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Micro checkvalves for integration into polymeric microfluidic devices

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Abstract. This paper describes the design, simulation, fabrication and characterization of micro checkvalves suitable for integration into polymeric microfluidic devices such as micropumps or test cartridges for biomedical analysis. The valves are fabricated by a polymeric surface micromachining process, which utilizes SU-8 as the functional material. The devices are assembled with the lamination technique. A micro checkvalve consists of 3 layers: an inlet layer, a valve layer and an outlet layer. The valve is a disc of 1-mm diameter. The disc is suspended on folded beams, which act as valve springs. Both valve disc and springs are fabricated in a 100- μ m SU-8 layer. The valves prove a clear flow rectification function. Relatively low pressure is required for opening the valve. The valves were tested and characterized with water. One of the valves are successfully integrated into a polymeric micropump. These valves prove the facile and reliable lamination technology for fabrication complex polymeric microfluidic devices for biomedical analysis.

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1. Introduction

Recently microfluidics has been emerging as an exciting research field with many applications [1]. One of the main growth thrust of microfluidics are microfluidic analysis devices - the so-called lab-on-a-chip. A number of chemical and biochemical assays were successfully implemented. Applications such as DNA sequencing [2], antigen detection [3] and high-throughput genotyping [4] were implemented in microfluidic platforms. Many of these devices were fabricated in glas and silicon, which require complex fabrication processes and pose possible problems with biocompability. Furthermore, the relatively large size of microfluidic systems utilize a lot of silicon surface that makes a microfluidic chip more expensive. Efforts have been made to fabricate polymeric microfluidic devices. Polymeric lab-on-a-chip has a potential of low cost and of high biocompability. Automated genotyping including purification, amplification and hybridization was implemented in a polymeric device [5]. Polymer analysis cartridges for blood analysis are commercially available [6]. Another polymer cartridge for automated pathogen detection was demonstrated [7]. A test elastomer cartridge was developed for multiplex detection of nucleic acid sequences [8]. The later cartridge has an integrated diffuser/nozzle pump for delivering fluids. Another concept combines glass and elastomer to realize peristaltic micropump with pneumatic actuation [9]. Elastomer cartridge was used as the interconnection solution for hybrid integration of off-the-shelf components, but no fluidic component was integrated in this platform [10].

The recent emerge of polymeric microfluidic cartridges show a trend of low-cost dispossable non-silicon microfluidic devices. The need of cheap polymeric devices leads to the development of new fabrication technologies. Our technology approach is to fabricate size-critical components with the conventional microtechnique of lithography, while large structures such as channels can be fabricated in cheaper ways such as molding, hot embossing or laser machining. Furthermore the polymeric technology can also include packaging techniques such as layer-by-layer lamination in order to solve the relatively costly interconnection problem. We have developed a technology which combines lithography of thick-film resist such as SU-8 with cheaper materials and technology such as laser cutting of polymethylmethacrylate (PMMA). This approach was proven by the realization of a number of microfluidic devices such as microchannels, micromixers, micro checkvalves, micro Tesla-valves, micro pumps and passive micro flow controllers. This paper focuses on the development of SU-8 micro check valves. Although the devices were stand-alone, the integration in a complex microfluidic cartridge is feasible.

Passive valve is one of the most important microfluidic components. Several micro check valve designs have been realized in the past. Although they differ in shapes and materials, all check valves have the same function of flow rectification. Micro check valve can be categorized by their forms such as ring mesa, cantilever, disc, V-shape and membrane [11]. Micro check valves can be used as a stand-alone microfluidic component. However their most popular application is the use in a reciprocating

micropump. Varieties of micro check valves were integrated in reciprocating micropumps [1]. The requirements on micro check valves for this application are low leakage in the reverse direction and low spring constant. Most of the silicon micropumps have check valves made off silicon and metals, which are relatively stiff [12]. Furthermore these check valves have hard sealing surfaces and may cause large leakage in the reverse direction.

Polymeric check valves offer unique advantages over silicon and metal counterparts. With a Young's modulus of two orders less than that of silicon and metals, polymeric microvalves have much lower spring constant allowing to open the valve with much less pressure. The soft sealing surface allows a zero-leakage design. A number of polymeric valves were fabricated in different materials such as polyimide [13], polysulfone (PSU) [14], polyester [15] and recently polydimethylsiloxane (PDMS) [9]. Many of the valves were fabricated on glass or silicon substrates, thus are not fully polymeric and may have chemical compatibility problems in lab-on-a-chip applications.

In this paper we offer a new alternative of making fully polymeric microvalves. We use SU-8 as the functional material which is structured by polymeric surface micromaching. SU-8 is coated over a sacrificial layer on a silicon substrate. Etching away the sacrificial layer releases the freely moveable SU-8 structures. This SU-8 layer is then considered as one functional layer in the subsequent multi-layer lamination. Other layers are made of laser-machined PMMA sheets and double-sided adhesive tapes.

Here we present the design, fabrication and characterization of SU-8 micro checkvalves. The successful operation of the valves facilitate the development of fully polymeric analysis cartridges for biomedical applications.

2. Design and simulation

The check valve is designed as a disc suspended on N folded beams, which work as valve springs. The folded beams can be considered as springs connected in parallel. Thus the force at each beam is:

$$F = \frac{F_0}{N} = \frac{p\pi r_d^2}{N}.\tag{1}$$

Where p is the pressure applied on the valve disc and r_d is the radius of the valve disc. Each folded beam can be seen as connected beam segments or springs connected in serial. The total deflection of the valve disc is the sum of all the deflections of the beam segments:

$$\delta = \sum_{i=1}^{N} \delta_i. \tag{2}$$

With a beam thickness of 100 μ m, the deflections can be assumed as small enough to be in the linear range. Thus linear beam equations are adequate for the analytical analysis. The deflection of each beam segment can be modelled as a straight beam, which is fixed

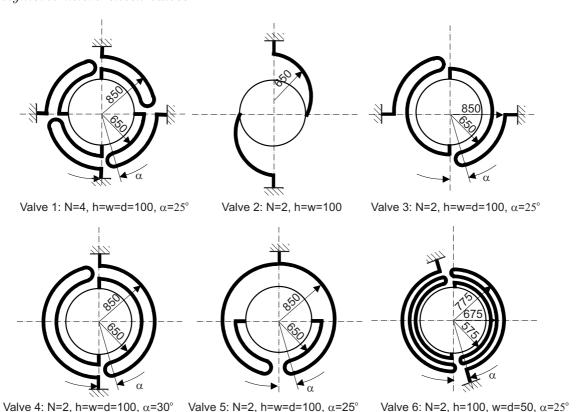


Figure 1. Different valve designs and their geometry parameters (in microns, h: layer

at the one end and guided at the other end [16]:

a diameter of 1mm.

$$\delta_i = \frac{FL_i^3}{12EI} \tag{3}$$

thickness, w: beam width, d: gap between folded beam sections). All valve discs have

where F is the force at the guided end, L_i is the length of the beam segment, E is the Yong's modulus of the beam's material and I is the moment of inertia:

$$I = \frac{wh^3}{12} \tag{4}$$

with w the width and h the thickness of the beam. Thus the total spring constant of the valve is calculated as [17]:

$$k_{Valve} = \frac{F_0}{\delta} = \frac{N}{\sum_{i=1}^{N} \frac{L_i^3}{12EI}}.$$
 (5)

The different designs investigated in this paper are depicted in Fig. 1. The curved segments of the arms can be modelled analytically as a beam with a fixed end an a guided end [17]. The smaller the curvature angle and the larger the radius of curvature of the segments the better is this assumption. With the space constrain, the length of the curved segments can be changed by varying the angle α .

While valve design 5 only has one suspension arm, valve designs 2, 3, 4 and 6 have two suspension arms. The advantage of the small number of arms (N = 1) and

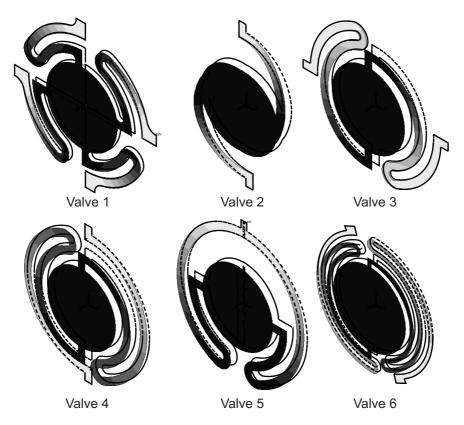


Figure 2. Deflection of different valves with pressure load on the valve discs (numerical simulation with ANSYS)

N=2) is the low spring constant. However, rotation around suspension axis may cause unexpected behavior of the disc displacement. With more then 3 arms $N \geq 3$, the valve disc can move parallel to its base plane with no rotation. The designs with three or more arms are called ortho-planar springs [17]. The advantage of ortho-planar design is the stable parallel out-of-plane deflection. The disadvantage is the much stiffer total spring constant, because the suspension arms can be considered as springs connected in parallel.

Figure 2 shows the simulation results with ANSYS using the element type SHELL 63. The valve disc in this models is loaded with a pressure p. The resulting deflection δ of the disc is used for calculation of the spring constant of the valve:

$$k_{Valve} = \frac{F_0}{\delta} = \frac{p\pi r_d^2}{\delta}.$$
 (6)

Table 1 compares the spring constants of the different valve designs using the analytical model (5) and the numerical simulation (6). The results show, that the analytical model can only be used for rough estimation of the spring constants.

The prediction of the behavior of the check valve requires a coupled fluid-structural simulation. The usual approach is to run the structural and flow analysis separately with two different solvers [19] [20]. The deflection of the valve is used for shaping the grid of the fluid domain. In turn, the pressure distribution from the flow analysis is

Table 1. Spring constants o	f the different	valve designs	(in N/m,	calculated for a
Young's modulus of 4.02×10^9	Pa [18])			

Calculation	Valve 1	Valve 2	Valve 3	Valve 4	Valve 5	Valve 6
Analytical		490	127	57	64	21
Simulation		499	95	65	59	31

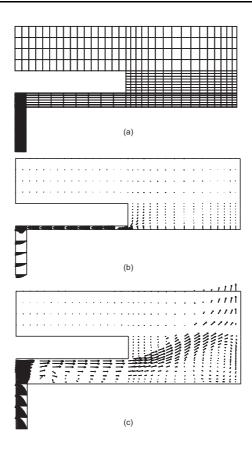


Figure 3. Semi-analytical coupled fluid-structural simulation of the microvalve: (a) grid model with the initial gap, (b) reverse direction, (c) forward direction (rotation-symmetric model, the velocity vectors in (b) and (c) are not to scale).

used as the boundary condition for the structural analysis. The multiphysics feature of ANSYS also allows this coupling approach. However, this simulation approach is very time consuming. For complicated structures such as our valve designs, a complex three-dimensional model is necessary for the simulation. The three-dimensional coupled fluid-structural simulation is even more complex and time consuming.

Because the valve disc in our case is assumed to move parallel to its surface, the behavior of the valve displacement is straight forward. A linear displacement with a constant spring constant can be calculated. Only one structural analysis needs to be carried out for determining the spring constant. This stiffness can then be used for a semi-analytical coupled simulation described next.

The coupling was realized in an iteration loop written as a macro script in the ANSYS batch file. First, the flow analysis was carried out for an initial gap between the valve disc and the inlet. The pressure distribution across the valve disc was used with the known stiffness for calculating the displacement analytically. The new gap between the valve disc and the inlet was then updated. The flow model was then meshed again with the new geometry, readied for the next iteration. The iteration loop stopped, if the displacement difference reached a given convergence condition which was in our analysis 10 nm. On average the coupled simulation stopped after 3 or 4 iterations. The simulation of our two-dimensional rotation-symmetric model with 14848 elements required about 20 minutes on a Pentium IV personal computer with a clock frequency of 1.8 GHz. Figure 3 shows the simulated flow field in forward and backward direction. The numerical models is only used for fitting the measurement results in section 4 because of the constraints of an unknown exact elastic modulus of SU-8, an unknown initial gap, and the possible kipping movement of the valve disc.

3. Fabrication

3.1. Polymeric surface micromachining with SU-8

The valves described above were fabricated in SU-8, a negative thick-film resist. We used SU-8 2100 (MicroChem, Corp, USA) for the fabrication of the 100- μ m-thick SU-8 layer. The SU-8 2000 family is a new formulation, which uses cyclopentanone (CP) as the solvent instead of γ -butyrolacetone (GBL) in the standard SU-8 family.

The valve designs were first drawn with a CAD-program and then printed on a polymer film with a high-resolution laser printer. The film was used as the lithography mask for the UV-exposure of the SU-8 layer. The layers for inlet and outlet holes are placed on separate masks. This low-cost approach allows us to have a very short prototyping cycle. We developed a polymeric surface micromachining process with SU-8 as the functional layer. The process starts with cleaning a polished silicon wafer. The silicon wafer works as the handling substrate. A 100-nm chromium layer was sputtered on silicon, Fig. 4a. This chromium layer is the sacrificial layer for the later release of the SU-8 structures. SU-8 2100 was then spincoated on the chromium layer. At a room temperature of 24° C, the spin speed was first ramped up in 5 seconds to 500 rpm. This speed remained constant for 5 seconds. The second ramp increased the speed from 500 rpm to 2100 rpm in 10 seconds. The high speed was kept constant for 22 seconds. The final ramp decreased the speed from 2100 rpm down to full stop in 20 seconds. This spin recipe results to a 100- μ m thick SU-8 layer with a thickness variation of less than $\pm 2\mu$ m, Fig. 4b.

A soft bake step follows the spin-coating step. Since SU-8 2100 still continued to reflow with a relatively low viscosity, care should be taken for levelling the wafer. The soft bake step was carried out in a convection oven. The wafer was kept at 65 o C for 10 minutes and then at 95 o C for 40 minutes. The wafer was then allowed to cool down to

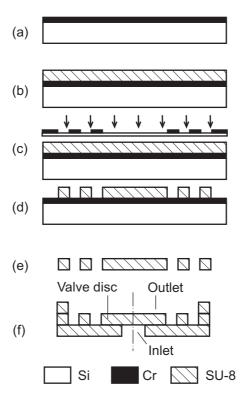


Figure 4. Fabrication process of SU-8 check valves using polymeric surface micromachining.

room temperature before the UV-exposure. The SU-8 layer was then exposed with an energy density of $525 \,\mathrm{mJ/cm^2}$ using near-UV wavelengths. The polymer mask described above was used for this step, Fig. 4c.

Following the exposure step, another bake process allows SU-8 to polymerize further. The wafer with the exposed SU-8 layer was baked at 65 °C for 5 minutes and then at 95 °C for 10 minutes. The wafer was allowed to cool down slowly in the switched-off oven to room temperature. This cooling step is crucial for avoiding microcracks and curling of the SU-8 film after release. SU-8 was then developed in propylene glycol methyl ether acetate (PGMEA), Fig. 4d. After cleaning with isopropyl alcohol (IPA), the wafer was blown dry with nitrogen. In the final step, chromium was etched away releasing the SU-8 layer, Fig. 3e. Fig. 5 shows the fabricated micro checkvalves. The SU-8 structures were coated with a thin aluminum layer for the measurement with scanning electron microscopy (SEM) measurement.

3.2. Device assembly and packaging

Fig.4 describes the assembly of a stand-alone micro checkvalve with three SU-8 layers: the outlet layer, the valve layer and the inlet layer. Each layer has a form of a disc with 5.5 mm diameter. The inlet has a diameter of 100 μ m, which is entirely covered by the valve disc (Fig. 6a). The outlet has a diameter of 2 mm (Fig. 6b), which is equal to the opening diameter of the valve layer (Fig. 5). This three-layers-design allows a

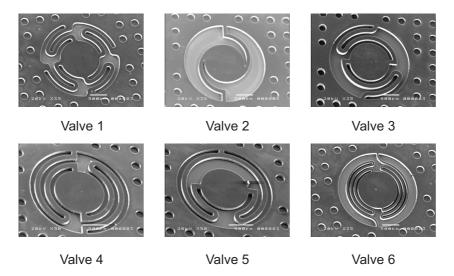


Figure 5. Fabricated SU-8 checkvalves.

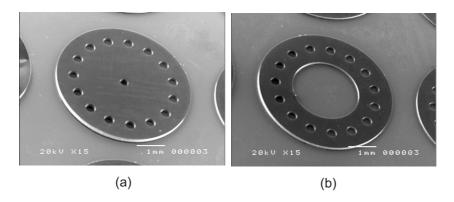


Figure 6. The inlet (a) and outlet (b) layers.

maximum deflection of 100 μ m of the valve disc. Thus the stress in the beams is kept at a relatively low value, which improves the fatique life of the valve. In a micropump application [21] valve number 1 can withstand few millions of deflection cycles.

The three-layer stack is pressed between two capillary tubes with an outer diameter 5.5 mm and an inner diameter of 2 mm by a spanner. Epoxy glue was the applied around the tubes. After the glue was hardened the device was released from the spanner readied for testing. Fig. 7 shows the picture of one of the assembled valves.

4. Characterization results

The flow rectification behavior of the checkvalves was tested with deionized (DI) water. The flow characteristics of the stand-alone valves was taken by measuring the pressure drop across the valve and the volumetric flow rate. The volumetric flow rate is determined by the travel speed of the water/air interface in a capillary with a known diameter. A big reservoir with different heights of the water surface emulates the inlet

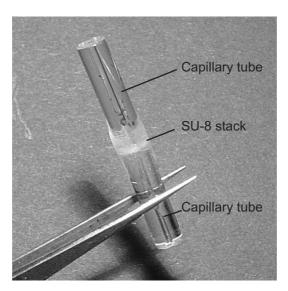


Figure 7. The assembled valve.

pressure and eliminates the error of height change during the measurement.

The pressure drop was measured with a differential pressure sensor (Honeywell 22PC-Series, \pm 1 psi) which was calibrated for a pressure range from 0 to 6000 Pa.

The outlet of the valve was connected to a horizontal capillary with a inner diameter of 0.8 mm. The travel speed of the meniscus was determined by measuring the travel time over a fixed distance and converted into flow rate. The measurement error can be estimated by:

$$\Delta \dot{Q} = \sqrt{\left(\frac{\Delta V}{t}\right)^2 + \left(\Delta t \frac{V}{t^2}\right)^2} \tag{7}$$

where V and t are the measured volume and time, ΔV and Δt are the worst-case errors of the measurement. An error of less then 5% can be estimated for our measurement.

Figure 8 shows the results of the valves discussed in this paper. Results of the numerical model described in section 2 was used as the fitting curve for the measurement. The parameters of the numerical models, the spring constant k and the initial gap g_0 , are given in the figure caption. It is obvious that due to the lower spring constant the valve with only 1 and 2 suspension arms allow higher flow rate in forward direction. However, they have large leakage flow rates in the reverse direction. This behavior may be caused by the kipping movement of the valve disc. Even a small particle or assembly misalignment may cause a large gap due to this kipping movement. The model of an initial gap and a parallel disc movement is not ideal for this case.

Valve 1 shows almost no leakage due to the tight closure with 4 suspension arms. A kipping movement is not possible with the ortho-planar design. Thus almost zero leakage can be achieved. The drawback is the stiff spring, which causes very small flow rates in the forward direction, Fig. 8a.

Figure 9 compares the flow characteristics of all the valves.

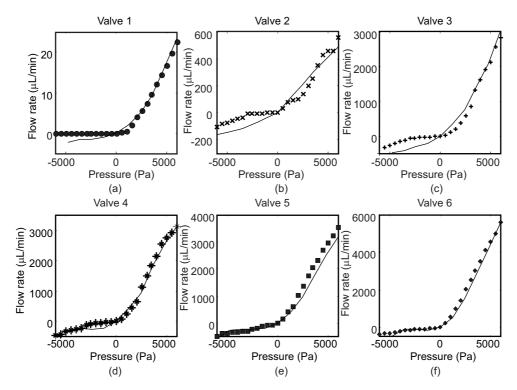


Figure 8. Measured flow behavior of each microvalve and its fitting curve from the numerical model: (a) Valve 1 (k = 613 N/m, $g_0 = 9 \mu\text{m}$), (b) Valve 2 (k = 499 N/m, $g_0 = 50 \mu\text{m}$), (c) Valve 3 (k = 95 N/m, $g_0 = 65 \mu\text{m}$, (d) Valve 4 (k = 65 N/m, $g_0 = 60 \mu\text{m}$), (e) Valve 5 (k = 59 N/m, $g_0 = 65 \mu\text{m}$), (f) Valve 6 (k = 31 N/m, $g_0 = 65 \mu\text{m}$).

5. Conclusions

Different micro checkvalves have been designed, fabricated in SU-8 and tested for functionality. The results show the feasibility of making a microfluidic system entirely of polymeric material. The check valves have a clear rectification behavior and can be opened with a relatively low inlet pressure of less than 1 kPa. Ortho-planar designs showed a better sealing characteristics due to the parallel out-of-plane motion. Valve designs with 1 and two suspension arms have large leakage flows, which are probably caused by the kipping motion of the valve discs. Valve design 1 was successfully used in a micropump, which can deliver up to 1 mL/min water flow and a back pressure of 2 kPa [21]. The results of the pump will be reported in a separated paper. The successful operation of the micro checkvalves and the pump indicate that our polymeric technology is relevant for a low-cost mass fabrication of polymeric microfludic cartridges. We are continuing efforts to design a low-cost, disposable cartridge for analytical chemistry. Only passive components such as micro checkvalves, micromixers, microchannels and pump chambers are integrated in the cartridge. Actuators are placed on the external evaluation device.

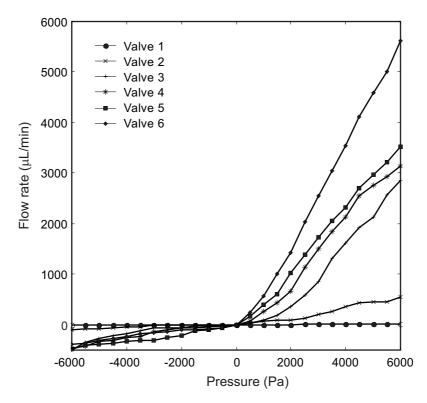


Figure 9. Flow characteristics of the stand-alone SU-8 micro check valves.

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