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A latchable microvalve using phase change of paraffin wax

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Abstract

This paper presents a novel latchable phase-change microvalve that can potentially be used for flow gating in portable lab-on-a-chip systems where minimal energy consumption is desired. The microvalve innovatively exploits paraffin wax of low melting point, whose solid–liquid phase changes allow the closing and opening of fluid flow through deformable microchannel ceiling. The latchable phase-change actuation scheme can be used in an active valve to shut off and open fluid flow. Valve switching is initiated by melting of paraffin through heating, with an additional pneumatic pressure used for valve switching from open to closed state. Energy consumption is only required during the valve switching. After paraffin solidifies the switched state is subsequently maintained passively without further consumption of energy. The microvalve can be fabricated from PDMS through the multilayer soft lithography technique. Testing results demonstrate that the valve has response times of 60 s for closing and 100 s for opening; when closed, the valve can passively withstand a pressure up to 35 kPa without significant leakage. The relatively slow time response of the proof-of-concept device can be readily improved by integrating on-chip heaters, while the latchability of the microvalve can be improved by optimizing the wax chamber and membrane design. Compared to existing latchable phase-change valves, the thin compliant channel ceiling of the valve separates the fluid channel from the wax chamber. Therefore, the microvalve is free of potential contamination of the fluid by the paraffin wax. In addition, the improved sealing offered by the compliant membrane makes the valve robust and flexible in operation, allowing large ranges of initiation pressure generated from various actuation schemes.

Keywords: Actuator; Flow control; Paraffin wax; Phase change; Valves

1. Introduction

Microvalves are used to direct fluid flow along required paths, and are an important building block in lab-on-a-chip systems. Microvalves generally can be divided into passive (check) valves and active valves [1,2]. Check valves are typically used to realize unidirectional flow without requiring actuation. Active valves can in general enable more versatile functionalities, but require actuation to open and shut off fluid flow. Such actuation has generally required continuous consumption of energy to maintain the microvalve in open or closed state. On the other hand, many practical applications, such as lab-on-a-chip systems, portable point-of-care medical systems, and drug delivery devices, favor minimized power consumption [2–5]. It is therefore of great interest to develop microvalves that are more energy-efficient than existing active valves.

Paraffin waxes have been used in microvalves recently [6–12]. Existing paraffin-based microvalves mostly utilize paraffin's relatively large volume change, as induced by paraffin's solid–liquid phase change, for actuation [6–10], and thus require continuous energy supply. Recently two latchable paraffin valves were reported, which utilize low-melting-point paraffin's ability to hold its shape at its solidified phase. The two latchable valves can maintain their latched open and closed states without energy consumption, which makes them promising for future lab-on-a-chip applications [11,12]. However, these devices are either single-use [11], or have difficulties in controlling paraffin motion in that a slightly excessive pressure could drive the melted wax completely into the liquid channel, leading to device failure [12]. In addition, for both valves the direct paraffin–liquid contact may induce contamination of biofluids by paraffin wax during operation [11,12].

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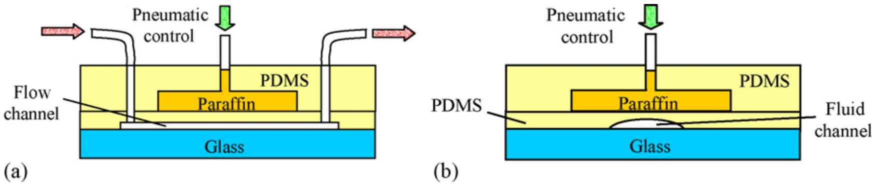


Fig. 1. Cross-sectional schematic of the phase-change microvalve: (a) along and (b) across the fluid channel. Paraffin is the latchable phase change actuation material. The channel has rounded cross-section to facilitate complete closure.

This paper presents proof-of-concept demonstration of a novel latchable phase-change microvalve, which exploits changes in abilities to maintain a solid shape (rather than changes in volume) of paraffin during its solid–liquid–solid phase transitions, and does not require continuous consumption of power, offering improved energy efficiency. The results in this paper were partially reported in Ref. [13], in which valve switching is initiated by melting of paraffin under heating. While valve switching from open to closed states requires an additional pneumatic pressure, switching in the reverse direction can be accomplished by melting of paraffin only, with the membrane's elastic force causing the valve to return to the open state. The switched state can be subsequently latched passively without further energy consumption after paraffin solidifies. Thus, energy consumption is only required during the valve switching. A major difference of our valve from the two existing latchable valves [11,12] is that our microvalve features a thin compliant membrane separating a fluid channel and a paraffin actuation chamber. Compared with the existing paraffin-based latchable microvalves [11,12], our device innovatively combines the solid–liquid–solid phase transitions with compliant sealing of paraffin, and is thus reusable and free of fluid contamination. In addition, the thin compliant membrane separates the paraffin and fluid, thus preventing melted wax from being driven to the fluid by large pressure. This also makes the valve more robust and flexible than existing latchable phase-change valves [11,12] by allowing large ranges of initiation pressure generated from various actuation schemes. Although the concept of our latchable valve using a compliant membrane to separate fluid from paraffin wax is demonstrated by a microvalve using external heater and external pneumatic pressure source, the valve's flow switching time response, total energy consumption, and size, can be drastically reduced by using integrated microheaters and on-chip pressure generation sources.

2. Design

Although our latchable phase-change valve concept of using a compliant membrane to separate fluid from paraffin wax can be implemented with various designs, we will focus on a simple valve design as shown in Fig. 1 for proof-of-concept demonstration. The valve design consists of three layers, *i.e.*, a paraffin microchamber layer, a thin fluid channel layer, and a substrate layer in Fig. 2. A microchamber is separated from a microchannel by a thin compliant membrane, which is the ceiling of the fluid channel. A low-meting-point paraffin wax (mp: 44–46 °C) is filled in the microchamber on the top layer as the phase-change material, while fluid flows through the microchannel in a compliant thin middle layer. A flat glass substrate, that is the bottom layer, sits underneath the thin middle layer to form the fluid channel as shown in Fig. 1a. To ensure complete closure of fluid flow, the microchannel is designed with rounded cross-section as shown in Fig. 1b. The fluid channel layer is designed to be fabricated from the elastomeric polymer polydimethylsiloxane (PDMS). Due to the large compliance of PDMS (nominal Young's modulus: 750 kPa [14]), the thin membrane (which is also the ceiling of the fluid channel) can deform easily under pressure, serving as the active valving element to allow the closing and opening of flow.

When the paraffin is heated to melt, a pressure applied at the pneumatic control port of the valve can be transferred through melted paraffin wax on the thin compliant membrane separating the paraffin and fluid flow, which is also the ceiling of the fluid channel. The thin membrane will then deform, resulting in a narrowed fluid passage. When certain pressure (*e.g.*, shut-off pressure) is reached, the fluid flow can be shut off completely due to the rounded channel cross-section (Fig. 1b [15]). In its deformed state, the membrane generates an elastic force that tends to cause the membrane to recover to the undeformed posi-

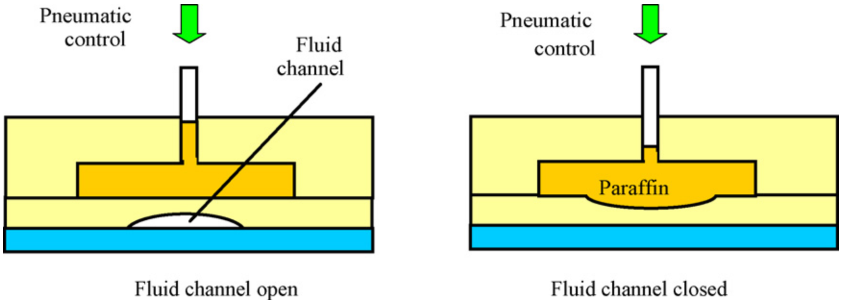


Fig. 2. Operating principle of the latchable phase-change valve. The open-to-closed valve switching can be realized by melting paraffin, applying pressure to shut off flow, and finally letting paraffin cool down. The closed-to-open switching is similar except that the compliant membrane recovers passively to the open state under elastic force when paraffin is melted.

tion. Therefore, when the paraffin is heated to melt, the channel will recover to its original open state passively without the action of an external pressure.

Fig. 2 illustrates the operation of the latchable phase-changing valve. To shut off the flow, the low-melting-point paraffin is first heated to melt. An external pneumatic pressure is then applied to the molten paraffin, forcing the compliant membrane to deform and close the fluid channels. After the paraffin cools down and solidifies, the pneumatic pressure can be removed. The device is thus in a latched, closed state without further consumption of energy. Reopening the valve is even simpler. The paraffin only needs to be heated to melt. The compliant membrane will recover to its originally open position automatically by the elastic force of the membrane, without needs for pneumatic pressure. After the paraffin cools down and solidifies, the device stays in latched, open state. Note that energy consumption is required only for the switching of the valve states and no energy is required after the switching is complete. Additionally, pneumatic pressure is only required for valve switching from open to closed state, and is not required for valve switching in the reverse direction. The valve's minimized energy consumption as well as the reduced risk of fluid contamination by paraffin wax make the valve potentially suitable for many practical applications, where minimal energy is required such as portable point-of-care biomedical devices and lab-on-a-chip systems. The principle of this latchable phase-change valve is also applicable to conventional large-scale actuation application where low power consumption is desired.

In the prototype design, an external heater is used to heat and melt the paraffin wax, and an external pneumatic pressure source is used for the actuation. As will be discussed below, the actuation energy consumption and time response of the device can be significantly reduced by using integrated heater and on-chip pressure generation sources.

3. Fabrication

Standard multilayer soft lithography techniques have been used to fabricate the proof-of-concept device from PDMS [15,16]. The fabrication process is illustrated in Fig. 3. PDMS (polydimethylsiloxane) was used to fabricate the wax chamber and fluid channel layers. A flat glass plate was finally bonded to the two PDMS layers to form the device.

Master molds for the chamber layer were made in two steps. First, a 350 μm-thick negative tone photoresist (SU-8 2100, MicroChem Corp., Newton, MA) was spin-coated on a silicon wafer. Then, the SU-8 photoresist was patterned with a high-resolution (3600 dpi) transparency photo mask. The PDMS prepolymer (5:1 part A:B, Sylgard 184 Silicone Elastomer Kit, Dow Corning) was then cast against the master and cured at 65 °C for 3 h in an oven. The wax chamber layer was relatively thick (∼5.5 mm) for mechanical stability. Master molds for the fluid channel layer were made by spin-coating a 9 μm-thick positive photoresist (Shipley SPR 220-7, Marlborough, MA) on silicon wafer and patterning with another transparency mask. The channels on the photoresist were rounded at 120 °C for 20 min to create a geometry that allows full valve closure. The maximum channel height was measured to be about 12 μm after rounding. The master mold was then spin-coated with 20:1 part A:B Sylgard 184 at 2500 rpm for 1 min. The resulting PDMS layer was about 30 μm thick, which was then cured at 80 °C for 30 min on a hot plate.

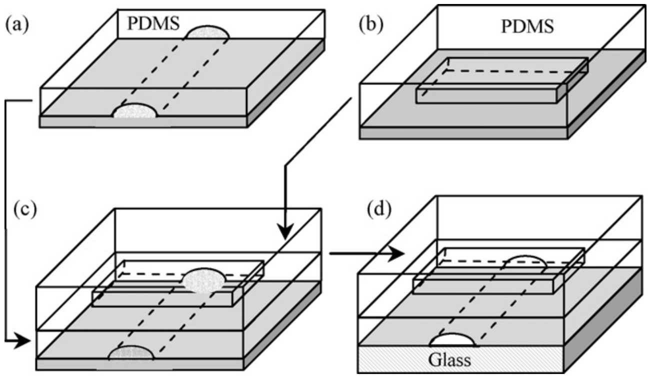


Fig. 3. Fabrication of the phase-change paraffin valve. (a) Spin-coating of PMDS on a mold containing reverse channel features. (b) Replica molding PDMS on a mold with reverse chamber features. (c) Bonding the PDMS layers together. (d) Bonding the bonded PDMS layers to a flat glass substrate, and injecting paraffin to the chamber.

Next, pneumatic pressure control holes were punched through the thick PDMS layer that had been released from the mold. After that, the thick chamber layer was sealed, with the chamber side facing down, on the thin fluid layer (before being retrieved from the SPR-220 mold), aligned with the fluid channel. Bonding between the assembled layers was accomplished by curing the device for an additional 30 min at 80 °C. The resulting two-layer silicone structure was then peeled off from the SPR-220 master mold, punched with the fluid inlet/outlet holes, and diced to the size (18 mm long, 9 mm wide and 5.5 mm thick). The diced silicone unit was next mounted on a flat slide glass (23 mm long, 12 mm wide and 0.5 mm thick) that was spin-coated with 5:1 part A:B Sylgard at 5000 rpm and cured at 80 °C for 30 min. The entire device was then baked at 80 °C on a hot plate overnight for improved bonding strength.

Finally, the pneumatic control ports and fluid interconnections were made using epoxy glue, and a low-melting-point paraffin wax (Sigma–Aldrich, mp: 44–46 °C) was manually injected into the chamber in a hot water bath (65 °C) using a syringe. Two pressure control ports were connected to the paraffin chamber. One port was used to introduce the melted paraffin wax into the chamber, while the other allowed air to be vented out during paraffin injection. After the chamber was completely filled with the paraffin, the second port was permanently sealed. Only the first port was used for pressure control during valve switching. Fig. 4 shows images of a fabricated device before and after packaging. Micrographs of a device are shown in Fig. 5 with three parallel fluid channels and a channel-like paraffin chamber before and after paraffin filling.

4. Results and discussion

This section presents the testing results from the proof-of-concept latchable phase-change microvalves. The devices were

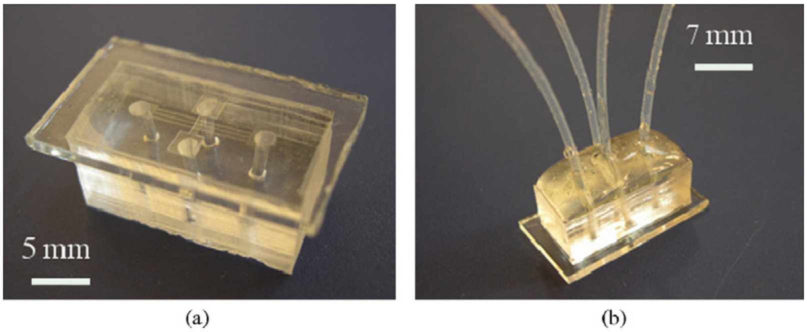


Fig. 4. Images of a fabricated valve: (a) before and (b) after packaging.

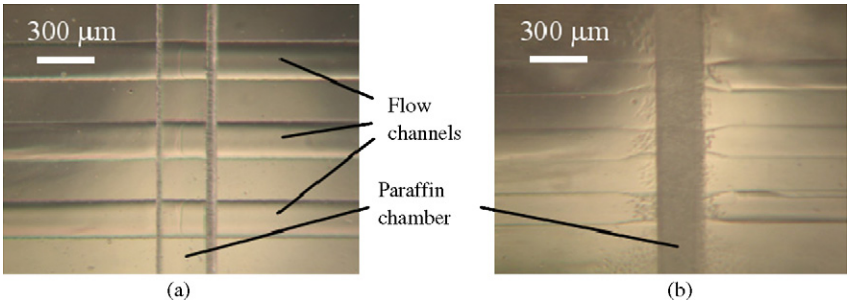


Fig. 5. Micrographs of a device with three parallel flow channels and a channel-like paraffin chamber: (a) before and (b) after paraffin filling. (Individual flow channel: 200 μm wide, 12 μm high and 6 mm long; wax chamber: 300 μm wide, 350 μm high and 2 mm long.)

tested using the setup shown in Fig. 6, in which deionized (DI) water was used as the testing fluid, and pressurized argon was used to apply pressure on the melted paraffin for valve switching from open to closed state. The pressure at the outlet of the device is estimated to be approximately atmospheric. A digital flowmeter (Alicat Scientific: L-1CCM-D, Tucson, AZ) with a resolution of 1 μl/min was used to measure flow rates. To demonstrate the actuation concept of the latchable microvalve, an ultrasonic cleaner with precise water temperature control capabilities (Branson: 1519R-DTH, Branson Ultrasonic Corp., Danbury, CT) was used as the isothermal water bath for heating and melting of paraffin wax. Isothermal water bath was used as it might be the simplest heating method that can precisely control the bulk heating temperature. In addition, using water bath provide a simpler convective heat transfer boundary condition (with constant convective heat transfer coefficient) in the future device operation simulation. Alternatively, an external strip heater could be used to heat the paraffin wax, which would more closely resemble a real lab-on-a-chip device with an integrated heater. Here, we specifically report the testing results for the fabricated device shown in Fig. 5, which has three parallel flow channels (200 μm wide, 12 μm high and 6 mm long) and a channel-like paraffin chamber (300 μm wide, 350 μm high and 2 mm long; total volume about 0.21 nl) that is perpendicular to the flow channels.

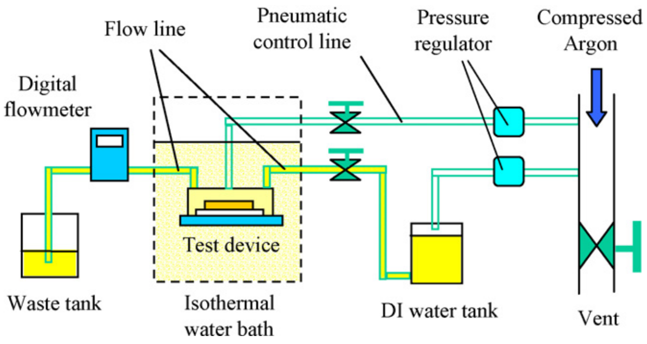


Fig. 6. Schematic of the experimental setup for testing the phase-change microvalve.

To date, most active valves require continuous energy consumption, which is typically on the order of tens of milliwatts to several watts depending on the actuation mechanism, functional material, and actuator sizes. The total power consumption level of portable medical devices largely depends on its functionality and complexity, *e.g.*, the number and type of active devices. It is highly desirable to minimize such power consumption for the practical microfluidic and lab-on-a-chip devices. We have estimated the thermal energy required for the microvalve to switch between the open and closed states using external water bath as heater and un-optimized wax chamber design. Using typical material properties (PDMS: density 970 kg/m^3 and specific heat 1.46 kJ/kg K [17]; glass: density 2500 kg/m^3 and specific heat 0.84 kJ/kg K [17]; paraffin: density 780 kg/m^3, specific heat 2.374 kJ/kg K and latent heat 195 kJ/kg [18]), the thermal energy required to heat the entire device from 25 to 63 °C is calculated to be about 58.9 J. While this appears to be large compared with the energy consumption of some existing phase-change latchable valves (*e.g.*, 1.5–4 J of Ref. [11] and 5 J of Ref. [12]), it is important that our heating method was not optimized. Indeed, the energy required just to melt the paraffin was only about 40 mJ. It is anticipated that a total energy on this order should be sufficient for an optimally improved device with on-chip heating and

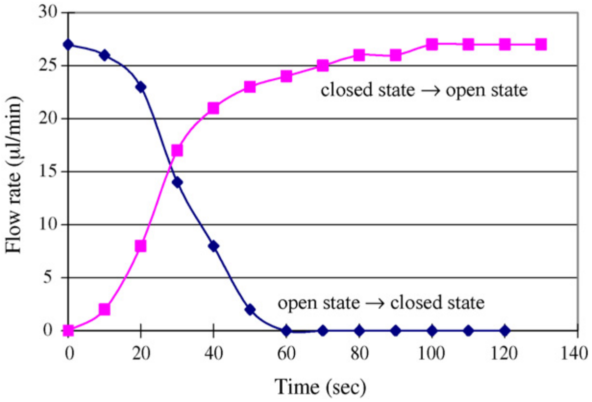


Fig. 7. Time course of valve switching between open and closed states with the driving pressure maintained at 40 kPa. At time zero, the device was immersed in 63 °C water. For valve switching from open to closed state, a pneumatic pressure of 70 kPa was also applied.

maximal thermal isolation. The development of such a device is a subject of future research.

Fig. 7 shows the tested time response of the microvalve switching between the open and closed states. The flow rate was measured to be about 27 μl/min when the valve was latched open. The valve was considered closed when the flow rate was under the detection limit of the flow meter (less than 1 μl/min). In the experiment, heating of the paraffin wax was achieved by immersing the device in an isothermal water bath (63 °C). The cooling-down and solidification of the wax in the device was passive at room temperature (25 °C). The driving pressure at the inlet of the microchannel was kept at 40 kPa in the experiment.

The time course of the valve switching from open to closed state can be seen in Fig. 7. The data points have been connected by solid lines to guide the eye here and in Fig. 8. At time zero, the open microvalve was taken from room-temperature ambient air and immersed in the water bath. In the meantime, a 70 kPa pneumatic control pressure was applied. It can be observed that the transition time was about 60 s for the valve to switch from open to closed state. Fig. 7 also shows the time course of the valve switching from closed to open state. At time zero, the originally closed microvalve was immersed in the water bath from ambient air. It was observed that the transition time was about 100 s to switch from closed to open state under the condition. Note that the shut-off time (60 s) was shorter than the opening time (100 s), which is possibly because the pneumatic pressure during valve shutting off was stronger than the passive membrane elastic force during valve opening. The device's relatively slow heating time response was mainly due to heating of the entire device in water bath, and can be drastically improved using on-chip integrated heaters.

We also measured time response for the cooling-down process of the paraffin wax associated with valve switching. It took roughly 3–5 min for the melted wax to cool down to room temperature in ambient air when a metal heat sink was placed underneath the valve. The exact response time for the cooling down process depends on the device's heat transfer characteristics. It was noticed that using a heat sink, *e.g.*, a metal plate placed underneath the bottom glass substrate of the valve could significantly reduce the cooling response time. Note that there is a trade-off between heating and cooling times. A longer cooling time would only imply that the holding pressure has to be maintained longer, and in general would not limit the utility of the device for a given application. Heating times are most important from a practical point of view, as they directly determine the device's switching time. This switching time was not optimized for the current proof-of-concept device, but can be drastically improved by including on-chip heaters in the design.

The latching capability of the device was also tested. Fig. 8 shows the measured flow rate versus pressure for the valve in either latched open or latched closed state under increasing driving pressures. As shown in Fig. 8, when the valve was latched open, the flow rate was largely linear with driving pressure. This indicates that the channel cross-section and flow resistance were largely constant under increasing driving pressures. When the valve was latched closed, the flow rate was zero within flowmeter detection limits under driving pressures up to 35 kPa. Leakage started to increase when the driving pressure further increased as the strength of the wax became insufficient to hold the valve in closed state. The two curves in Fig. 8 approach each other more and more closely with increasing driving pressure. Specifically, at a given driving pressure higher than 70 kPa, the flow rates differ by only about 3 μl/min.

Fig. 8 shows that when the inlet flow pressure is higher than 35 kPa, the valve loses its capability to remain closed passively. Experiments also showed that the fluid channel can be completely sealed by the thin membrane at about 28 kPa. In the closed state, the thin membrane is subject to an elastic restoring pressure (∼28 kPa), independent of the driving pressure at the channel inlet. This critical latching pressure, *i.e.*, 35 kPa for this specific device, depends on several factors, such as stiffness of the paraffin wax, thickness and stiffness of the thin PDMS membrane, height, shape and rigidity of the chamber. Precise estimation of this latching pressure will be considered in future work.

We believe that the originally latched closed microvalve start to leak at high inlet fluid pressure mainly due to the paraffin wax's low mechanical strength to retain its solid shape. This is because paraffin has a relatively low melting point and a low Young's

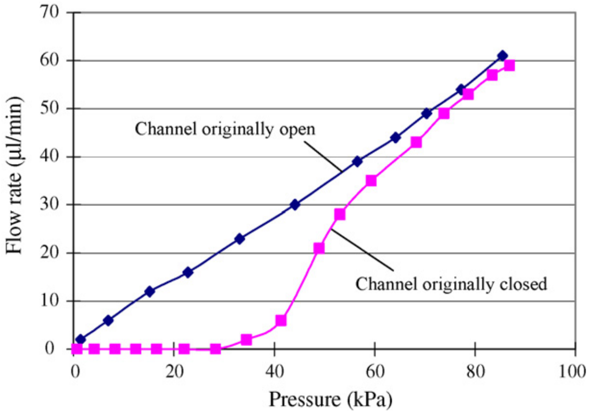
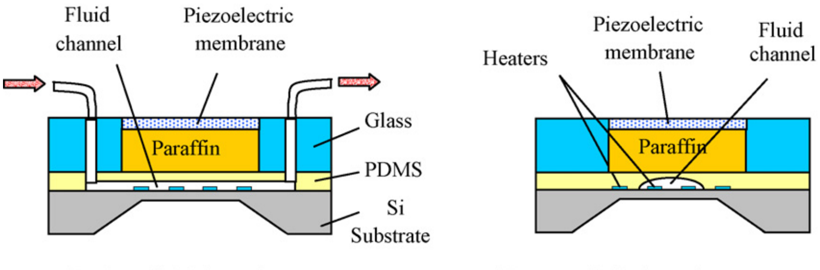


Fig. 8. Latching characteristics of the microvalve at room temperature. The fluid channels were originally either open or closed, and were subject to an increasing driving pressure.



(a) Along fluid channel (b) Across fluid channel

Fig. 9. Schematic of a conceptual latchable valve design that features integrated microheaters and an on-chip piezoelectric pressure generation source.

modulus. Thus, using a more compliant membrane (which has smaller elastic restoring pressure) and a deeper paraffin chamber could increase this critical latching pressure. In addition, how easily the wax can change its shape at high inlet flow pressures, and thus the valve's latching capability, is related to the rigidity of the paraffin chamber. Using a rigid wax chamber, *i.e.*, glass, instead of deformable PDMS, could also significantly improve this critical latching pressure. While for simplicity in the current device, PDMS was used to fabricate the paraffin wax chamber, more rigid materials (*e.g.*, glass) may be used in future designs. Using a paraffin wax of higher melting point could also increase the latching capability at the expense of larger heating power and higher operating temperature.

The experimental results above have demonstrated that the concept of this paraffin-based microvalve can be used to realize latchable flow control. While microheaters and a freestanding channel and chamber design can be used to improve the device's switching time response and latching capability, it will also be highly desirable to integrate on-chip pressure sources for practical lab-on-a-chip applications. Various pressure generation schemes, *i.e.*, piezoelectric, electromagnetic and thermopneumatic actuation, can possibly be used for this purpose. Of course, additional power consumption will in general be necessary for the on-chip pressure generation source.

Fig. 9 shows the schematic of such a conceptual design, which contains integrated microheaters and an on-chip piezoelectric pressure generation source. In the design, a thin PDMS layer containing a compliant fluid channel similar to that in Figs. 1–3, is sandwiched between a rigid top paraffin chamber layer and a bottom substrate. An integrated piezoelectric membrane that forms the ceiling of the wax chamber is used to generate on-chip actuation pressure. Although piezoelectric actuation typically has relative small displacement, the stroke volume change generated by piezoelectrically actuated membrane deflection may still be sufficiently large to shut off the fluid flow, since the total volume change of the fluid channel associated with the switching process is much smaller than the total volume of the paraffin wax. After the chamber is filled with paraffin wax, it is then completely sealed. Microheaters are patterned directly on a free-standing silicon diaphragm that is back-side etched from the bottom silicon substrate. The thin silicon substrate allows a smaller thermal mass, thus a short heating time response. Fabrication and testing of such device will be pursued in the near future.

To further shorten the heating time response of the latchable valve, the microheaters could alternatively be fabricated on the thin compliant PDMS layer, so that it directly contacts the paraffin wax. Note that one needs to develop fabrication process such that there is adequate adhesion between metal heaters and PDMS. Conceptually, it is also possible to design the microheaters embedded or suspended inside the paraffin chambers. However, the fabrication of such microheaters would be rather challenging.

5. Conclusions

A novel latchable phase-change valve has been presented in this paper. The valve consists of three layers, featuring a paraffin-filled microchamber separated from a fluid channel by a thin compliant membrane. The valve exploits a low-melting-point paraffin wax as the phase-changing material to shut off and reopen fluid flow through a deformable microchannel. Valve switching is initiated by melting of paraffin through heating. We have successfully demonstrated a proof-of-concept phase-change microvalve, which was fabricated from PDMS using multilayer soft lithography techniques. Experimental data has shown that the device has response times of 60 s for closing and 100 s for opening; the valve in the latched closed state can passively withstand an inlet pressure up to 35 kPa without significant leakage. While these valve switching times are not optimized for the current proof-of-concept device, they can be drastically improved by including on-chip heaters in the design.

There are several distinct advantages to the latchable phase-change valve. First, during operation, energy is required only to initiate valve switching, and the switched state can be maintained passively without further consumption of energy. In addition, while pneumatic pressure is required for switching the valve from open to closed state, no external pressure is needed for valve switching from closed to open state as the compliant membrane's spring force allows the fluid channel to passively return to its natural open state. Second, the membrane separates the fluid from the paraffin wax, eliminating any potential contamination of the fluid by the wax. Finally, due to the improved sealing offered by the compliant membrane, large ranges of initiation pressure, generated from various actuation schemes, can be used for valve actuation during switching from open to closed state. Thus, the valve is more robust and flexible than the existing latchable phase-change valves. Because of these advantages, the valve is attractive for flow control in applications, such as portable lab-on-a-chip systems, where minimal energy consumption is desired. It is believed that the switching time response, energy consumption and size of the latchable valve can be further reduced by using integrated microheaters and an on-chip piezoelectric pressure generation source.

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