A method for reducing pressure induced deformation in silicone microfluidics

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Abstract

Poly(dimethylsiloxane) or PDMS is an excellent material for replica molding, widely used in microfluidics research. Its low elastic modulus, or high deformability, assists its release from challenging molds, such as those with high feature density, high aspect ratios and even negative sidewalls. However, owing to the same properties, PDMS-based microfluidic devices stretch and change shape when fluid is pushed or pulled through them. This paper shows how severe this change can be, and gives a simple method for limiting this change that sacrifices few of the desirable characteristics of PDMS. A thin layer of PDMS between two rigid glass substrates is shown to drastically reduce pressure induced shape changes while preserving deformability during mold separation and gas permeability.

Keywords: PDMS, microfluidics, deformability, fabrication, poly(dimethylsiloxane)
**Introduction**

Poly(dimethylsiloxane) is an elastic polymer well suited for microfluidic and soft lithography research.\(^1,2\) Numerous authors have emphasized its desirable properties; these include optical transparency, chemical inertness, low surface free energy, good adhesion to surfaces, non-toxicity, and gas permeability.\(^3-5\) In particular, its high elasticity and high elongation-at-break assist in mold release.\(^6\) However this same high elasticity leads to features that deform significantly under pressure, altering the performance of devices built to rigorous specifications.

This paper reviews the typical construction technique for PDMS microfluidic devices, highlighting relevant material properties and shows experimental and simulated deformation due to fluid pressure in an array of pillars. Strategies to limit this change, including: UV-cured optical glue and hard PDMS (h-PDMS) are discussed. Finally it describes the fabrication method of a glass-PDMS-glass device that shows greatly reduced deformation under pressure. Glass-PDMS-glass devices have been described before but the mechanical advantages of this construction have not been described.\(^7\)

The most common formulations of PDMS used in microfluidics research labs are Silgard 184 (Dow Corning) and RTV615 (GE Silicones). A notable but less common formulation, RG01 (Gelest Inc.), is claimed to be suitable for mass production. All three are supplied as two component siloxane liquids, to be mixed 10:1 w/w and cured at room temperature or with heat. The prepolymers are mixtures of siloxanes (Si–O–Si–O–...) having combinations of methyl (–CH\(_3\)), vinyl (–CH\(_2\)), and/or hydro (–H) side and terminal groups. The curing agent is a shorter siloxane polymer with an excess of hydro groups and a platinum catalyst. The catalyst crosslinks the siloxane polymers by converting hydro and vinyl groups into methyl groups.

For chip fabrication the two components of PDMS are mixed together then placed in a vacuum chamber for about 20 minutes. The mixture is then poured onto a master mold where it creates a conformal coating. As the diffusivity and permeability of air in PDMS is high \((D_{N2} = 3400 \text{ } \mu\text{m}^2/\text{s})\), comparable to that of air.
in water\textsuperscript{5}) it dissolves any trapped pockets of air and the polymer is able to
completely fill dead-end structures such as deep, narrow holes. After curing, the
daughter mold is peeled off the master. Here its high elongation-at-break (50\%),\textsuperscript{6}
high Poisson ratio (0.5),\textsuperscript{8} and low surface free energy allow challenging chip
features such as pillars to elongate, narrow and release from the master with
relatively little force. After its release the daughter mold can be reversibly or
permanently sealed to a variety of surfaces.

The strength of a reversible bond depends on the cleanliness and history of the
surfaces, but typically such bonds leak at a few psi (a few 10s of kPa). A
permanent bond can be made by treating both surfaces with a low power air or
oxygen plasma. An alternative and more reliable permanent bond is made using a
thin layer of uncured or partially cured PDMS.\textsuperscript{9} If the mold is permanently sealed
the bond will hold positive pressure up to the breaking limit of PDMS, around 700
kPa. However, even at pressures well below the breaking strength, the
deformation of PDMS chip features is significant. The deformation will depend
on the local geometry, but will generally lead to deeper and wider microfluidic
channels than originally intended.

Figure 1 shows top-view micrographs of a PDMS device designed for particle
separation, before and while a pressure of 173 kPa (25 psi) is applied. The pillars
are 22 µm in diameter and 40 µm tall with a pitch of 44 µm. The gap is a critical
dimension and it has increased by more than 15\%. Figure 2a shows a plot of the
change in the post to post gap under various pressures for those same devices.
Clearly significant changes are occurring at typical operating pressures, and these
need to be addressed.

Alternatives to Standard PDMS

There are a number of alternatives to molding in standard PDMS that overcome
the problem of pressure-induced stretching. These include optical glue molding
and the use of h-PDMS. Optical glue molding from either a PDMS master or from
a hard master like SU-8 resist produces hard devices that can be bonded to
the Young's modulus of NOA 81 is $1.4 \times 10^3$ MPa, roughly 3 orders of magnitude higher than that of PDMS. This approach is relatively easy with a low set-up cost. The procedure is also well suited for multilayer structures. One problem is that the polymer is not gas permeable, so wetting the device has to be done by fluid flow and not all structures wet in this way without trapping air. The devices used here, when built in NOA 81 could not be completely wet. The suitability of these types of polymers for various biological work is also largely unknown.

An immediately available option is to increase the hardness of standard PDMS by adding additional cross linking agent. Armani et al. 1999 measured the elastic modulus of an un-named brand of PDMS at the standard 10:1 ratio, and with twice the amount of cross-linker, a 10:2 ratio. The Young's modulus increased from 750 to 870 kPa, only a 16% increase. This should lead to a similarly modest percent decrease in the deformation. Figure 2b shows the change in post to post gap for the structures when molded in 5:1 PDMS. The harder PDMS deforms less at smaller pressures, but by 200 kPa the degree of deformation is the same as standard PDMS.

Hard PDMS or h-PDMS was developed at IBM Zurich for nanoscale soft lithography.\textsuperscript{6,12} It is a highly cross-linked, 4 component version of poly(dimethylsiloxane). The tensile modulus of h-PDMS is 4.5 times that of standard PDMS.\textsuperscript{13} For convenience, the recipe is reproduced here\textsuperscript{1}. The mixture is typically spun onto a master mold to form a thin layer. Once the h-PDMS has cured, a thicker layer of standard PDMS is formed on top of the h-PDMS, allowing removal of the rigid layer without unacceptable amounts of cracking in the h-PDMS layer.

Hard-PDMS molds failed to release from the structures used here. Many of the 22 um diameter, 40 um tall posts broke off and were left in the mold. This may be because h-PDMS has a deformation-at-break value of only 6%, compared to 50%.

\textsuperscript{1} 3.4 g vinyl prepolymer VDT-731 (Gelest Inc.), 0.5\% w/w or 18 uL platinum catalyst SIP6831.2LC (Gelest Inc.), 0.1\% w/w (one drop, assumed to be 50 uL) modulator 2,4,6,8-tetramethyltetrahydrodicyclosiloxane (Sigma Aldrich), and 1 g hydro prepolymer HMS-301
to 100% for standard PDMS. It would be highly desirable to have a two stage curable PDMS: one that could be heat cured to something similar to standard PMDS, then UV cured after mold separation and device construction to a rigid material like h-PDMS; however, such material is not available. In the meantime a simple change in construction can dramatically reduce pressure-induced deformation in standard PDMS devices without sacrificing desirable properties such as gas permeability or deformability during mold separation. Additionally, the method also enhances the strength of reversible bonding and eliminates the possibility of a sagging or collapsing roof.

**Experimental**

The method for creating a glass-PDMS-glass device is as follows: A line of standard degassed PDMS is poured onto the master mold and squished under a glass microscope slide. The amount of PDMS should be just enough for the area under the glass slide to be filled by capillary forces before or during curing. After curing, a razor blade is used to separate the glass slide and PDMS film from the master mold. The resulting PDMS layer is typically around 200 µm thick. The steps of this procedure are shown in a movie in the electronic supplementary information. Separating the rigid device from the mold requires much more force than for bulk PDMS. So it is recommended that the master mold be supported on a flat surface. If the master is a wafer then taping or gluing it to a glass plate can prevent it from breaking. These glass supported, thin PDMS devices can now be reversibly or permanently sealed to a flat surface.

Fluidic connections in standard devices are usually made by boring a hole in the PDMS then press fitting or gluing in a tube. In the glass-PDMS-glass construction holes must be made in one of the glass plates and these are typically made by micro sandblasting, but diamond grinding, or ultrasonic drilling are also possible.

The major benefit of this new technique can be seen by comparing the pressure induced changes in permanently bonded PDMS-on-glass devices to those in glass-

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(Gelest Inc.). Gelest no longer makes SIP6831.1, but SIP6831.2LC is reported to be a suitable replacement.
PDMS-glass devices. Devices are made using RTV615 mixed at the intended 10:1 ratio (or 10:2 as indicated for Figure 2b), degassed for 20 minutes and cured for 2 hours at 75°C. Each device is created from the same master mold, that contains six 1-mm wide, 5-cm long arrays of pillars. The pillars are 22-µm diameter, 40-µm high with a period 44-µm in an approximately square array.

The first construction that was tested is the standard one. A 1 to 2 mm thick PDMS device is bonded to a glass slide. The bond was made by spin coating a thin (~50 µm) layer of 10:1 PDMS onto a glass slide, curing that for 8.5 minutes in a 75°C oven, then bonding to the device. A single hole was bored through the PDMS using a blunt syringe. A tube was inserted into the hole and glued, then connected to a syringe and manometer. A bright-field microscope using a 5 times objective and digital camera were used to record images of the pillar arrays at various pressures. Figure 1a shows what the pillars look like with no applied pressure and Figure 1b shows the same region at 173 kPa (25 psi). The posts have narrowed and other deformations are apparent. Figure 2a shows the percent change in the gap between the posts as the pressure is increased. Measurements were taken between posts in the middle of the array. Upon releasing the pressure the original dimensions were recovered. Figure 2b shows the change in post-to-post gap for the same device when molded using twice the recommended amount of cross linker, 10:2 RTV 615.

The lower set of images in Figure 1 and the data in Figure 2c are from glass-PDMS-glass devices. These figures show that there is little or no change in the appearance of the PDMS features after applying 173 kPa. Comparing the standard construction to the glass-PDMS-glass devices we see roughly 10 times less deformation at 70 kPa (10 psi).

Two of the glass-PDMS-glass devices were permanently bonded to glass slides by air plasma, while the other six were not treated in any way to enhance the natural PDMS-glass bond. The last measured pressure before leakage occurred is shown by a large dot that terminates their curves in Figure 2c. After cleaning with detergent and water, the devices were resealed and re-used. No correlation
between re-use and bond strength was seen. This shows the second advantage of the glass-PDMS-glass construction, an increased reversible bond strength.

Devices constructed here using standard PDMS on glass without bonding leaked and delaminated at 21 ±1.6 kPa (3.0±0.2 psi) N=5, consistent with previous observations. The same devices in the glass-PDMS-glass format help pressures ranging from 97 to 250 kPa (14 to 36 psi). This increase of at least five fold means permanent bonding is usually not necessary and devices can be used, disassembled, cleaned and used again.

**Modeling**

Models were created in COMSOL Multiphysics to clarify the deformation that has been observed. The models were created to match device geometry and used material properties from the literature. RTV-615 PDMS: Elastic Modulus (E) is 1.54 MPa for strain less than 0.45, \( \nu = 0.499 \). Glass: \( E = 2 \times 10^5 \) MPa, \( \nu = 0.33 \). Recent work by Schneider et al. gives precise data for modulus vs strain, which increases rapidly for strain larger than 0.5. Due to the difficulty in modeling rubber-like materials this non-linear character was not included in the model; however, the models presented here are limited to a regime where strain is less than 0.45.

It is clearly not reasonable to model the entire microfluidic device with its 155 thousand pillars. For the PDMS glass device, the reduced model (Fig 3a) contains 3 pillars, the glass beneath them and 200 \( \mu \)m of PDMS above. The PDMS is allowed to move vertically (\( z \)) but the outer boundary is not allowed to swell in the \( x \) or \( y \) directions because of the surrounding material. The model is one period, 44 um deep (\( x \)), and 3 x 44 um wide (\( y \)). The bottom surface of the glass block is constrained in all directions. Pressure is applied in a direction normal to each surface in the microfluidic channel: the floor, roof, and post surfaces.

The reduced model of the glass-PDMS-glass device (Fig 3b) contains three pillars at the bottom of a 200 um PDMS slab sandwiched between two glass plates. The depth of the model is 44 um. The width of the device is 682 um. It is made this
way so that the ratio of pillared area to bonded area is 6/25, the same as the full device that contain six, 1-mm wide arrays under a 25-mm wide glass slide. Again the bottom surface of the lower glass block is completely constrained, the walls are constrained in the x and y directions.

The nature of the deformation is always the same, for positive pressure the uppermost surface of the model moves upward, the fluidic channel roof also moves up in the z-direction becoming curved and the posts narrow. For negative pressure the posts become shorter and fatten through most of their height and the roof begins to curve inwards. Figure 3 shows the solutions to both models at 100 kPa with coloring to indicate displacement of between 0 (blue) and 14 (red) microns. Figure 4a shows the change in the post to post gap for the two models. The glass-PDMS-glass model shows 3 times less deformation than the standard PDMS on glass model. Because PDMS is incompressible any change in the height of the top surface comes through an increase in the microfluidic channel volume. This change in fluid volume is shown in Figure 4b.

**Discussion**

The deformation seen in the standard PDMS-glass devices (Fig. 2a) is highly non-linear, with most of it occurring by 70 kPa (10 psi). Clearly the elastic modulus increases with increasing strain, and the onset of this non-linearity is occurring earlier than predicted by the model. The deformation remains more linear in the glass-PDMS-glass device as the total strain is smaller and the modulus has not yet begun to increase. The model and experiment both show significantly reduced deformation when using the glass-PDMS-glass construction compared to conventional bulk PDMS on glass.

The good performance of the glass-PDMS-glass device is explained by the thin PDMS layer and the close proximity of the features to the plates. The plates distribute the load over a large area so that any change in channel height come at the cost of stretching a large area of PDMS. And a thin PDMS layer stretches less in absolute terms than a thick layer under the same load. Thinner PDMS layers can be made but have lead to unacceptably high mold separation forces. It is also crucial that PDMS is not compressible. Small air bubbles in the material would
allow it to compress, negating the effect of the glass-PDMS-glass construction. It may be possible to reduce deformation by layering a glass slide over thin PDMS, but thin PDMS films are fragile and air bubbles between the layers would reduce the effectiveness of the method.

There are other benefits to this glass-PDMS-glass construction as well. The reversible bond formed between PDMS and glass normally de-laminates and leaks at low pressures, but when a thin PDMS layer is sandwiched between two rigid planes, de-lamination occurs at higher pressures. Furthermore, this construction prevents roof sagging, allowing fabrication of wide unsupported chambers.

**Conclusion**

Pressure induced deformation is not a universal disadvantage; it is of course the essential material property enabling the Quake valve. But especially when syringe pumps are used or when high throughputs are sought, it should not be overlooked as pressures can be quite high. Deformability is especially problematic in deterministic lateral displacement devices where the distance between pillars is a critical dimension that should not change during operation or throughout the device as the pressure drops from input to output. It is also relevant to other cell separation approaches that use obstacles and to microfluidic sheathing in PDMS.

The significance of PDMS to the field of microfluidics cannot be overstated, but it has limitations. The deformability of PDMS clearly assists in its mold replication capabilities, but is a shortcoming once the device has been constructed. PDMS devices may never be mass-produced but PDMS is very convenient for prototyping and research applications where it will continue to be the dominant material for prototype chip fabrication. This paper shows how to manage the tendency of PDMS to change shape under pressure. Until a better material is developed, this method will help the research community to take advantage of the strengths and work with the weaknesses of PDMS.
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References

Figure Legends

Figure 1. Micrographs of two PDMS devices before and during pressurization. (a) A PDMS-glass device at 0 kPa and (b) at 173 kPa. (c) A glass-PDMS-glass device at 0 kPa and (d) at 173 kPa.
Figure 2. Experimental plots of deformations in the PDMS devices under fluid pressure. Each curve represents one device. (a) Percent change in post-to-post gap for devices formed by bonding a 1.5-mm thick, unsupported PDMS mold to a glass slide. (b) Percent change for devices formed by bonding a 1.5-mm thick, unsupported, over-crosslinked (10:2) PDMS block to a glass slide. (c) Percent change for devices formed by the method described in this article. These devices show much less deformation than devices made using the other methods. Large circles indicate the leak pressure for reversibly sealed devices.
Figure 3. Results of structural simulations of fluid pressure induced deformation. (a) The reduced model for the PDMS on glass construction. (b) The reduced model for the glass-PDMS-glass construction. In each case the wire frame shows the initial shape/position and the deformed shape is colored using the scale shown to show total displacement. The fluid pressure in each is 100 kPa.
**Figure 4.** Plots showing: (a) the percent change in post to post gap, and (b) fluid volume for the two models. Error bars in (b) are ± 0.3% and are smaller than the data points.