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A bi-polymer micro one-way valve

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Abstract

We have developed an in-plane bi-polymer check-valve for controlling microfluidic flow and preventing contamination between solutions by utilizing the elastic force of a swollen hydrogel. The valve was created using microfluidic tectonics, a fabrication procedure that allows construction of microscale components and autonomous systems using liquid-phase photopolymerization and *in situ* fabrication. The valve is composed of rigid parts (poly isobornyl acrylate) that provide a base frame, and compliant parts (hydrogel) that seal off the channel. The rigid part was fabricated by filling a polycarbonate cartridge with the isobornyl acrylate based prepolymer followed by UV light exposure through a photomask forming a chamber. To obtain well-defined chamber walls, a double exposing method (first exposure under lower UV dosage, then second exposure after filling the formed channel with DI water) was applied. Next, the chamber was filled with the hydrogel prepolymer mixture and exposed to UV light through a valve mask to define the compliant component of the device, resulting in an in-plane bi-polymer structure. The valve is actively assembled *in situ* providing precise sealing using low resolution lithography fabrication methods. Valve performance can be adjusted by varying the device geometry. Due to its in-plane structure and *in situ* fabrication process, microfluidic devices incorporating microvalves can be designed and fabricated conveniently.

Keywords: Hydrogel; In-plane; Microvalve; Photopolymerization

1. Introduction

A wide variety of microfluidic components have been developed thus far and portable bioassay or 'lab on a chip' continues to populate recent bioMEMS research [1–6]. To date, most practical functions (pumping, valving, sensing) have been demonstrated in a variety of forms. A growing issue is the complexity of the platform with respect to the ability to manufacture microfluidic systems. From this perspective, devices made using two-dimensional or in-plane geometry provides advantages over devices made using multi-layered fabrication. Along with the simplified fabrication process, one of the important benefits of an in-plane structure is adaptability. Unlike a multi-layered structure, planar components can be freely arranged in any location by simply changing the photomask and thus, provides many design options to the researchers in developing microfluidic systems.

A microvalve is an important component in several micro assay devices for many reasons, including preventing contamination between reagents and provision of precise amounts of reagents. Thus, many researchers have been interested in developing a micro check-valve. Wang and Tai developed a check-valve based on flexible Parylene membrane with good sealing for operation under relatively low pressures (20–40 kPa) [7]. While this valve can provide good valving function, it is not easy to merge concurrently with other components because of its multi-layered design and hybrid composition. Nguyen *et al.* also developed a micro check-valve using a valve plate and spring structure made of SU-8 [8]. Although, it is monolithic, their valve also has limitation in developing a device incorporating many components due to its multi-layered structure. Seidemann *et al.* introduced an in-plane micro check-valve design, which has a valve head and spring body made from SU-8 [9]. However, sealing difficulty is expected for the valve due to the rigidity of SU-8 and its design. To overcome the sealing difficulty, alternative fabrication technology incorporating compliant materials or appropriate polymers is recommended. Previously, our research group has introduced the use of functional microfluidic

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structures made from hydrogels, which are compliant materials [10]. Recently, Hasselbrink *et al.* created a micro passive check-valve, which uses a piston made of nonstick polymer in a silica capillary [11]. It has fast response times and good sealing; however, the minimum actuation pressure limits its application operating in low pressure.

In addition to the difficulty of fabricating multi-layered structures (often modeled after existing macro scale valves), the complexity of the multi-step fabrication processes gives rise to increased likelihood of defects. The potentially high failure rate and lengthy fabrication time result in fabrication costs that can be prohibitive for disposable or portable microfluidic devices.

After reviewing the above, the criteria for an improved micro check-valve can be summarized as: (1) structurally simple, (2) good sealing, (3) quick restoration of valve head or plate, (4) low leakage rate and (5) convenience to incorporate with other microfluidic components.

In this paper, we present the description and characterization of a hydrogel micro check-valve, which can be made conveniently and rapidly using an in-plane structure and *in situ* fabrication [12]. The valve provides precise sealing by taking advantage of a compliant material (hydrogel) and uses minimal space compared to the whole device due to the in-plane design. The in-plane structure also provides good adaptability (*i.e.*, the hydrogel valves can be located anywhere they are required). It has quick restoration of the valve head as a result of the elastic force created by swollen hydrogel [13]. The valve is robust and can be fabricated concurrently with other components because of its sturdy and simple geometry. The micro check-valve is assembled actively by the swelling of the hydrogel and operates passively as a one-way valve.

2. Materials and methods

A simple microfluidic device containing a straight channel, valve and inlet/outlet was designed and fabricated to test the valve function. Microfluidic tectonics was used in fabrication and a complete device can be constructed in approximately 30 min. The following sections describe the design and materials used in the fabrication of the device.

2.1. Valve design

The valve is composed of two different polymers—PIBA (poly isobornyl acrylate) and HEMA (hydroxy–ethyl–methacrylate). PIBA is used to construct the rigid parts of the device (microfluidic channels, rigid valve components) that serve as a base frame while the hydrogel forms the compliant parts that provide sealing of the valve. The hydrogel inherently possesses flexibility and an elastic force when it is swollen. These two materials are combined *in situ*, with appropriate geometric dimensions, to create a functional valve.

The base frame includes a valve neck and post as shown in Fig. 1. The valve neck has the same profile as the valve head such that the contact surface area is maximized. The valve post serves to support the valve body as well as confine the swelling direction of the valve body such that the valve body swells toward the valve neck. The valve head makes contact with the valve neck as the elastic force of the valve body pushes the valve head.

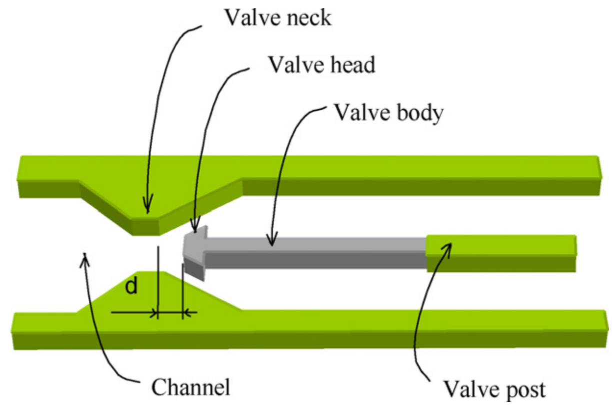

Fig. 1. Schematic representation of the hydrogel check-valve. The valve neck has the same contour profile as the valve head such that the contact surface is maximized. The valve post serves to support the valve body as well as confine the swelling direction of the valve body such that the valve body swells toward the valve neck. The valve head makes contact with the valve neck as the valve body pushes the valve head with the spring force. Pre-swelling gap ‘d’ represents the distance that the valve head travels to make contact with the valve neck.

The valve described in this paper is a normally closed check-valve. The valve opens when forward flow is sufficient to create a flow path between the valve head and valve seat. The force generated by the pressure from the forward flow causes the valve head to retract once this force becomes greater than the elastic force of the hydrogel. Once the forward flow stops, the elastic force of the hydrogel valve body restores the initial state (*i.e.*, closed state) by pushing the hydrogel valve head toward the valve neck. Under reverse flow, the valve geometry causes the valve to become more tightly sealed to stop flow. The continuing reverse flow increases pressure of valve chamber, which pushes valve head toward the valve neck, a rigid confinement. The sealing pressure between the valve neck and valve head can be tuned by adjusting the pre-swelling gap, which is the distance the valve head travels to make good contact with the valve neck during swelling (see Fig. 1). If the pre-swelling gap is reduced, the sealing pressure is increased and vice versa.

2.2. Fabrication of microfluidic channel and valve

Channel structures, including rigid parts of the valve (valve neck and post), are fabricated from the IBA-based prepolymer (see Table 1) using the tectonics platform, a fabrication procedure for constructing microscale components and autonomous systems using liquid-phase photopolymerization [14]. Since the

Table 1
PIBA mixture for channel and rigid part of the valve

Material	Ratio (in weight)
Isobornyl acrylate (IBA)—monomer	1.9
Tetraethylene glycol dimethacrylate (TeGDMA)—crosslinker	0.1
2,2-Dimethoxy-2-phenyl-acetophenone (DMPA)—photoinitiator	0.06

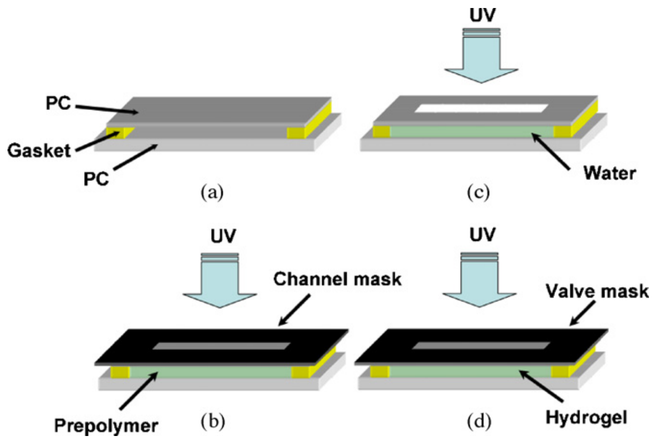

Fig. 2. Fabrication procedures and structure of the micro one-way valve. (a) A polycarbonate sheet with an adhesive gasket is attached to a polycarbonate plate and filled with the IBA-based prepolymer. (b) A film photomask with the channel and rigid part of valve is placed on top of the device and exposed to UV light for 12 s using a wavelength of 365 nm and an intensity of 12 mW/cm^2. (c) After removing the unpolymerized prepolymer with a vacuum pump, the channel was washed with 4 mL of deionized (DI) water. After washing, the channel was filled with DI water and exposed again to UV light (12 mW/cm^2, 12 s) to obtain well-defined channel walls and rigid part of valve. (d) The hydrogel mixture was flowed into the channel network. A photomask with the valve body and head was placed on top of the cartridge and exposed to UV light (25 mW/cm^2, 60 s).

valve neck and post are defined in the same photomask as the channel network, all three components are polymerized at the same time.

The surface property (*i.e.*, hydrophilicity or hydrophobicity) of the channel is a significant factor that affects the performance of the hydrogel valve body. If the property of the top and bottom surfaces are different (*i.e.*, glass and polycarbonate surfaces), the swelling movement of the hydrogel valve body is asymmetric, resulting in incomplete swelling. Thus, it is important to use the same material on the top and bottom for proper function of the hydrogel valve. This condition can be easily achieved in the microfluidic tectonics platform by using the same polycarbonate layers for both the top and bottom. The hydrophobicity of the polycarbonate surface is made apparent by the high contact angle (103°). A glass top and bottom surface also provides identical surface conditions; however, it is not recommended due to the hydrophilic property of glass. Hydrophilicity of glass surfaces increase the frictional stress between the glass and hydrogel, and thus, result in slower and/or shorter swelling distances [15]. It is possible to use glass if the surface is modified to be hydrophobic using surface treatment [16].

The detailed fabrication procedure and final structure of the device are described in Fig. 2. A polycarbonate cartridge with an adhesive gasket at the boundary (125 μm thick for corresponding channel and hydrogel thickness; Grace Bio-Labs, Inc., Bend, OR) is attached to a polycarbonate sheet, previously fixed on a microscope slide glass. The cartridge cavity is filled with the IBA-based prepolymer. The film photomask with the channel and rigid part of valve is placed on top of the device and exposed to UV light (EXFO Acticure 4000, Mississauga, Ontario, Canada) for 12 s using a wavelength of 365 nm and intensity of 12 mW/cm^2. After removing the unpolymerized monomer solution material with a vacuum pump, the channel was flushed with 4 mL of deionized (DI) water. After washing, the channel was filled with DI water and exposed again to UV light (12 mW/cm^2, 12 s) to obtain well-defined channel walls and rigid part of valve. This is called double exposing method and is described next.

2.2.1. Double exposing method

To obtain well-defined channel walls, the UV exposing and washing should be done very carefully as these parameters affect the final channel characteristics. Overexposure results in smaller channel size than mask dimensions as well as difficulty in subsequent washing. In liquid-phase photopolymerization, this is of critical concern for small channel sizes (*e.g.*, 200 μm wide), which generate more fluid resistance during washing. The increased viscosity of the channel region caused by the overexposure makes subsequent washing more difficult. Underexposure results in bigger channel size than mask dimensions. Thus, it is important to find an appropriate exposure condition for each target channel dimension.

During liquid-phase photopolymerization, a lower UV intensity is found to be better. The lower UV intensity facilitates washing by affecting the viscosity of channel region to a lesser degree. However, the channel wall is likely to be in an underpolymerized state. Further UV exposure does not fully polymerize the channel wall since the presence of oxygen inside channel prevents the polymerizing reaction. Oxygen reacts with many free radicals at a much faster rate than monomers do, resulting in significant retarded polymerization [17]. There are several ways to avoid direct contact with oxygen in the channel and the simplest way is to fill the channel with DI water. To demonstrate the double exposing method, two different devices were exposed to UV light (12 mW/cm^2 for 12 s) for their first exposure. After washing, device 'B' was exposed to UV light for an additional 48 s under dry channel condition while device 'A' was exposed to UV for an additional 12 s under wet channel conditions, *i.e.*, after filling the channel network with DI water to remove air inside the channel. The completed devices were washed with methanol and dried with nitrogen gas. To examine the cross-section of the channel wall, the two device cartridges (with and without the double exposing method) were carefully opened and pieces of the channel wall were taken out. The sample pieces were qualitatively compared under a stereo microscope.

The hydrogel prepolymer mixture was flowed into the channel network. The mask representing valve body and head was placed on top of the cartridge. The mask was aligned to have a designated pre-swelling gap and exposed to UV light (25 mW/cm^2, 60 s). After removing the unpolymerized hydrogel material with a vacuum pump, the channel was washed with 4 mL of DI water. The hydrogel mixture used for the valve is shown in Table 2. DI water was flowed into the channel network and allowed it to swell for 20 min.

2.3. Valve test

One of the well-known methods to characterize a valve is to draw the characteristic curve which compares the back-

Table 2
The hydrogel recipe used for the body material

Material	Ratio (in weight)
2-Hydroxyethyl methacrylate (HEMA)—monomer	1
Ethyleneglycol dimethacrylate (EGDMA)—crosslinker	0.03
2,2-Dimethoxy-2-phenyl-acetophenone (DMPA)—photoinitiator	0.03

ward and forward flow rate versus pressure. The flow rates of two devices, which have two different pre-swelling gaps (*i.e.*, 350 and 400 μm) were measured using the flow rate measurement method developed previously [18]. The measurement setup includes two pressure sensors and a glass tube, which provides series resistance. The principle of the method is based on the fluidic equivalent of Ohm's law. If a known value of series resistance is used, it is possible to obtain the flow rate of a device connected to the series resistance by measuring the pressure. A water column provides the constant inlet pressure and a designated level of pressure is obtained by adjusting the air pressure regulator. To verify the repeatability of the valve, the flow rate of a device, which had the pre-swelling gap of 400 μm was measured during a period of 50 h. This same device was used to evaluate the response time of the valve using the same aforementioned experimental setup.

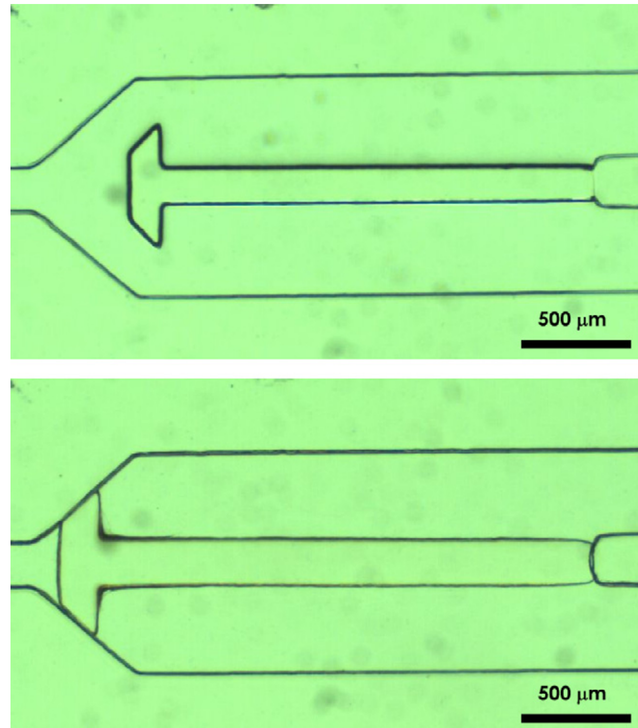

Fig. 3. A completed hydrogel check-valve before (top) and after swelling (bottom). DI water was flowed into the channel network and the device was put in a Petri dish for swelling under ambient room temperature for 20 min.

3. Results and discussion

The compliant valve head for sealing and flexible body structure to provide an elastic force for restoration of valve head produce a practical micro check-valve when they are combined. The valve head was able to seal the channel as long as the pressure never reached the operating pressure. Once the pressure of the inlet channel reached the operating pressure, the valve opened and bypassed the fluid. When the inlet pressure was released, the valve head reverted to the sealing position. Hence, the contamination by the backward flow was prevented. In the following sections, the characteristics of the individual experiments are described.

3.1. Valve design and fabrication

Fig. 3 shows the completed check-valve before and after the hydrogel swelling. The valve design was optimized to maximize the usage of the inherent elastic force of the swollen hydrogel. The valve head should not be thicker than valve body, else swelling was incomplete because of increase in frictional stress stemming from the wider contacting surface of the valve head. The greater frictional stress of the valve head limited the longitudinal swelling of the valve body toward valve neck. The valve body confined between the valve head and post buckled and thus, failed to make a functional check-valve.

A double exposure polymerization procedure was developed to improve the dimensional resolution and repeatability of the fabrication process. Fig. 4 shows two devices that were exposed to UV light under different conditions. The interface between the polymerized and unpolymerized region typically had an underpolymerized property after the first exposure (Fig. 4a). The underpolymerized property was thought to be caused by two factors: *i.e.*, light interference at the mask edge and the transition of the polymerization reaction at the interface. This boundary region could be easily removed by solvents during the washing process, but then results in a channel geometry which is larger than the film photomask. Instead, device 'A', which was exposed to UV light under wet channel conditions, shows significant reduction of the underpolymerized region (Fig. 4b) compared to device 'B' (Fig. 4c). Later, device 'B' was also filled with DI water and exposed to UV light under similar conditions as device 'A'. The resulting device 'B' was identical to device 'A' (Fig. 4d). A smooth and straight channel wall was observed at the cross-section for the device fabricated using the double exposing method (Fig. 5-top). Channel walls showed an undercut profile, which is a typical characteristic of photolithography using a negative photo-resist [19]. There are cleavages at the corner of the channel wall fabricated without the double exposing method (Fig. 5-bottom). The cleavages may both cause the leakage of the valve and be the origin of the delamination when a high pressure flow is introduced inside the microchannel. The perpendicularity of the channel wall was important to create a well-sealed hydrogel check-valve. Also, a fully polymerized valve neck helped the operation of the hydrogel valve. The channel network fabricated without the double exposing method left the surface of the valve neck in an underpolymerized state, which

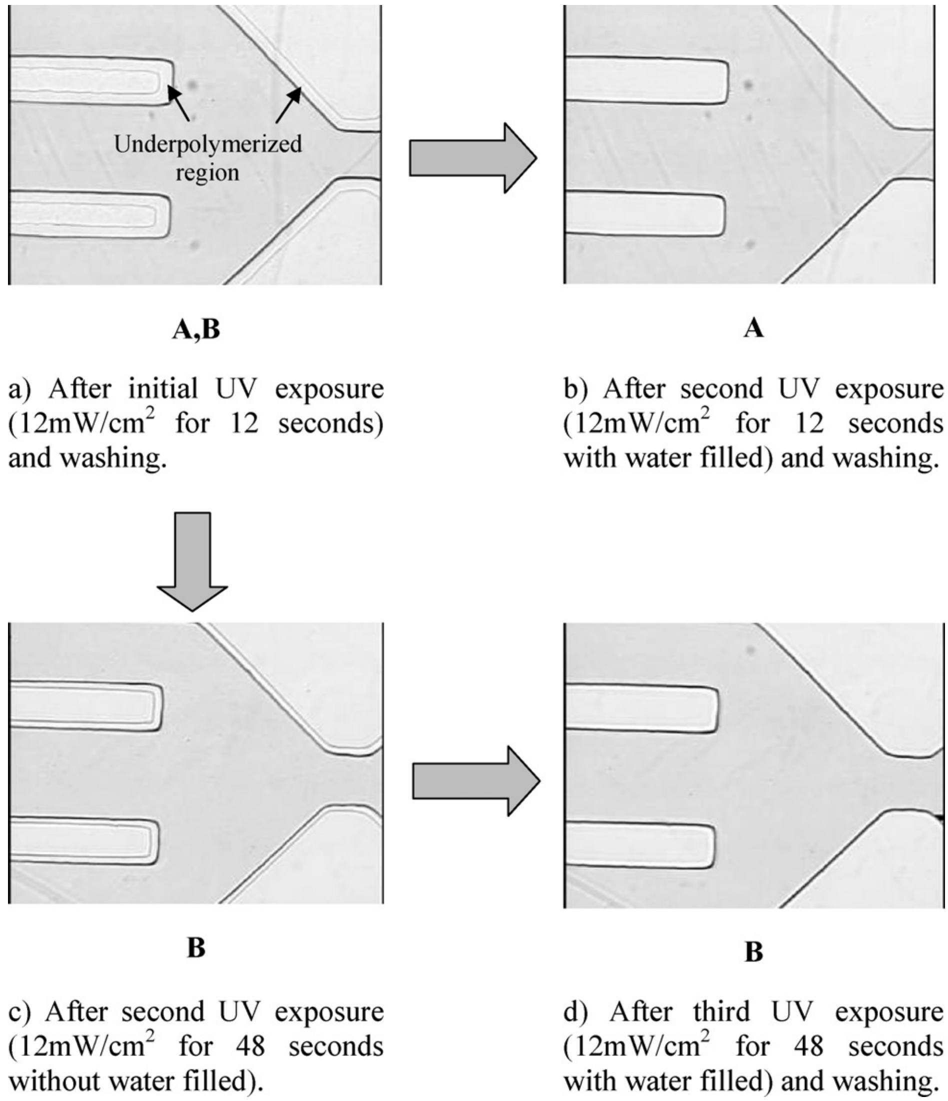

Fig. 4. (a–d) Pictures showing different UV light processing treatments during the fabrication of a microchannel. Device 'A', which was exposed to UV light under wet channel conditions, showed significant reduction of the underpolymerized region after a second exposure.

became bound with the valve head of the hydrogel and resulted in an inoperable valve.

The identical physical property of both the top and bottom surfaces of the hydrogel valve was another important factor of the valve operation. With different surface properties (*i.e.*, glass and polycarbonate), the hydrogel valve head and body easily flipped and collapsed due to the different interfacial properties at each surface. It is believed that the hydrophobicity of the polycarbonate surface helps achieve maximum swelling of the hydrogel valve by reducing the frictional force at the contact interface [15].

The hydrogel check-valve used the elastic force of the swollen hydrogel body. Thus, the swelling ratio of the hydrogel was a dominant parameter in determining the magnitude of the restoration force. If the valve head was fabricated too close to the valve neck for realizing higher sealing pressures, the valve body would buckle between the valve neck and post due to excessive swelling (see Fig. 6-top). If the valve head was fabricated too far back from the valve neck, the valve head would not make sufficient contact with the valve neck (see Fig. 6-bottom). Normally, the valve head was fabricated to have a pre-swelling gap of 350–400 μm. The average swelling distance was 450 μm, and thus, the compressed length of the hydrogel body ranged from 50 to 100 μm for the current design.

3.2. Valve test

The resulting characteristic curve of the valve is shown in Fig. 7. The negative side indicates the backward flow data and the positive side shows the forward flow data. As expected, the resistance of the valve (*i.e.*, the inverse of the slope of the flow rate) becomes larger when the pre-swelling gap 'd' becomes smaller. Thus, it is possible to design check-valves, which have different operating pressure by adjusting the pre-swelling gap. Various microfluidic applications require a wide range of flow regulation. The range of the operating pressure may be different depending on the application and pressure source (*i.e.*, pump). A microfluidic HPLC system may typically require high pres-

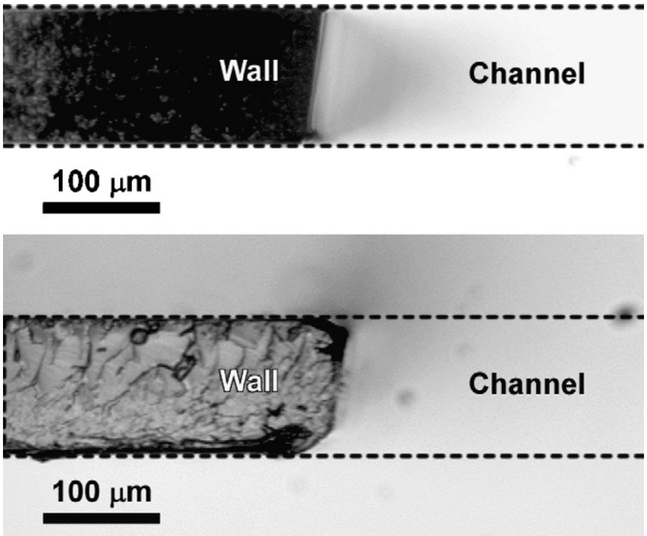

Fig. 5. Cross-sectional view of channel walls fabricated with (top photo) and without (bottom photo) the double exposing method. Both channel walls show an undercut profile, which is a typical characteristic of photolithography using a negative photo-resist. There are cleavages at the corner of the channel wall fabricated without the double exposing method. The cleavages may both cause the leakage of the valve and be the origin of the delamination as well.

sure flow regulation and a valve operating at a pressure greater than 2 MPa. Other applications use lower or modest pressure ranges (0.1–200 kPa) [20]. A device which uses a manual pressure source, *e.g.*, thumb actuated pump, may require a valve,

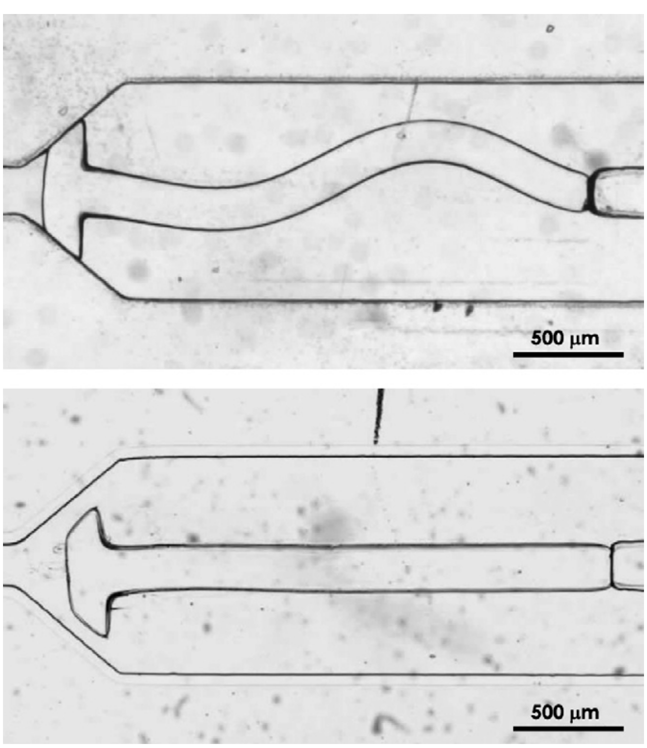

Fig. 6. Examples of failed hydrogel check-valves. If the valve head is fabricated too close to the valve neck for realizing higher sealing pressures, the valve body buckles between the valve neck and post due to the excessive swelling (top). If the valve head is fabricated too far back from the valve neck, the valve head is unable to make sufficient contact with the valve neck (bottom).

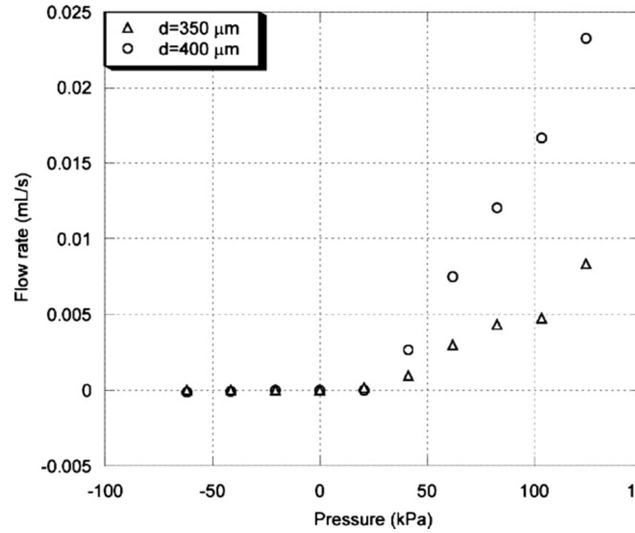

Fig. 7. Characteristic flow rate curve vs. pressure of the hydrogel check-valve. The negative side indicates the backward flow data and the positive side shows the forward flow data. As expected, the resistance of the valve (*i.e.*, the inverse of the slope of the flow rate) becomes larger when the pre-swelling gap ‘d’ becomes smaller.

which operates at a pressure less than 172 kPa [12]. The valve developed in this paper will be appropriate for applications that have pressure sources ranging up to 200 kPa.

When the backward flow was applied, the leakage was negligible with newly made valves. However, after several operations, a small amount of leakage was observed and verified through the repeatability test (see Fig. 8). As the operation was repeated, the sealing pressure or resistance of the valve for the forward flow does not change significantly whereas leakage is observed for the backward flow, especially at low pressures. However, the leakage after repeated operations is not a concern for disposable and portable device applications since the valve will

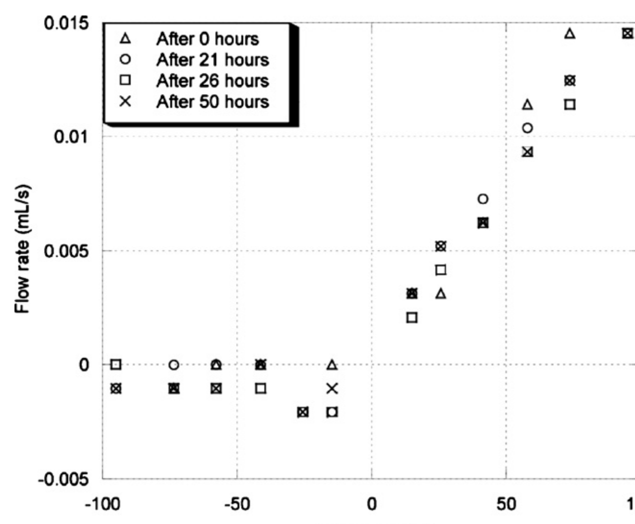

Fig. 8. The repeatability test of the hydrogel check-valve, which has the pre-swelling gap of 400 μm. As the operation repeats, the sealing pressure or resistance of the valve for the forward flow does not change significantly whereas there is leakage for the backward flow, especially at low pressures.

only operate once. Repeatability was not tested with multiple devices; however, because the slope of the graph does not change significantly over the relatively long experimental time (50 h), it suggests that for a given pre-swelling gap this behavior is deterministic to a considerable extent. The response and restoration times of the valve, which had 400-μm swelling gap were 0.51 and 0.47 s, respectively. The valve's performance was sufficient to prevent contamination between solutions for purposes of a portable ELISA device [12]. To investigate the leakage, the cross-section of the valve neck area was examined under a confocal microscope with fluorescein-dyed water applied at a flow rate of 50 μL/min using a syringe pump. The major leakage flow was observed at four corners of the valve head and neck contacting area. The leakage was thought to be caused by incomplete sealing at the sharp corners after repeated operations in spite of the flexibility of the hydrogel. The leakage can be decreased if either circular channels or channels which have round corners are used [11]. Currently, technologies to make curved structures are emerging and could be used to enhance the performance of the hydrogel valve presented in this paper [21,22].

4. Conclusion

An in-plane micro check-valve was made using hydrogels and PIBA. The elastic force generated from a swollen hydrogel was utilized to provide a sealing pressure of the valve head. The ability of the compliant hydrogel to seal allows the use of a simple low resolution fabrication process. To obtain well-defined channel network, a double exposing method was applied which resulted in a smooth and straight channel wall. The fabrication process was rapid and the device was fabricated within 30 min. The in-plane structure of the hydrogel valve makes it easy to design and/or modify the valve so that the valve can have appropriate operating pressure. Using microfluidic tectonics, the fabrication process was able to be done without a clean room facility [14], thus enabling easier and cost-saving development of microfluidic systems.

One of the key features of the hydrogel valve is its adaptability and ease of integration into microfluidic systems. Due to the in-plane structure and capability of *in situ* fabrication, the hydrogel valve can be made in any location as long as there is sufficient space to hold the valve structure. The adaptability will provide options in future developments of microfluidic systems. For example, if one needs to add several valves to the device, a user must only modify the check-valve film photomask, which is usually a simple 'copy and paste' task. Other additional changes (*e.g.*, modifying other layer mask, adjusting process steps) are not required. A user may also just use a single valve mask to add valves at arbitrary locations in pre-fabricated channel networks by repeating the *in situ* valve liquid-phase photopolymerization fabrication process sequentially.

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Biographies

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