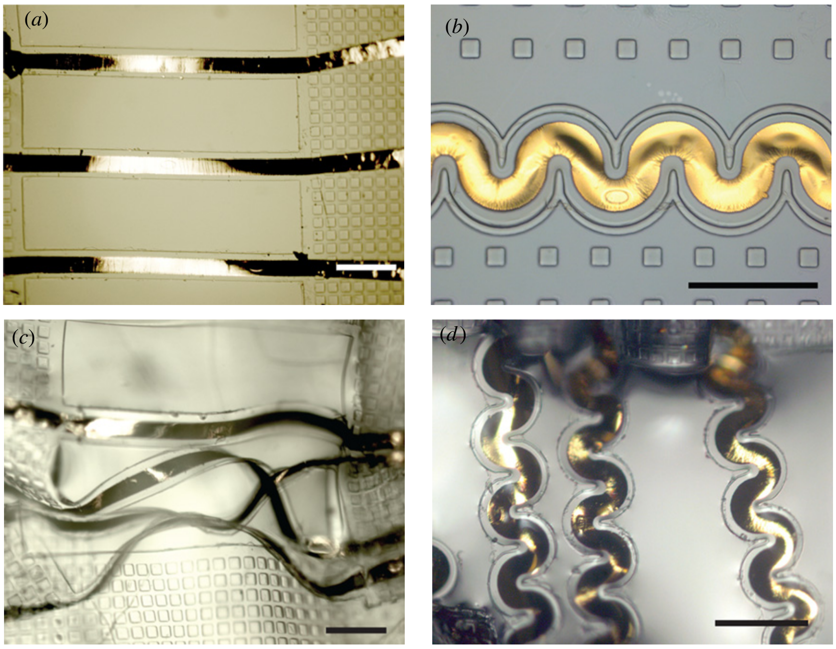

Figure 6. The photomicrographs show completed devices of straight (left) and sinusoidal (right) microcables. The arrays are shown laid flat (*a*) and (*b*) and in a water bath (*c*) and (*d*). The microcables are flexible and compliant after release, as demonstrated by their free-form conformation in the water bath. The scale bars are 400 μm.

and through holes in the spun-cast, PDMS membrane. As an example of the types of structures that can be fabricated using this process, consider the free-floating microcables shown in figure 6. The straight microcables have been placed flat on a slide to show their planar shape (figures 6(*a*) and (*b*)) and in a water bath to demonstrate their mechanical compliance (figures 6(*c*) and (*d*)). The membrane framing the microcable array has through holes from a subset of the posts, demonstrating that the SCμM process can be used to make micropatterned membranes several square centimeters in area. The through holes are shown corresponding to the subset of taller SU-8 posts (figure 7).

The microcables are 2 mm long and 200 μm wide. The access sites on the top insulated area are ovals 50 $\times$ 100 μm (approximate area: 4000 μm^2). The gold leads on the microcables are 100 μm wide. The average resistance for each lead is 136 Ω ($n = 6$, SD $= 30\ \Omega$). The resistance measurements were made with a multimeter after the array was released. The thickness (50 nm) was determined by measuring the resistance via a four-point probe (Signatone; Gilroy, CA) and Wyko optical profilometer (Veeco; Plainview, NY) of a film deposited on a glass slide under the same conditions. The calculated value for the leads, with 50 nm of gold is 50 Ω. Prior to release the average resistance was 89 Ω ($n = 6$; SD $= 6\ \Omega$). The resistance and standard deviation increases suggest microcracks. Cracking of gold thin-films on elastomers has been documented in the literature [14]; because of the compliance of the substrate the cracks do not tend to propagate to the point of full discontinuity. The larger than expected resistance in the unreleased leads (i.e. prior to any microcracking) may be due to the thinness of the film. The grain size of the film ranged from approximately 25 to 40 nm as measured via scanning electron microscope imaging (data not shown). The proximity of the grain size values to the film thickness, combined with the stress induced from the wrinkled topography of the gold and may have contributed to the increased resistance because this disparity was not found in films deposited on glass slides.

5. Applications

The use of patterned photoresist and SU-8 to create multi-layered devices with through holes is a useful technique when applied to multi-layer electronic devices, but it also has potential applications in mechanical and micro-fluidic PDMS-based devices where a patterned film of PDMS is desirable. Its compliance makes it a potentially useful microscale 'seal' for protection of electronic components from integrated fluidics or conversely, for directly facilitating fluidic containment. The substrate conformability and suitability for spin-casting were motivations for using Sylgard 184 (Dow Corning; Midland, MI), which has a low Young's modulus (1.78 MPa, [13]) and can be spun-cast and thermally cured *in situ*.

The spin times are on the order of a few minutes, the metallization uses a standard process and the SU-8 molds are reusable. The arrays can be fabricated relatively rapidly, allowing large quantities to be easily made. This is beneficial because biological studies require a relatively large number of samples. To encourage the use of elastomer-based electrodes as a biological tool, it is desirable to minimize supply limitations that might impact their implementation.

The microcable nets can also be made in a range of geometries to fit the requirements of the application. The sinusoidal leads shown in figure 6 provide more conformability than the straight leads. The extra extension or deformation available in the sinusoidal microcables may be useful; alternately accurate placement may be easier with straight microcables.

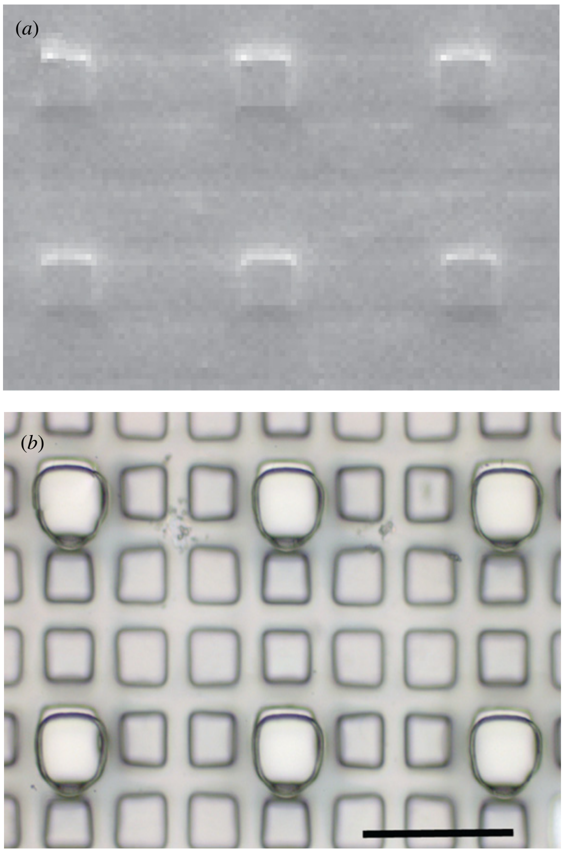

Figure 7. The SU-8 mold is coated with PDMS (*a*). The mold features are still visible through the cured layer of PDMS. As seen in the released PDMS membrane (*b*), the taller posts make through holes, while the shorter surrounding posts do not. The shorter set of posts created a substrate with a thickness and durability suitable for handling and metallization patterning. The taller set of posts was designed to allow fluid and gas exchange across the membrane. The scale bar is 200 μm.

The microcables have potential applications in neural and cardiac recording applications. The access sites are available for surface modification and could be used to stimulate or record electrophysiological signals. The compliance and open web-like conformation of the microcable array are conducive to recording over irregular or curved bodies, such as the surface of the brain or heart and do not create a barrier to fluid and gas exchange. The microcables are adaptable for use as shank-style electrodes (providing a temporary coating is applied to aid insertion [9]), wrapping around small features, such as peripheral nerves, or integration into cell and tissue culture systems.

6. Conclusion

Our implementation of SCμM in a multilayer electronic structure demonstrates a simple, relatively rapid process that can be used to make highly compliant electronic structures with patternable geometries. Spun-cast micromolding techniques can also be used to create multilayer structures, enabling electrical components to be insulated with selectively exposed sites. The sizes of features demonstrated here include 16 μm thick membranes featuring through holes with diameters of tens of microns and microcables with millimeter lengths and 10:1 aspect ratios. The multilayer feature allows conductive material to be insulated and selectively exposed. The SCμM process can be used to make components such as the microcable array that have applicability in biological applications.

Acknowledgments

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