FABRICATION OF PNEUMATIC MICROVALVES FOR PDMS MICROFLUIDIC DEVICES

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Abstract. *Investigations on microfluidic and lab-on-a-chip (LOC) technology are originating a great variety of devices for general analysis, such as chemical and biological. In general, the components of microfluidic devices are channels, reservoirs, valves and pumps. In this work, the fabrication process of a pneumatic actuated microvalve will be presented. Manufacturing steps involve the fabrication of control channels and working channels molds using photolithography for further replication using polydimethylsiloxane (PDMS), in a process called soft lithography. The bonding between different PDMS layers is obtained via oxygen plasma. A simple, resistant and integrated process of connection with the macro world was also developed. Experiments have demonstrated that the microvalves obtained works satisfactorily.*

Keywords*: Microfabrication, multi-layer soft lithography, microvalves, lab-on-a-chip.*

1. INTRODUCTION

Microfluidics refers to the science and technology of manipulating fluids in networks of channels with dimensions of ~5-500 µm, in order to transport volumes of fluid that vary from microliters to femtoliters (Whitesides and Weibel, 2006). This technology has been applied to micro total analysis system (µ-TAS) in chemical and biological studies. A µ-TAS, sometimes called lab-on-a-chip (LOC), is a device that can perform, when it is totally developed, all functions of large analytical laboratory, since acquisition until detection of distinct samples in small scale (Manz *et al.*, 1990). Miniaturized systems significantly reduce the cycle times, reagent costs and disposal (Yoo *et al.*, 2008). It allows the fabrication of portable devices, which can be used for *in situ* analysis, reducing contaminating problems. Micropumps and microvalves are essential for fluid control in a LOC, because they can control the transportation of fluids or biological entities accurately inside of a LOC.

Most of the methods used in micro total analysis device manufacturing were developed in the 1970s and 1980s in the silicon microelectronic industry. To cover a broader range of microfluidic applications, new materials and microfabrication procedures were tested and added to the existing repertoire (Reyes *et al.*, 2002). Recently, the technique that has most been used in this field is soft lithography. The principle of this technique is to pattern structures on the surface of an elastomeric block. Photoresists can be patterned directly to the elastomeric stamp (Whitesides and Xia, 1998) hence combination of photolithography and soft lithography can be used to fabricate microfluidic devices.

The material most commonly employed in soft lithography is polydimethylsiloxane (PDMS). It presents several attractive properties over silicone, for example, to fabricate microfluidic devices, such as low cost, low Young's modulus, transparence to visible and UV light. Another important characteristic is the facility to bond PDMS layers, favoring multilayer fabrication (Multilayer soft lithography -MSL). On the other hand, PDMS has the disadvantage of being incompatible with many organic solvents, and exposure can lead to adverse effects (including swelling) that are specially pronounced in microscale channels due to the high surface to volume ratio (van Dam, 2005). However, this polymer is also biocompatible, what makes it useful for study biological systems, such as cells and small organisms (Whitesides *et al.*, 2001).

There are several actuation methods for microvalves such as piezoelectric (Roberts *et al.*, 2003), electrostatic (Vandelli *et al.*, 1998), thermopneumatic (Yang *et al.*, 1999), and pneumatic (Chen *et al.*, 2005). Each one has advantages and disadvantages, depending on the desired application.

Mathematical modeling of microvalves and micropumps has been implemented since 1989. The first model for a thermo-pneumatic micropump with bond graphs was made by van der Pol *et al.* (1990). Analytical model were developed by Zengerle and Richter (1994) and after their publications many others work in these field started appear for several kinds of microvalves and micropumps. In particular, for a peristaltic micropump, Goulpeau *et al.* (2005) published recently a model that describes a behavior of that device. Our microvalve was based in a mathematical model developed by Espíndola (2006). This work is an expansion of the Goulpeau model using an electro-mechanical-analogies technique was made, which shows better interaction of the microvalves that composes the whole micropump.

Pneumatic microvalves can be produced on chip based on multilayer soft lithography using PDMS to fabricate microchannels in a cross-configuration separated with a thin film of PDMS. Applying a pressure in one of the channels, the PDMS film is deformed so that the cross section of the adjacent channel is changed, resulting in an effective actuation or valving (Chen *et al.*, 2005).

In this paper, we fabricated and tested valves with pneumatic actuation based on PDMS, using a technique known as multilayer soft lithography that combines soft lithography with the capability to bond multiple patterned layers of PDMS.

2. WORKING PRINCIPLE

Pneumatic microvalves are obtained by a cross configuration of two channels, separated by a thin membrane of PDMS (Fig. 1a). One of the channels contains liquid, what means samples and reagents in a μ-TAS, and it is called working channel or fluid channel. The other channel is called actuation channel, by where a gas is injected in order to increase the pressure inside it (Fig. 1b). If the pressure applied in the actuation channel is larger than the pressure in the working channel, the PDMS membrane will deflect upwards and seals the working channel (Fig. 1c), blocking the liquid flow. The flexibility of PDMS is essential for this upward movement.

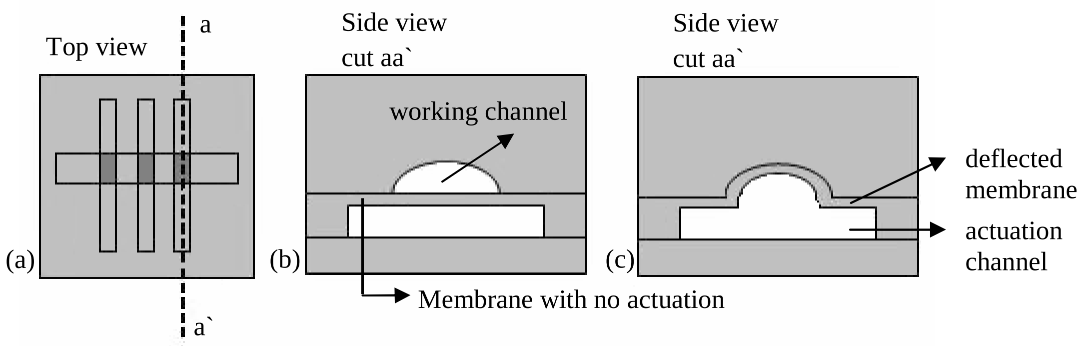

Figure 1. Part (a) and (b) shows an intersection between working channel and actuation channel. These channels are separate by thin and flexible membrane. Cut view of the microvalve is shown in (b) and (c). In (c) a pressure applied in the actuation channel deflects the membrane and blocks the liquid flow rate in the working channel.

The working channel has a rounded profile (Fig. 2) to prevent leaks when the microvalve is closed. With a square contour, for example, the membrane would not seal the corners, allowing leaks. When the pressure is relieved, the membrane returns to its original configuration and opens the working channel.

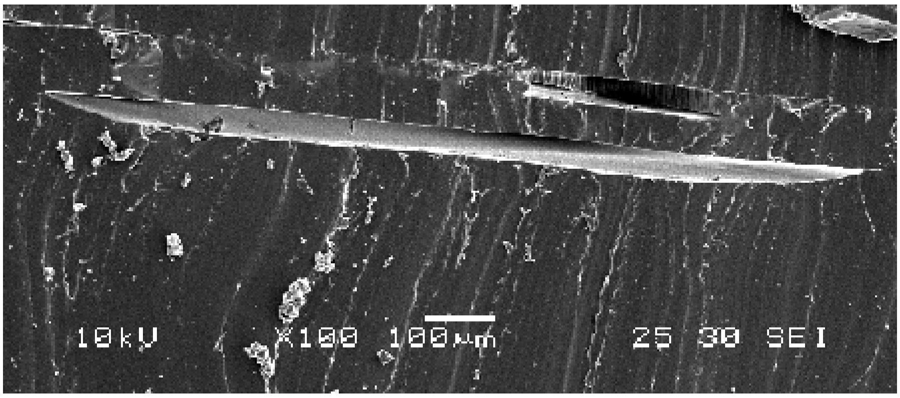

Figure 2. Scanning electron microscopy (SEM) micrograph of the PDMS membrane between working (rounded profile) and the actuation channels.

3. FABRICATION PROCESS

The fabrication method of microchannels in PDMS is based on the manufacturing approach of casting a liquid pre-polymer on a mold. The mold is structured by UV lithographic techniques to form the negative of the intended shape. The cast pre-polymer assumes the shape of the model and is cured to form an elastomeric replica that can subsequently be peeled off (Subramani and Selvaganapathy, 2009). The microfluidic device is obtained fabricating multiple layers of PDMS (multilayer soft lithography), each one containing different components. Further the layers are bonded to obtain the final device.

3.1. Photolithography

To obtain the working and actuation channels, two different molds were fabricated by optical lithography. The AZ®50XT positive photoresist (from AZ Electronic Materials) was used to fabricate the working channel mold and SU-8 negative photoresist (from Microchem) was used to obtain the actuation channel mold. The process is showed in Fig. 3. For both molds, 40 µm-thick photoresist was spin-coated, soft baked, exposed to UV light, developed and hard baked. The rounded profile required by the working channel was obtained during the hard bake, where the positive photoresist reflows and forms rounded shape channels (Yoo *et al.*, 2008). This negative photoresist does not reflow at the temperatures required for the curing of the elastomer, maintaining the squared profile (Studer *at el.*, 2002). We observed that the width of the working channel mold shrank about 6% to 8% during the hard bake, while the thickness in the center expanded about 15% to 18%.

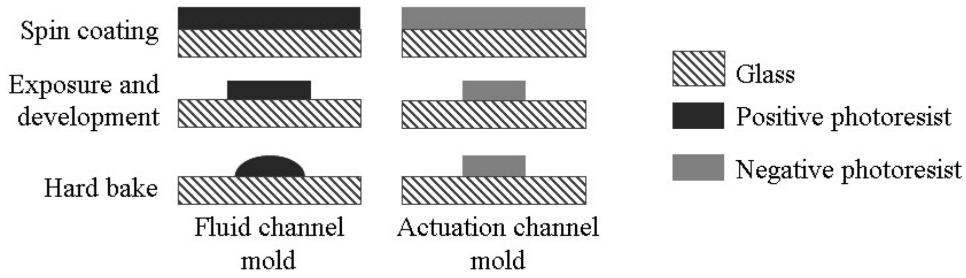

Figure 3. Fabrication of actuation and working channels molds using photolithography.

3.2. Soft Lithography

Silicone elastomer Sylgard® 184 (PDMS) from Dow Corning, containing vinyldimethyl-terminated polydimethylsiloxane as base and methyl-hydro-dimethylsiloxane as crosslinker (curing agent) was used. The ratio used was 10:1. The mixture of base and curing agent was stirred strongly and degassed in a vacuum chamber, in order to remove the air bubbles.

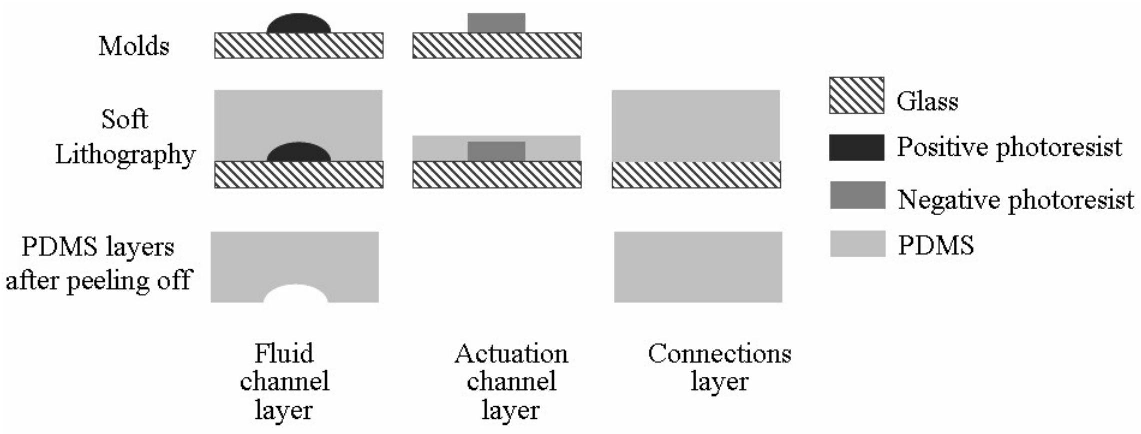

Figure 4. Fabrication process of the PDMS layers by soft lithography.

Three layers were prepared to microfabricate that device (Fig. 4). In the first layer, connections to macro world were fixed on the mold to obtain the reservoirs and then PDMS was poured onto the working channel mold wafer. After curing the polymer (100 ºC/30 min), this thick layer (~ 4 mm) was peeled off from the mold. The layer containing the actuation channel and the membrane was obtained by spin-coating the PDMS on the appropriated mold at 2000 rpm for 30 seconds, providing a membrane of about 50 μm after a soft bake (95 ºC/5 min).The membrane was not peeled off from the glass substrate. The third layer, also thick (~4 mm), containing the connections for the actuation channel was fabricated by pouring PDMS onto a glass where the tubes were fixed. After cure reaction, it was also peeled off.

3.3. Oxygen plasma treatment

The complete device was obtained by bonding the three layers via oxygen plasma (Fig. 5). The hydrophobic surface of the PDMS is modified during the plasma treatment because silanol groups and SiO*x* (i.e. silicon bonded to three or four oxygen atoms) are formed (Hillborg *et al.*, 2000). The surface of the PDMS turns hydrophilic while the bulk properties remain unchanged. The oxidized surface allows Si-O-Si covalent bonding between the layers (Yoo *et al.*, 2008). The strength of these bonds makes the sealing irreversible. The working channel layer was bonded to the membrane in a cross configuration, obtaining the microvalve in the intersection of the microchannels. After that, this bi-layer was peeled off from the glass and bonded to the third layer of PDMS hence obtaining the microfluidic device.

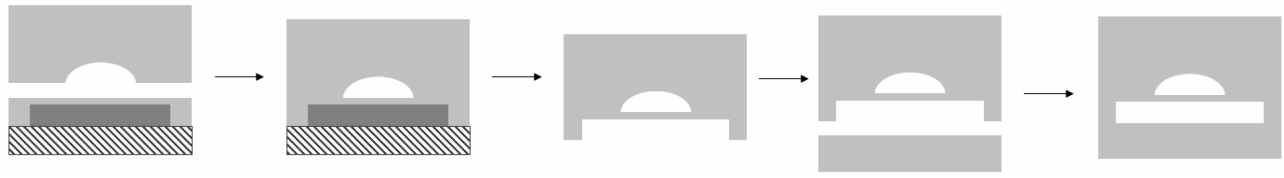

Figure 5. Multilayer PDMS bonding sequence via oxygen plasma treatment.

Figure 6 shows the final device in 3D, where it is possible to view the PDMS multilayers configuration, the working and the three actuation channels. The device contains three microvalves in order to be used as a micropump in further studies.

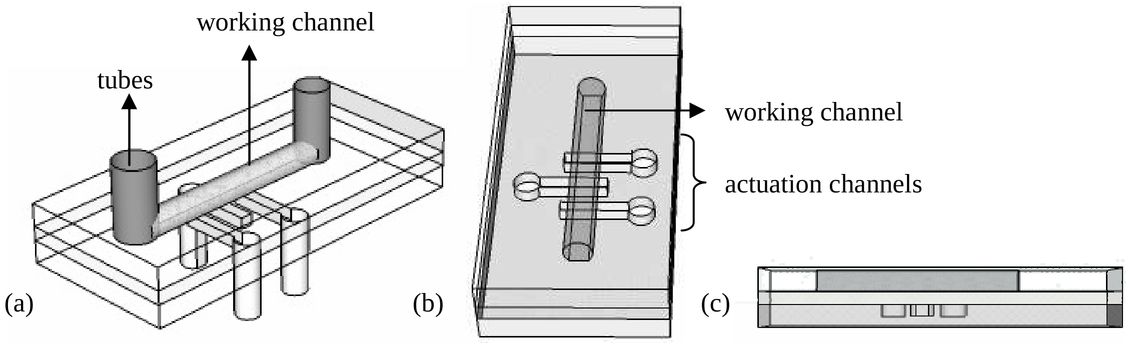

Figure 6. Schematic of the microfluidic device. (a) Connections (tubes) to macro world. (b) Showed without the tubes. (c) Side view of the three layers.

4. RESULTS

Microvalves with dimensions of 200 µm x 700 µm were obtained. The thickness of the microvalve membrane has about 50 µm. The microvalves have been tested applying different pressures in the actuation channel while a colored liquid passed through the working channel. The transparence of the PDMS allows the usage of a CCD camera to verify the working principle of the microvalves. When the microvalve closes, the working fluid flow was blocked by the membrane's deflection. It can be observed by the displacement of working fluid volume under the membrane as shown in Fig. 7. The PDMS membrane deflection is a function of the pressure applied in the actuation channel. The deflection of the membrane to completely close the valve is about 40 µm when a 180 kPa pressure is applied to the actuation channel. Lower actuation pressure (100 kPa) results in controlled leaks, as shown in Fig. 7b.

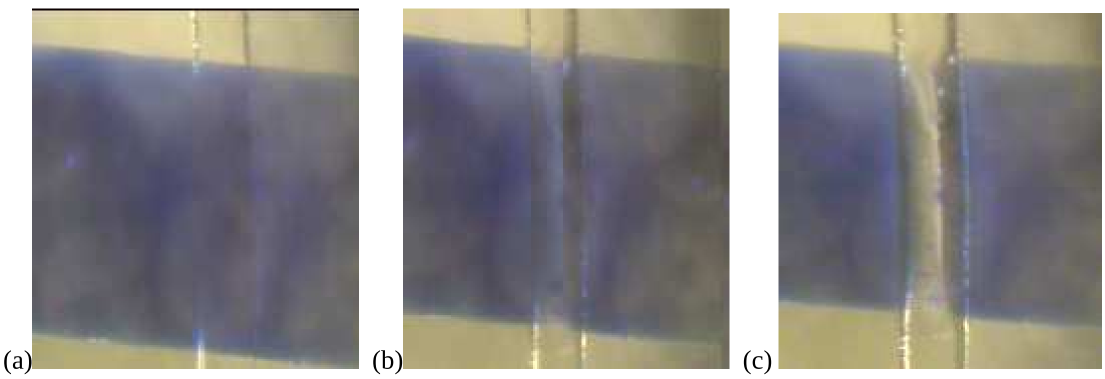

Figure 7. Top-view of the microvalve. (a) microvalve opened, and (b) partially closed with 100 kPa. (c) Microvalve closed with 180 kPa, pressure which the liquid flow was completely blocked.

In our experimental setup, the microvalves were tested using a syringe pump and deionized water. The output from the syringe pump (New Era Pump System) was connected to the inlet of valve and a gauge pressure sensor (Freescale MPX4100G) through a T type tube. The actuation channel was pressurized with nitrogen gas and the applied pressure was controlled by a gauge pressure sensor (Freescale MPX 4250). A data acquisition (DAQ) provided the connections between computer and system measurement, as shown in Fig. 8.

The valve opening and closing could be precisely controlled by varying the pressure in the actuation channel. The time response for a valve immersed in water is in the order of 50 milliseconds. The valve described in this paper presents many advantages when compared to the valves manufactured in silicon. The major advantage is related to the lower Young's modulus of PDMS. This implies that the active area of the valve can have the same dimensions of the working channel, resulting in a small dead volume (Unger *et al.*, 2000). Furthermore, the softness of the membrane allows the complete sealing of the valve at low pressure and in the presence of particulates.

In order to characterize the devices we use the experimental setup described in Fig. 8, with a syringe pump that maintained a constant flow during the measurement. Keeping a constant pumping rate, we could measure the specific pressure related to that flow, using the external pressure sensor connected to the inlet working channel tube. The actuation system was controlled by a computer using a LabView program that allowed us to obtain the control over the 3 way actuation valve. In this case, the duty cycle and the actuation pressure on the membrane could be controlled. As a result, it was possible to control the conductance into the channel when the actuation pressure is switched off and switched on.

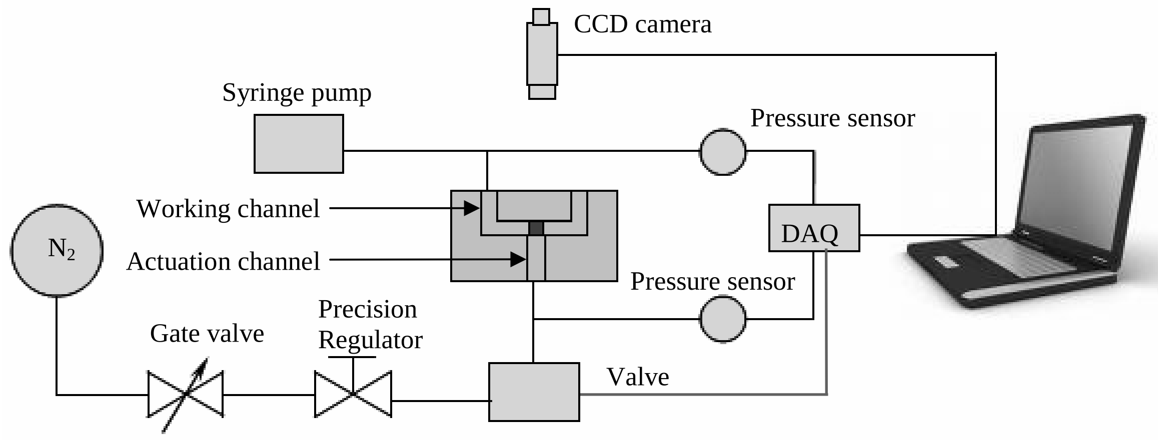

Figure 8. Experimental set up for microvalve characterization.

Figure 9 shows the pressure measurements in actuation and working channels, monitored by the gauge pressure sensor, for two different flow rates. The flow rate was kept constant during the experiments. The actuation pressure was changed from 60 to 180 kPa. A duty cycle of 66% and a frequency of 12 mHz were used. The measurements for all flow rate started when the pressure reached constant value of pressure, that we will call baseline pressure.

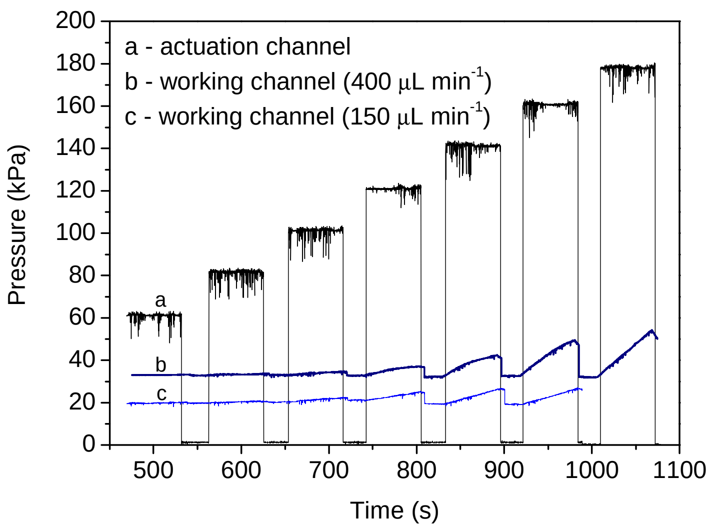

Figure 9. Relationships between the actuation pressure to close the valve and the pressure into the working channel for two different pumping rates (150 µL min^{-1} and 400 µL min^{-1}).

Starting the measurements with a constant flow rate of 150 µL min^{-1} (baseline pressure of 20 kPa) and varying the applied pressure on the membrane, the conductance into the channel decreased with the valve partially closed. When the valve was activated (60 kPa) the fluid pressure rose until it overcame the deflection exerted by the gas on the membrane and produced a controlled leakage. When the valve was switched off, the pressure of the fluid decreased until the baseline pressure. Increasing the actuation pressure on the membrane, the conductance of fluid was reduced, hence increasing the pressure into the working channel. When the membrane was deflected by a pressure of 160 kPa, the valve closed completely and the fluid pressure increased continuously.

Similar results could be observed for a 400 μL min^{-1} pumping rate (baseline pressure of 32 kPa). In this case the valve was not fully closed at 160 kPa. The increase of pressure into the working channel saturates with time, indicating the starting of a leakage. Increasing the actuation pressure to 180 kPa, we observe a fast increase in pressure into the working channel, showing a complete valve closing.

5. CONCLUSIONS

The use of non-traditional materials and processing methods such as soft lithography has enabled the manufacture of microdevices with advantages over conventional methods. They allow the construction of devices without the traditional disadvantages related to the problems of interlayer adhesion. The bonding of the microvalve layers was easily got by oxygen plasma treatment of the PDMS surfaces that would be bonded, with manual alignment between them. Such processes can be used to build complex structures such as valves and pumps with a very low-cost and rapid turnaround time to implement new modification in mechanical design of those devices. Besides the biocompatibility and flexibility of the PDMS, it also has transparency to visible light, which allows better observation of the working principle of these devices.

Herein we presented the fabrication process of a PDMS pneumatic microvalve, which is actuated by a deflection of PDMS membrane. Moreover, it is a low-cost material that combined with the easiness of sealing layers, results in a simple process of manufacture. We observed that to block the passage of liquid flow through the valve, the shape of the working channel is an important design parameter.

The manufacturing process used allowed the achievement of efficient operating valves for work pressures between 100 and 180 kPa, depending on the flow of liquid injected into the working channel. The flow varies from 100 μL min^{-1} to 500 μL min^{-1}. The response time measured was in the order of milliseconds.

6. ACKNOWLEDGEMENTS

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