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Planar micro-check valves exploiting large polymer compliance

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Abstract

This paper presents three types of microfabricated planar check valves that exploit the large compliance of elastomeric polymers such as polydimethylsiloxane (PDMS). The micro-check valves consist of a thin compliant flap and a rigid stopper embedded in close proximity in a microchannel. The flap is perpendicular to the flow and lies near the stopper, forming a restricted fluid path inside the channel. The shape and size of the gap between the flap and stopper vary with the applied pressure, resulting in large flow resistance for forward flow and small resistance for reverse flow. The microfabricated valves have either two- or three-dimensional arrangement of valving elements. In particular, a interesting normally closed check valve was designed and fabricated using a special technique, in which the flap and stopper are, as fabricated, in intimate contact and form a zero-gap between them. Testing results show that the check valves can achieve a diodicity up to 10^5. The check valves can be fabricated using the standard replica molding technique from PDMS, and thus are highly amenable to integration with other PDMS-based microfluidic systems. The planar check valves can potentially used in lab-on-a-chip systems due to its high diodicity, planar configuration and use of inexpensive polymeric materials.

Keywords: Check valves; Compliance; Flow control; Lab-on-a-chip; Microfluidics

1. Introduction

Micro-check valves that allow unidirectional flow are among the simplest passive flow control devices for miniaturized lab-on-a-chip systems [1–3]. Such devices can be used to guide and gate fluid flows, as well as to enable mechanical micropumps. For integrated lab-on-a-chip systems, check valves are desired to have a simple planar configuration and large diodicity (*i.e.*, the ratio of forward flow to reverse flow rates), and be of low cost. Micro-check valves have commonly been fabricated on silicon substrates using micromachining technology [1–3]. Various polymers, such as parylene [4], polyimide [5], polysulfone [6], polyester [7], SU-8 [8] and polydimethylsiloxane (PDMS) [9–13], have also been used to fabricate microvalves. Polymer materials generally have advantages of low cost compared with semiconductor materials. In particular, PDMS has been widely used due to its large compliance and ease of fabrication. Currently, most PDMS-based valves are active devices consisting of hybrid structures of PDMS and other materials [9–12], having complex designs that require rather complicated fabrication procedures. The few existing PDMS-based micro-check valves [13,14] are non-planar, using a thin PDMS membrane covering a valve seat.

To date, check valves with simple planar geometry and high diodicity have been rather scarce. Existing microfabricated check valves generally have complex, non-planar configurations, and are thus difficult for integration. Their diodicity varies significantly depending on the design and configuration [1–3]. The no-moving-parts (NMP) valves, such as Tesla and diffuser valves, are typically planar, but they usually have very low diodicity (typically less than 4), which significantly limits their applications [15]. Recently, an in-plane SU-8 structure was reported for check valves, although no testing data were provided [16]. Two other planar check valves were also reported that use moving micro-pistons [17] and responsive hydrogels [18] as passive valving components, respectively. The valve with a moving piston shows a high diodicity up to 1.25×10^5 [17]. However, both these valves were fabricated using unconventional fabrication methods, such as *in situ* laser polymerization. Recently, a micro-gate valve with a planar configuration was also reported

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that demonstrated a diodicity up to 2.7×10^4 [19]. The diodicity of this valve may be further increased as the valve seats can completely encompass the fluid flow, leading to minimal leakage.

This paper presents three types of planar check valves that uniquely utilize the large compliance of an elastomeric polymer such as PDMS. The results on these valves were partially reported in Ref. [20]. The valves feature a thin compliant flap and a rigid stopper located closely within a microchannel. Under applied pressures, the gap between the flap and the stopper changes its shape and size, thus, leading to a small flow resistance for forward flow and large resistance for reverse flow. The microfabricated valves have either two- or three-dimensional arrangement of valving elements. In particular a zero-gap device, in which the flap and stopper are in contact when there is no flow, has demonstrated much reduced leakage under low reverse pressures. The high diodicity of the valve (*i.e.*, up to 10^5 for the types-B and -C) is because the flap can completely seal the valve seat (*i.e.*, the stopper), which is similar to the sealing approach used in the recent reported gate valve [19]. Compared with existing devices [16–18], these check valves have a simple planar configuration, and can be fabricated completely using standard low-cost PDMS replica molding techniques [21,22]. These valves are also highly robust due to their simple planar geometry, and can be readily integrated in PDMS-based complex microfluidic systems.

2. Design

The micro-check valves are designed based on the large compliance of PDMS. For all three valve designs, a compliant microstructure is embedded in a microchannel and consists of a thin compliant flap and a rigid stopper located in close proximity. The flap is perpendicular to the flow and lies near the stopper, forming a restricted in-plane fluid path. For forward flow, hydrodynamic force pushes the flap away from the restriction step, allowing fluid passage. For reverse flow, the flap is pushed toward and contacts the stopper, blocking the flow passage and shutting off the flow. Therefore, unidirectional flow can be achieved by the in-plane flap movement.

Three valve designs have been investigated (types-A, -B and -C designs shown in Figs. 1 and 2). The type-A valve has two-dimensional (2D) arrangement of valving elements, where the flap is fixed to one sidewall of the microchannel (300 μm wide and 275 μm high), and the stopper to the other sidewall, forming a flow restriction 40 μm wide and 500 μm long. Bonding a PDMS sheet fabricated with such microfluidic features to a flat PDMS or glass plate forms the devices (Fig. 1), where the flap and stopper are both anchored at the three channel walls, *i.e.*, ceiling, floor and one side wall of the channel. Only one end of the flap is not anchored. Therefore, the flap deflection under hydraulic pressure is symmetric about the middle horizontal plane of the fluid channel. For the type-B valve, the stopper sits inside the microchannel (300 μm wide and 400 μm high) on the bottom PDMS sheet, forming a flow restriction 75 μm wide and 200 μm high, while the flap is anchored to the cover PDMS sheet. Thus, type-B valve has three-dimensional (3D) arrangement of valving elements. To further improve the check valve performance, the device design can be varied in such a way that the flap is in direct contact with the stopper in the absence of an applied pressure (Fig. 2). Specifically, the type-C valve contains a thin PDMS through-hole structure layer sandwiched between two flat PDMS or glass plates. It has the same channel cross-section dimensions (300 μm wide and 275 μm high) as the type-A valve, except that the as-fabricated flap and stopper are in contact and form a zero-gap, allowing virtually zero leakage. While this design has minimal impact on the device's forward flow resistance due to the large flap compliance, the leakage rate under reverse pressure can be drastically reduced. This zero-gap device will be referred to the type-C valve. Note that the specific device dimensions, chosen for demonstration of principle, can be readily varied to satisfy given performance specifications.

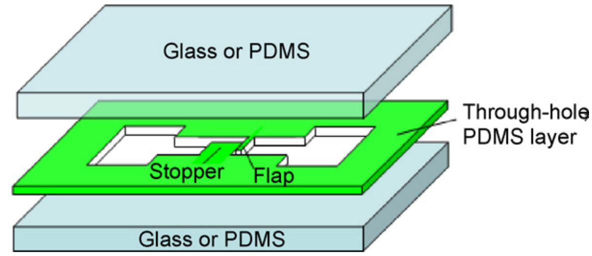


Fig. 2. Schematic of the type-C valve design featuring a zero flap–stopper gap for improved diodicity. The valve contains three layers, where the stopper and the flap are in close contact in the absence of a fluid flow.

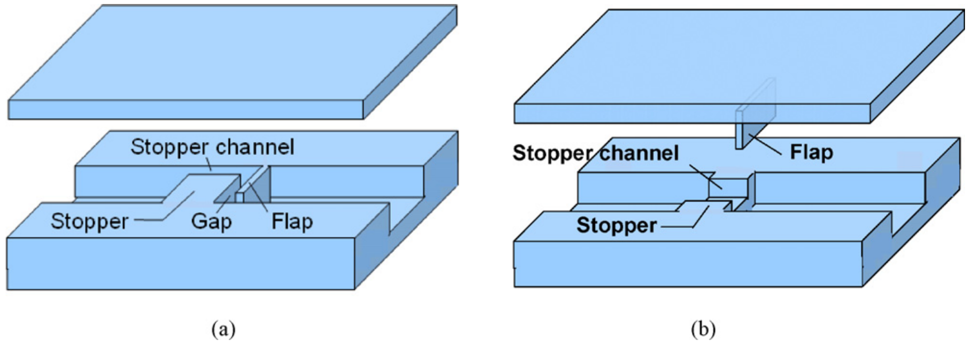


Fig. 1. Schematics of types-A and -B design check valves. (a) Type-A valve design with a 2D arrangement of valving elements. The flap will be anchored to three channel walls. (b) Type-B valve design with a 3D arrangement of valving elements. The flap is fixed to the PDMS cover sheet.

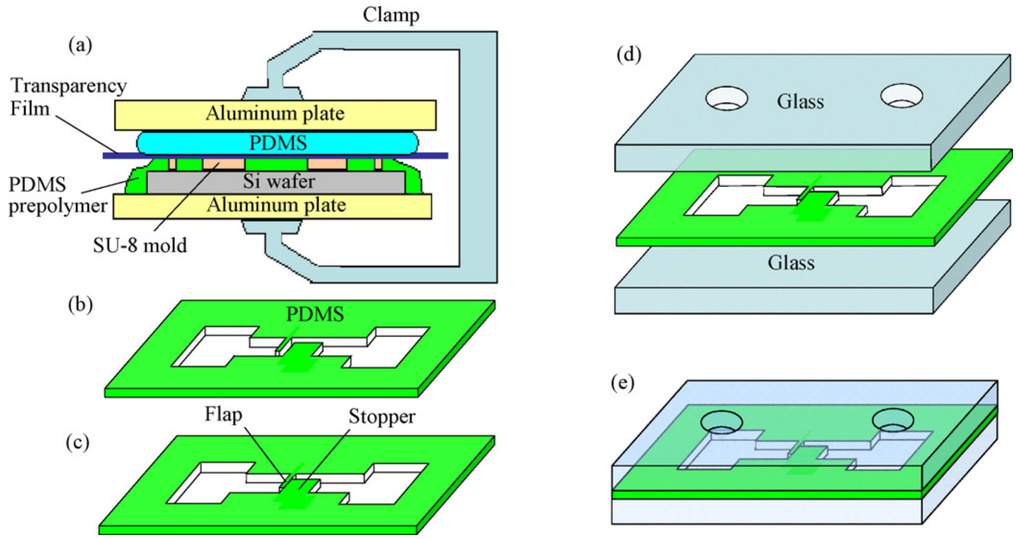


Fig. 3. Fabrication process of the type-C valve. (a) Replica molding of thin through-hole PDMS layer by clamping method [22,24]. (b) Demolding of the thin PDMS layer (a gap exists between the flap and stopper). (c) Manipulation of the flap to contact the stopper by spontaneous adhesion or using tweezers. (d) Bonding of the three layers. (e) An assembled device.

3. Fabrication

The replica molding technique [21,22] was used to fabricate the prototype devices from PDMS (with a nominal Young's modulus of 750 kPa [23]). The molding master was fabricated from the SU-8 photoresist (SU-8 2100, MicroChem Corp., Newton, MA) on a silicon wafer using transparency film as photo masks.

A curing agent and PDMS prepolymer (Sylgard 184 Silicone Elastomer Kit, Dow Corning) were thoroughly mixed in a 1:10 weight ratio. After being degassed for 1 h to remove any air bubbles, the mixture was poured onto the master mold. A transparency film was carefully lowered onto the prepolymer mixture to prevent bubbles from forming at the interface. It also served as a handling tool to remove the PDMS replica from the master mold after curing. The master-prepolymer-transparent film stack is clamped between two plates and cured for 3 h at 100 °C. The resulting PDMS replica containing microfluidic features was finally peeled off the master mold, and was permanently bonded to a flat PDMS sheet or glass plate to complete the fabrication process.

While the PDMS fabrication followed the standard procedure [21,22] for the type-A device, the assembly of the type-B valves involved more demanding alignment of the cover and bottom PDMS sheet. After the bottom and cover PDMS sheets were cleaned by ultrasonic agitation with acetone, they were placed into a shallow container with methanol, which served as a surfactant for alignment. Five small holes and pillars of the same size (100 μm diameter) were fabricated on the bottom and cover sheets, respectively, to facilitate the alignment during assembly. The two sheets were aligned and assembled manually under a microscope. After methanol evaporated, the surfaces of the two sheets were reversibly bonded together. The accuracy of the alignment is estimated to be about 3–5 μm, by comparing the actual distance measured by microscope to the designed distance.

Furthermore, a special technique was developed to fabricate of the type-C valves (Fig. 3). Here, a thin through-hole PDMS sheet was fabricated that contained the flap–stopper structure. Specifically, the PDMS prepolymer was poured onto the SU-8/Si master mold and degassed. A transparency film and a thick cured flat PDMS plate were then placed onto the prepolymer, and the resulting stack was tightly clamped between two aluminum plates and cured. The resulting PDMS sheet contained the desired microfluidic features, along with an unwanted, residual film formed between the master and the transparency film. The residual PDMS film was very thin (with sub-μm or μm thickness) thanks to the conformal contact between the transparency film and the SU-8 master allowed by the thick PDMS plate in the molding assembly [22,24]. As a result, the residual fractured at the edges where it joined the solid microfluidic features when the PDMS sheet was peeled off the master, and was hence easily removed. Thus, the microfluidic features extended through the entire PDMS sheet thickness, with the flap and the stopper still separated by a small gap (determined by design layout). Only constrained at one side of the channel, the highly compliant flap tended to deflect and adhere to the stopper spontaneously. Alternatively, using methanol as a surfactant, the PDMS sheet could be slightly stretched at its two diagonally opposing corners. Due to the large compliance of PDMS, considerable deformations occurred near the flap, resulting in deflection and contact of the flap with the stopper. The contact persisted due to spontaneous adhesion between the surfaces. Figs. 4 and 5 show some three-dimensional micrographs of fabricated PDMS sheets.

For the fabrication of the SU-8 mold for the type-A and -C valves, the practically attainable smallest gap between flap and stopper was about 15 μm when the channel height was 275 μm. If the gap was smaller than 15 μm, fabrication of the SU-8 mold became difficult due to the high aspect ratio.

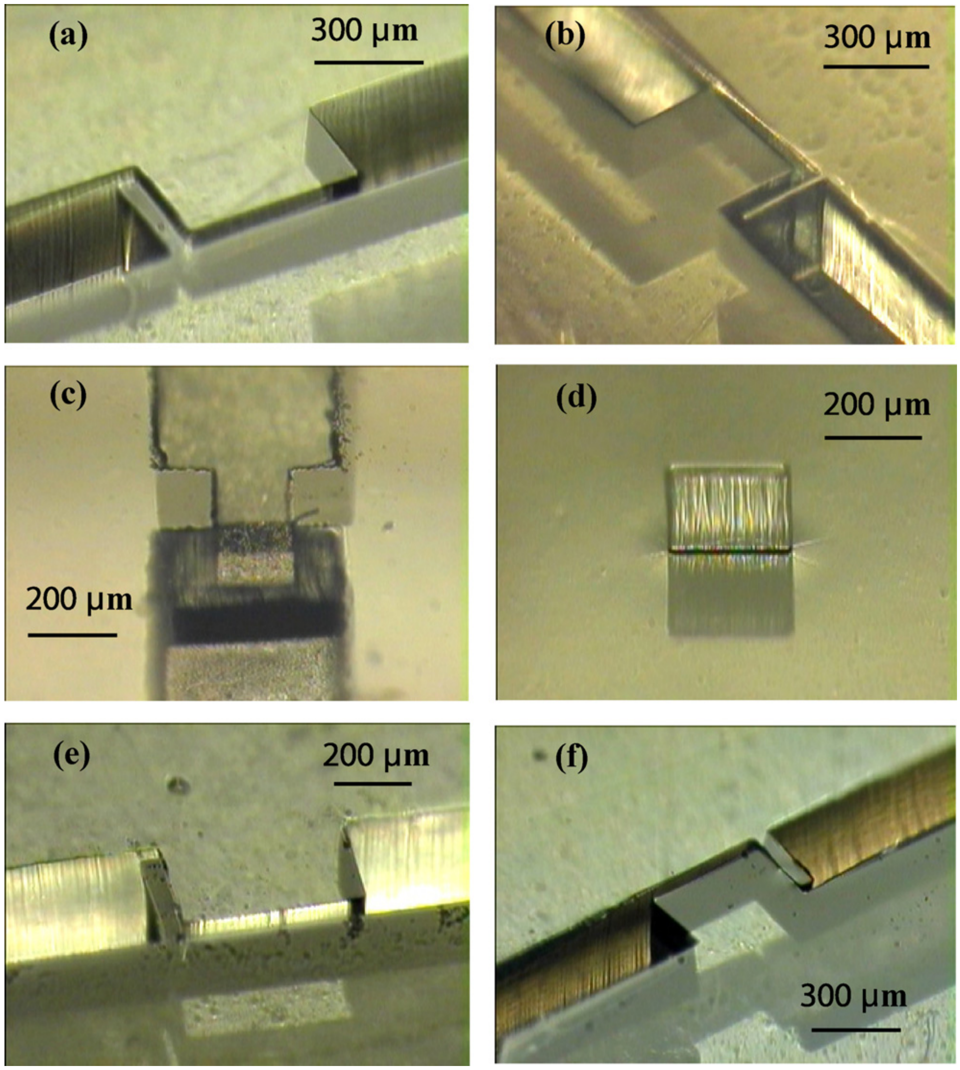


Fig. 4. Micrographs of fabricated PDMS sheets before assembly. (a and b) Type-A valves, (c) bottom and (d) cover sheets of type-B valves. (e and f) Type-C valves (with a zero-gap between the flap and the stopper).

4. Results and discussion

The fabricated microvalves were tested using the setup shown in Fig. 6, in which pressurized argon was used to drive de-ionized water through the device. Pressure was measured upstream of the water tank. The total pressure loss in the test setup and the microchannels leading to the compliant flap–stopper structure was estimated on the order of hundreds of Pascals, thus, was

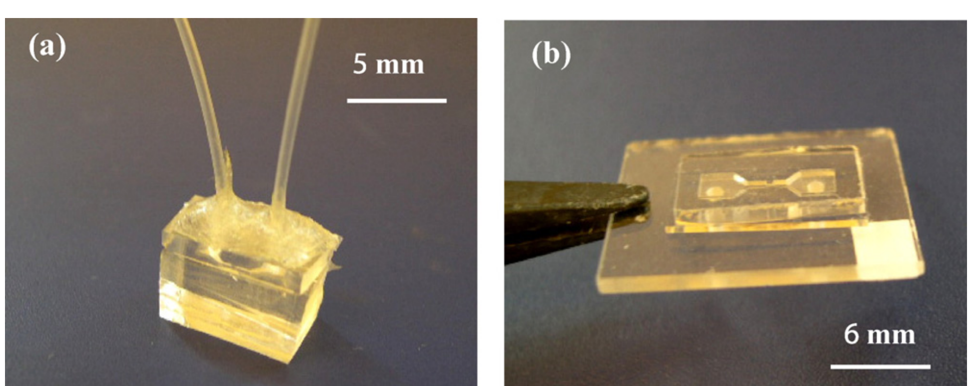
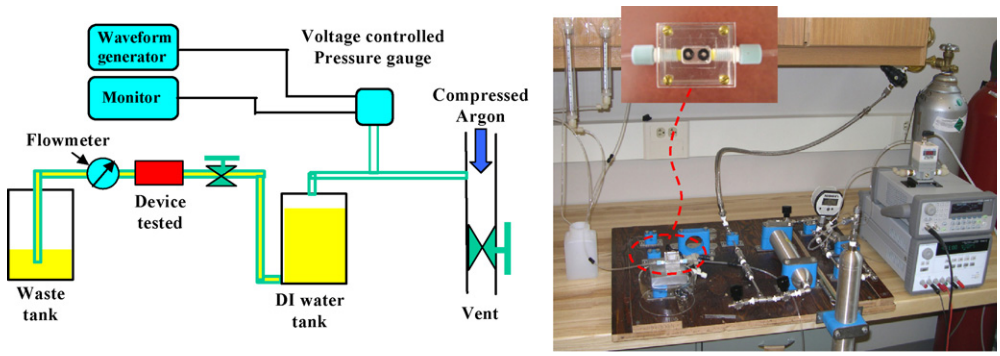


Fig. 5. Images of the packaged valves: (a) a packaged type-A valve; (b) a packaged type-C valve bonded with two glass plates.



(a) (b)

Fig. 6. (a) Schematic and (b) image of the experimental setup.

negligibly small compared to the driving pressure. A digital flowmeter (Alicat Scientific: L-1CCM-D, Tucson, AZ) was used to measure flow rates.

4.1. Type-A valves

Testing results of two type-A valves (A1 and A2) were shown in Fig. 7. The flap thickness was 60, 75 μm, and the flap–stopper distance was 36 and 25 μm for valves A1 and A2, respectively. It can be seen that as pressure increases the forward flow rate increases much faster than the leakage rate. The forward flow rate reaches 4 ml/min at about 80 and 170 kPa for valves A1 and A2, respectively. The nonlinear behavior of the forward flow can be explained by the continuous decrease of the flow resistance as pressure increases. For reverse flow, the leakage rate increases only slowly with pressure. The leakage first increases with the applied pressure at lower pressures. After reverse pressure becomes larger than approximately 60 and 90 kPa, the leakage rate saturates to nearly constant values, about 0.2 and 1.2 ml/min for A1 and A2, respectively.

It can be seen in Fig. 7 (here and in the following figures, data points have been connected by solid lines to guide the eye) that valve A1 has larger forward and reverse flow rates than those of valve A2. This is mainly because the flap–stopper distance for A1 (36 μm) is larger than for A2 (25 μm). The flap thickness may also play a role as a thinner flap (*i.e.*, 60 μm for A1) is easier to deflect than a thicker flap (*i.e.*, 75 μm for A2). Generally, larger flap–stopper distances with thinner flap lead to larger forward flow rates, while smaller flap–stopper distances with thinner flap are desired for low leakage rate. Testing results demonstrate that type-A valves indeed function as check valves. However, since the flap is anchored to three channel walls for type-A valves, they cannot shut off completely under reverse pressures.

It is believed that using a thicker PDMS layer (*i.e.*, a deeper fluid channel) will improve the sealing of the type-A valve, leading to a larger diodicity. This is because a higher flap associated with a deeper fluid channel is more compliant, and allows a larger portion of the PDMS flap to contact the stopper. In the further work we plan to fabricate valves with thinner and higher flap to further improve the diodicity. However, fabrication of high-aspect-ratio PDMS flaps (AR > 10) faces challenging PDMS demolding problems.

4.2. Type-B valves

Shown in Fig. 8 is the measured flow rate versus applied pressure for two type-B valves (B1 and B2). In the design, the flap thicknesses were 65 and 45 μm, and the flap–stopper distances were 10 and 5 μm for valves B1 and B2, respectively. The actual flap–stopper distances for both valves were measured

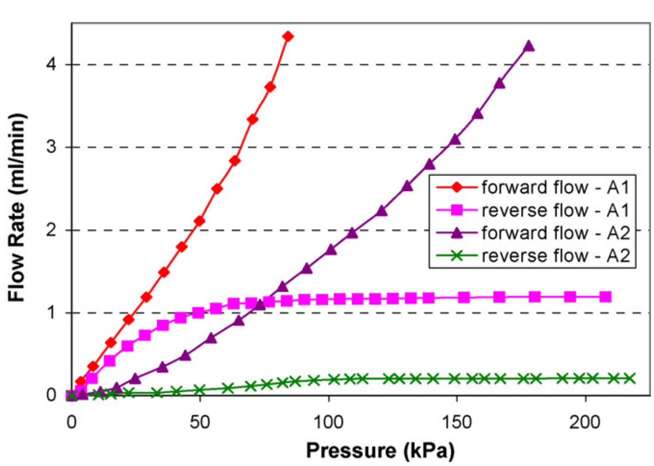


Fig. 7. Testing results for two type-A valves (A1 and A2).

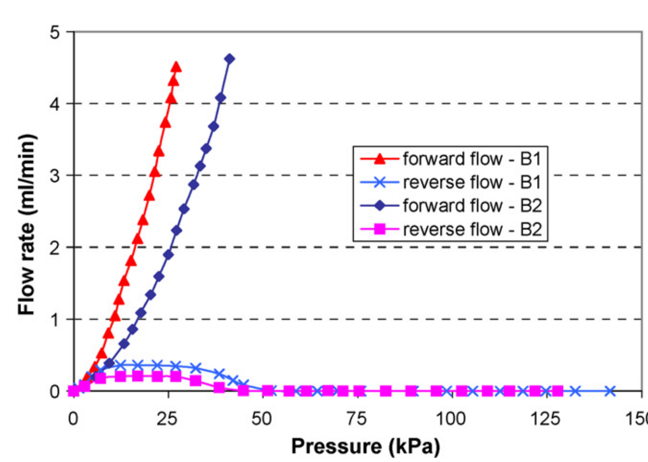


Fig. 8. Testing results for two type-B valves (B1 and B2).

under a microscope, which were about 8 and 4 μm for valves B1 and B2 when no pressure was applied. The flap was 300 μm in height and 200 μm in width for both valves. The main channel is 400 μm in height. Therefore, there is a 100 μm overlap between the flap and the stopper channel for both valves, which ensures that there is no leakage from the bottom of the stopper channel. This overlap between the flap and channel is important to ensure minimal leakage under reverse pressure. Fig. 8 shows that the forward flow rate reaches 4 ml/min at only about 25 and 37.5 kPa for B1 and B2, respectively. The forward flow rate for type-B valve is much larger than that of type-A valves, possibly because of its more flexible flap as well as the larger channel dimensions. For both valves, the leakage rate first increases with driving pressure at low pressures. While the pressure varies from 10 to 30 kPa, the leakage rate remains almost constant, at about 0.36 and 0.20 ml/min for valves B1 and B2, respectively. When the reverse pressure is further increased to higher than 35 kPa, the leakage rate decreases continuously until reaching a level below the detection limit of the measurement instrument (1 μl/min). We measured the leakage rate by timed observation of fluid accumulation using a fine pipette, from which it was determined that when the driving pressure was higher than 100 kPa, the reverse flow rate was zero within measurement error (a few nl/min). That is, at sufficiently high pressures, the device exhibits a diodicity on the order of 10^5. The shut-off pressure is about 58.7 and 44.8 kPa for B1 and B2, respectively. Testing results show that type-B valves have improved performance than type-A, as they allow the stopper channel to be completely sealed by the flap, thus, drastically reducing the leak rate under reverse pressure.

The difference in the shut-off pressure for valves B1 and B2 can be attributed to the fact that the flap–stopper distance for B2 (5 μm) is smaller than that of B1 (10 μm). In addition, the flap thickness of B2 (45 μm) is smaller than that of B1 (65 μm), which possibly also impacts the shut-off pressure.

4.3. Type-C valves

Fig. 9 shows the testing results for two type-C valves (C1 and C2). The flap thickness was 45, 60 μm for valve C1 and C2,

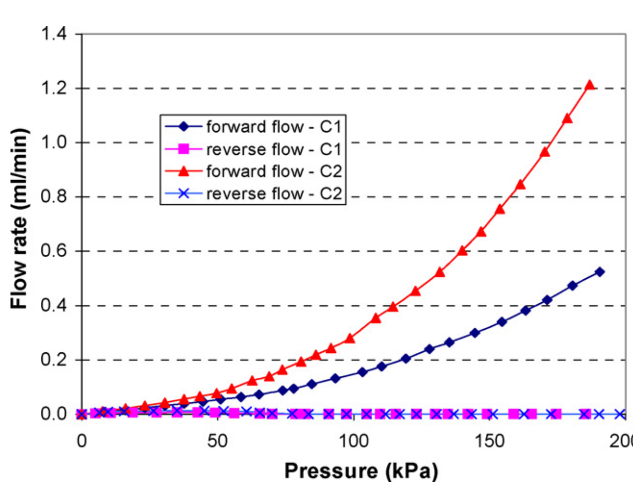


Fig. 9. Testing results for two type-C valves (C1 and C2).

respectively, and the flap–stopper distance was nominally zero for both valves. It can be observed from Fig. 9 that the type-C valves have the smallest leakage rate of all the three valve designs. Since the type-C valves are normally closed (due to the zero flap–stopper distance), the forward flow resistance is higher than those for the type-A and -B valves at low driving pressures. As forward pressure increases, the forward flow rate increases rapidly, indicating that the flap can be easily deflected away from the stopper. Note that under a given pressure, the forward flow rate of the type-C valves is much smaller than that of type-A and -B valves. This is because the type-C valves are normally closed, thus, have much larger flow resistance. It is believed that using a thinner flap in type-C valves can reduce forward flow resistance, thus leading to an even larger diodicity.

It can be observed from Fig. 9 that leakage is indeed drastically reduced over a large reverse pressure range for type-C devices. The maximum leakage rate is about 6 and 13 μl/min, at 39 and 52 kPa for valves C1 and C2, respectively. The valves shut off completely at reverse pressures higher than 60 and 65 kPa for C1 and C2, respectively. As the reverse pressure further increases to cause the flap into intimate contact with the stopper, the leakage rate drops to a level below the detection limit of the measurement instrument (1 μl/min). That is, at sufficiently high pressures larger than 60 kPa, the device exhibits a diodicity on the order of 10^5.

When the valve closes off the flow at sufficiently high pressures, its flap contacts the stopper. Hysteresis would occur if the flap cannot recover to its original position after the reverse pressure is removed, which is possibly caused by flap–stopper stiction (for all types of valves) or flap entering stopper channel (for type-B valves). It is thus highly desired that the flap can recover to its original position once the pressure is removed.

In the experiment, we did not observe any hysteresis effects, *i.e.*, flap–stopper stiction, for both type-A and -C valves in the pressure ranges (100–300 kPa) investigated, and the measured flow rate versus pressure relationship was repeatable. Observation of flap deflection under a microscope also confirmed that the flap could recover to its original position once pressure was removed. This suggests that the flap–stopper stiction force was negligible compared with the elastic force of the deformed flap. However, for type-B valves, we observed hysteresis at reverse pressures higher than 280 kPa, which forced the flap to into the stopper channel, causing the leakage flow rate to increase. When this happens, using a forward flow could easily push the flap out of the stopper channel, allowing the flap to recover to its original shape and position.

The experimental results confirm that the type-B and -C valves generally have higher diodicity (up to 10^5) than the type-A valves (diodicity up to 20). Type-B valves require low pressures to achieve high diodicity than type-C valves (*i.e.*, type-B and -C valves reach diodicity of 10^5 at higher pressures larger than 44–58 and 60–65 kPa, respectively). Type-C valves have smaller leakage than type-B valves at lower pressures.

For the measurement results shown in Figs. 7–9, the Reynolds number was estimated to be less than 1000 under both forward and reverse pressures for all the six valves, and the flow was therefore laminar in nature. Additionally, we observe a nonlinear dependence of the forward flow rate on the pressure for all devices tested. This can be explained by the continuous decrease of the resistance of each device to fluid flow as pressure increases, which can be attributed to the widening of the gap between the flap and the stopper as a result of the flap being increasingly deflected away from the stopper. This nonlinearity is particularly pronounced for type-C valves, as the flap–stopper gap widens from a virtually closed initial configuration.

5. Conclusions

The design, fabrication and characterization of three types of planar check valves have been presented that allow unidirectional fluid flow by exploiting the large compliance of elastomeric polymers such as PDMS. For all three valves, a compliant microstructure is embedded in a microchannel and consists of a flexible, vertical flap and a largely rigid stopper located in close proximity. The gap between the flap and stopper decreased or even closed almost completely for reverse flow, and enlarged for forward flow. This generates different resistances depending on flow direction.

The valves can be fabricated completely using the standard replica molding techniques from PDMS. In particular, we have developed a special technique to fabricate the type-C valves (with a zero flap–stopper gap), in which the flap and stopper were manipulated to contact before the final permanent bonding step. Testing results from prototype devices have verified the function of all three valve designs. The type-A valves (with 2D arrangement of valving elements) demonstrate an interesting flow saturation behavior at reverse pressure higher than 80 kPa, which can be exploited for such applications as producing constant flow rates under variable pressure. Both type-B (with 3D arrangement of valving elements) and type-C (with zero flap–stopper gap) valves can shut off flow completely within measurement error because the flap and stopper can be sealed virtually completely, *i.e.*, there is no leakage at the top and bottom of the flap–stopper sealing surface. While for all three designs forward flow increases with applied pressure, reverse flow for type-B and -C valves can be limited to zero under sufficiently high pressures within measurement error on the order of several nl/min, or a diodicity on the order of 10^5 under driving pressure up to 200 kPa.

The device is well suited to flow control in lab-on-a-chip systems due to several important features. First, the device is passive and simple in design, which leads to reduced power consumption requirements and improved robustness and reliability. In addition, the planar geometry of the device and use of inexpensive polymers will greatly facilitate cost reduction and system integration. To understand the device operation quantitatively, it will be interesting to perform a theoretical investigation of the physical phenomena involved. The key challenge is posed by the complex interaction between fluid flow inside the flap–stopper gap, and the deflection of flap. This challenge can be addressed by numerical simulations using a software package with capabilities to simultaneously consider fluid flow, structural deflections, and elastic contact. Alternatively, a more efficient approach may be based on modeling the fluid flow in the flap–stopper gap using lubrication theory [25], and determining the gap shape and size using theory of elastic plates [26,27].

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