Supplementary Materials

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1 Additional Characterization

1.1 Packaging bioMEMS/CMOS chips

3340 and ethanol

Exposure to ethanol (used in standard protocols to sterilize the chip, package, and working area) causes package failure (and therefore chip failure). Figure 1 shows cracks in the 3340 package after exposure to saturated ethanol vapor at 37° C for 1 hour. Exposure to ethanol in liquid form causes immediate cracking.

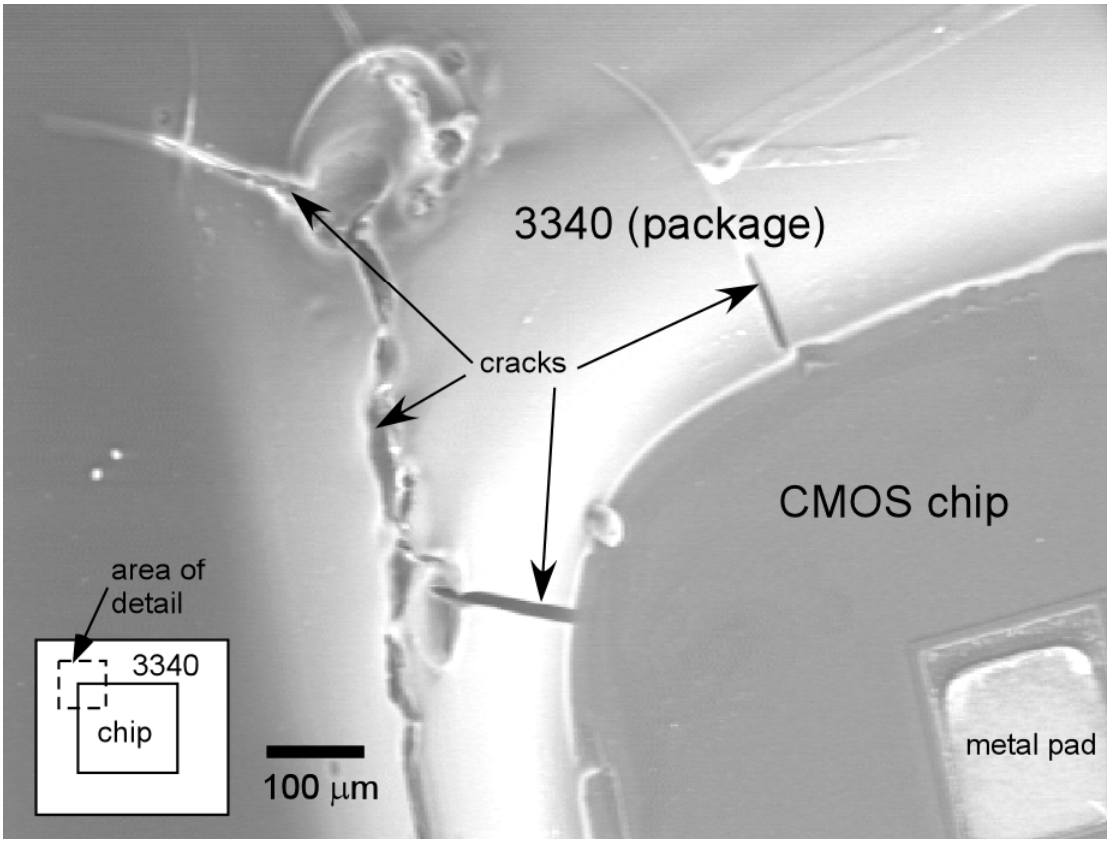

Figure 1. Scanning electron micrograph of a chip packaged with 3340 (see section 4.1 of the paper) after exposure to ethanol vapor.

3108

Based on the 30-day swelling data, one would expect the performance of a chip packaged with 3108 to be comparable to that packaged with 3340. However, this is not the case, as shown in Figure 2. Hank's buffered saline solution containing bovine aortic smooth muscle cells and neutral red was placed over a chip packaged with two levels of 3108, and the cells allowed to plate over 24 hours. The chip was then powered up, but after 1-2 minutes the chip short circuited. Both the water and the dye had penetrated the polymer, and electrochemistry occurred at several of the wires, as seen by the dark red and green colors. The reason for the difference in performance between 3340 and 3108 is probably that the former swells more slowly (see Figure 10 below), and can thus be used for some time before the water content becomes too high or the polymer cracks.

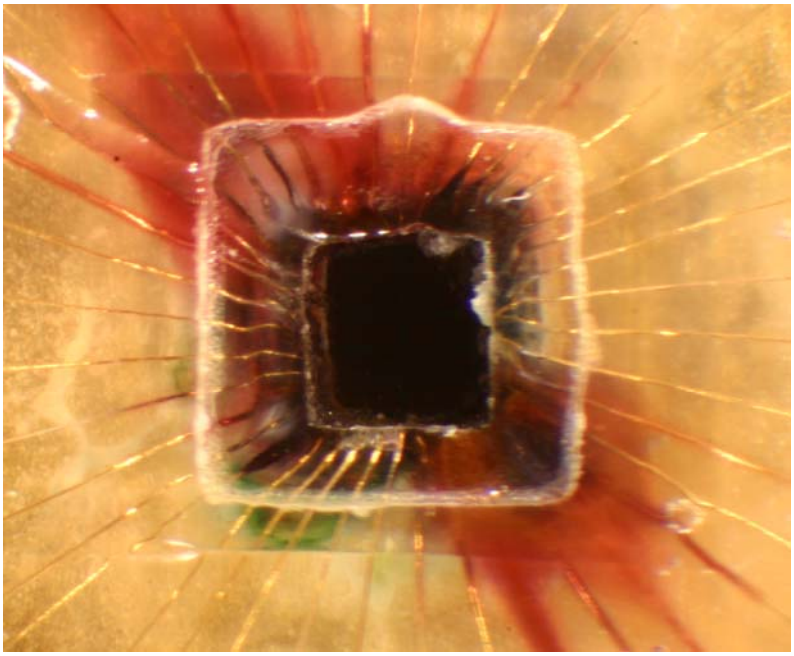

Figure 2. Overhead view of a chip (dark square in the middle) packaged in a DIP40 using two levels of 3108 and the same procedure as described in section 4.1 (except using 3108 instead of 3340).

1.2 Electroplated metal films formed in Loctite templates

Molds with high sidewalls are required to electroplate thick metal layers, and the mold material should be easily removable. Most photoresists are limited to tens of μm in thickness. SU8 is a negative photoresist that can be patterned to thicknesses up to 250 μm, but it can be difficult to remove. Therefore, we tested whether 3340 and 3108 could serve as plating molds, and both could. Because these adhesives can be patterned as thick films, they could potentially mold several mm-high electroplated features.

Figure 3 shows a 350 μm thick patterned nickel film electroplated on a chromium/gold seed layer. Dark field molds like this are more successful than light field molds such as the one shown in Figure 4 *if the template is removed by swelling* because Loctite features are wider at the bottom than at the top (see Figure 7 below). Therefore, the template polymer should be removed by dry etching in O_2 plasma.

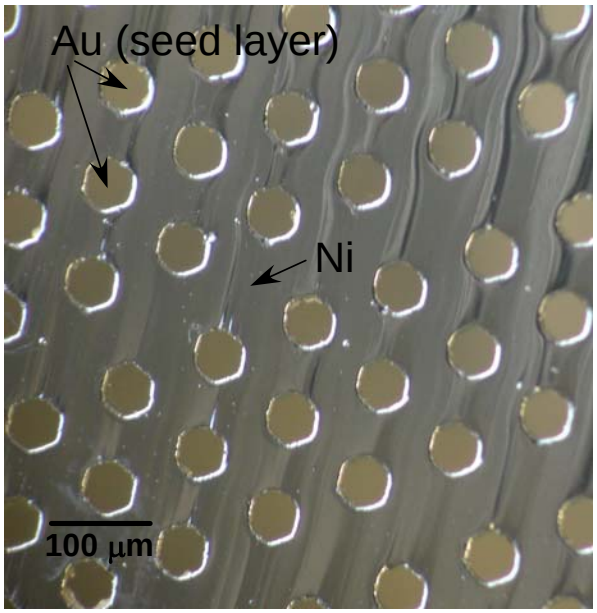

Figure 3. Electroplated nickel film 350 μm thick on chromium/gold. An array of Loctite 3108 hexagons was patterned on top of the gold prior to electroplating. (The waviness on the film is normal for such large areas and thicknesses.)

Electroplating was performed at current densities between 10 and 30 mA/cm^2 at room temperature using a commercial plating solution (Bright Nickel JB, Technic Inc.). An EcoChemie Autolab pgstat 30 controlled the electroplating current. The reference electrode lead was connected to the counter electrode lead, which was connected to a piece of graphite. The working electrode was connected to the sample.

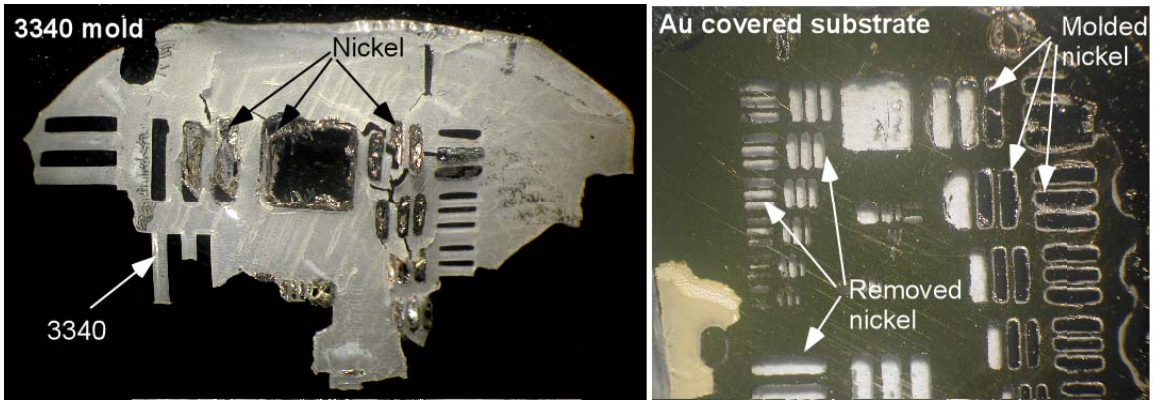

Figure 4. (left) Loctite 3340 template used for electroplating nickel, shown after removal from the substrate by swelling. The Ni features that were removed with the template are readily apparent. (right) The substrate, showing the Ni features that remained and the white patches where the Ni was removed.

1.3 Additional SEMs of patterned Loctite features

1.3.1 Loctite 3108

One of the difficulties with 3108 is re-deposition of residue following rinsing in a good solvent such as acetone. To circumvent this problem, sonication can be performed to leave clean surfaces (Figure 5). However, the width of the features is reduced by approximately 20% by the sonication step.

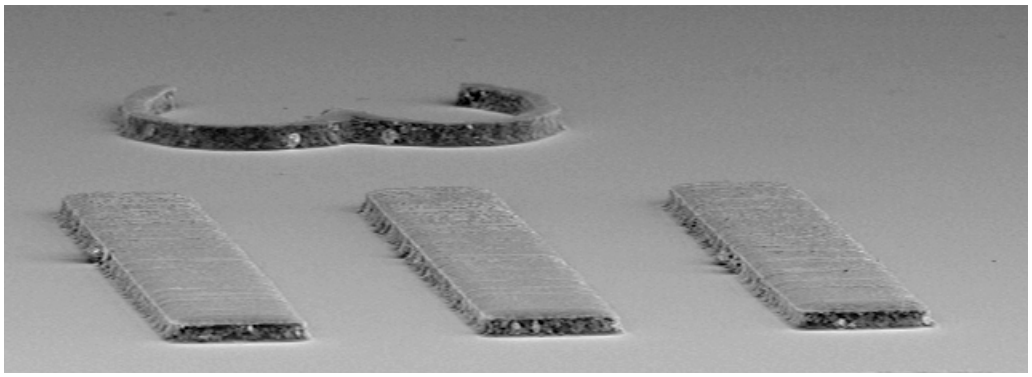

Figure 5. 3108 features 400 µm wide rinsed with ethyl acetate and then sonicated in ethyl acetate for one minute.

Molding 3108 over an SU8 template results in features of the desired size with no significant shrinkage (Figure 6).

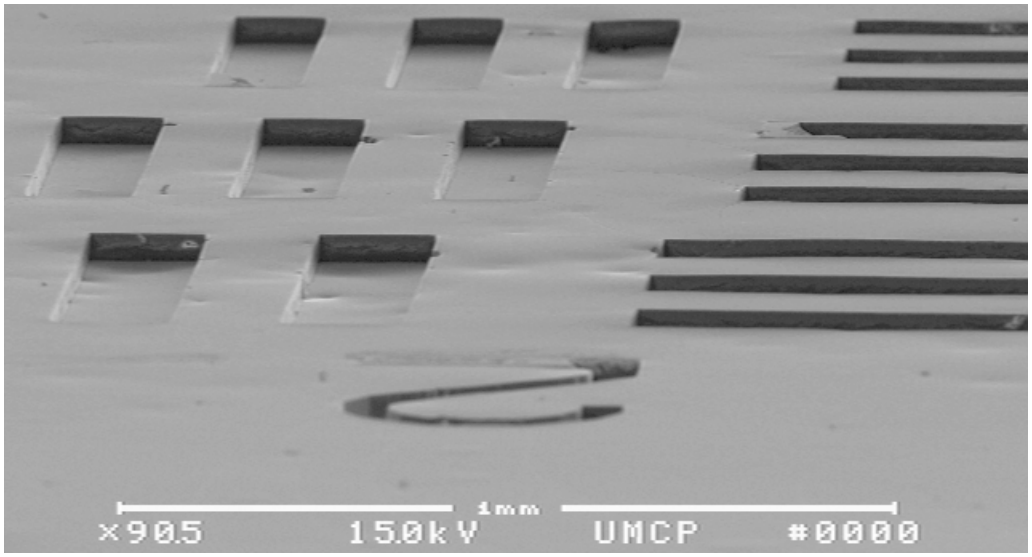

Figure 6. 3108 features molded with an SU8 template.

1.3.2 Loctite 3340

As mentioned previously, the sidewalls of 3340 features rinsed with ethyl acetate are sloped, so the features are smaller at the top than at the bottom (Figure 7). After an RIE descum, however, sidewall verticality improves, as illustrated in Figure 8. Care must be taken not to leave the polymer in the RIE too long, however, or the features may lift off.

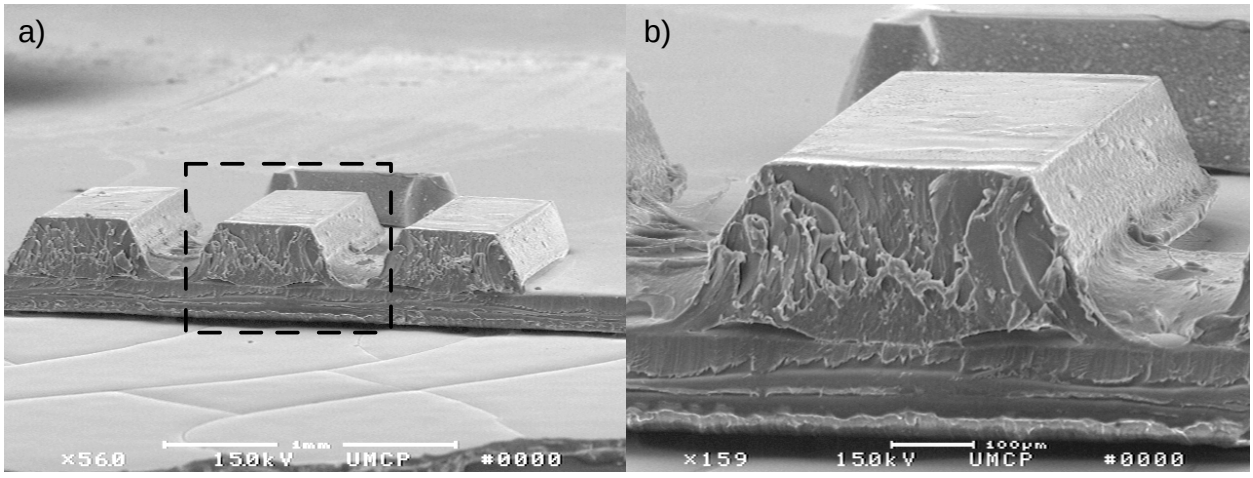

Figure 7. a) Cross-sectional view and b) close-up of 3340 features 250 µm wide rinsed with ethyl acetate.

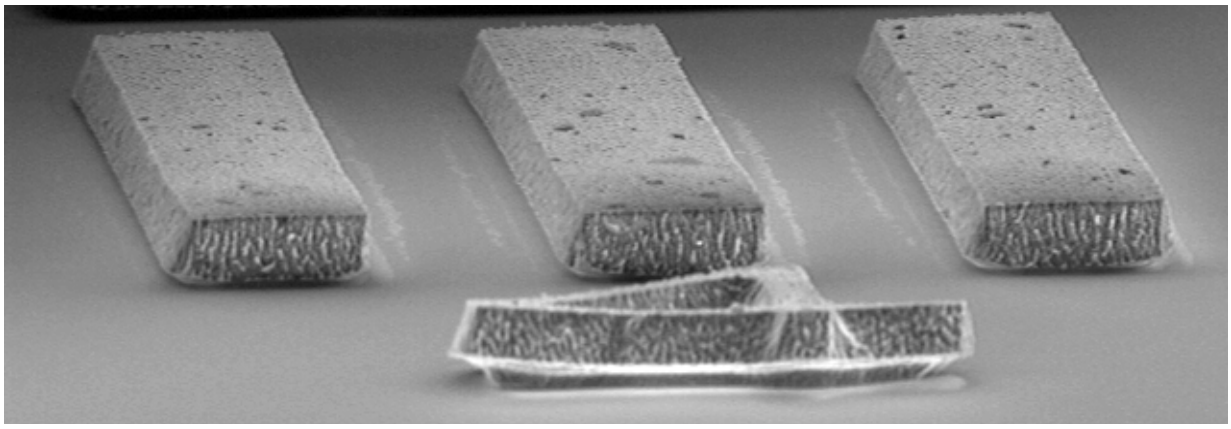

Figure 8. Loctite 3340 features 175 µm wide following a 10 minute RIE descum.

1.3.3 Loctite 3525

Loctite 3525 (as opposed to 3108) does not need a sonication step to leave clean surfaces. A rinse in a good solvent is sufficient to provide features with no redeposited residue, as illustrated in Figure 9.

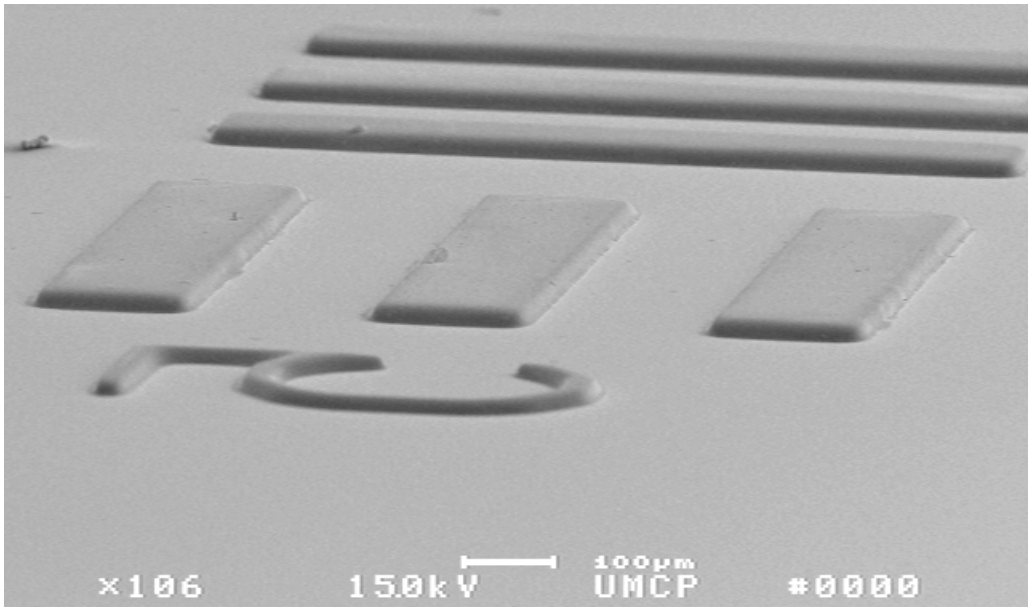

Figure 9. 3525 features 160 µm wide rinsed with ethyl acetate.

1.4 Swelling in solvents

Swelling in water

Figure 10 shows swelling in water over time for 3340 and 3108. The amount of swelling of the two polymers after a month is not significantly different. However, 3108 swells *faster* than 3340, which may explain why 3340 can be used as a packaging material and 3108 cannot. (The 3340 packages fail after approximately one week under cell culture conditions.) At room temperature in water, the weight change of 3340 increases from approximately 6% to 10% after 100 hours, whereas Loctite 3108 swells 10% after only 10 hours.

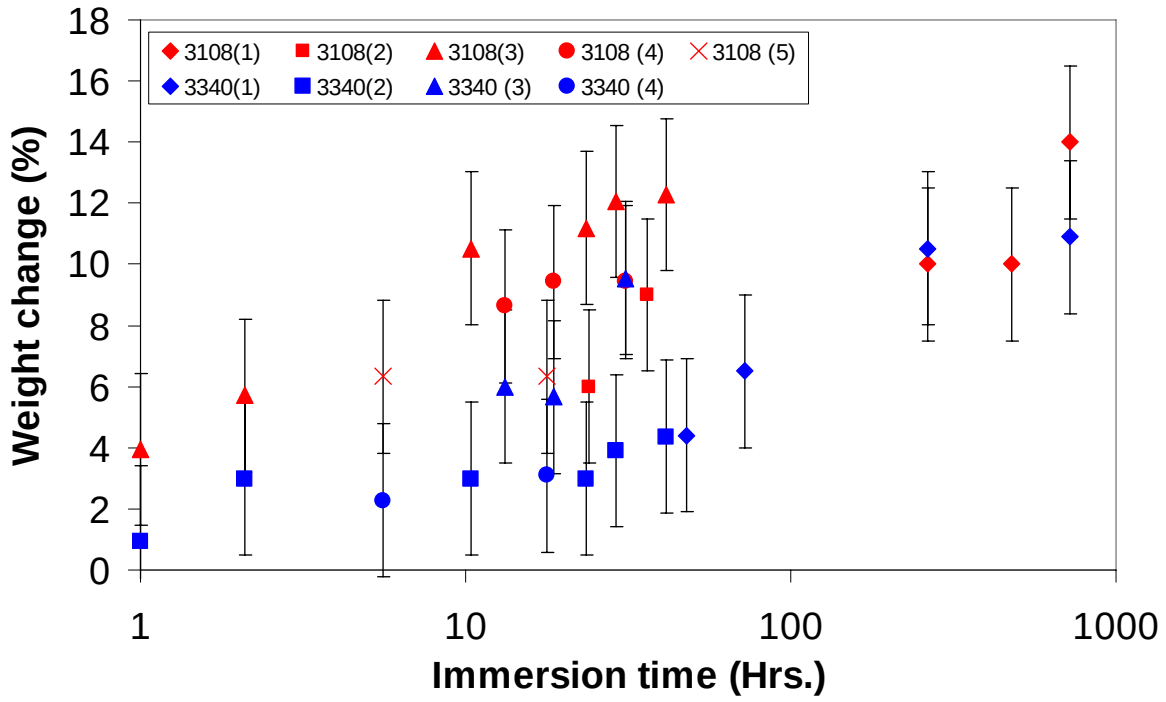

Figure 10. Swelling of 3108 and 3340 in water as a function of time, log scale.

Swelling in solvents

In organic solvents, Loctite 3108 swells the most and 3340 the least. For all three polymers, the swelling in hexane (solubility parameter = 15) was low, and swelling in solvents with solubility parameters around 18-19 was highest. Loctite 3108 also swelled significantly in xylene, and 3340 swelled more than expected in methanol. In Figure 13, line colors indicate a solubility parameter below (blue), at (red), or above (black) that for maximum solubility of the precursor; water is indicated in green. Swelling does not increase significantly after the first 72 hours, except for the case of 3340 in water.

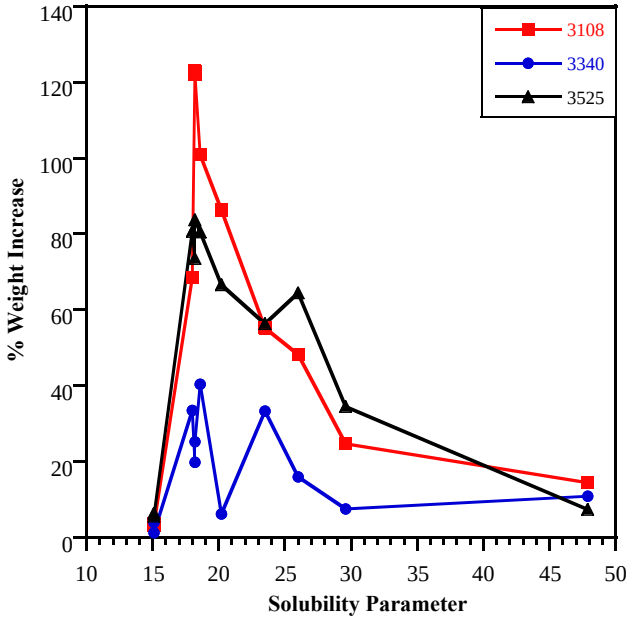

Figure 11. Swelling as a function of the Hildebrand parameter after 30 days (720 hours).

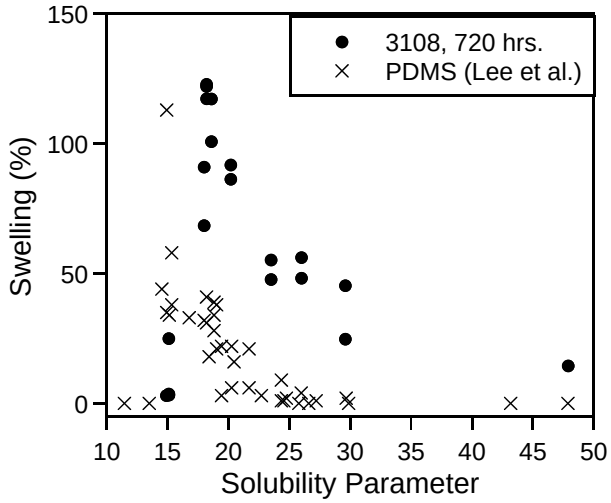

Figure 12. Swelling of 3108 after 30 days compared with published swelling data for PDMS [*]. The swelling for 3108 is a weight change, but for PDMS it is a volume change.

[*] J. N. Lee, C. Park, and G. Whitesides, "Solvent compatibility of poly(dimethylsiloxane)-based microfluidic devices," Anal. Chem., 75 (23), 6544-6554 (2003).

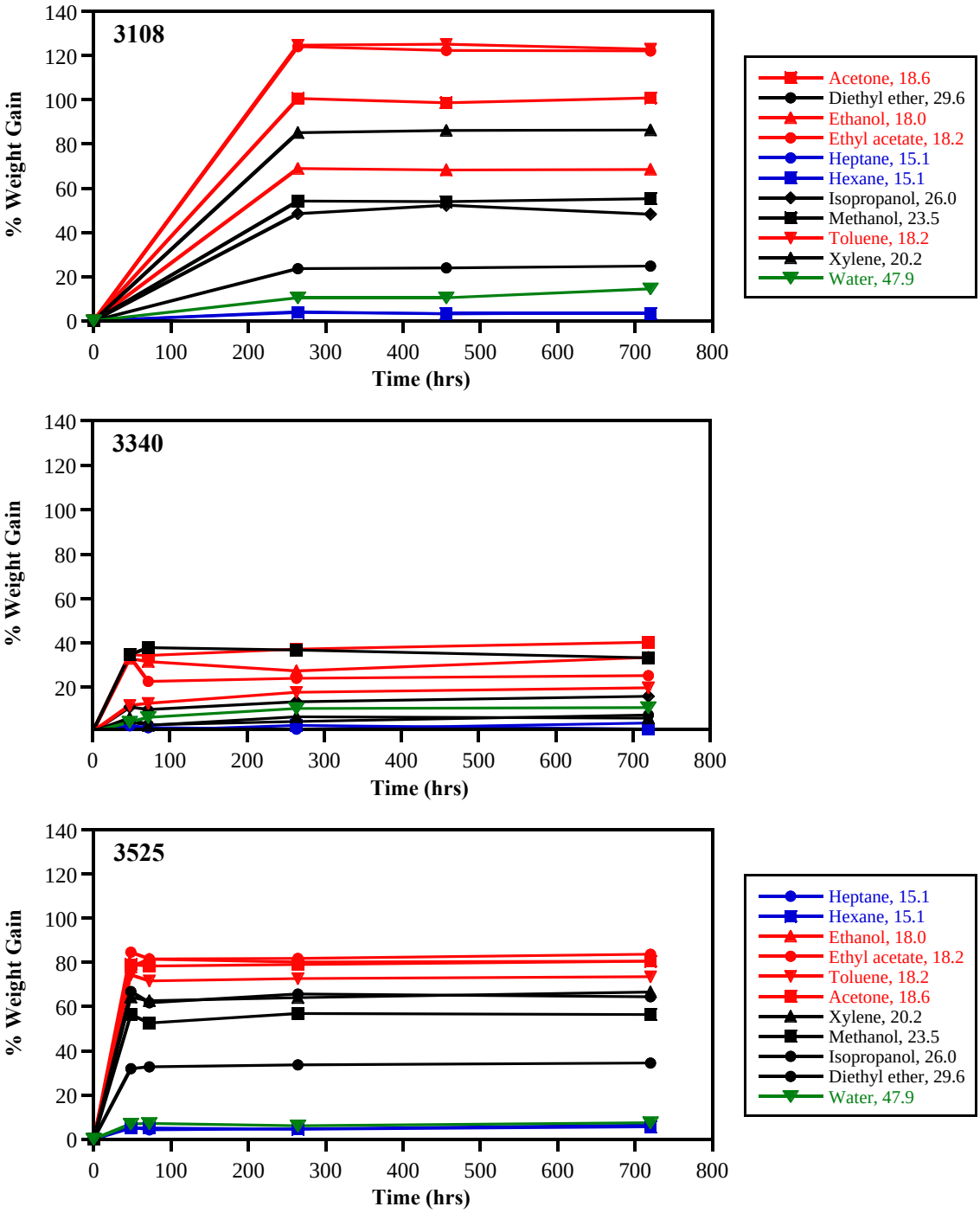

Figure 13. Swelling of 3108, 3340, and 3525 in various solvents over time. The top legend lists solvents alphabetically, the bottom by solubility parameter.

2 Illustrated Guide for Employing Benchtop MEMS Techniques

2.1 Materials required

The fabrication of films and devices using Loctite polymers can be accomplished with a few inexpensive, commercially available materials. Figure 14 displays several of these items.

Loctite polymer precursor
low resolution transparency masks (or high resolution Cr on glass)
gloves
solvent (acetone or ethyl acetate) in squirt-bottles
water
waster container
tissue paper
UV lamp
PDMS or SealView
glass and/or plastic slides
connectors, hoses, syringe
electric drill
Plexiglas
tweezers
gelatin

Processing can be done in normal room light, even if the room has windows.

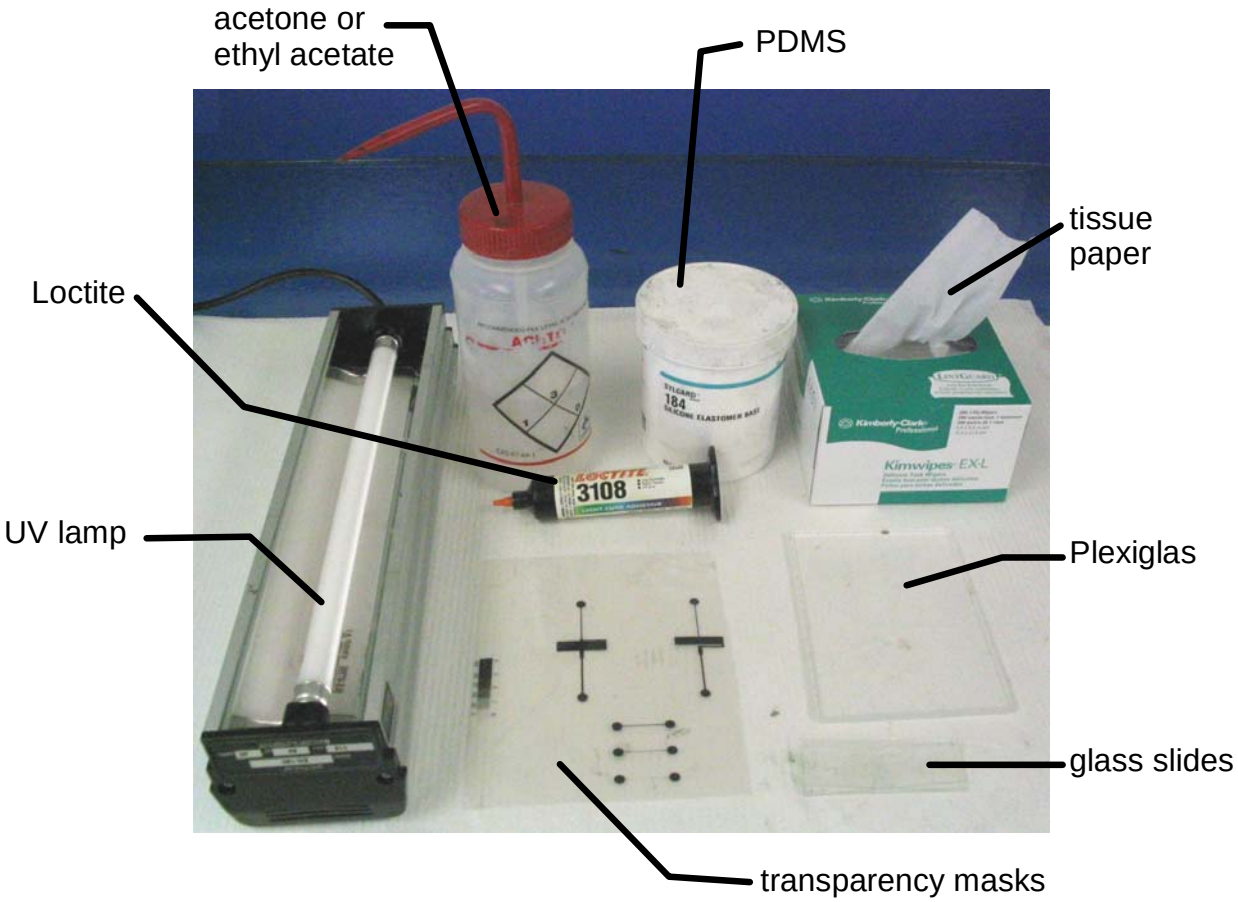

Figure 14. Materials and tools needed to fabricate benchtop MEMS.

2.2 Use of Loctite 3108 as an etch mask for patterning a metal film

This example shows how to use the Loctite 3108 as a negative photoresist. In the figures, a Cr/Au-coated Kapton film was used as the substrate. Metal film-coated polymer sheets are available commercially.

Step 1. Dispense the polymer precursor onto the surface (Figure 15). In the photographs, the metal-coated Kapton is lying on a piece of glass. Any metal whose etchant is withstood by the Loctite (see Table 5 in the paper) can be etched. As mentioned in the article, the Loctites can also be used as masks for etching glass.

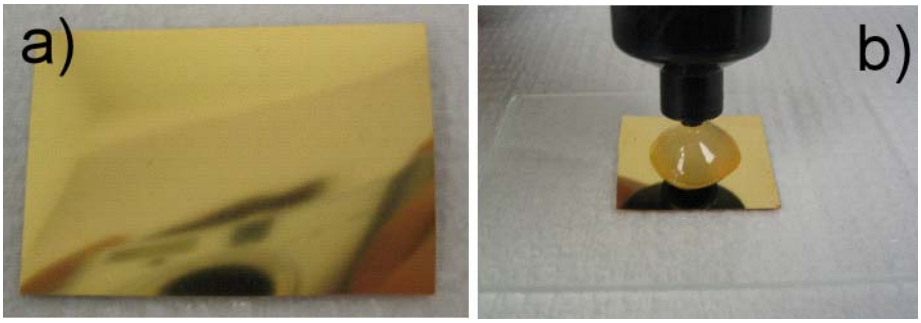

Figure 15. a) Starting with a gold-covered Kapton film, b) the Loctite is dispensed onto the substrate.

Step 2. Cover the surface of the 3108 with a piece of plastic wrap, such as Saran or SealView (Norton Performance Plastics Corp.) (Figure 16a). The cured Loctite will not stick to these plastics, but they will stick to the transparency mask. Place the mask ink-side-down over the film and cover it with a piece of glass to ensure flatness (Figure 16b). The mask shown here is a transparency printed on a high-resolution (3000 dpi) commercial printer. The adhesive is a negative resist, cross-linking where it is exposed.

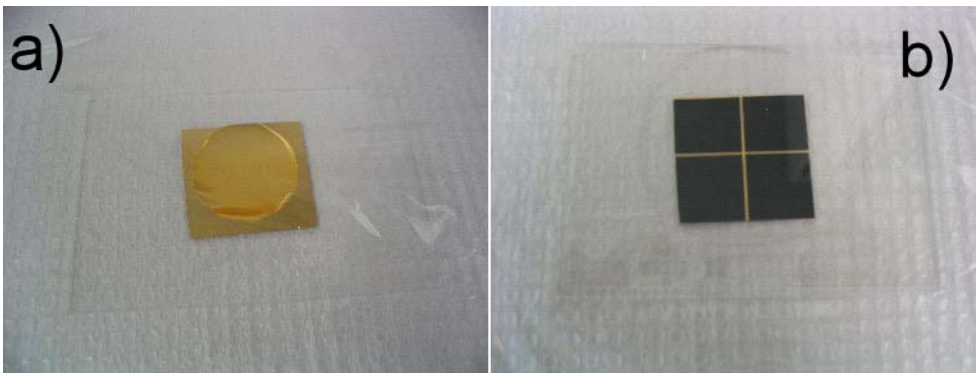

Figure 16. a) Cover the Loctite with Sealview or Saran wrap to prevent the adhesive from sticking to the mask. b) Place the mask face-down on the non-stick surface and cover with a piece of glass.

Step 3. Expose the Loctite 3108 with a UV lamp (for example with a Spectroline model EN 180 or in a mask aligner) (Figure 17). Exposure times are given in the paper for several thicknesses; for this procedure the time is approximately 40 seconds.

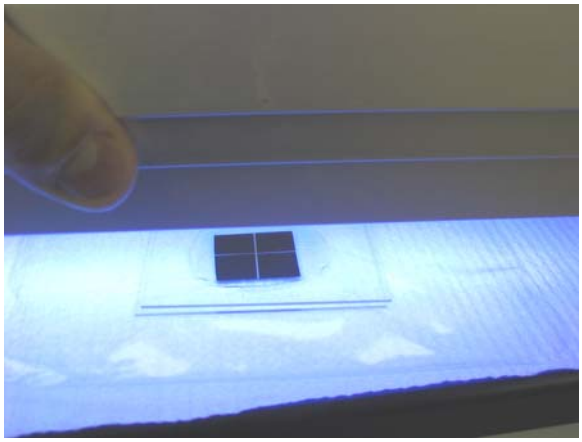

Figure 17. Expose the substrate to UV light.

Step 4. Remove the glass slides, mask, and Saran wrap. Rinse the sample with acetone or ethyl acetate in a squirt bottle to remove the unexposed 3108 and develop the pattern. The cured polymer serves as an etch mask in the following step.

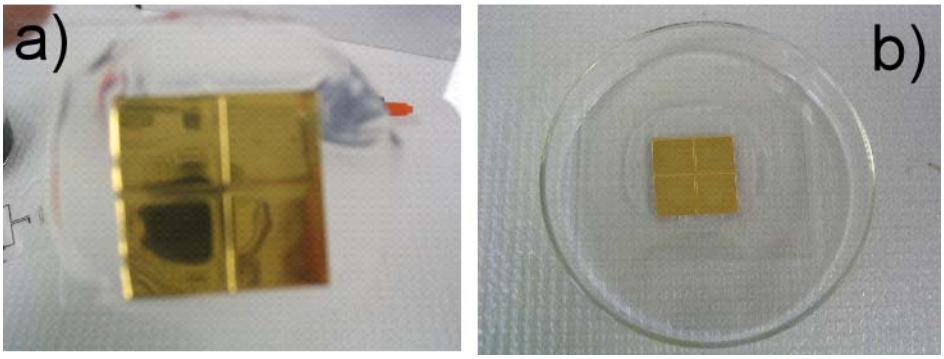

Figure 18. Take off the glass, mask, and non-stick plastic and rinse with a solvent. The polymer cures where it is exposed and is removed elsewhere by the solvent.

Step 5. Place the sample in the etchant (Figure 19a). Gold etch is a harmless aqueous solution of iodine and potassium iodide, which can be mixed or purchased commercially (Transene). After the gold has been etched, rinse the sample in water (Figure 19b).

Figure 19. a) Etch the gold and b) rinse.

Step 6. Dry the sample and peel the 3108 from the substrate (Figure 20). (Soaking the 3108 in acetone for some time makes it easier to peel off.)

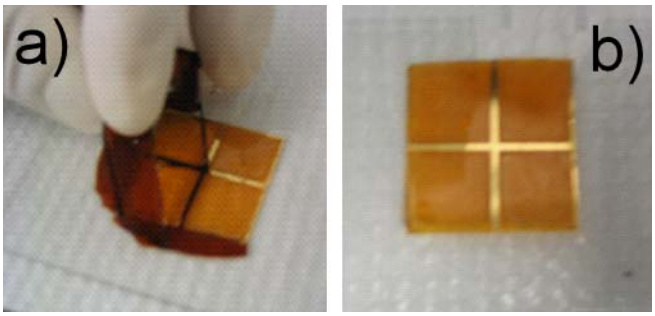

Figure 20. a) Remove the mask to b) obtain the etched film.

2.3 How to make a microfluidic channel using 3108

This example shows how to make a channel on a Plexiglas substrate with a transparency mask as the top cover.

Step 1. Drill two holes 1/16" in diameter into a piece of Plexiglas; the holes will serve as inlet and outlet for the finished channel (Figure 21). Use the mask as a guide to position the holes.

Figure 21. Plexiglas substrate used in the fabrication of microfluidic devices with two holes.

Step 2. Apply a bead of 3108 to the surface of the substrate (Figure 22a). Place a transparency mask directly on the surface of the 3108 (Figure 22b). Note that the dark circles of the transparency overlie the holes.

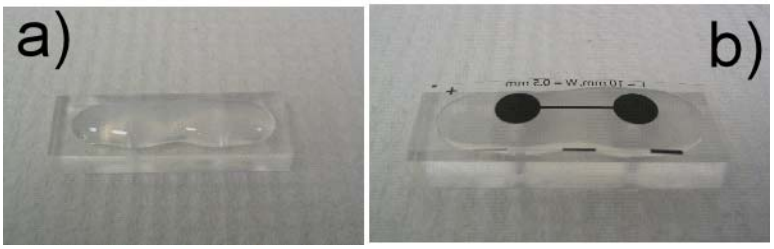

Figure 22. a) The 3108 is applied to the substrate, and b) the mask is placed directly on top, to be permanently bonded.

Step 3. Expose to UV light for approximately 40 seconds (Figure 23).

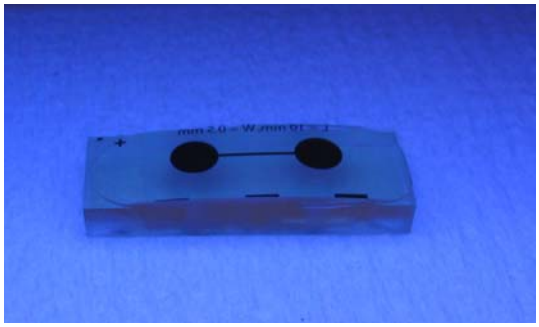

Figure 23. Expose the 3108 to UV light through the mask.

Step 4. Connect commercially available fittings (nylon, 1/16" McMaster Carr 5117k41) into the holes drilled in the substrate and attach tubing to the fittings (Tygon super-soft high purity tubing,

McMaster Carr 9449k11, OD 3/16”, ID 1/16”, wall 1/16”). Place one tube into a beaker of acetone and connect the other to a syringe (10 ml disposable). Create suction with the syringe and draw acetone through the channel, clearing out the undeveloped 3108 (Figure 24).

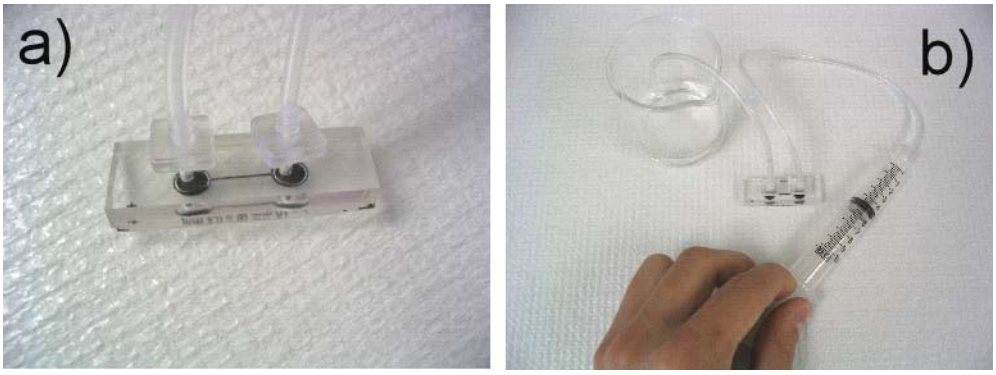

Figure 24. a) Connect tubing and b) remove the uncured 3108 by flushing acetone through the channel.

Step 5. The acetone can be cleared from the channel by drawing water through the channel. To demonstrate channel operation, water containing food coloring (McCormick) can be drawn through the channel (Figure 25).

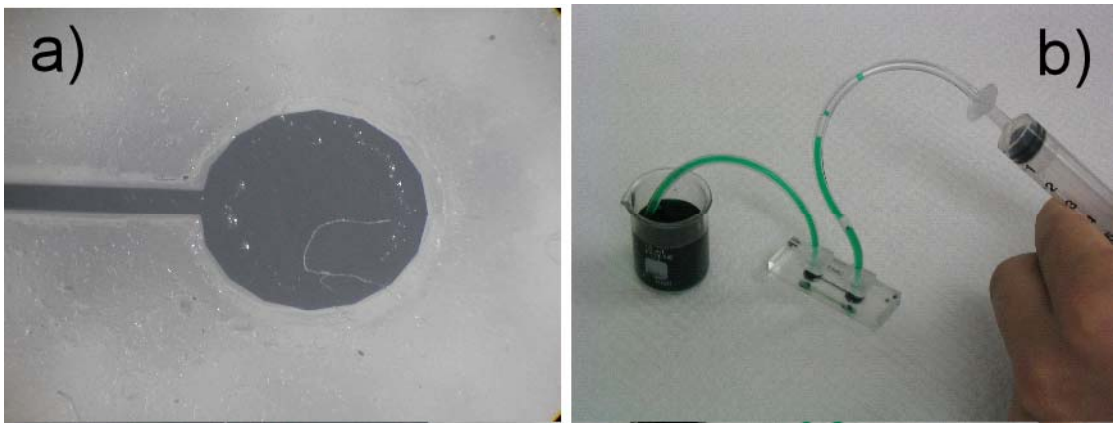

Figure 25. a) A cleared channel. b) Fluid running though the microfluidic channel.

2.4 How to make multi-level channels using Loctite 3108

The use of a sacrificial layer is one of the cornerstones of surface-micromachining. Gelatin is a suitable sacrificial layer material for use with the elastomers and benchtop fabrication. This example shows the fabrication of a two-level microchannel on a glass substrate, but more complex structures are of course also possible.

Step 1. Drill a hole into a piece of glass using a drill bit designed for glass (1/8" Glass & Tile Drill Bit, Black & Decker) and attach a fluid connector as described in section 2.3. Dispense 3108 onto the substrate (Figure 26a). Rub Sylgard 184 elastomer base onto the printed side of the mask to prevent it from sticking to the cured polymer. Place spacers onto the substrate to define the channel height. Align the left-hand reservoir on the mask over the hole in the substrate (Figure 26b). Cover with glass and push down to the level of the spacers. Expose the 3108 to UV light for 40 seconds, peel off the mask, and develop the channel with acetone.

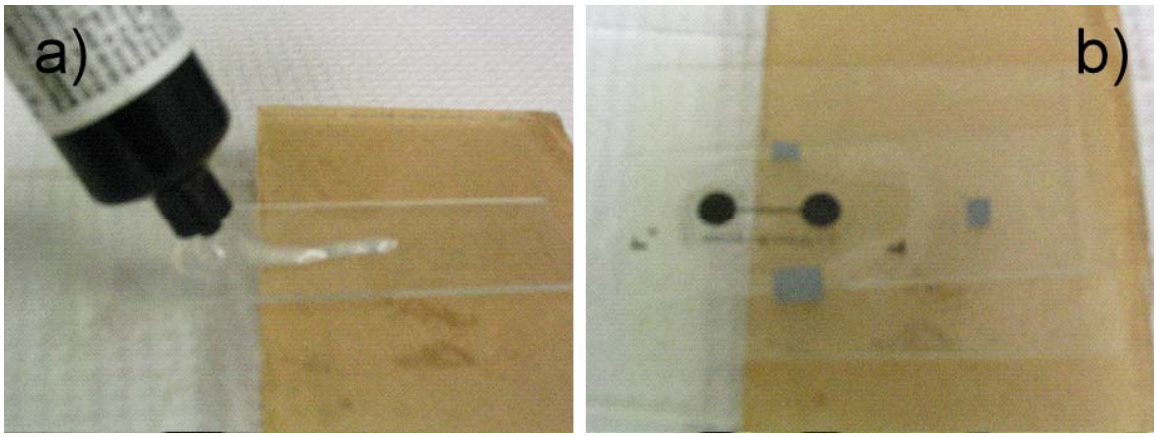

Figure 26. a) The 3108 is dispensed onto the glass substrate and b) covered with a mask coated with Sylgard 184 elastomer base. The gray squares are silicon wafer pieces 500 μm thick that are used as spacers to give a defined film thickness.

Step 2. Fill the patterned channel with gelatin and let it solidify in the refrigerator. The thin layer takes only a few minutes to solidify.

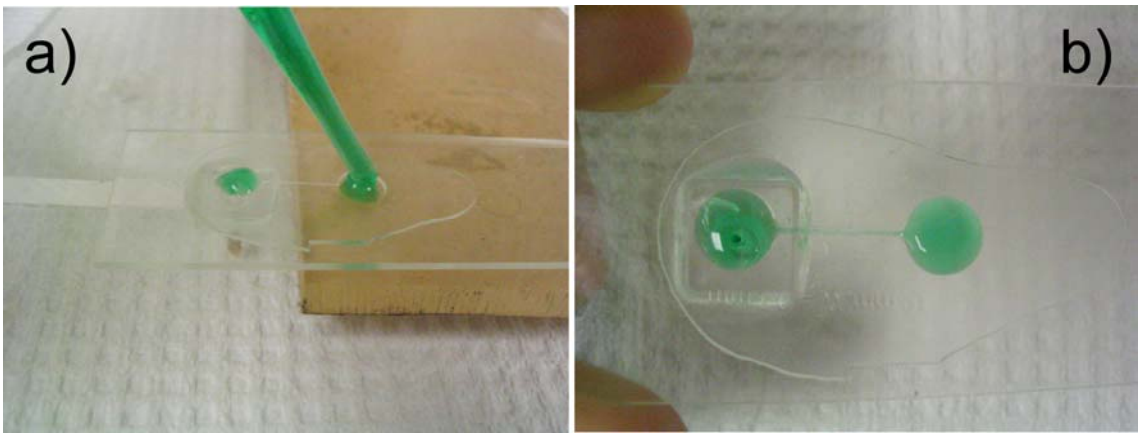

Figure 27. a) The channels are filled with gelatin which is b) solidified in a refrigerator. The gelatin has been dyed with green food coloring.

Step 3. Dispense additional 3108 over the first channel. Align the coated mask over the glass so that the left-side reservoir for this layer lies over the right-side reservoir of the lower channel. Expose this layer for only 30 seconds to underexpose the polymer. Peel off the mask, but note that the polymer is not completely cured. This will allow sealing of the two-layer channel with a second glass slide.

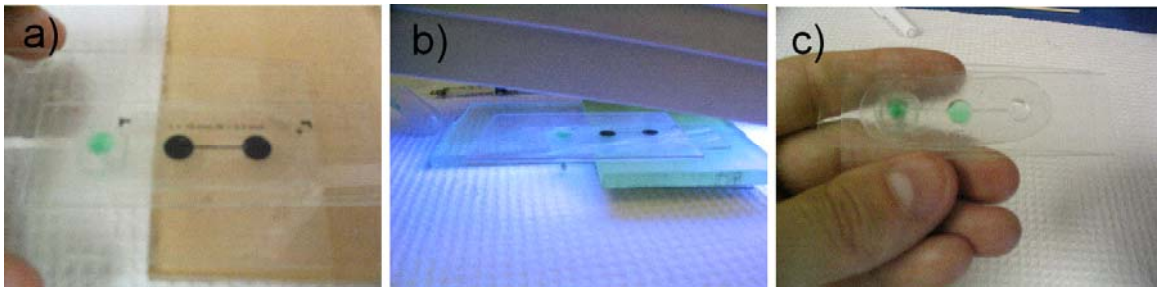

Figure 28. a) Apply 3108 over the solid gelatin and align the mask for the second channel. b) Expose to only partially cure the polymer. c) After removing the mask, the polymer is still sticky.

Step 4. Place a second glass slide with one hole drilled in it on top of the underexposed 3108. Place the mask face down on the glass and align it with the previously exposed pattern. Expose again for approximately 15 seconds.

Step 5. Heat the gelatin in a bowl of hot water to melt the gelatin. The temperature at which the gelatin melts can be controlled by the amount of water in the gelatin mix.

Step 6. Push slightly on the left-side reservoir to force the unexposed 3108 and gelatin out of the channel. Put a connector into the hole of the top glass slide and pull acetone through the channel (from the lower channel to the upper channel) using a syringe. This completes the 2-level channel.

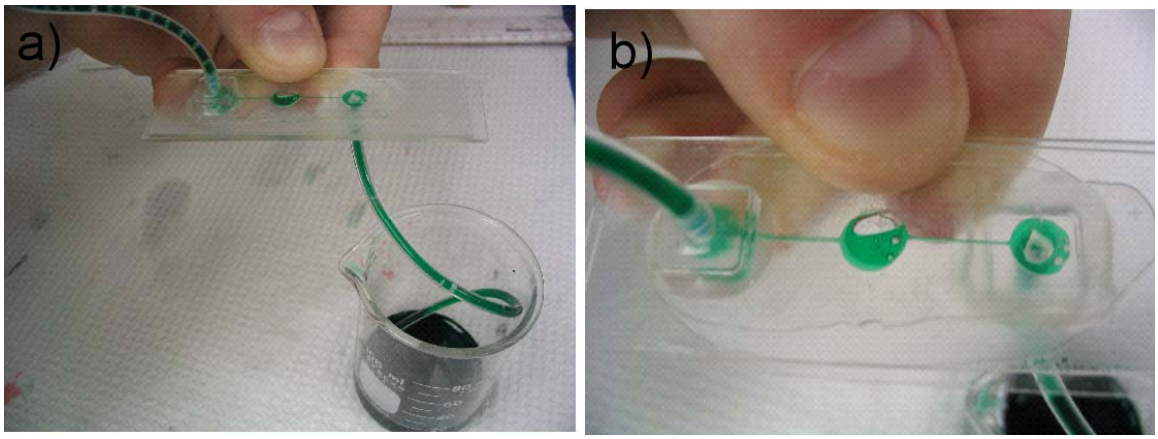

Figure 29. a) A complete two-level microfluidic channel shown with dyed fluid. b) A close-up.

2.5 Use of Loctite 3108 for fabricating a magnetic composite

Mix magnetic particles (strontium ferrite powder HM410, particle size ~2 μm, Hoosier Magnetics, Inc.) into the 3108 and stir. Place the mixture onto a substrate and cure with UV light.

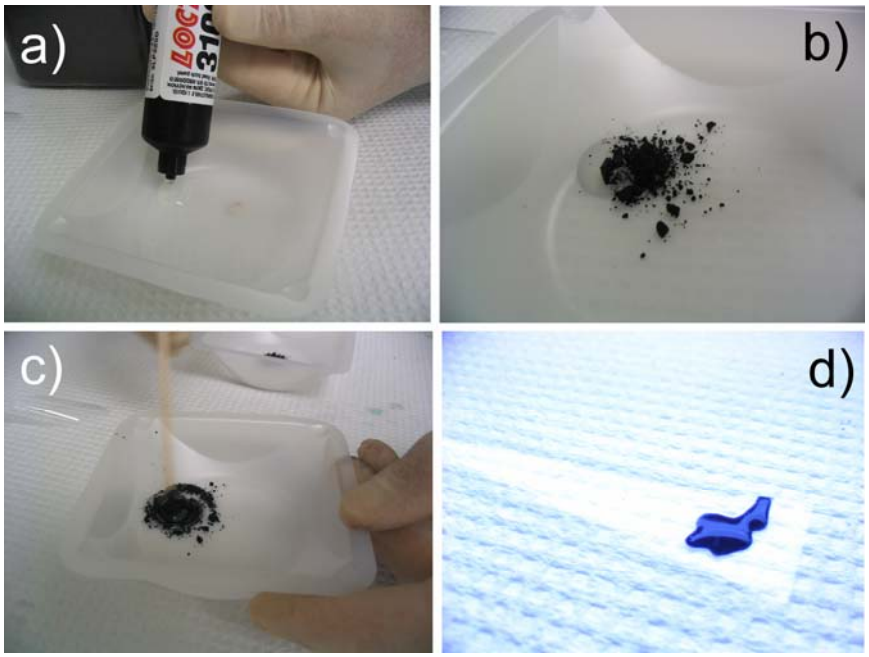

Figure 30. a) Dispense Loctite into a plastic dish. b) Add magnetic particles. c) Mix by hand or use a homogenizer. d) Transfer the mixture to a substrate and expose to UV light.

In order to demonstrate that the mixture is magnetic, the composite was cured on a piece of SealView and brought near a magnet, as shown in Figure 31.

Figure 31. The magnetic composite is attracted to a magnet.

The precursor with magnetic particles in it remains patternable (Figure 32).

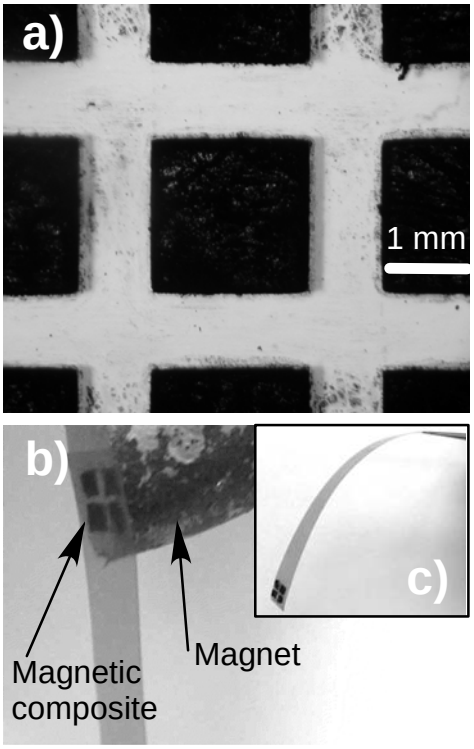

Figure 32. a) Patterned film with 10% $^{v}/_{v}$ strontium ferrite on Kapton; the squares are 2 mm on a side. b) Patterned composite attracted by a magnet, overcoming gravity. c) Kapton strip bending under gravity.

2.6 How to make a film with varying stiffness

Loctite that is exposed to UV light for different amounts of time has different stiffness. Exposing some areas for longer times and others for shorter times through different masks gives areas of varying stiffness, forming a functionally graded material.

Figure 33 shows a strip 500 μm thick and 7 mm wide that was exposed for a longer time on the outer edges (45 sec) and a shorter time in the center (15 sec). The strip bends under its own weight at the region with lower Young's modulus. In order to retain the low modulus upon further exposure to light, it is necessary to rinse crosslinking species out of the material in a good solvent. Immersion in acetone for a day will accomplish this.

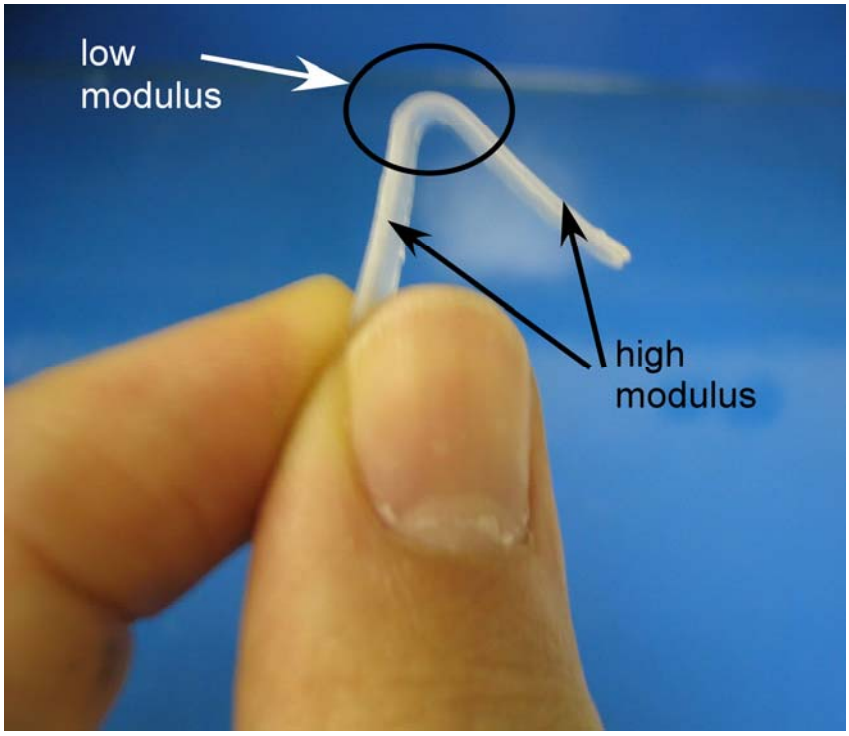

Figure 33. 3108 with varying mechanical stiffness.

2.7 How to make a structure with continuously changing sidewall properties

The ability to fabricate channels with sidewalls that have continuously changing properties, such as zeta potential, may be of interest. This example shows the feasibility of doing this through the fabrication of a slab which has material 1 (3108) in a wedge shape and material 2 (3108 with coloring) in another wedge pointing in the opposite direction (Figure 34a). The technique could be used with a mask to make a channel with walls whose average properties change along its length. Features of graded height using spacers have been presented by Harrison et al.*

Step 1. Dispense 3108 on a substrate and cover it with a glass slide coated with PDMS elastomer base. Tilt the slide at an angle by putting a spacer on only one side.

Step 2. Expose the polymer to UV light and peel off the slide.

Step 3. Dispense 3108 with coloring on top of the cured wedge, cover with a glass slide, but place the slide horizontal to the substrate by using spacers on both ends. Expose to UV light, and peel off the top slide.

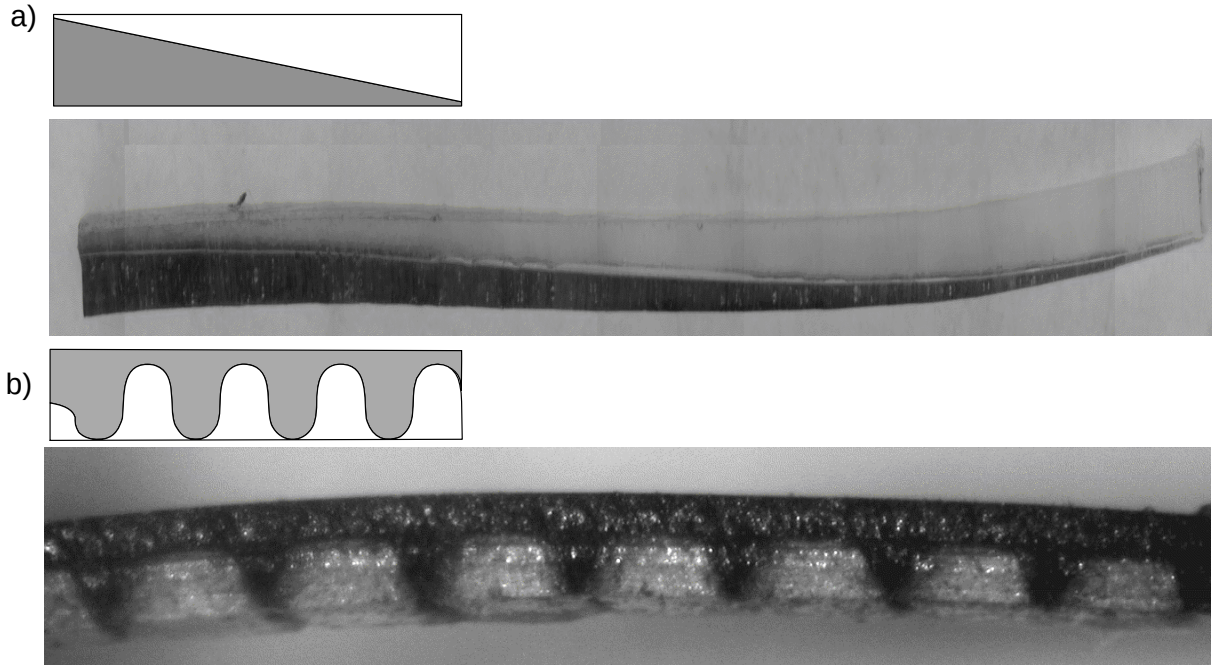

Figure 34. a) Cross section of a film with varying composition along its length formed by tilting the top cover during the first polymerization. b) Example of using a relief mask with a grating to pattern the bottom layer (clear). The top layer (colored) was poured over that, and cured under a flat plate.

* C. Harrison, J. T. Cabral, C. M. Stafford, A. Karim, and E. J. Amis, "A rapid prototyping technique for the fabrication of solvent-resistant structures," J. Micromech. Microeng., 14 (1), 153-158 (2004).