

Benchtop Polymer MEMS

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Abstract—Loctite photopatternable adhesives 3108, 3340, and 3525 are introduced for microelectromechanical systems (MEMS) applications. These materials are patterned within minutes by exposure to ultraviolet (UV) light followed by rinsing with a solvent; no further processing is required. Because the uncured fluid is relatively insensitive to room light, this can be done on any lab bench without the requirement for a clean room. The materials can be spin-coated to obtain films, or cast between spacers for layers up to 1 cm thick, and the cured polymers range from elastomeric to rigid. These adhesives are of interest for rapid, inexpensive fabrication of relatively low-resolution features (tens to hundreds of micrometers) by photocuring. They can alternatively be molded, like polydimethylsiloxane (PDMS), to achieve high resolution, as well as irreversibly bonded after an O₂ plasma treatment. In addition, like SU8, they can be used as molds for patterning PDMS. Initial characterization of resolution, swelling, and biocompatibility were performed. One of the polymers, 3340, can be used for packaging bioMEMS-on-complementary-metal-oxide-semiconductor (CMOS) chips, exploiting its biocompatibility and its photopatternability at thicknesses of 1500 μm to cover the bond wires while exposing the chip surface. As further demonstrations of the versatility of these materials, multilevel, interconnected channel structures were fabricated with a gelatin sacrificial layer, and magnetic films were prepared, since the polymers remain patternable even with additives. [1535]

Index Terms—Polymer, photopatternable, negative resist, elastomer, packaging, rapid prototyping.

I. INTRODUCTION

MICROELECTROMECHANICAL systems (MEMS) began as silicon-based devices, but have since been transformed through the development of new fabrication processes based on polymers, including micromolding [1], microcontact printing [2], microfluidic tectonics [3], and decal transfer [4], to name a few. BioMEMS in particular has benefited from the innovative use of polymers [4]–[7], especially photocurable polymers [8]–[11]. These developments have been largely motivated by microfluidics [12], a field driven by the requirement for low-cost disposable devices with relatively

large areas but nanometer to micrometer size features in one or two dimensions. When such devices are fabricated using classical MEMS techniques, however, the required clean-room facilities and semiconductor equipment raise the cost of manufacture. There is thus a continued interest in alternative, low-cost microfabrication methods. For example, macroscale processes like hot embossing can be applied to polymer substrates such as compact disks for forming microfluidic channels [13]. Ink-jet printing has also been introduced to form microstructures [14], [15], which has proven popular in fields such as chemical sensing. It has even been discovered that laser-printed toner can serve as an etch mask [16]. Cost and simplicity are also important for developing lab-on-a-chip systems for medicine, yet there has been little effort in developing polymers other than polydimethylsiloxane (PDMS) [17]. Yet another motivation for such new microfabrication techniques is the possibility of using them to realize large-area, nonplanar MEMS [4], which would be prohibitively expensive with conventional microfabrication techniques, as well as the possibility of ultrarapid prototyping [18]. Finally, low-cost demonstrations of basic microfabrication concepts would benefit MEMS education if they could be performed outside a clean room.

In this paper, we introduce a further development of low-cost polymer MEMS based on photopatternable adhesives that are commercially available as Loctite 3108, 3340, 3525, and related products. These materials are most appropriate for relatively large features, but with some of the techniques presented here, small features can also be realized. As we show, these materials have attractive characteristics for MEMS that are not currently available. For example, 3108 combines the photopatternability of SU8 with the mechanical flexibility of PDMS.

Loctite offers a line of photopatternable adhesives with a range of material properties; the mechanical properties, for instance, range from flexible to rigid. This paper focuses on three of these, chosen to represent the different types of polymers and to span the range of mechanical stiffness: loctite 3108, an acrylated polyurethane adhesive, 3340, an epoxy, and 3525, a modified acrylic. Loctite 3108 is an elastomer, 3525 is stiff but flexible, and 3340 is rigid; their Young's moduli are 19, 175, and 2.5 GPa, respectively [19]–[21]. Selecting three polymers with a range of stiffness allows the investigation of various applications and processing techniques. For example, the low modulus of 3108 is conducive to molding and to fabricating flexible or free-standing structures because the cured films can be easily peeled from the template and formed into different shapes. Loctite 3340, on the other hand, makes a good packaging material.

The uncured polymers are packaged ready for use and are viscous fluids with a consistency similar to honey (approximately 5×10^3 cP for 3108 and 3340 and 15×10^3 cP for 3525

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This paper has supplementary PDF material available at <http://ieeexplore.ieee.org>, provided by the authors. This material is 2 MB in file size. The Supplementary Materials contain additional characterization information on the polymers, including additional SEM images and swelling data as well as descriptions of their use for packaging and as molds for electroplating metal. In addition, the Supplementary Materials include an illustrated guide for using the materials, showing step by step their use as masks for wet etching, the fabrication of multi-level channels, the fabrication of magnetic films, and the fabrication of films with varying stiffness and varying sidewall properties.

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TABLE I
THICKNESS AND ROUGHNESS OF SPIN-COATED FILMS

Polymer	Avg. thickness (μm) \pm standard deviation (μm) ¹		Roughness ³ (μm)		
	1000 rpm	3000 rpm	1000 rpm	3000 rpm	5000 rpm
3108	145 \pm 14	42 \pm 7.4 ²	20	dewets	dewets
3340	151 \pm 10	41 \pm 3.7	0.06	0.09	dewets
3525	174 \pm 13	62 \pm 5.4	0.40	0.40	0.40

¹ Based on at least 4 samples, with 5 measurements taken on each sample at different locations.

² On the polymer-coated areas.

³ Average of peak-to-peak maximum of 5 scans each 1 mm long.

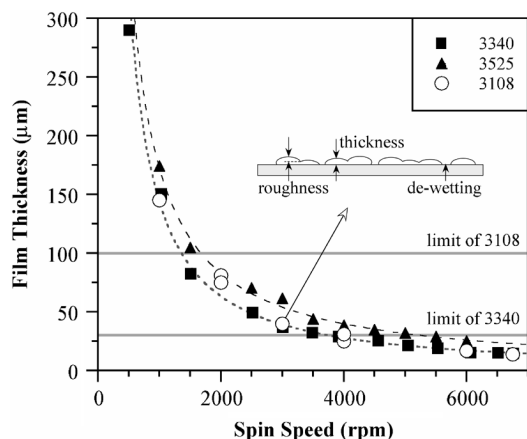


Fig. 1. Average thickness of cured films as a function of spin speed. The speed was ramped up at 1000 rpm/s, and the total spin time was 30 s. The dashed lines are guides for the eye for 3340 and 3525. The error bars are equal to or smaller than the size of the symbols, and thus cannot be seen in the figure; they are quantified in Table I. Due to roughness and dewetting, 3108 and 3340 should not be spin-coated below the indicated thicknesses. The schematic illustrates the dewetting of 3108.

[19]–[21]). They present no known health hazards [19]–[21] and can thus be used with minimum protection.

In Section II, the procedures for using these polymers are detailed. Section III discusses achievable linewidths, swelling behavior, and biocompatibility. Section IV illustrates techniques that demonstrate the versatility of the approach.

II. METHODS

A. Formation of a Patterned Film of Defined Thickness

All patterning steps were carried out under normal lighting conditions, which included natural light entering through two large windows and fluorescent ceiling lights. The polymers were applied onto the substrates directly from 25-ml opaque, syringe-like dispensers. To obtain defined thicknesses, the polymers were either spin-coated or pressed between two flat surfaces separated by spacers (spacers were also used to obtain films of graded thickness).

Fig. 1 shows the thickness of cured films on glass substrates as a function of spin speed, and Table I provides standard deviations and roughness for selected spin speeds. Thicknesses were determined with a profilometer (Dektak 3ST) or, for films thicker than 130 μm , digital calipers. Loctite 3340 and 3108 formed films of approximately equal thickness at the same

spin speeds, but 3525 formed somewhat thicker films due to its higher viscosity.

Spin-coated Loctite 3525 films were smooth and uniform down to a thickness of 20 μm , which was limited by the maximum spinner speed. The other two polymers, 3108 and 3340, dewetted on glass and silicon at higher spin speeds (Table I). Neither hexamethyldisilazane (HMDS) treatment of the substrate nor dilution of the precursor in a solvent remedied this problem. These thin spun layers were also rough due to the variable consistency of the precursor fluid at that scale. As a result, the lower limit of thickness for satisfactory spun films of 3108 and 3340 is approximately 100 and 30 μm , respectively. Films of 3108 and 3340 thinner than 100 μm , characterized later in this paper, were fabricated using spacers instead of spin coating.

Patterning was performed using both transparency masks (3000 dpi, from a commercial print shop) and higher resolution chromium-on-glass masks [U.S. Air Force (USAF) 1951 test masks, Edmund Optics Inc., Blackwood, NJ]. The polymers were cured 2 cm away from the bulb of a hand-held UV lamp (Spectroline, EN-180, center wavelength 365 nm), which delivered a power flux of 5 mW/cm². (Exposing the polymers to direct sunlight through a window for an hour also cured them.) The required exposure times for all three polymers are given in Table II for several film thicknesses. The reported curing times are those that yielded the best feature resolution (Section III-A). The curing time-dependence on film thickness for 3108 was consistent with Loctite's published information for a fusion *D* light bulb [19]. Therefore, for thicknesses not included in Table II, this curve can be consulted. The relationship between exposure time and thickness is approximately linear up to 1 mm; above that it is exponential. (No fusion *D* bulb results are given for the other two polymers, and only times for thicker films are reported). Since exposure times depend on the power spectrum, they need to be calibrated for the particular type of lamp one is using. The exposure times for 3340 were significantly longer than for the other two. The numbers in Table II were from one batch of this polymer, but it must be noted that the times vary by up to 20% among different batches of 3340, which are produced on demand at the time of purchase; 3108 and 3525 did not have such variations and were consistent from batch to batch. The user should, therefore, calibrate exposure times for each batch of 3340 before use.

Since these materials are adhesives, direct contact with the polymer results in bonding the mask to the substrate. To prevent

TABLE II
EXPOSURE TIMES OPTIMIZED FOR LINE RESOLUTION

Polymer	Thickness	Exposure ¹
3108	50 μm	23 seconds
	100 μm	24 seconds
	1 mm	33 seconds
3340	50 μm	4.00 minutes
	100 μm	5.75 minutes
	200 μm	6.50 minutes
3525	20 μm	20 seconds
	100 μm	46 seconds

¹ Exposures for 3108 and 3525 were tested in 1 second increments. Because of the longer exposure times required, 3340 was tested in 15 second increments. Measurements are based on at least five samples, and curing times were repeatable within 2 seconds for 3108 and 3525, and within 15 seconds for each batch of 3340. However, batch-to-batch variations in exposure time for 3340 were up to 20%.

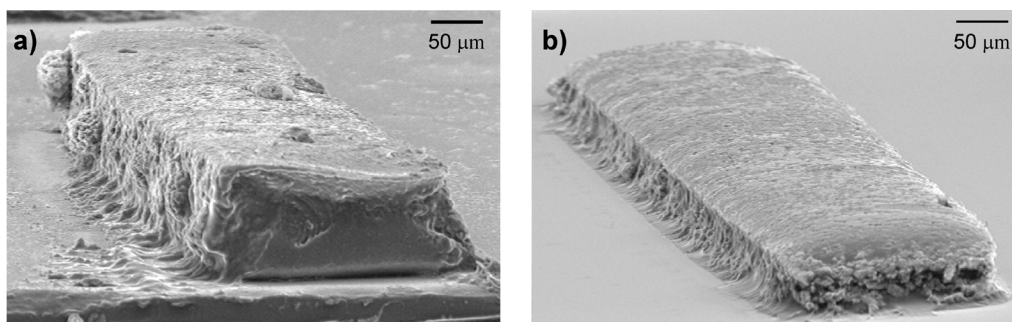


Fig. 2. SEM photographs of 3108 features obtained by contact-exposure through a mask coated with Sylgard 184 elastomer base followed by developing. (a) Features were developed with acetone, resulting in redeposition of residue. (b) Features were developed with ethyl acetate and water, and then sonicated in ethyl acetate for one minute, leaving cleaner surfaces. [This pattern was fabricated to be half as thick as the one in (a)].

this, one can use proximity lithography in an aligner. However, outside a clean room contact lithography is more practical, and to do this one can take advantage of the fact that the cured adhesives do not stick to all materials. PDMS (Sylgard 184, Dow Corning Company, Midland, MI), for example, acts as a non-stick surface treatment. Wiping Sylgard 184 elastomer base (no curing agent added), onto a surface, or curing a thin layer of PDMS on a substrate, renders it nonadhesive. A mask treated with PDMS can thereby be brought into direct contact with the Loctite polymers during exposure and subsequently removed. Sylgard 184 elastomer base must be reapplied after each use, whereas a thin layer of cured PDMS will withstand 11 exposures (standard deviation of 2.3, based on five samples) before the coating delaminates and needs to be replaced. Other examples of materials that do not adhere to Loctite include SealView [a transparent poly(olefin) wrap, Norton Performance Plastics Corp., Wayne, NJ] and Teflon. Polymer films that have been cured on nonadhesive substrates can be peeled off to form free-standing patterned films, as will be discussed in Section II-C.

Many photocurable polymers must be protected from oxygen during exposure for the crosslinking reactions to occur. Crosslinking of the three Loctites was tested in an air atmosphere to determine if protection was required for these materials. Loctite 3340 could be completely cured while uncovered in air. When the surfaces of 3108 or 3525 were exposed to air during crosslinking, on the other hand, the bulk of the polymer cured, but a layer of uncrosslinked material (approximately 30 and 5 μm thick, respectively) was left on the surface. This layer remained curable if the film was initially exposed for less than a minute, however, and could subsequently be cured

in the absence of oxygen [this is exploited in the fabrication of microchannels, (Section IV-B)]. Loctite 3108 and 3525 were completely cured when exposed under a blanket of nitrogen or when covered by an oxygen-impermeable material such as glass or Mylar.

Uncrosslinked polymer remains liquid and can be rinsed away with a stream of an appropriate solvent, thereby developing the pattern. The best results were obtained by starting with a stream of poor¹ solvent, such as water, to remove uncured polymer by mechanical action, followed by rinsing with a good solvent, such as ethyl acetate or acetone. Overexposure to a good solvent (e.g., approximately 10 min) led to swelling and delamination, as well as to adhesive residue redepositing on unexposed areas [Fig. 2(a)]. Redeposited residue can be removed by following a rinse in a good solvent by another rinse in a poor solvent or by sonication for one minute in a good solvent [Fig. 2(b)].

The surface morphology and sidewall profiles were highly dependent on exposure time, rinsing solvent, and postprocessing [such as sonication or descum by reactive ion etching (RIE)]. For example, 3340 films rinsed with ethyl acetate had smooth sidewalls that were slightly sloped in the direction of adjacent features. After RIE etching with O_2 plasma for 10 min, the sidewalls became nearly vertical [Fig. 5(b)]. Good line resolution in 3108 was achieved using acetone, but uncured residue redeposited on the features [Fig. 2(a)], while with ethyl acetate the

¹In this paper the terms “good” and “poor” are used in an informal way to express the degree of solubility relative to the other solvents used in this paper, in contrast to the formal thermodynamic definition of good ($\chi < 0.5$) and bad ($\chi > 0.5$) solvents for a solvent-polymer system.

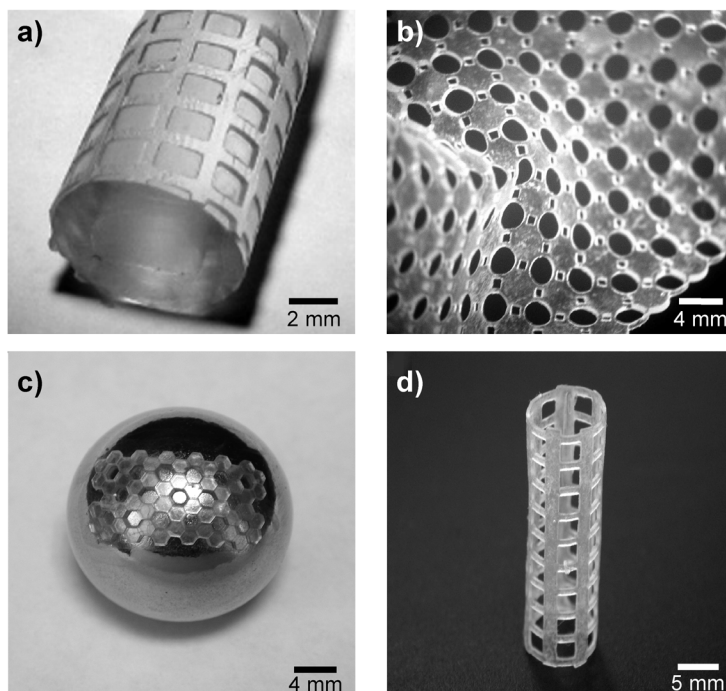


Fig. 3. Patterned Loctite 3108 elastomer films. (a) Grid patterned on 8 mm diameter plastic tubing; each square is 1 mm \times 1.5 mm. (b) Flexible, 550- μ m thick free-standing film patterned on a nonstick surface and later removed, as inspired by [1]. (c) Free-standing patterned film subsequently adhered onto a metal sphere. (d) Free-standing version of (a).

sidewalls were clean at the expense of residue between features. In order to optimize morphology and resolution simultaneously, developing in ethyl acetate was followed by one minute of sonication in ethyl acetate [Figs. 2(b) and 5(b)–(d)]. However, sonication decreased the width of the features. Therefore, additional research is needed to optimize the procedures for each of the Loctite polymers, depending on the desired outcome for a particular application.

B. Photopatterning on Nonplanar Surfaces

Because of their high viscosity, it is possible to photopattern the polymers on curved surfaces. Fig. 3(a) shows 3108 patterned on the outside of a piece of plastic tubing 8 mm in diameter. This was done by wrapping a plastic mask around a 6-mm diameter cylinder and warming it with a heat gun (Wagner HT1000) to deform it into a cylindrical shape. The mask was then coated with Sylgard 184 elastomer base and wrapped around the tubing, onto which 3108 had been applied. The polymer was exposed by rotating the tube relative to the lamp during UV exposure (45° increments, 20 s each). (Films of 3108 were patterned this way also on wooden pencils.)

C. Photopatterning Free-Standing Films

Loctite 3108 can be patterned and released to form flexible free-standing films [Fig. 3(b)] similar to the structures presented by Whitesides *et al.* [1]. Release from the substrate was achieved after exposure and development either by swelling in a good solvent or by curing the polymer on a nonstick surface.

Free-standing structures could subsequently be applied and adhered to other surfaces, flat or contoured, by applying a thin layer of uncured polymer between the new substrate and the freestanding structure and then curing a second time. Fig. 3(c)

shows a 3108 structure adhered to the surface of a metal sphere. Oxygen plasma treatment can also be used to effect adhesion (see Section II-D). These films can be stacked together and laminated, as demonstrated previously using micromolding in capillaries (MIMIC) [1], but without the need for molds in their formation.

To produce the cylindrical structure in Fig. 3(d), a narrow, inflated balloon was coated with Sylgard 184 elastomer base, covered with 3108, and surrounded by a cylindrically shaped transparency mask. After exposure and rinsing with acetone, the balloon was deflated to release the structure.

D. Micromolding and Bonding

PDMS is patterned by micromolding on a template [1], where the template is typically an etched Si surface or a Si substrate with SU8 features. The molding step can be followed by an oxygen plasma treatment of the PDMS and a Si, SiO₂, quartz, or glass substrate to achieve permanent bonding [22]. The same techniques can be applied to 3108 because it is elastomeric and can easily be peeled from the molding template, but fabrication is faster than with PDMS because of the photocuring.

Molds were fabricated on silicon wafers with SU8-10 (MicroChem Corp., Newton, MA) exposed using the negative USAF 1951 test mask. The SU8 molds were 28 μ m thick, measured by surface profilometry. The mold surface was covered with a thin layer of teflon-containing tri-flow superior lubricant (Sherwin Williams Consumer Group, Cleveland, OH) to serve as a mold release; the excess was removed with a jet of nitrogen gas. Loctite 3108 was applied to the surface, covered with a transparency sheet, and exposed using the UV lamp. To aid subsequent release, the substrate was placed in a sonication

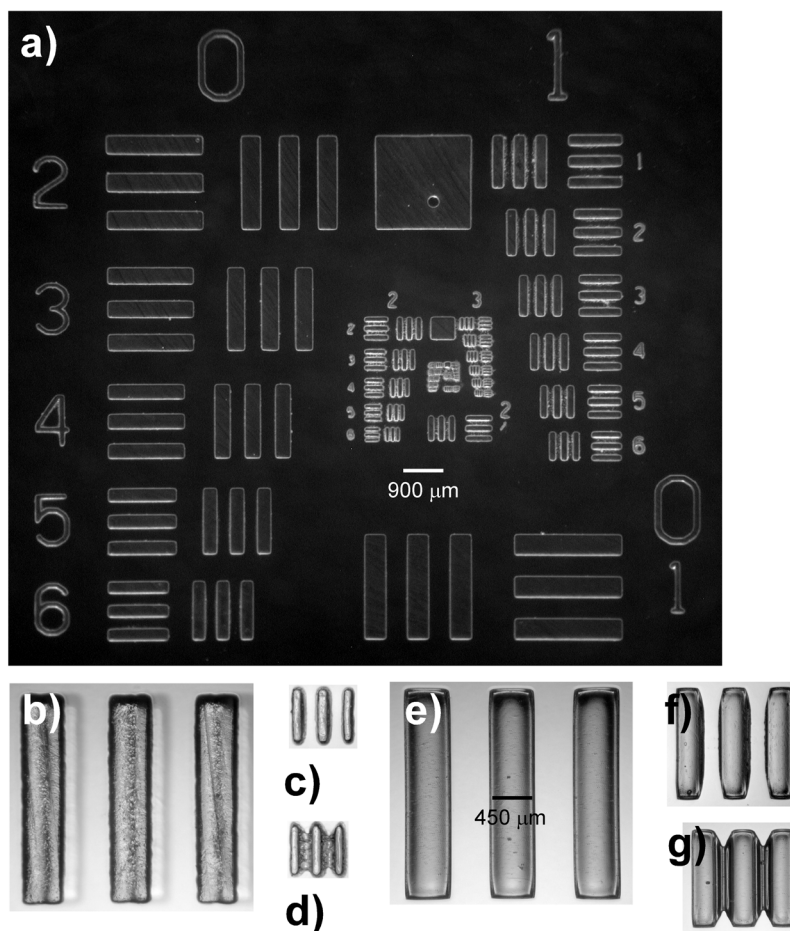


Fig. 4. (a) Overview of a Loctite 3108 film $110\ \mu\text{m}$ thick, patterned using the negative USAF 1951 test mask and developed in ethyl acetate. (b)–(d) Representative features of a 3108 film $50\ \mu\text{m}$ thick, developed and sonicated in ethyl acetate, for the purpose of illustrating resolution definition. (b) Bars located to the right of the “2” at the upper left of (a). (c) The smallest resolved features ($125\text{-}\mu\text{m}$ width and spacing). (d) First unresolved features ($110\ \mu\text{m}$). (e)–(g) Representative 3525 features in a film $80\ \mu\text{m}$ thick [(e) is in the same location as (b)]. (f) Smallest resolved features ($250\ \mu\text{m}$). (g) First unresolved features ($220\ \mu\text{m}$).

bath of methanol for 3 min; the film was then removed by peeling from one corner. (This step failed if the mold release compound was not properly applied.) Wiping a small amount of Sylgard 184 elastomer base on the mold, in a region away from the features of interest, created a tab that aided in removal of the cured polymer. Even the smallest features in the mold ($2\ \mu\text{m}$) were transferred into the 3108 films. Micromolding resulted in higher resolution features than photopatterning [Section III-A and Fig. 5(a)] and decreased surface roughness ($1.6\ \mu\text{m}$ compared with $200+\ \mu\text{m}$). The surfaces of the SU8 molds had a peak-to-peak surface roughness of only $80\ \text{nm}$; the greater roughness of the 3108 may have been due to poor release. Additional research is needed to develop a more satisfactory mold-release coating. One possibility is fluorocarbon coatings applied by an inductively coupled plasma (ICP); these have been used as mold-release layers for PDMS [23].

Following the same procedures as used for PDMS, 3108 can be treated with oxygen plasma to irreversibly bond it to a substrate, as well as to itself. This was accomplished by treating the 3108 and a glass substrate, or another 3108 layer, in RIE [120-mtorr pressure, 40-W radio frequency (RF) power, 100-sccm O_2 flow rate, 80 s] and immediately bringing the surfaces into contact. The bonded pair was then placed on a hotplate at 110°C for 10 min. Attempting to remove the film resulted in cohesive me-

chanical failure but not debonding (the tensile strength at break of 3108 is $7.8\ \text{MPa}$ [19]–[21]). This material could, therefore, in principle be used for decal transfer microlithography [4].

Furthermore, PDMS can be patterned by molding using any of the three Loctites as a master, in place of SU8 or Si. No mold release is needed because these materials do not adhere. This is a significant capability because PDMS is frequently used for bioMEMS, but mold fabrication has proven to be the rate-limiting step in the fabrication of devices by soft lithography [18]. Use of photopatternable Loctite for the production of masters would transfer the entire soft lithography fabrication process into ordinary laboratories, which would be particularly welcome to researchers in life sciences, and it would reduce the time required for prototyping.

III. CHARACTERIZATION

A. Feature Resolution

Because 3108 and 3340 formed poor-quality spun films, their resolution was examined in samples $100\text{--}200\ \mu\text{m}$ thick, fabricated using spacers. The resolution of 3525 was examined in thinner, spin-coated samples approximately $20\ \mu\text{m}$ thick. Minimum achievable line-widths were determined using the negative USAF 1951 test mask pretreated with a layer of Sylgard 184

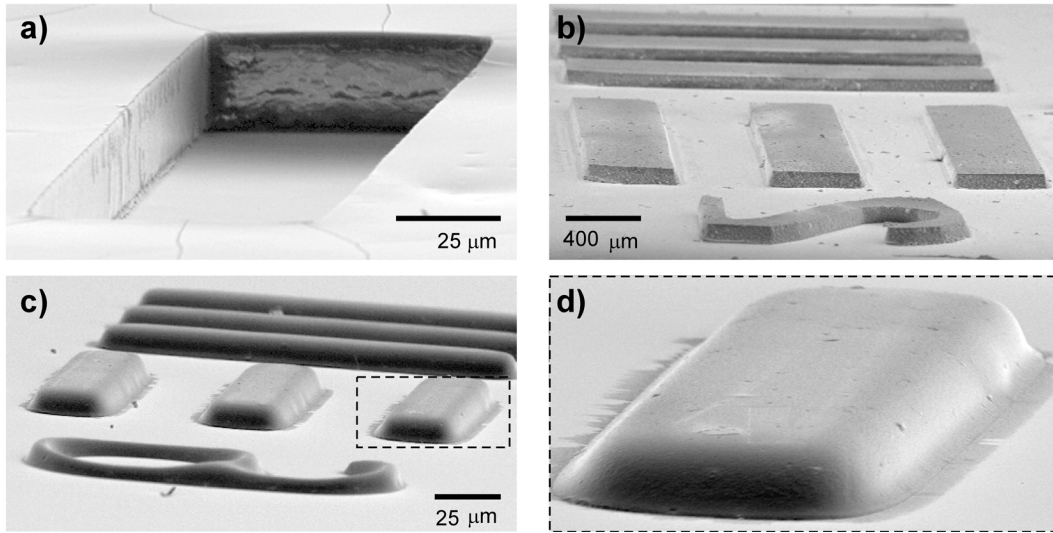


Fig. 5. SEM photographs of (a) a 3108 pit 28 μm deep, fabricated using an SU8 mold, (b) photopatterned 3340 features 80 μm thick after a 10-min RIE descum, (c) photopatterned 3525 features 18 μm thick, and (d) a close-up of a feature in (c) as indicated by the dashed rectangle. Magnifications differ in these images.

TABLE III
RESOLUTION BEFORE AND AFTER A 10-MIN OXYGEN PLASMA RIE DESCUM. THE POLYMERS WERE DEVELOPED IN ALTERNATING SOLVENT STREAMS OF ETHYL ACETATE AND ISOPROPNOL

Polymer	Thickness	Resolution After Developing	Resolution After Descum
3108	100 μm	200 μm	140 μm
	200 μm	350 μm	250 μm
3340	100 μm	100 μm	80 μm
	200 μm	250 μm	220 μm
3525	20 μm	30 μm	---
	80 μm	250 μm	---

elastomer base. A UV light-absorbing material, such as plexiglas or kapton, was placed under the glass substrate to improve pattern definition by preventing light reflections. A representative result for 3108 is shown in Fig. 4(a).

The definition of minimum achievable line-width used in this paper was the smallest feature set without residue connecting the features after developing [Fig. 4(c) and (f) versus Fig. 4(d) and (g)]. This is considerably more stringent than the smallest features that can be patterned, since pattern fidelity was better at the top of the film than at the base. Although not directly shown in this paper, Loctite polymers should thus allow production of significantly finer ink patterns in applications such as microcontact printing than the resolutions reported here (Table III).

The resolution of the polymer films depended on their thickness (Table III). The 30- μm resolution for thin 3525 films is comparable to that previously reported for liquid phase polymerization [18]. Using a mask aligner (Karl Suss MJ-B3) instead of the UV lamp did not significantly improve the line resolution. It should also be noted that we did not systematically attempt to optimize resolution, and some improvement upon further research is to be expected.

Line resolution was examined for 3108 and 3340 films before and after an RIE descum in an oxygen plasma (120 mtorr, 150 W, 10 sccm of O_2 , 10 min); results are shown in Table III.

The line resolution increased by as much as 30% after the descum step, which etched through the thin layer of polymer that intruded into the areas between features. RIE descum was not performed on 3525 films; however, results with 3340 suggest that RIE descum of 3525 would yield more vertical sidewalls and improved line resolution.

One of the limits on resolution is that 3108, 3340, and 3525 have low contrast in comparison with polymers such as SU8. Gray-scale lithography, which is employed with high-contrast polymers, was not successful with these Loctites, and exposure of a thick layer of uncured polymer for different times through the back of a transparent substrate did not yield cured features with different heights. Rather, exposure times that were shorter than those given in Table II simply led to poorer resolution or partially cured films that rinsed away during development.

B. Swelling

Swelling by solvents impacts the possible applications of a polymer. Understanding swelling is important because solvents are used to develop the pattern and to remove cured films from surfaces, and also as thinners and cosolvents for incorporating soluble additives. Following prior work on PDMS [24], [25], we measured the swelling of the three polymers. A strip of each polymer approximately 7.5 mm \times 50 mm \times 1 mm (400 mg) was immersed in 14.5 ml of solvent. After 11 and 30 days, the

TABLE IV
WEIGHT INCREASE AFTER IMMERSION IN VARIOUS SOLVENTS

Solvent	Solvent Hildebrand Parameter [24,26] (MPa ^{0.5})	Weight Change (%)					
		3108		3525		3340	
		11 days	30 days	11 days	30 days	11 days	30 days
Acetone	18.6	101	101	79	80	37*	40*
Chloroform	18.8	456	460	315	311	broke apart	
Diethyl ether	29.6	24	25	34	34	5	8
Ethanol	18	69	68	80	81	27	34
Ethyl acetate	18.2	124	122	82	84	24	25
Heptane	15.1	4	4	5	6	1	4
Hexane	15.1	4	3	4	6	3	1
Isopropanol	26	48	48	66	64	14	16
Methanol	23.5	54	55	57	56	37*	33*
Methylene chloride	20.3	361	429	214	219	broke apart	
Toluene	18.2	125	123	73	74	18*	20*
Water	47.9	10	14	6	7	11	11
Xylene	20.2	85	86	64	67	7	6

* Cracks formed in the samples.

TABLE V
WEIGHT INCREASE AFTER IMMERSION IN VARIOUS AQUEOUS SOLUTIONS

Aqueous Solution	Weight Change (%)					
	3108		3525		3340	
	11 days	30 days	11 days	30 days	11 days	30 days
CD30 ¹ developer	18	40	34	33	12	14
Au electroplating solution (Oromerse SO Part B ³)	8	11	17	21	9	10
Ni electroplating solution ^{3,4}	10	9	8	10	10	11
Au etchant (type TFA ²)	71	78	35	43	14	19
Ni etchant (type TFG ^{2,4})	chemical reactions		20	21	20	21
Cr etchant (type TFD ²)			13	11	11	16
Al etch (type A ^{2,5})			chemical reactions		chemical reactions	
sulfuric acid (H ₂ SO ₄), 95.8% ⁶	chemical reactions		chemical reactions		chemical reactions	
nitric acid (HNO ₃), 69-71% ⁷			chemical reactions		chemical reactions	
phosphoric acid, (H ₃ PO ₄), 85% ⁷			chemical reactions		chemical reactions	

Sources: ¹ Shipley Co., ² Transene Co., ³ Technic Inc.

⁴ Contains small quantities of sulfuric and/or nitric acid.

⁵ The main ingredient is phosphoric acid, with small quantities of nitric acid and acetic acid.

⁶ Caused foaming and change in solution color. ⁷ Turned the polymer film brown.

samples were lightly blotted with absorbent paper and weighed (Explorer, Ohaus Corp., Pine Brook, NJ) within 5 s of being removed from the solvent.² The results are summarized in Tables IV and V.

The solubility, or Hildebrand, parameter provides a method of predicting swelling in solvents [26]. It is generally assumed that the swelling of crosslinked polymers is greatest when the solvent and polymer solubility parameters are equal. The solvents that were used to develop the polymers after photocuring (see Section II-A) have Hildebrand parameter values δ of 18–19 MPa^{0.5}. Moreover, the solvents within this range of δ swelled the cured polymers the most, according to Table IV. (Plots are included in the supplementary materials, section 1.4. See the first footnote, after author affiliations, for the URL.) For comparison, PDMS swells the most in solvents with δ from 15 to 18 MPa^{0.5} [24]. Because the Hildebrand parameters favored by the Loctites and PDMS are different, one polymer could re-

²The same-sample weight reproducibility was 2%. To examine the repeatability of the procedure, five different samples of 3108 were tested in acetone; these measurements agreed to within 3%.

place the other in applications for which swelling is undesirable. For example, hexane ($\delta \sim 15$ MPa^{0.5}) swells PDMS 30% [24], whereas it swells 3108, 3525, and 3340 under 7%. Water is the most important solvent for microfluidics and bioMEMS applications. Water swells 3108 by 14%, 3340 by 11%, and 3525 by 7%. PDMS, on the other hand, swells less than 1% in water [24]; this value was verified using the aforementioned method.

Swelling of the polymers was also examined in various aqueous etchants to ascertain whether they could be used as a negative photoresist for wet etching (Table V). The results show that it withstood numerous etchants, but reacted with some acids.

Swelling in Ni electroplating solution, also of importance for MEMS, was tested as well and found to be relatively low at approximately 10% for all the polymers. Loctite 3108 can be used as a mold for Ni electroplating (see the supplementary materials, section 1.4, for a figure showing features 350 μ m tall. Go to the first footnote, after author affiliations, for the URL). To remove the polymer, an oxygen plasma etch should be used for thick electroplated films. (Since the walls are not perfectly ver-

tical but sloped somewhat, removing the 3108 by swelling can lift off the metal features.)

C. Biocompatibility

When fabricating devices for microfluidics or bioMEMS applications, the biocompatibility of materials must be considered. In our own research [27], [28], we make use of bovine aortic smooth muscle cells (BAOSMC) [27], so we tested the ability of these cells to be cultured on the adhesives. Square vials of the polymers were fabricated onto cover slides. Five such slides of each polymer were attached to the bottoms of five wells of a 48-well culture flask using an adhesive known to be biocompatible (Novagard RTV-200–257). The slides were rinsed under a sterile flow hood with ethanol, sterile water, and warm growth medium. A suspension of BAOSMC cells that had been cultured in a T-25 flask was placed into each well; the quantity of cells corresponded to 50% confluence on the surface of the slides. The 48-well culture flask was then placed into an incubator at 37 °C and 5% CO₂. Cell coverage was measured every two days for two weeks. For 3108 and 3340 samples, the growth curve showed the expected logarithmic relation with time, and the population doubled in two weeks, matching the growth pattern of BAOSMC cells in culture flasks. In the wells with 3525, cells were slower to grow on the polymer than on the surrounding glass substrate, suggesting that the material is not preferred by the cells, though not necessarily nonbiocompatible.

IV. EXAMPLE APPLICATIONS

To illustrate the versatility of the Loctite adhesives, we discuss a few examples of their use. The application for which we are now using one of these polymers is packaging lab-on-a-chip devices comprising MEMS structures and actuators built over CMOS sensors [29], [30]. Other examples are given that, while not original, were made within minutes on a lab bench using inexpensive materials and equipment. The purpose of these demonstrations was not to characterize the resulting devices, but to suggest possibilities for future exploration.

A. Packaging BioMEMS/CMOS Chips

Packaging can be a formidable challenge in the realization of lab-on-a-chip applications. Fluids and cells may require contact with sensors and actuators on the surface of an electrically active chip, but the bond wires must be kept dry. This is particularly difficult if the transducers are located close to the bond pads. Some researchers have glued a precision-machined well to the chip [31]–[33], which requires the chip to have a wide nonactive perimeter [34].

We have found that Loctite 3340 works well for packaging bioMEMS/CMOS chips. In contrast, Loctite 3108 permits water and small molecules access to the bond wires, resulting in electrochemistry, as shown in the supplementary materials, section 1.1 (see the first footnote, after author affiliations, for the URL). Packaging was not attempted with 3525 due to a lack of clear biocompatibility. Although 3340 and 3108 swell approximately the same amount after a month in water, 3340 swells more slowly (see the supplementary materials, section 1.4. Go to the first footnote, after author affiliations, for the URL). Loctite 3340 protects the chip for approximately a week in cell culture conditions.

The 3340 has significant advantages over SU8, benzocyclobutene (BCB), PDMS, and other polymers for this application. Unlike millimeter-thick SU8, this adhesive requires no mold and undergoes no appreciable shrinkage upon curing [35]. Packaging with 3340 is performed at room temperature, which was important to us because temperatures above ~120 °C damage the electroactive polymers used in our actuators. In addition, it takes only a few minutes. Films of 700- μm thickness can be readily patterned (thick enough to cover the bond wires) with tolerances of 25 μm [Fig. 6(a) and (c)]. Since the polymer is biocompatible and can be cured in air, it is an ideal candidate for this application.

The procedure for packaging $1.5 \times 1.5 \text{ mm}^2$ or $3 \times 3 \text{ mm}^2$ chips wire-bonded to a DIP40 is as follows. The DIP40 cavity is filled with 3340. A $5 \times 5 \text{ mm}^2$ mask, consisting of a black square (either $1.2 \times 1.2 \text{ mm}^2$ or $2.5 \times 2.5 \text{ mm}^2$, the size of the area to be left open), is placed on the polymer directly over the chip and inside the DIP40 cavity, with one corner folded 90° out-of-plane. The mask can be manipulated by the folded corner under a microscope for alignment. After exposure (6 min, optimized to reduce residue) and development (alternating streams of ethyl acetate and water), which does not damage the MEMS structures, the wirebonds are completely encapsulated and thus mechanically supported. Prior to cell plating, the chip is left at ambient temperature and pressure for 24 h to ensure that any remaining solvent has evaporated.

Encapsulation can be done using two layers of polymer [Fig. 6(b) and (d)], which has a couple of advantages. For one, the first layer (300 μm above the chip surface) can be patterned with greater accuracy since visualization through the microscope is improved when the mask is closer to the surface. For another, the second layer can have a larger opening than the bottom layer so that it holds more fluid and gives easier access to the chip.

The accuracy achieved with this method allows packaging of the chips with more than 90% of the active chip area exposed to the cell medium and a gap of only 25 μm between the pads and the MEMS features. The main limitation of this technique lies not in the verticality of the polymer walls, but in the alignment of the mask.

Cells have been cultured inside an incubator at 37 °C and 5% CO₂ on chips with packaged CMOS capacitance sensors. Capacitance data were collected for a week without package failure. The capacitance readings were correlated with cell contact with the substrate, spreading and adhesion, and subsequent death [30]. It is important to note that exposure to ethanol (any exposure to liquid and prolonged exposure to concentrated vapor), used for sterilization in cell culture protocols, produced cracks in the 3340 and led to package failure (see the supplementary materials, section 1.1. Go to the first footnote, after the author affiliations, for the URL). The chips were, therefore, sterilized with UV light.

B. Microchannels

In 2000, Beebe *et al.* [3] presented microfluidic tectonics (μFT), a method for fabricating complete microfluidic channel systems based on liquid-phase photopolymerization of hydrogels. *In situ* creation of the channels in a packaged system offered an important advantage over prior microfluidic fabrication methods: the structures and components were fabricated si-

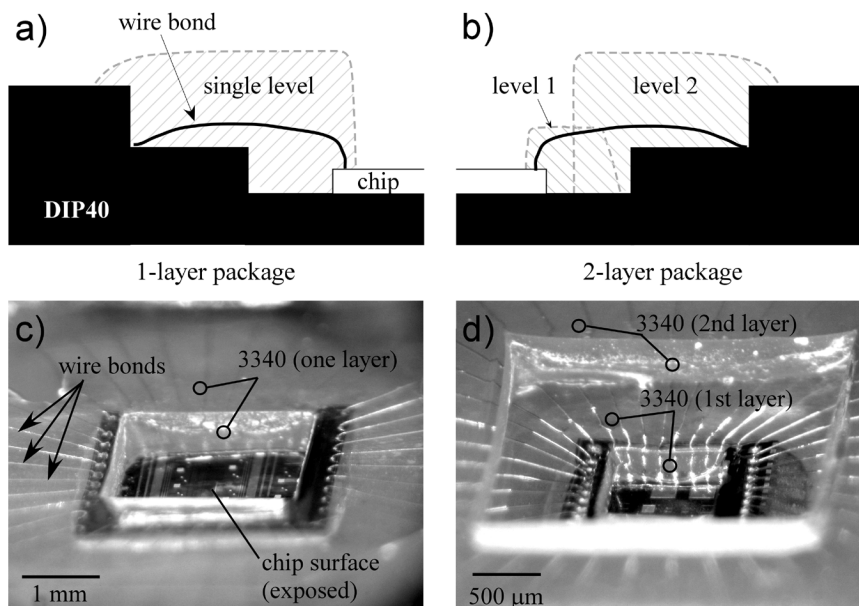


Fig. 6. (a) and (b) Schematic of the cross-section of the Loctite packaging of a chip in a DIP40. One or two layers can be used to cover the wire bonds while keeping the chip surface exposed. (c) A packaged CMOS chip with overlying MEMS structures using a single layer of 3340. This layer is ~ 1.5 mm thick ($700 \mu\text{m}$ above the chip surface). (d) A two-layer packaged chip with a first layer $300 \mu\text{m}$ above the chip surface and a second that goes to $700 \mu\text{m}$.

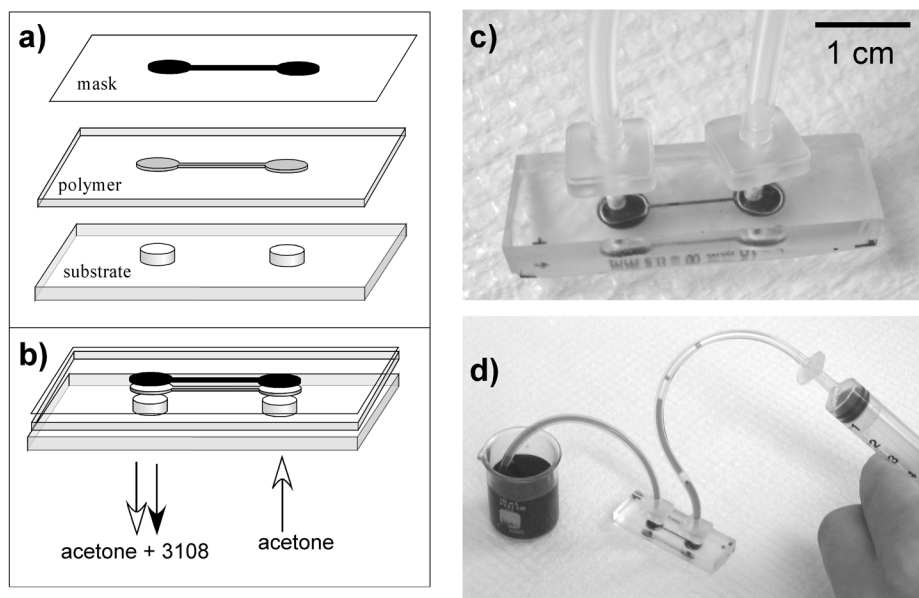


Fig. 7. Microfluidic channels formed in 3108 using μFT [3]. (a) The polymer is placed on a substrate with port holes, covered with a glass slide, topped with a mask, and exposed. (b) The uncrosslinked polymer is flushed out of the channel using acetone. (c) Completed microchannel, face-down, with a mask top cover. (d) Water, dyed for better visualization, drawn through the channel after fabrication.

multaneously without the need for later alignment and bonding. In 2004, Harrison *et al.* [36] published a modification of this method based on thiolene-based optical adhesives. These channels had some advantages, including solvent-resistance and the use of a commercially available product; however, this process required a long curing time.

To fabricate microchannels, we adapted the techniques developed by these authors, as illustrated in Fig. 7. A substrate was prepared by drilling holes into a glass slide (glass drill bit, 1/16 Black & Decker, Towson, MD) or a piece of plexiglas. The substrate was placed over a UV-absorbing material, and

3108 was dispensed onto the surface. A transparency channel mask was placed over the adhesive and aligned by eye to place the reservoirs over the holes. A glass slide was placed over the mask to ensure flatness, and this stack was exposed to UV light. Spacers between the substrate and the mask defined the channel depth. Commercially available nylon fittings (1/16 McMaster Carr 5117k41, Los Angeles, CA) were pressed into the holes in the substrate, and tubes (Tygon super-soft high purity tubing, McMaster Carr 9449k11, ID 1/16) were slipped onto the connectors. A syringe (10 ml, disposable) filled with acetone was plugged into one of the hoses, while the other tube was placed

into a container of acetone. The unexposed polymer was flushed out by drawing acetone from the container through the channel using the syringe to create negative pressure. The result was a sealed channel utilizing the mask as the top cover [see Fig. 7(c)]. The total time to complete such a device was less than 10 min.

If a top cover other than the mask was desired, the mask was placed on top of that cover. For an open channel, the mask was treated with Sylgard 184 elastomer base and removed after exposure.

Certain microchannel features, such as sudden expansions and blind alleys, posed flushing problems. In this case, an open channel was first fabricated to allow complete rinsing. A cap substrate was covered with 3108 and exposed without a mask, leaving an uncured layer $30\ \mu\text{m}$ thick (see Section II-A). This layer was placed, 3108-side down, onto the channel layer and exposed for an additional 30 s, gluing the two slides together.

C. Multilevel Channels Using Gelatin Sacrificial Layers

In order to make interconnected multilevel channels, a compatible sacrificial layer is needed. Gelatin (Gelatine, Knox: Kraft Foods, Northfield, IL) is a good candidate for a low-tech, bench-top approach because it is harmless, easy to prepare, and has a melting temperature near ambient that can be controlled by the amount of water used. Open channels and reservoirs were filled with gelatin dissolved in water. Channels with a top cover were filled by injecting the mixture through the fluid ports. The channels were placed in a refrigerator and, because of the small size of the devices, the gelatin solidified within minutes, coplanar with the first channel height. A second layer of 3108 was patterned over the Loctite/gelatin base, and the sacrificial layer was removed by immersing the assembly in warm water. Multiple layers could be built up using this technique. The components of a two-layer channel fabricated with this method are illustrated in Fig. 8(a), and a fluid-filled two-layer channel is shown in Fig. 8(b). (A series of photographs showing the fabrication and testing of this device is included in the supplementary materials, section 2.4. Go to the first footnote, after the author affiliations, for the URL)

D. Use of Patterned Films as Resist for Etching

The polymers discussed in this paper are sufficiently impermeable to aqueous solutions (Table V) that they could be used as a negative photoresist for wet etching. There is every reason to expect that they would also serve as effective resists for dry etching. After the exposed portions of the underlying material are etched, the polymer is removed by immersion in a good solvent. Sonication accelerates removal. Loctite 3108 is preferred over 3340 and 3525 for this application because of its mechanical flexibility.

Use of 3108 as a wet etch mask is illustrated in Fig. 9 with large features that can be readily seen in the photograph (the technique also works with small features). A Kapton film substrate was coated with a Cr adhesion layer and $1000\ \text{\AA}$ of Au by thermal evaporation. The 3108 was applied onto the Au surface, exposed to UV light for 40 s through a transparency mask treated with Sylgard 184 elastomer base, and developed with acetone. The Au was wet chemically etched (TFA, Transene Company Inc., Danvers, MA), the substrate was immersed in acetone for 10 min, and the 3108 resist layer was peeled off.

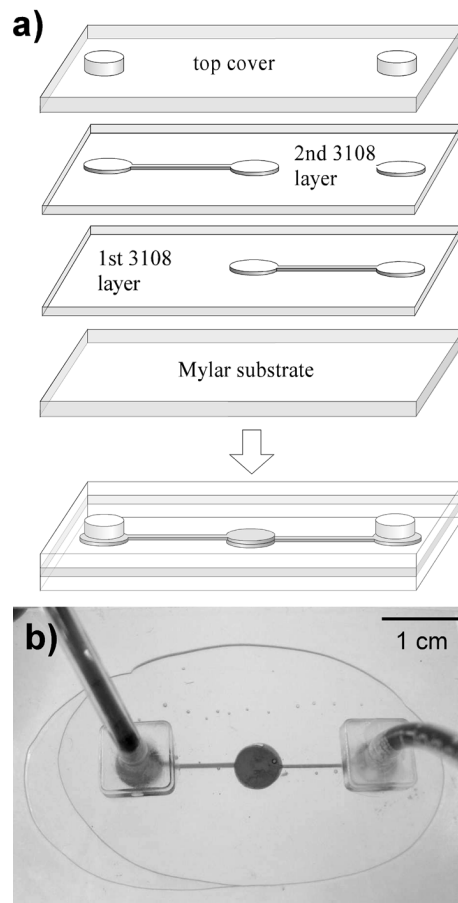


Fig. 8. (a) Schematic illustration of a two-level microchannel formed with a gelatin sacrificial layer. (b) Water, dyed for better visualization, drawn through the channel after fabrication.

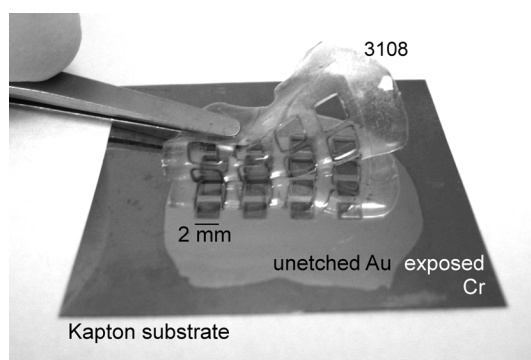


Fig. 9. Use of Loctite 3108 as an etch mask. After patterning the polymer, the uncovered underlying Au was wet etched down to the Cr adhesion layer. The $250\text{-}\mu\text{m}$ -thick polymer mask was then swelled in acetone and peeled off.

The Loctite polymers were particularly useful as masks for the wet etching of glass (performed in a chemical hood) with buffered oxide etchant (J. T. Baker, Philipsburg, NJ: 7 : 1 with surfactant): both 3108 and 3340 allowed etching several times deeper than is usually possible with conventional photoresists. Loctite 3108 withstood 75 min before parts of the mask started to peel off the glass, allowing an etch depth of $40\ \mu\text{m}$. Loctite 3340 began delaminating from the glass after 48 min, resulting in features $27\ \mu\text{m}$ deep.

E. Formation of Magnetic Films

One of the advantages of working with polymeric materials is that they can be readily mixed with other materials, as shown in a number of recent papers. For example, carbon [37] or silver [38] particles mixed into SU8 render it conducting, and this has been used in the formation of piezoresistors for measurement of cantilever deflection [37]. In another example, dye mixed into SU8 has been used for the formation of lasers [39]. The Loctite materials also remain patternable with additives, if they are not too absorbing or scattering, as shown by the formation of elastomeric magnetic composites using 3108 and strontium ferrite.

Magnetic materials are typically deposited by electroplating [40] or sputtering [41]. Composites [42]–[44] formed by mixing magnetic particles into a polymeric matrix have been screen-printed [43], [45], molded [43], squeegee-coated [43], and wet-etched [45]. One of the polymers that has been used as a matrix is polyimide, which can be screen printed to form 250- μm -wide features or wet-etched to form 200- μm features [45]. Since polyimide is cured at 300 °C, it is incompatible with some substrates; epoxy resin (bisphenol-A-based, Shell, Houston, TX) has also been demonstrated for 2-mm features patterned by screen-printing, and it cures at 80 °C for 2 h [42]. PDMS has also been used to form magnetic composites [43], [44], but patterning is limited to molding. It would be more convenient to use a photopatternable polymer as a host matrix. It may also be attractive in some cases, such as for the formation of compliant magnetic gaskets, for the host to be an elastomer such as Loctite 3108.

Strontium ferrite powder (HM410, particle size 1.8–2.3 μm , Hoosier Magnetics, Inc., Ogdensburg, NY) was added to 3108 and mixed with a homogenizer (T18 Ultra Turrax) until the particles were uniformly dispersed (approximately 5 min). The homogenizer gave significantly greater particle uniformity than hand-mixing. Additives to improve particle dispersion such as those suggested in [46] were not used. The mixture was deposited and exposed in the usual ways, although light absorption/scattering by the particles increased the exposure times linearly with the volume percent of particles. Films 150 μm thick required approximate curing times of 4.5 min for 5% V/V strontium ferrite (4.9 g/ml), 9 min for 10%, 18 min for 20%, and 27 min for 30%. Uncured material between features less than 300 μm apart was difficult to remove during development. Increasing volume fractions of strontium ferrite resulted in thicker layers of such residue. Mixtures of 20% and 30% patterned but tended to adhere to the patterning mask, even with a nonadhesive layer of Sylgard 184 elastomer base. The magnetic composites could be etched using an RIE O_2 plasma; however, the etch rate dropped with increasing volume fraction of particles: a 10% mixture had half the etch rate of 3108 alone (0.35 $\mu\text{m}/\text{min}$ at 120 mtorr, 150 W, 10 sccm of O_2 , compared to 0.7 $\mu\text{m}/\text{min}$ for the unloaded polymer).

A magnetically aligned film was formed by photocuring over a magnet. The magnetic moment of samples with 10% strontium ferrite was measured as a function of magnetic field using a superconducting quantum interference device (SQUID) (MPSM-5,

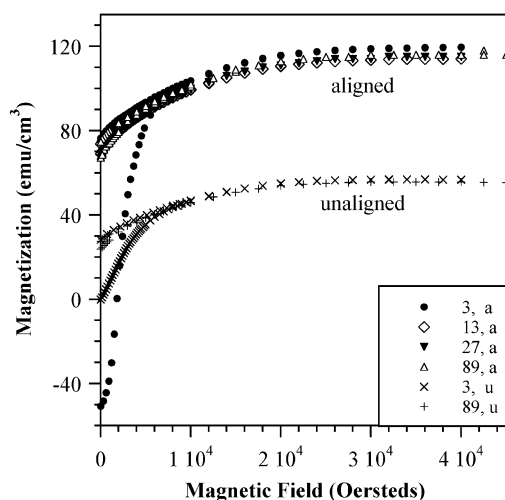


Fig. 10. Magnetic saturation and remanence of 10% strontium ferrite/3108 composites. Magnetic measurements were performed 3, 13, 27, and 89 days after curing for the aligned composite sample (indicated by “a” in the legend), and 3 and 89 d after curing for the unaligned composite sample (indicated by “u”).

Quantum Design, Inc., San Diego, CA). Magnetization was calculated by dividing the magnetic moment by the sample volume (samples were 2.25 mm² in area and 100 μm thick).

Fig. 10 shows curves for aligned and unaligned composite samples. The aligned composite had a remanent magnetization ($-50 \text{ emu}/\text{cm}^3$) at the start of the measurement, thus indicating that either the particles, their magnetic moment, or both, had been aligned by the field during curing, whereas the unaligned composite had no net magnetization. The aligned sample exhibited greater saturation ($117 \text{ emu}/\text{cm}^3$) than the unaligned sample ($56 \text{ emu}/\text{cm}^3$) (saturation obtained using the $1/H^2$ method [47]) and greater remanence after saturation (74 versus $28 \text{ emu}/\text{cm}^3$). The magnetization did not decrease with time for either the aligned or unaligned samples; variations in Fig. 10 were within experimental error. The magnetic properties were thus improved by aligning the magnetic particles in the composite during curing, and the effect was semipermanent.

V. CONCLUSION

We have presented a bench-top microfabrication methodology that utilizes low-cost, commercially available, photocurable adhesives. We have also performed initial characterization and demonstrated their utility as promising new MEMS materials through several examples of their use, so that others who may find these capabilities of interest can further explore their potential applications.

There are several notable contributions that these Loctite materials, and potentially also other photopatternable adhesives (such as the Norland family of UV optical adhesives [48]), can make to MEMS. The wide variety of formulations considerably extends the range of material properties available for structures and devices, as well as the upper limit of film thickness (up to an unprecedented 1 cm). Patterning is fast compared with SU8 since no baking steps are required. Furthermore, patterning

is possible on nonplanar surfaces and on nontraditional materials. All three polymers can be used to fabricate templates for molding mesoscale features in PDMS with simple equipment and fast turn-around time. The fact that the materials are good adhesives for a wide variety of materials can be exploited, for example in the fabrication of microchannels. Yet the adhesivity can also be easily prevented by coating the surface with PDMS or other surface modifications. By patterning the nonstick areas over the substrate, the principle of differential adhesion [49] could be applied in device fabrication.

Two of these polymers have features that are of particular interest. The 3108 shares some of the attractive capabilities of PDMS, including its elastomeric properties, the formation of irreversible bonds to Si and glass by oxygen plasma treatment, and biocompatibility with cells. However, the fact that 3108 is also photopatternable decreases the turn-around time for prototyping compared with PDMS. Likewise, the 3340 shares many of the attractive characteristics of SU8, but even thicker films can be produced, and a wide range of thicknesses (30–1000 μm) is achievable with a single formulation and at substantially lower cost. The 3340 has proven useful for packaging bioMEMS/CMOS chips because it is biocompatible and prevents electrolyte from reaching the bond wires.

The ability to work with all of these materials under room light and at room temperature allows photolithographic techniques to be employed virtually anywhere, and thus to be exploited by a wider community. It also opens the way for educational institutions that do not have microfabrication facilities, such as community colleges and even high schools, to demonstrate MEMS fabrication techniques and devices.

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