Electronic Supplemental Material for

Reconfigurable Microfluidic Systems with Reversible Seals Compatible with 2D and 3D Surfaces of Arbitrary Chemical Composition

Abhiteja Konda,[a] Jay M. Taylor,[a] Michael A. Stoller,[a] and Stephen A. Morin*[ab]

[a] Department of Chemistry, University of Nebraska-Lincoln,
Lincoln, NE 68588.

[b] Nebraska Center for Materials and Nanoscience, University of Nebraska-Lincoln,
Lincoln, NE 68588.

* To whom correspondence should be addressed. E-mail: smorin2@unl.edu.

Materials and Methods

Materials

The microfluidic stamps were fabricated in PDMS or Ecoflex® or combination of both. PDMS was purchased from Dow Corning Co., and Ecoflex® (00-30 and 00-50) two part platinum cure silicone kits were purchased from Smooth-On, Inc. Polymer films and sheets, t-slot struts, screws, nuts, and barbed fittings were purchased from McMaster-Carr®. Solutions flowed through the devices were prepared using various water-soluble dyes in different concentrations ranging from 25, 50 and 100 mG/100 mL. Tartrazine-FD&C Yellow #5 (CAS 1934-21-0), Allura Red AC-FD&C Red #40 (CAS 25956-17-6), Fast Green FCF-FD&C Green #3 (CAS 2353-45-9) and Erioglaucine-FD&C Blue #1 (CAS 3844-45-9) were purchased from VWR®. Flow through the microfluidic devices was controlled using peristaltic (MasterFlex L/S, Cole-parmer) and syringe (KD Scientific) pumps and silicone/polyethylene tubing (Masterflex Tygon® Lab E-3603 Tubing, and Polyethylene (PE) tubing, Intramedic™ PE60 and PE160 by BD & Co) were used to connect the devices to these pumps. The materials testing system used was an Instron® 5944 (2kN capacity).

Methods

Fabrication of masters and microfluidic stamps

We made our masters using solid object printing.[1] We designed a master in a CAD program and then printed it in ABS using a 3D-printer (Dimension Elite, Stratasys Ltd.). Technical drawings of a typical master used are shown in Fig. S1. The ABS masters were textured and can be smoothened using acetone vapor. The ABS mold is then used to fabricate micro-channel network in PDMS, Ecoflex® and combinations of both through soft lithography. All the devices (except those used in materials testing and replication for profilometry) have a layer of Ecoflex at the bottom (~1 mm thick). The thickness of this layer depends on the substrate, for example when seals are to be made to extremely rough substrates (e.g., sand paper or corrugated surfaces) a thicker (~2 mm) layer of Ecoflex is used. Barbed fittings are used as inlets/outlets to connect the tubing and the device. The devices are fabricated using PDMS with a 10:1

base:catalyst ratio and Ecoflex® 00-30 with a 1:1 ratio of "part A":"part B". The devices used to seal over 3D objects are fabricated either using only Ecoflex®, or combination of 25% Ecoflex® and 75% PDMS with a 20:1 base:catalyst ratio. The curing time for PDMS (10:1) is 60 mins at 70^{o}C, PDMS (20:1) is 90 mins at 70^{o}C, and for Ecoflex® is 45 mins at 65^{o}C. Following curing the channels can be treated with oxygen plasma to increase wettability to aqueous solutions.

Sealing Methods and Compression Devices

The bi-layer devices fabricated in PDMS and Ecoflex via soft lithography are reversibly sealed by compressing against an arbitrary substrate (e.g. polycarbonate (PC), polyethylene terephthalate (PET), teflon, PDMS, acrylonitrile butadiene styrene (ABS), aluminum sheet, glass, etc). The inlets/outlets connected the device to the peristaltic/syringe pumps for controlled flow through PE/Silicone tubing. We used a manual or a semi-automatic compression setup for creating the compression-based seals. The manual compression set-up (Fig. S2) is fabricated in ABS using 3D printing and has two parts: a top part that holds a transparent acrylic sheet to provide uniform compression, and the bottom part that supports the substrates. Screws and wing nuts are used to compress the device over a desired substrate between the top and bottom ABS parts. An equal number of turns to each wing nut was used to ensure uniform distribution of compressive stress on the device, and verified by measuring the height (the distance from the top frame to the bottom frame) around the entire perimeter of the set-up using a digital caliper. If necessary tension was adjusted by tightening and/or loosening the appropriate wing nuts. A semi-automatic compression unit set-up (Fig. S3) facilitates rapid switching between devices and compression states (Video S1 and Video S2). It is made of aluminum t-slot universal struts, a steel plate at the bottom, and an acrylic top. The acrylic top provides uniform pressure over the entire channel network and is transparent for visualization of the device. A hydraulic piston was used to tune compressive stress.

Operation of the Devices

The flow through the devices was controlled using peristaltic/syringe pumps. All the experiments were run at room temperature at flow rates ranging from 0.25 – 6.00 mL/min. High temperature (100^{o}C) and flow rates (300 mL/min) were used to test the compression-based seals under extreme operating conditions.

Determination of Channel Dimensions

We quantitatively investigated the change in the morphology and dimensions of the channel under various degrees of compression. Five devices of different heights (H_0 = 6.75, 7.0, 7.25, 7.75, and 8.15mm) were fabricated in PDMS using a single master. These devices were compressed to a final height (H_f) of 6.25 mm, by exerting a force of 50 – 1650 kPa (measurement described below). To assess the interior dimensions of the channel when compressed, replicates of the channels for these devices were generated by flowing a liquid low-melting temperature bismuth alloy into the devices under two different compression states: one, compressed to seal, and two, fully compressed (Fig. S4a,b). The material was delivered using a syringe, and disposable plastic microscopic slides were used as substrates. The alloy was allowed to solidify at room temperature while under compression. We expect minimal (-0.07%) thermal contraction. These replicates were then characterized using profilometry (Fig. S4c-f and Fig. S5). Bowing of the ABS set-up at very high compressive stress (> 900kPa) can cause the channels to bend/curve (Fig. S4d,f), therefore these stresses are avoided in typical operation. The change in width and height of the channels is tabulated in Fig. S5 and used in the plots shown in Fig. 3 of the main text. The stress exerted on the devices can be estimated as shown below:

$$\sigma = \frac{F}{A_0} = \frac{E\Delta H}{H_0} \quad (1)$$

Where, F = Force, A_0 = Cross-sectional Area, $\Delta H = |H_f - H_0|$, σ = stress, and E = Young's Modulus of PDMS, 7.5 x 10^5 Pa taken as from Ref. S2. Using this calculation the pressures for various ΔH values have been estimated (Fig. S6e).

The stresses estimated using the above formula and for the given Young's modulus of PDMS agree with those measured using the material testing system (MTS) at low stress, but deviate at high stress because eq. 1 does not account for friction and barreling (Fig. 3 and Fig. S6). A set of control devices of PDMS and PDMS/EcoFlex bilayer (36 mm x 24 mm x 9 mm) was used in these measurements. Two sets of data were collected: one with an ABS frame around the device to duplicate the scenario used in all the experiments with 2D substrates and the other iteration without the frame. From these data, it is evident that the stress required to compress a device with a frame to a particular strain ($\varepsilon = H_f/H_0$) was equal to or more than devices without the frame as expected because a frame prevents expansion in the lateral directions.

The stress vs. ΔH relationship measured using the materials testing system was fit using an exponential function ($y = y_0 + A\, e^{(R_0 x)}$, Fig. S6). This exponential fit was used to estimate pressure throughout the manuscript. This method was adopted as simultaneously measuring pressure and observing the device would have been experimentally difficult.

Rapid Switching

The ability of compression to rapidly reconfigure the fluid pathways based on the compression state of the device (sealed or sealed and further compressed) is shown in the Video S1 and Video S2 (also see main text Fig. 4). A flow rate of 3–6 mL/min was used. The stress exerted on the device varied from ∼120 kPa in compressed to seal state to ∼430 kPa in the futher compressed state that results in the formation of independent channels. Video S3 illustrates the fluid flow in multi-mode devices (Fig. 4e, also see main text).

UV-Visible Spectroscopy

The “cross-talk” between channels in multi-mode device was obtained by analyzing the colored solutions using UV spectroscopy. Stock solutions (1000μM) of Blue #1 and Yellow #5 were prepared. Standard solutions were prepared in concentrations of 100, 50, 25, 12.5 and 6.25 μM by serial dilution of the stocks and the absorption spectra were recorded to determine the λ_{max}. Absorbance values were then recorded at λ_{max} to calculate the concentrations using the Beer-Lambert Law. The Absorption spectra for Blue #1, Yellow #5 and Mixture (50% Blue & 50% Yellow) are shown in Fig. 5b. The spectra for the samples collected at three different compression states (state 1: ∼120 kPa, state 2: ∼300 kpa, and state 3: ∼680 kPa) and the corresponding concentrations calculated are shown in Fig. 5c-i.

Sealing over 3D Substrates

We demonstrated the ability of our approach to seal over three-dimensional objects. We wrapped the devices that are fabricated using a combination of 25% Ecoflex® and 75% PDMS with a base:catalyst ratio of 20:1 around an 8 mL vial, a gloved hand, round bottomed flask, and a rectangular ABS frame. The ends of the devices were held in position using a transparent tape for the vial, flask, and frame. The stress exerted through this wrapping process was estimated as follows:

A device (180 mm x 18 mm x 6 mm) was wrapped around a 3D cylinder and colored aqueous solutions were flowed through the device at a flow rate of 6 mL/min for 5 mins. After verifying that the device was leak-free, the length of the device was measured (238 mm) and compared to its initial length (180 mm) to calculate the tensile stress. The device was then strained to the same degree using the MTS and the required tensile stress was found to be 460 kPa. We assume the stress on the surface of the rod to be equal to the measured tensile stress. As the measured quantity (460 kPa), has a magnitude similar to all planar demonstrations, we feel this estimate was a reasonable one.

Fig. S1 Technical Drawing of an ABS Master: The drawing shows the dimensions (mm) of the ABS mold and the typical channel dimensions (mm) for the various devices shown in the main text. This specific master was used in the fabrication of the device shown in Fig. 1i of the main text, and is a ABS mold for a device with parallel channels.

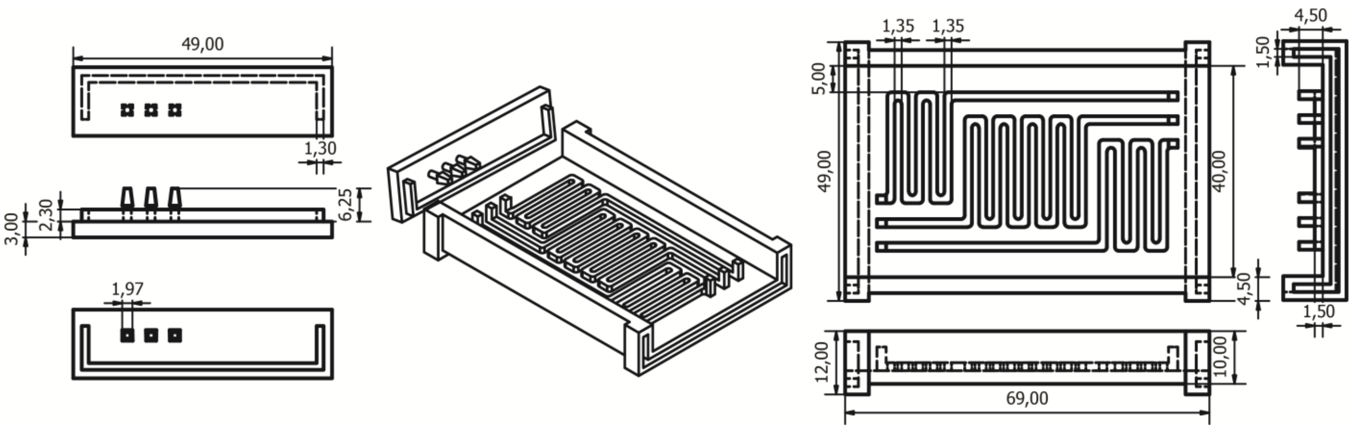


Fig. S2 Manual Compression Set-up: a) Technical Drawing (dimensions in mm) for the set-up (top and bottom part), and b) A photo of a device with PDMS inserts as inlets/outlets sealed over a polycarbonate film while flowing colored aqueous solutions and penny is shown for scale.

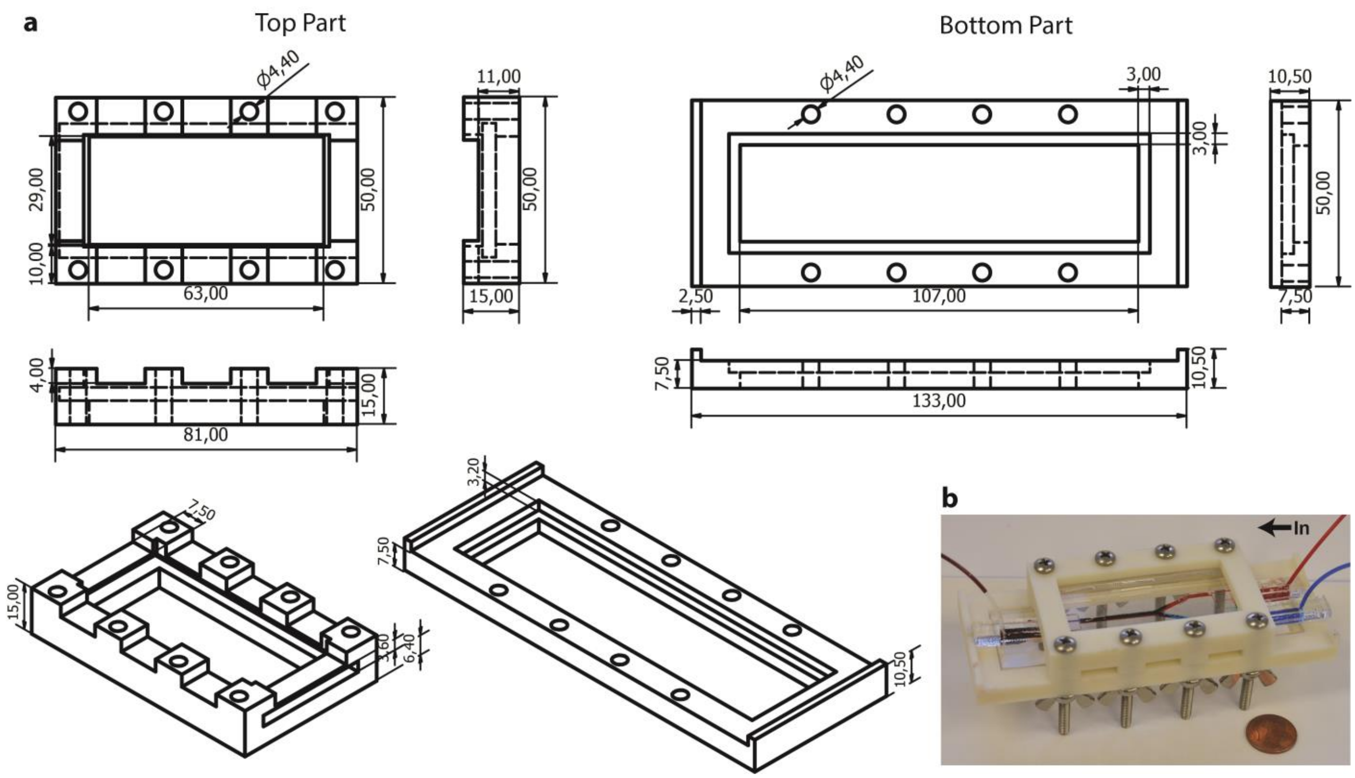


Fig. S3 Semi-Automatic Compression Set-up

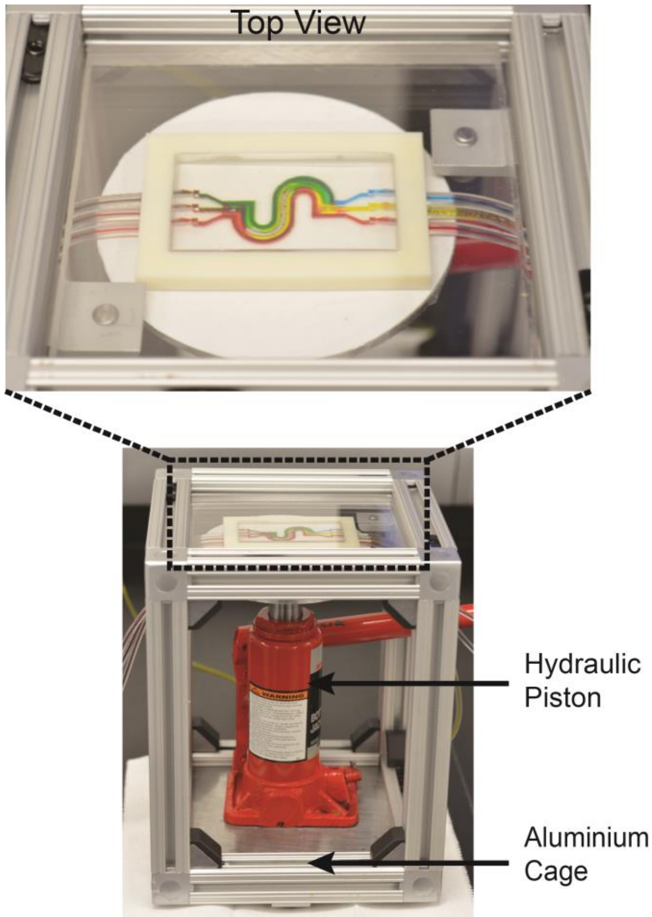


Figure S4. Change in channel morphology and dimensions. (a,b) Schematic showing the cross-sectional view of the device and channel in two compression states, sealed or sealed and compressed. Profilometric images of metal replicates formed from a sealed device (c), and a sealed and compressed device (d). Shown is a ~600 μm (c) wide channel (at ∼120 kPa) reduced to ~250 μm (d) under compression (at ∼340 kPa). Profilometric images of a channel which is ~1290 μm tall and ~1120 μm wide at ∼120 kPa (e) reduced to ~500 μm in height and ~400 μm in width at ∼1600 kPa (f).

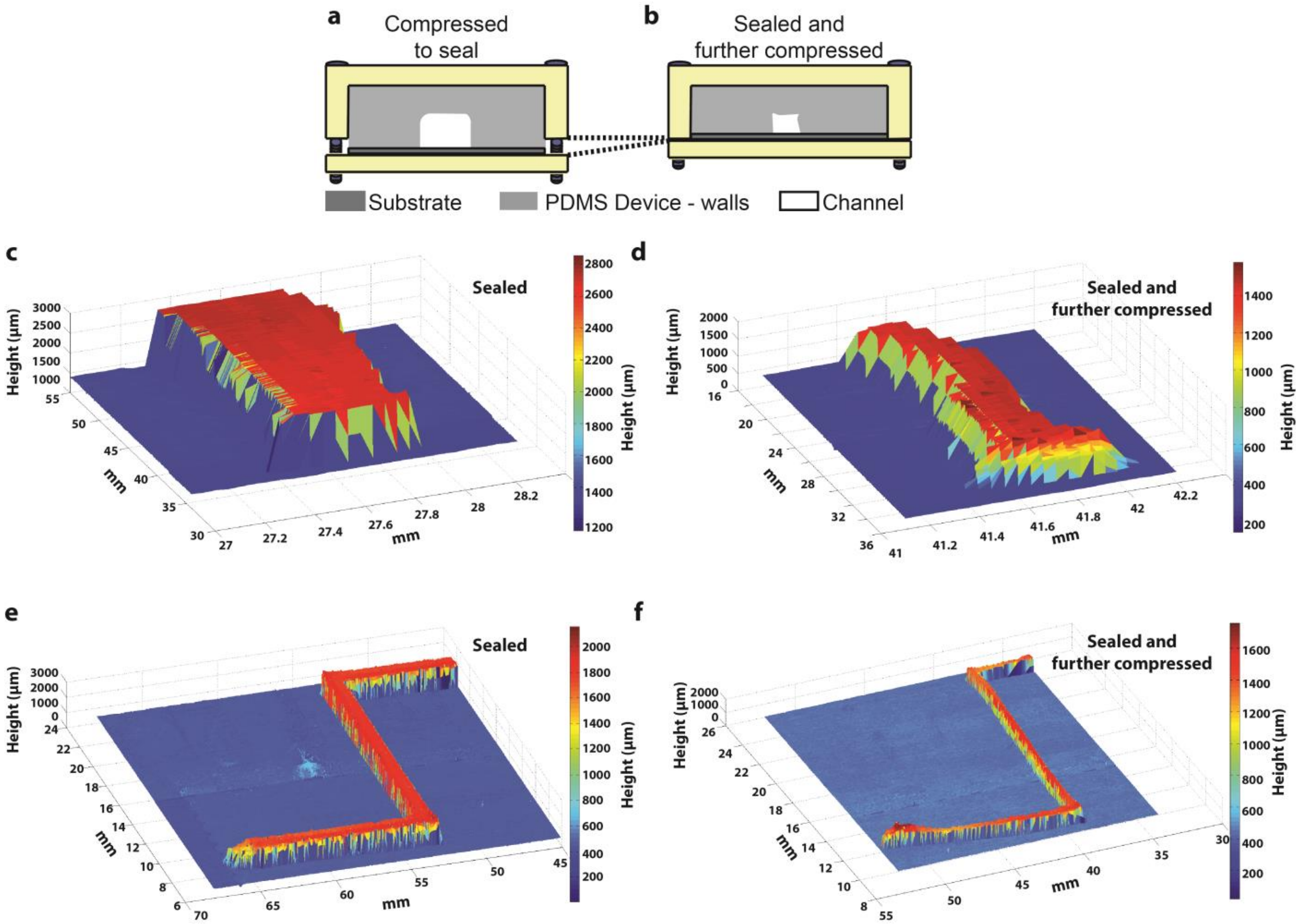
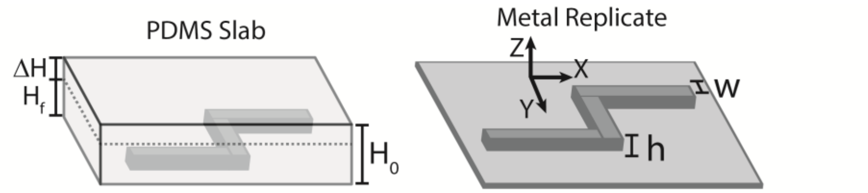


Figure S5. Data for the height and width measurements of the channels along XZ and YZ axes (a) plotted in Fig. 3. Tabulated data was obtained through profilometry of bismuth alloy replicates cast at varying pressures. Z is the height, XZ-width is the width of the channel perpendicular to X-axis and YZ-width is the width of the channel perpendicular to Y-axis. The devices used were fabricated in PDMS.



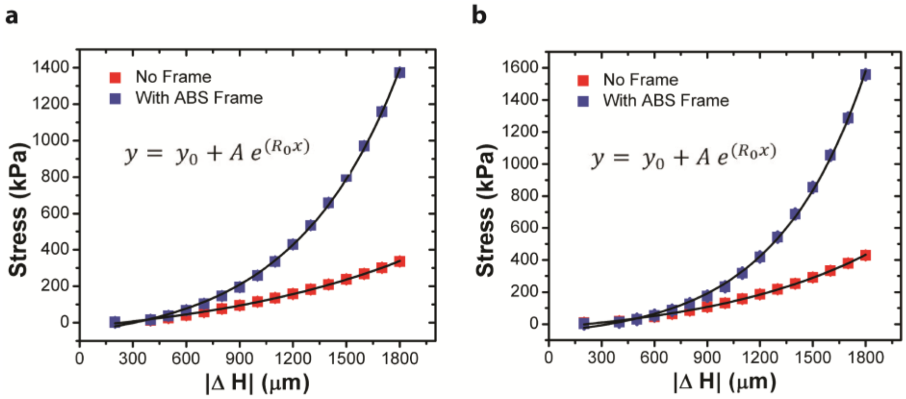
Along XZ-axis

Pressure (kPa)	Height (µm)			Width (µm)		
	Control	Test	Δh	Control	Test	Δw
~50	1230	1100	-130 ± 20	1120	910	-210 ± 20
~135	1330	1080	-240 ± 10	1360	780	-580 ± 20
~270	1430	900	-530 ± 20	1070	660	-410 ± 10
~790	1240	480	-760 ± 20	1040	510	-520 ± 10
~1660	1210	380	-830 ± 30	1080	370	-710 ± 10

Along YZ-axis

Pressure (kPa)	Height (µm)			Width (µm)		
	Control	Test	Δh	Control	Test	Δw
~50	1380	1130	-250 ± 20	1220	970	-240 ± 20
~135	1370	850	-520 ± 10	1270	930	-340 ± 20
~270	1410	850	-560 ± 20	1170	610	-570 ± 10
~790	1280	620	-670 ± 30	1050	370	-680 ± 10
~1660	1370	620	-750 ± 40	1150	430	-720 ± 20

Figure S6. Graph showing the pressure exerted on a PDMS only (a) and a bi-layer (PDMS, 10:1, with a ~1 mm thick EcoFlex layer at the bottom) device (b). The pressures exerted on each device are measured by compressing the device using a materials testing system, and data is collected with and without a frame. These data are fit using the following exponential equation: $y = y_0 + A\,e^{(R_0 x)}$. The fitting parameters are tabulated for the PDMS device (c) and the bilayer device (d). The lines of fit are shown in the plots (a,b). (e) Tabulated data comparing forces estimated with eq. 1 and the values estimated (for device without frame) using the line of fit for the data measured using materials testing system.



c

PDMS Device Only			
No Frame		With Frame	
R^2	0.9988	R^2	0.9990
y_0	130 ± 10	y_0	120 ± 10
A	110 ± 10	A	72 ± 6
R_0	$8.2 \times 10^{-4} \pm 0.4 \times 10^{-4}$	R_0	$1.69 \times 10^{-3} \pm 0.04 \times 10^{-3}$

d

PDMS & EcoFlex Device			
No Frame		With Frame	
R^2	0.9991	R^2	0.9987
y_0	102 ± 9	y_0	96 ± 14
A	82 ± 7	A	50 ± 5
R_0	$1.04 \times 10^{-3} \pm 0.04 \times 10^{-3}$	R_0	$1.96 \times 10^{-3} \pm 0.05 \times 10^{-3}$

e

ΔH (μm)	Compressive Stress Estimated Using Young's Modulus (kPa)	Compressive Stress Calculated Using the Fit from b (No frame, kPa)
500	~55	~35
750	~80	~75
1000	~105	~120
1500	~145	~245
1900	~175	~390

Supporting Videos:

Video S1. Rapid switching of a device between two compression states (4X actual speed).

Video S2. A device switching between a mixing and non-mixing state by varying the compression (4X actual speed).

Video S3. A "multi-mode" device cycled between three compression states (2X actual speed). Each state represents a unique configuration of the channels.

Video S4. A device sealed to a piece of sand paper maintaining leak-free operation as colored solutions were flowed through it.

References

S1 J. C. McDonald, M. L. Chabinyc, S. J. Metallo, J. R. Anderson, A. D. Stroock and G. M. Whitesides, *Anal. Chem.*, 2002, **74**, 1537-1545.

S2 D. Armani, C. Liu and N. Aluru, Re-configurable fluid circuits by pdms elastomer micromachining. In *Micro Electro Mechanical Systems*, 1999, 222-227.