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Flow restrictor silicon membrane microvalve actuated by optically controlled paraffin phase transition

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Abstract

Restrictor valves allow proportional control of fluid flow but are rarely integrated in microfluidic systems. In this study, an optically actuated silicon membrane restrictor microvalve is demonstrated. Its actuation is based on the phase transition of paraffin, using a paraffin wax mixed with a suitable concentration of optically absorbing nanographite particles. Backing up the membrane with oil (the melted paraffin) allows for a compliant yet strong contact to the valve seat, which enables handling of high pressures. At flow rates up to 30 μL min^{-1} and at a pressure of 2 bars, the valve can successfully be closed and control the flow level by restriction. The use of this paraffin composite as an adhesive layer sandwiched between the silicon valve and glass eases fabrication. This type of restrictor valve is best suited for high pressure, low volume flow silicon-based nanofluidic systems.

Keywords: microfluidic, silicon-on-insulator, paraffin, wafer bonding.

(Some figures may appear in colour only in the online journal)

1. Introduction

Microfluidic actuators and valves have been studied as a means to regulate and control flow in miniaturized artificial environments e.g. portable lab-on-chip or environmental analysis devices [1,2]. Pressure-driven fluid handling allows for more robust active valving than capillary or electrodynamically-driven valving. A good introduction to various kinds of microvalves is given in the review by Oh and Ahn [2].

One particular actuation method is to drive and mechanically support a membrane valve with the phase change material paraffin. This is justified by its large expansion at the phase transition i.e. it expands some 10% in volume when the wax is melting to oil, and by the fact that the low compressibility of paraffin oil is close to that of hydraulic oil. Hence, melting paraffin exerts very high force and large displacement, which can be used to generate or act against high pressures. The system is rather slow and not power-efficient, since it is controlled by thermal actuation, and cools over a steep thermal gradient, with paraffin having both low thermal conductivity and high latent heat. More details on paraffin as an actuator material and different solutions of paraffin actuated microvalves are discussed in [3].

In this demonstration, the actuating material is a paraffin wax mixed with a suitable concentration of optically absorbing nanographite particles. Similar composite materials have been used to increase thermal conduction and to allow other actuation mechanisms than direct thermal actuation. For example, other types of miniaturized valves, where a composite of paraffin and iron oxide nanoparticles (in ferrofluid) has been used as a melting plug actuated wirelessly by absorbing optical power [4], or magnetically positioned after being melted by a microheater [5].

Most microvalves are suited only for discreet control, opening or closing the flow. However, in fluidic handling it is often preferred to control the magnitude of flow by restricting its path. Hence, in this work a restrictor membrane valve

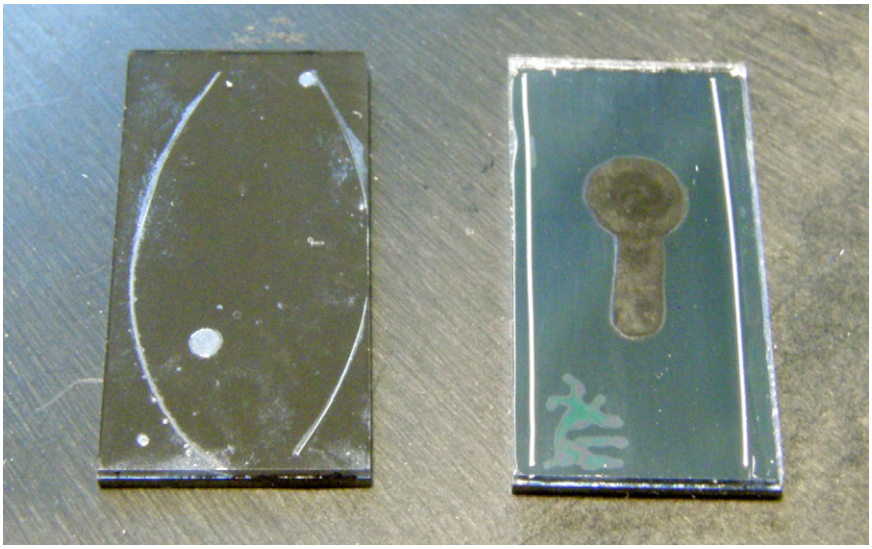

Figure 1. Assembled membrane valves photographed from the glass side. Design with edgeless paraffin layer (left) and with paraffin enclosed in acrylate (right).

enables integration of optically operated microfluidic restrictors in silicon-based microfluidics. This omits the need for electrical interconnects or global heating, facilitating the interfacing for actuation of disposable chips in analytical instruments. Wireless actuation without electromagnetic interference for microfluidics is a new concept and, to the best of our knowledge, this is the first time it is used for hydraulic silicon membrane valves as well as for restrictor valves, enabling both high-end remotely controlled applications for e.g. space but also disposable cartridges for environmental sampling or healthcare.

2. Experimental

2.1. Fabrication of the Si-SOI chip

A standard 150 mm Si wafer was first oxidized to 200 nm depth for subsequent deep reactive ion etching (DRIE) of the 1–3 mm wide valving cavity (see figure 2). After DRIE to 6–14 μm depth, 2.8 μm thick chemical vapour deposited (CVD) SiO_2 was patterned on the back side of the wafer to act as the mask for subsequent opening of holes through 600 μm Si wafer by DRIE. Two different diameters for the holes were used: 100 and 250 μm. Having the thermal oxide under the CVD oxide and removing them both in concentrated hydrofluoric acid (HF) left the silicon surface pristine for subsequent fusion bonding.

Three different methods were studied for bonding the Si cavities and the silicon-on-insulator (SOI) wafers containing 400 μm thick Si handle wafer, buried oxide of 2 μm and the SOI layer of 8 μm, the latter being bonded onto the cavities. Among these methods e.g. high temperature fusion bonding, Al_2O_3-assisted bonding and low temperature plasma-assisted wafer fusion bonding, the latter was used for its superb yield with oxide thickness of 750 nm and nitrogen plasma activation for 30 s (230 W, 150 mTorr, 30 sccm N_2) in a reactive ion etcher (RIE), followed by annealing at 300 °C for 1 h [6].

As the last steps, to release the strained SOI membranes the 400 μm handle wafer was removed with the buried oxide by combining grinding and DRIE.

2.2. Assembly of the restrictor valve

The individual silicon structures were diced to 10×20 mm Si-SOI chips which were then paired with glass plates of the same dimensions. The assembled restrictor valves were of two different designs:

(1) assembly with an edgeless paraffin layer that in principle provides adhesive coupling at the same time as a smaller part can be used for actuation;
(2) 2 mg of paraffin enclosed in UV-curable acrylate adhesive (DSM DeSolite 3471), forming a traditional enclosed actuation body on the membrane.

For the actuation material, we chose paraffin with a melting range from 45 to 49 °C, and added graphite 5% by weight. To assemble design (1), the Si chip was placed on a hotplate and an excessive amount of paraffin on the chip, together with 200 μm thick wires acting as spacers. After heating the paraffin to about 47 °C, the glass lid was placed on top of it and the system was allowed to settle by gravity and solidify by cooling. In design (2), spacer wires were first fixed to the Si chip with UV-curing acrylate. The fixed wires were then sanded with a fine sharpening tool to remove excess acrylate and to flatten possible bends in the wires. After removing sanding debris, a piece of paraffin, manually formed under microscope, was placed onto the chip to cover the membrane. The chip was then heated slightly so that the paraffin stuck to the chip. A glass lid was placed on top of the assembly and the paraffin heated through the glass by exposing it to a hot soldering iron. Once molten paraffin had visibly wetted

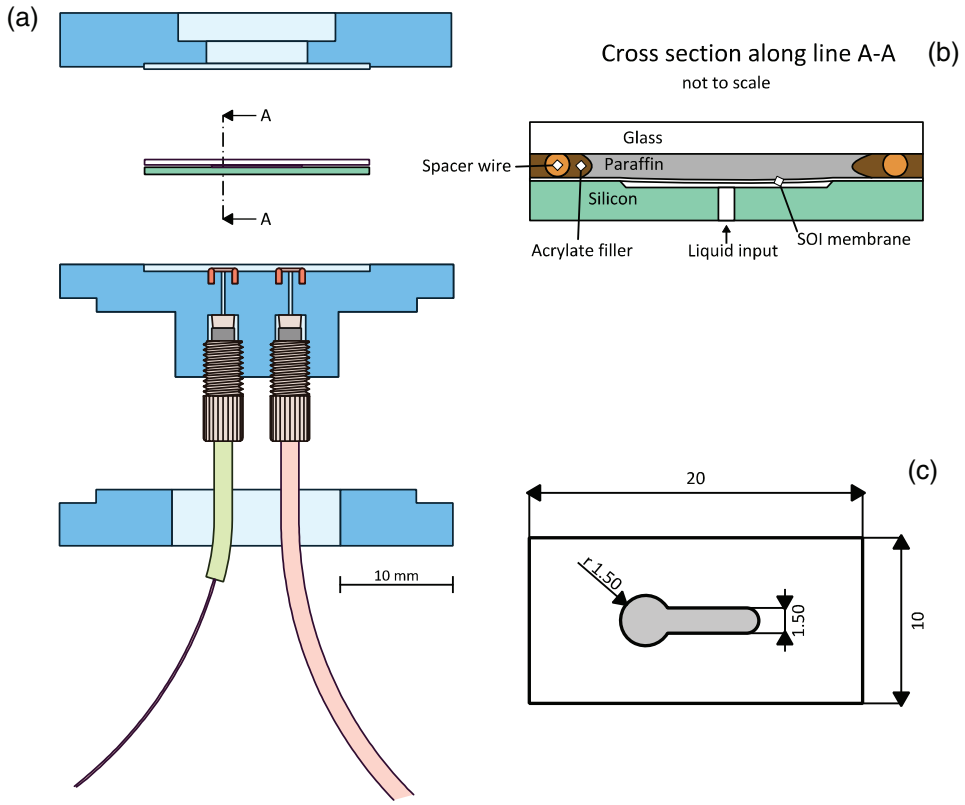

Figure 2. (*a*) Cross-section of the microvalve restrictor setup with chip. (*b*) The chip cross section is orthogonal to the clamp, showing only the inlet with the membrane and the epoxy-enclosed paraffin body. (*c*) Location and dimensions of the membrane (grey) on the chip.

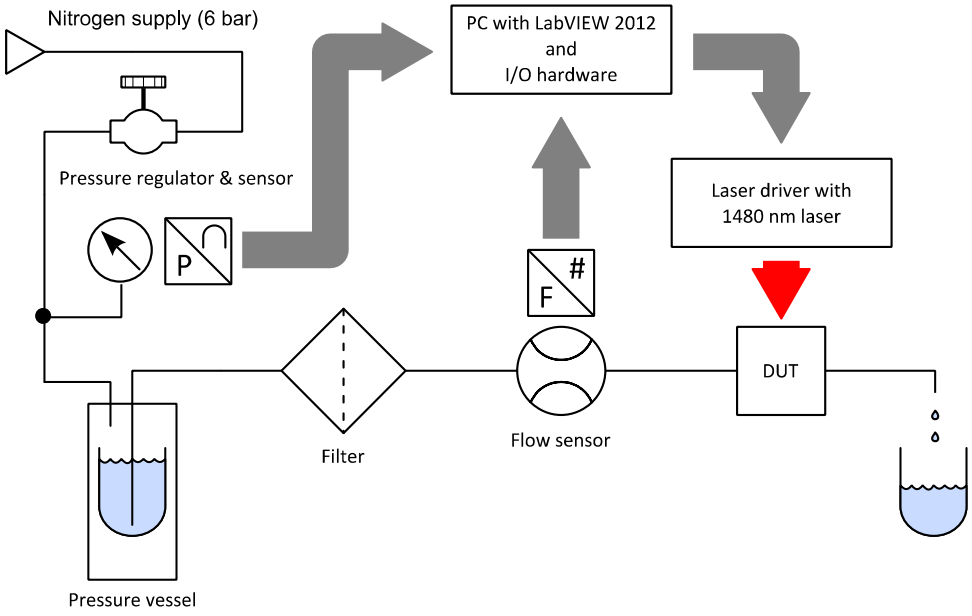

Figure 3. Sketch of the setup for testing the restrictor valves.

the underside of the glass, the soldering iron was removed. Finally, adhesive was introduced from the sides into the gap between the Si and glass. It was allowed to fill the gap by capillary action and was then cured in UV light.

The results of these two different procedures can be seen in the photograph in figure 1. There is also a sketch of the chip cross section of the enclosed structure in figure 2. In the other case, paraffin would cover the whole volume sandwiched between the silicon valve and the glass.

2.3. Restrictor valve evaluation

The glass-paraffin-Si block was assembled and clamped in a home-made polycarbonate chip holder with fluidic

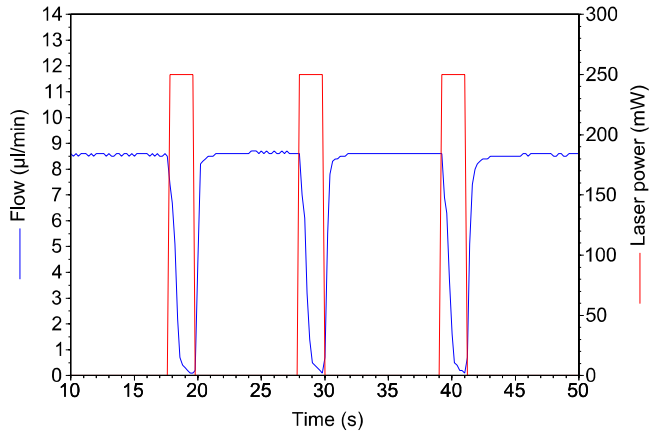

Figure 4. Switching an optical paraffin-mediated flow restrictor.

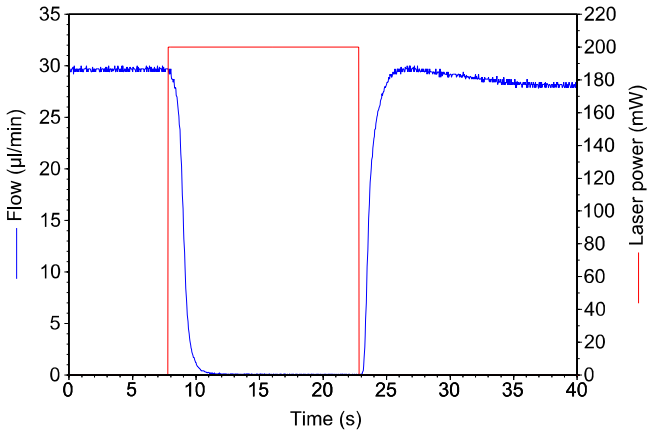

Figure 5. Complete cycle of the valve.

connections. The holder of the restrictor valve is sketched in figure 2. Threaded fittings made of poly-etheretherketone (PEEK) were used in the holder, with silicone gaskets sealing the fluidic interface to the Si through-holes.

The test setup for the optically actuated restrictor valves is depicted in figure 3. A laptop PC running NI LabVIEW 2012 connected to a NI DAQCard 6024E was used to record measurement data and modulate the laser. For optical actuation, we used a laser whose size and power intensity were suitable for microfluidics without the need for complex optics. A semiconductor fibre laser (Alcatel A1948PLI) providing up to 250 mW at 1480 nm was set up to heat the paraffin through the glass. The laser was driven by an ILX Lightwave LDC-3900 controller and its optical power modulated with an analogue signal from the PC. Distance between fibre and lid varied from 0.5 to 10 mm in our experiments, exposing an area 3 mm in diameter at most. Throughout the experiments, the room temperature was 21 °C.

The valves were tested with DI water fed from a vessel pressurized with nitrogen. Gas pressure was held constant with a mechanical regulator (Festo LRMA-QS-6), whose output was monitored with a pressure sensor (Honeywell 40PC150G) providing an analogue voltage signal to the PC. Drive pressures of up to 3 bar were used in the experiments. Flow reading was obtained by using a Sensirion SLG-1430-048 flow sensor. To prevent solid particles from entering the sensor or valve device, a PEEK filter with 2 μm pores was placed upstream of the sensor.

3. Results and discussion

3.1. Speed and reproducibility of operation

By switching the laser beam on and off a repeatable operation was observed, as shown in figure 4. With initial flow of 8.5 μL min^{-1}, laser pulses of 250 mW and 2.0 s restricted flow to less than 10% of the original, recovery being faster than closure. Longer laser pulses resulted in completely cutting off flow even at high flow rates, as can be seen in figure 5. The time to reach 10% of the maximum flow rate was 2.0 s. Recovery to 90% flow rate occurred in 1.7 s, see figure 5.

From the paraffin volume and its specific heat [7], it can be calculated that roughly 0.3 J is required for the epoxy-confined 2 mg of paraffin contained in 0.2×Ø3 mm to heat up from room temperature to the steepest slope in density (50 °C), consisting mostly of melted paraffin. Of course, more energy is needed to be absorbed by the paraffin since thermal transport will only allow for a smaller portion of energy to be stored in it. To better understand the thermal transport and time constants, a simple system was studied with IR thermography (Fluke Ti32). This was made without the PEEK clamping and with the design (1) with paraffin spread between lump chips.

From the thermography it was observed that both the top glass plate and the edge of the Si chip start to heat up almost immediately. This thermal leakage to Si presumably starts at the edges of the channel when the partially molten and hence more heat-conductive paraffin reaches there, making any further increase in the paraffin's temperature much slower due to the high thermal capacity of the Si (about 200 mJ K^{-1}). Additionally the convective loss by the flowing water was considered significant. After about 15–20 s, the temperatures of the Si, paraffin and bottom of the glass increase in concert and most of the thermal expansion occur, see figure 6. However, at the same time, the melt is expanding out of the gap between Si and glass making repeatable operation impossible once the valve is fully closed.

In the second design, lateral confinement of paraffin was used along with a flow rate feedback to tune the laser input. The result is presented in figure 7. This valve has a much slower response than the previous although less paraffin is present. A possible reason for the slow closing may be a gas bubble between the paraffin and epoxy, while for the slow opening a solid layer of paraffin formed at the water-cooled SOI membrane should be the main limitation.

The steady-state flow rate measured for a chip with 14 μm deep channel and 1.8 bars is shown in figure 8. After stabilizing for 3 min at each point, two flow restrictors showed plateaus in both low and high power end and close-to-linear response between 50 and 100 mW. The two different devices were measured subsequently with random power order, both having two repeat points for checking the possible presence of any drift. As a first approximation this can be explained by the direct linear relationship between input energy and volume of paraffin when melting. Since the flow restrictor is defined

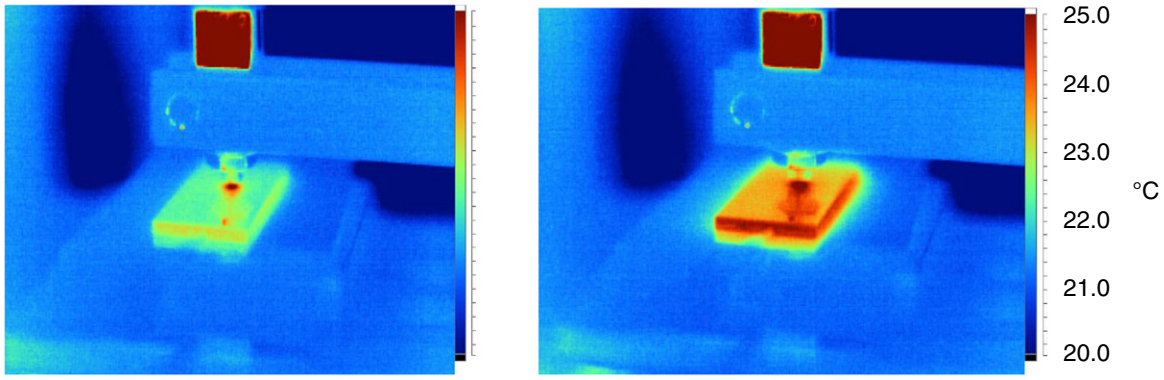

Figure 6. Left: thermal map 5 s after switching the laser on. Centre of the beam is 26.2 °C, surrounding glass plate is 21.2 °C, top of the cover glass is 22.3 °C with the exception of a small area in front (maybe a reflection from the fibre mount) and the edge of the chip stack is 23.1 °C. Right: Thermal map 24 s after switching the laser on. Bottom plate temperature is still unchanged, beam maximum is 26.8 °C, but the top glass is now 23.5 °C (reflection image still heating up) and the edge is 24.6 °C.

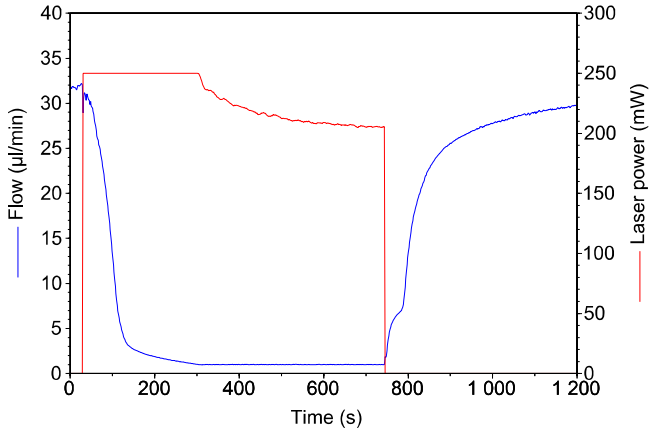

Figure 7. Flow restrictor with laterally confined paraffin volume and feedback control. A setpoint of 1.0 μL min^{-1} was used for the controller.

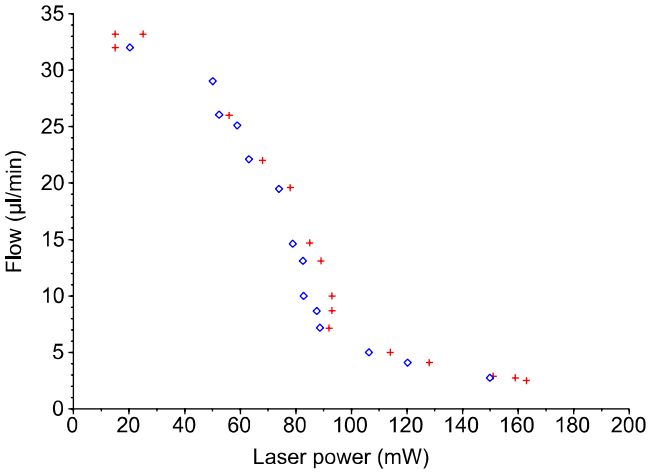

Figure 8. Flow restrictor with laterally confined paraffin volume.

by the circumference of the orifice and the height between it and the membrane, the flow is controlled by the height, which decreases linearly in relation to increasing volume of liquid paraffin. The weak response of the flow below 50 mW is presumably caused by thermal loss from softened and hence more thermally conductive paraffin layer to glass. Another constraint is probably the steep thermal gradient formed between water and glass at the steady-state. On the other hand, closing the valve requires power in excess of the loss to water and glass, but also to silicon at the channel's edges. Maintaining the full height of the melt zone demands a significantly higher power than just having the glass and water as the thermal sinks.

An important limitation with thermally actuated systems like this is that they need a feedback control. The ambient temperature affects how much power is needed to reach the right actuation. Power demand is also partly determined by actuation history, as thermal gradients expand or contract following every step change in set-point (power decreases with time in figure 7). This is more important for a restrictor valve than for a discreet on/off valve that can overcompensate for thermal variations. In this case we use a flow sensor, but it is also possible to integrate temperature control, as was recently made for a paraffin actuated micropump [8].

4. Conclusion

From the experiments, we conclude that the first restrictor membrane valve with optically actuated paraffin has been demonstrated. The use of paraffin as an adhesive sandwiched between the glass and silicon chips eases fabrication. However, the system is not optimized. It is clear that if paraffin is spread all over the interface between the silicon and glass chips, the optical power is not sufficient as the high thermal conductivity of silicon transfers thermal energy to a wider area of paraffin consuming the energy with its high latent heat. Thicker paraffin allows for a thermal insulation between silicon and the melt but the resulting viscoelastic wax layer will then make the system slow and support the membrane so that it needs a higher force to retake its original position after being actuated. Also, with a high flow of water, the major thermal transport will be through convection, and thermal insulation will be even more important. All in all, this type of optically actuated, paraffin-based membrane valve is best suited for high pressure, low volume flow silicon-based nanofluidic systems.

The optically actuated restrictor microvalve based on phase-transition of paraffin was implemented with silicon membrane,

allowing for parallelisation and integration of other microelectronic and micromachined features in the same device. At flow rate up to 30μL min^{-1} at 2 bar pressure, the valve can successfully be closed and used to control the flow by restricting it. However, given the limitations in its opto-thermal control, this type of restrictor valve is best suited for high pressure, low volume flow silicon-based nanofluidic systems.

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