Electronically Controllable Microvalves Based on Smart Hydrogels: Magnitudes and Potential Applications

Andreas Richter, Dirk Kuckling, Steffen Howitz, Thomas Gehring, and Karl-Friedrich Arndt

Abstract—Electronically controllable microvalves based on temperature sensitive hydrogels as actuators are described. A thermal–electronic interface was used for electronic control of the liquid flow. The hydrogel actuators were directly placed in a flow channel. They used the process medium as the swelling agent. Because of the direct placement into the channel the elastic properties of the hydrogel actuator were utilized to improve the pressure insensitivity, to achieve high particle tolerance and to avoid a leakage flow. The microvalves show an extremely simple structure. They can be fabricated using conventional micro technology within a few technical steps. The microvalves can also be miniaturized to a currently unrivalled extent of about 4 $\mu m \times 4 \, \mu m \times 1 \, \mu m$. Valves for "laboratory on chip" applications can already be obtained. The switching times of the electronically controllable microvalves based on hydrogels are 0.3 s to 10 s.

[844]

Index Terms—Flow-control, hydrogel actuator, hydrogel application, microvalve, particle tolerance, temperature-induced phase transition.

I. INTRODUCTION

S TIMULI-RESPONSIVE or "smart" hydrogels are able to change their volume reversible and reproducible by more than one order of magnitude even through very small alterations of certain environmental parameters. Due to this property their enormous importance for many technological and scientific applications was expected [1]. However, they have not lived up to these expectations until now. Current research trends show a stronger orientation toward special mostly biomedical applications [2]–[7]. The volume change of smart hydrogels can be induced by a change in the properties of the liquid phase such as temperature, pH, solvent composition and ionic strength. In an earlier work [8], we utilized these characteristic properties of smart gels to fabricate a hydrogel-based valve of average performance (p: 0 bar to 10 bar, flow rate ($\Delta V/\Delta t$): 0 L/min to 5

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L/min). The automatic valve responds to changes of temperature, of pH values and of alcohol concentration. The valve behavior is reversible and reproducible with cycle times of 25 s (opening) and 35 s (shut-off).

Polymers which perform sensing and actuation functions well are of interest for application in microsystems, esp. in microfluidic. For these application it is necessary to synthesize and to pattern films or to form small particles of the sensitive polymer, both in the μ m-range. The synthesis of thin layers of a thermosensitive polymer and their photo-patterning for microactuator purposes are described in [9] and [17].

The autonomous flow control in microfluidic channels based on a film of sensitive hydrogel is possible. By using the photopolymerization and crosslinking of water-soluble polymer microfluidic actuators (hydrogel-coated posts) that respond automatically to a change in the pH were obtained. A flow sorter with an actuator size of approximately 1000 μ m × 700 μ m × 250 μ m and opening and closing times of about 8 s is described in [10].

Common conventional microvalves couple a distortable diaphragm to a piezoelectric [11], electrostatic [12], electromagnetic [13], thermopneumatic [14] and shape memory alloy [15], or other actuators. The diaphragm is bent in order to open or to close the flow channel. These microvalves offer generally three advantages. Due to the electronic controllability of the actuators they are switchable at any time, therefore they are universally applicable. Conventional microvalves possess small cycle times in millisecond-ranges. They can also be fabricated using established microtechnological processes and, therefore, they can be manufactured in large quantities.

Due to the complicated diaphragm based set-up and the difficulty to scale the forces of common actuators to the micro scale, conventional microvalves can only be miniaturized to a very limited extent. Dimensions smaller than 1 mm^3 are hardly obtainable. From a functional point of view the conventional microvalves have to face the problems of pressure sensitivity, of particle intolerance and of leakage.

The aim of the presented work was the design, development, fabricating and testing of a gel-based microvalve. Particle intolerance and leakage can be avoided using hydrogel actuators, because hydrogels are soft materials with excellent sealing properties. By renunciation of a distortable diaphragm and direct placement of the hydrogel actuator in the flow channel a very simple microvalve structure with a very small dead volume can be obtained. These features can be combined with the advantages of conventional microvalves, particularly an

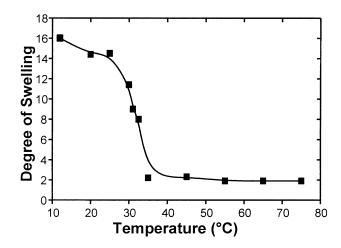


Fig. 1. Volume phase transition behavior of the temperature sensitive hydrogel poly(*N*-isopropylacrylamide) BIS 4 (details of synthesis see Section II-B). At a temperature of about 33° C the gel changes from the swollen to the shrunken state and vice versa.

electronic interface, tenable cycle times and potential complete manufacturing using existing microsystem technology. The direct electrical stimulation of the swelling/deswelling process of smart hydrogels is very difficult. A thermal stimulation via a thermal-electronic interface is possible, if a temperature sensitive hydrogel is used.

II. DESIGN AND FABRICATION

The most known temperature sensitive hydrogel poly(*N*-isopropylacrylamide) (PNIPAAm) exhibits lower critical solution temperature (LCST) behavior with a volume phase transition temperature (T_C) at approximately 33°C. Below T_C , e.g., at room temperature, the hydrogel is swollen and above T_C the hydrogel is deswollen (see Fig. 1).

The gel based microvalves (see Figs. 2 and 3) are three component devices consisting of the channel structure support, a Pyrex glass cover and a circuit card for electrical contacting (not shown in the figures). The channel geometry [width (100 μm to $800 \,\mu\text{m}$ × height (50 μm to 200 μm)] and the actuator chamber [width (100 μ m to 800 μ m) × height (50 μ m to 300 μ m)] are generated by a two-side process (wet etching with 30 wt-% KOH at 80°C; plasma etching, ASE-Bosch process). Applied materials were Si wafers (4", orientation $\langle 100 \rangle$, N-type, specific resistance = $250 \ \Omega \text{cm}$, $500 \ \mu \text{m}$ thickness, obtained from SICO Wafer GmbH Heiningen), or SiO₂ wafers (4", type Pyrex 7740, 500 μ m thickness). Heating elements (110 nm thickness, resistance 50 Ω) and temperature sensors, both prepared by a platinum-thin-film system (plated by GeSiM mbH with MSBA-400SP, from Malz and Schmidt) with lift-off patterning, are located on the rear of the Pyrex glass cover. The valve can be electronically controlled using these elements. The channel structure, the cover layer and circuit card were coupled by a combination of flip-chip and gluing technology [16].

A. Functional Principle

The hydrogel actuator was directly placed in a flow channel and remains in contact with the process medium. The actuator swells by absorption of the medium. In the normal case the

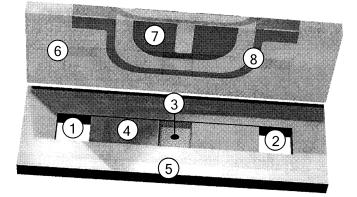


Fig. 2. Sketch of a microvalve with a filling of hydrogel particles. An actuator chamber is integrated in the flow channel. 1—Inlet, 2—Outlet, 3—Actuator chamber filled with gel particles, 4—Flow channel, 5—Structure layer, 6—Pyrex glass cover, 7—Heating element, 8—Temperature sensor.

medium temperature is below the T_C and the hydrogel seals the flow channel completely ("normally closed" function).

The electronic control of the valve was achieved by a heating element. To open the valve the gel actuator was warmed up above $T_{\rm C}$ with the heating element. The hydrogel actuator deswells and allows the fluid to flow through the channel. In order to obtain controllability of the valve, between completely opened and closed, a temperature sensor (thin meander structure) was integrated into the set-up to maintain a standard temperature.

A hydrogel, which possesses an upper critical solution temperature, would have to be used to obtain a valve that can be controllably closed ("normally open" function). These gels are swollen above $T_{\rm C}$ and shrunken below $T_{\rm C}$. However, they are not available yet.

We considered two different valve designs in present research: a microvalve with an actuator chamber and a photopatterned microvalve.

B. Microvalve With an Actuator Chamber

This valve design (see Fig. 2) possesses a separate actuator chamber to fix the hydrogel. For integration of hydrogels in this design no special hydrogel properties are required. Theoretically, all available hydrogels can be used. A very simple method to realize an actuator is to fill the chamber with hydrogel particles. Particles must be easily producible and doseable.

The lower side of the setup shown in Fig. 2 contains the channel structure as well as the fluid connecting pipe. An actuator chamber which is located within the channel is filled with temperature sensitive poly(*N*-isopropylacrylamide) (PNIPAAm) hydrogel particles. The hydrogel was prepared as follows. The *N*-isopropylacrylamide (NIPAAm) (Aldrich Chemical Co.) was recrystallized from n-hexane solution. The crosslinking agent was N, N'-methylenebisacrylamide (BIS). The initiator and accelerator for the polymerization reaction were potassium peroxidisulfate (KPS) and N, N, N', N'-tetramethyl-ethylenediamine (TEMED) (both from Aldrich Chemical Co.). NIPAAm and 4 mol-% BIS (BIS 4) were dissolved in deionized water. The total monomer concentration was 0.53 mol/l. To initiate the polymerization reaction 0.3

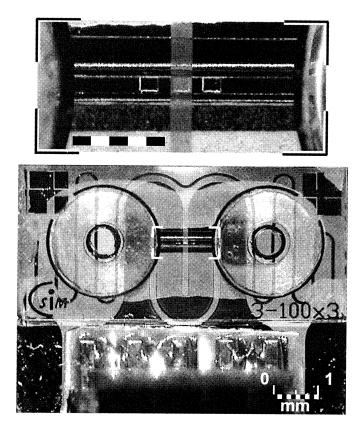


Fig. 3. Photograph of a microvalve with photopatterned hydrogel actuator dots. The circular structures are fluid connectors. The meander structures in the background act as temperature sensor (thin) and heating element (thick). The upper picture shows three gel dots within the channel (dimensions of a dot in the dry state: $100 \ \mu m \times 100 \ \mu m \times 100 \ \mu m$); scale bar $100 \ \mu m$.

mol-% of KPS and TEMED, respectively, were added to the oxygen free (bubbled with N_2) solution. After polymerization (about 12 h at room temperature) the PNIPAAm gel was immersed in deionized water for about one week to wash out nonreacted reagents. After drying the PNIPAAm BIS 4 gel the particles were obtained by milling and subsequent fractionating into different particle sizes using test sieves. The particles possess an irregular shape.

The fabrication of these valves is expensive due to the manual incorporation of the particle fraction.

C. Photopatterned Microvalve

In the second design (see Fig. 3), a PNIPAAm-based hydrogel dot was placed directly into the channel by photopatterning. This valve set-up does not require an actuator chamber. A satisfying adhesion of the hydrogel dot to the underground structure was achieved by an adhesion promoter [9].

The actuator dots were prepared from PNIPAAm copolymers bearing 4.5 mol-% light sensitive chromophores based on dimethyl maleinimide. The copolymer solution (20 wt-% in butanone), containing 2 wt-% thioxanthone with respect to the polymer weight as photosensibilisator was spin coated onto the SiO₂-support pretreated with 1,1,1,3,3,3-hexamethyldisilazane (HMDS) as an adhesion promoter. The film was subsequently dried and irradiated with a UV lamp (Hg lamp 400 W, wavelength 360 to 450 nm). Irradiation of the polymer resulted in an irreversible crosslinking by a [2+2]-cycloaddition. The

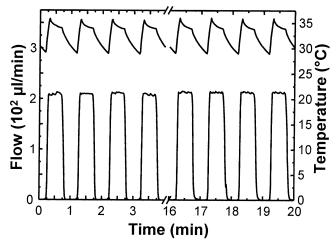


Fig. 4. Behavior of a microvalve with PNIPAAm BIS 4 particle actuator (size $500 \times 500 \times 50 \ \mu m^3$) at power modulated work (power peak at 380 mW for 500 ms, retaining power 150 mW). Upper curve: temperature versus time, lower curve: flow rate versus time. The fastest switching times for actuators based on PNIPAAm particles are presently for opening 300 ms and for closing about 2 s. The operating pressure was about 1.5 bar.

noncrosslinked polymer was removed with a water alcohol mixture (20 wt-% ethanol, 80 wt-% water).

The most important difference to the former principle is that the use of hydrogels, which can be patterned in the dry state by light, allows the continuous manufacturing of microvalves using normal microsystem technology. These actuator dots can be placed in almost every existing channel layout with very little additional work. Possessing a minimal lateral resolution of 2 μ m spacing and 4 μ m structure width at layer thicknesses in the μ m-range the gels are also applicable in all current channel geometries.

III. VALVE BEHAVIOR

The valves were designed to regulate the flow of a liquid. The flow-rate (volume per time) was measured with a flow sensor with an integrated temperature sensor (GeSiM). The resolution was 1 μ L/min. The flow with nl-precision was generated by a piezoelectric microdroplet pump (MEP, GeSiM).

Fig. 4 shows the opening and shut-off behavior of a microvalve with an actuator chamber size of 500 μ m × 500 μ m × 200 μ m filled with PNIPAAm BIS 4 particles (diameter of (82 ± 8) μ m).

In order to control the water flow rate the valve was temporarily warmed up with 380 mW to 35°C. Subsequently the temperature was kept constant within 1 K with 150 mW.

The valve showed no leakage in the closed state up to the maximal pressure limit of the test set-up of 1.8 bar. Water diffusion through the gel actuator was not observed. With a bigger valve (chamber size \emptyset 5 mm and 4 mm length, filled with PNI-PAAm BIS 4 particles, see [8]) a leakage free switching for a maximal pressure drop of 5 bar was obtained. Generally, the maximum value of the pressure drop is a function of the ratio of the volume of the dry hydrogel in the chamber to chamber volume. The higher this ratio, the bigger is the maximal pressure drop, which can be obtained without leakage. The ratio hydrogel volume to chamber volume influences not only the leakage and

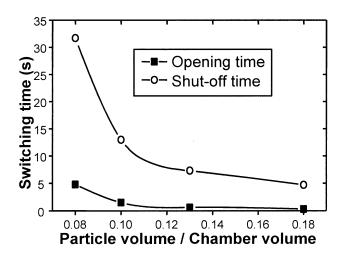


Fig. 5. Dependence of the switching time on the ratio particle volume to chamber volume. The microvalve was filled with PNIPAAm BIS 10 particles; chamber size 500 μ m × 500 μ m × 200 μ m; spherical particle size (135 ± 15) μ m.

pressure behavior, but also it determines the switching time of microvalves (see Fig. 5). It has to be optimized.

During the swelling/deswelling process the hydrogels change their volume by one order of magnitude, e. g., for the photo patternable gel between 10-fold and 1.6-fold of the dry volume, for the gel particles between 16 fold and 1.6-fold of the dry volume, respectively. Following the statement from Fig. 5 only a part of this swellability is constructively usable. The best possible working behavior gives a filling of 10 to 50% of the actuator chamber volume with dry actuator material.

An increase of the response time, especially the shut-off time, was observed at smaller values of filling. The influence of higher values is small.

Another important design parameter is the particle size. The influence of the particle diameter on the closing time is shown in Fig. 6. These measurements were done with a valve scaled up by a factor of 10 [8]. The application of a particle fraction with smaller diameter (faster swell kinetic of a single particle) did not result generally in a decrease of the response time. This is an effect of the packing density. The measured ratio of the volume of the particles to the sum of volume of particles and void volume dependent on the particle diameter shows local minima at $(450 \pm 50) \,\mu\text{m}$. This correlates strongly with the experimental results shown in Fig. 6.

Furthermore, the response time was found to be dependent on the chamber size, the swelling and mechanical properties of the hydrogel.

A relevant operating parameter, which effects the switching time of the microvalve, is the heating power. Particularly, the opening time of the valve is a function of this value (see Fig. 7). An optimum was reached at 350 mW with an opening time of 300 ms. A further power increase led only to a small time advantage. The time required to close the valve is determined by the difference between the temperature at which the valve is normally maintained and the T_C , as well as the cooling rate of the microvalve due to heat transfer to the environment. Presently, the spontaneous shut-off time was approximately 2 s. The valve closes within 1 s when an external fan for cooling is used. An

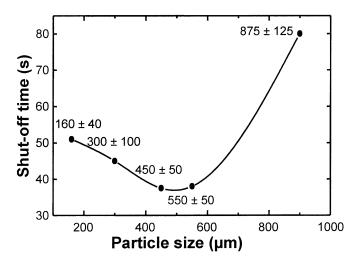


Fig. 6. Particle size dependence of the shut-off time. The valve [8] was filled with a constant mass (17 mg, dry state) of PNIPAAm BIS 4 particles of different size; chamber size \emptyset 4 mm × 5 mm. The particle sizes are given as mean values and maximum deviations.

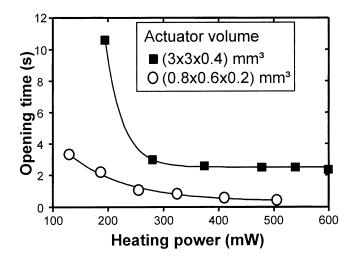


Fig. 7. Dependence of the opening times of the microvalve with a PNIPAAm BIS 4 particle actuator on applied heating power.

improvement can be achieved by decreasing the heat capacity of the valve body. In this case we think that closing time can be reduced to 500 ms. The time behavior of the valves was also influenced by the amount of liquid in the channel.

The behavior of the hydrogel actuator is influenced by components of the fluid, especially by the content of organic solvents and salts. This problem can be avoided by choosing an appropriate hydrogel for a specific fluid. As the T_C can be properly adjusted [18], microvalves with switch temperatures between 10 and 50°C could be obtained.

For examination of the particle tolerance an aqueous styrene-butadiene dispersion with 40 wt-% solid content (Dow Chemicals XZ 94466.01, particle size about 300 nm) was used. It could be shown that owing to the viscoelastic properties of the gel the valve was also very tolerant against particles in the fluid. They were enclosed by the hydrogel and the valve did not show a leakage flow. Given the possibility that a few particles might remain in the actuator chamber, a flushing step is necessary.

Fig. 8. Behavior of a microvalve with photo patternable hydrogel actuator (size 250 μ m \times 250 μ m \times 50 μ m) at nonpower modulated work (heating power 300 mW). Upper curve: temperature versus time, lower curve: flow rate versus time. The fastest switching times for photo patterned actuator dots are presently for opening 4 s and for closing 10 s.

The microvalve behavior is reproducible and shows a maximum error in reproducibility of less than 1%.

The explorations of the microvalves with photopatterned actuators showed that the dots adhere very well when the adhesion promoter HMDS was used. A strip off of the dots up to a pressure drop of 1.8 bar was not observed. The actuator dynamic of the photopatterned valves, size 250 μ m × 250 μ m × 50 μ m, was strongly influenced by the sharpness of the volume phase transition. This can be seen in Fig. 8. The phase transition of the photopatterned actuator dots is less immediate. This leads to a longer switching time, i.e., 4 s for the opening and 10 s for the valve closing.

IV. CONCLUSION AND OUTLOOK

Presented microvalves can be used in microfluidic processors due to their capability of miniaturization combined with other advantages like a small dead volume, a simple setup, the integrability into microsystems, the particle tolerance and the leakage free behavior. The hydrogel actuators used show a good chemical and long-term resistance. Only concentrated acids and bases affect the hydrogel stability.

The application potential of the valves is limited by two special circumstances. The switch behavior of the hydrogel actuators is a diffusion controlled process. Thus, switch frequencies of > 10 Hz are hardly realizable. Hence, hydrogel based microvalves cannot be used in highly dynamic applications. The second limitation results from the fact that hydrogels need direct contact with the process medium as the swelling agent. Process media in this case have to be aqueous solutions because LCST behavior of temperature sensitive hydrogel in other solvents is not known. Through contact with the process medium especially salts and pH may shift the phase transition temperature. Also cross sensitivities to a number of organic solvents are known. This might cause serious malfunction if the composition of the process medium is not properly chosen. The hydrogel based microvalves basic element is the "throttle valve" or "shut-off valve" only. In our opinion the development of hydrogel based microvalves will go in two directions.

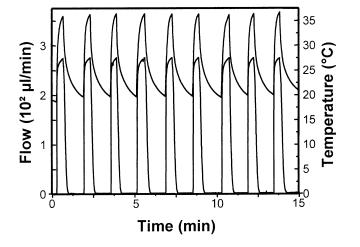
• In automatic microvalves the hydrogel can act as an integrated actuator-sensor. In this kind of application a critical concentration, e.g., nutrient broth for cell cultivation, can be kept constant by opening or shuting-off by activating a threshold value. The combination of self regulating properties with the electronic controllability via the thermal interface allows an external intervention. For automatic function microvalves with an actuator chamber are of particular interest, because this chamber can host nearly every smart hydrogel.

For possible medical applications the interactions of the hydrogel actuator with fluids, such as blood, have to be investigated. Most of the hydrogels are biocompatible but additional tests have to be performed. Hydrogel materials may serve then as a basis for the development of new automatic intra- or extracorporeal drug delivery systems. If the actuator and the medium materials are not compatible a semipermeable membrane or an auxiliary circuit have to be used. Of course, this would drastically effect the valve setup as well as the time and actuatoric behavior of the hydrogels.

Electronically controllable microvalves as toggle elements for microfluidic processors can be developed. By appropriate combination of these microvalves different operations can be realized. The parallel connection of two microactuators with different sensitivities results in a mixer (one exit, two inlets) or a flow sorter (one inlet, two exits), connection in series leads to a comparator. In particular the processor functions such as flow switching, sample injecting or dispensing, mixing and sample separation are destined to be the future for microvalves. Due to their properties hydrogel based microvalves are employed for the handling of small liquid volumes as well as liquids containing particles. A microscopy processor for the observation of the response of living cells to supplied reagents is currently under construction.

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