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An active valve incorporated into a microchip using a high strain electroactive polymer

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ABSTRACT

Recently, various kinds of microvalves have been developed in the field of micro total analysis systems. Most are pneumatic or piezo electric valves, which require significant space on a microchip and hence make the system complicated. This study reports the use of an electroactive polymer-based microvalve that occupies only a small space on a microchip. An electroactive polymer membrane sandwiched between soft electrode sheets was placed on a dome-shaped diaphragm. The polymer actuator was installed in a glass microchip and the valve function was demonstrated. First, the displacement of the diaphragm was measured without fluid, and sufficient displacement (over 50 μm) was obtained for valve closing. Second, flow in a linear microchannel was stopped and then restarted by the valve. The flow in the microchannel produced by the constant pressure from a microfluidic controller (1.0 kPa) was completely stopped by applying a 50 V/μm electric field. This valve functioned well at pressures up to 4.0 kPa. The response time was about 0.7 s, similar to the time required for piezo electric actuator valves in channels of this size. This type of valve is very suitable for portable devices filed because of its compactness.

1. Introduction

In the last several decades, integrated chemistry, some of which is often referred to as either as a micro total analysis system (μ-TAS) or as a lab-on-a-chip, has generated substantial interest due to the desirable characteristics of these systems, which include reduced reagent consumption, space requirements and analysis times. Microdevices exhibit several distinct performance advantages including short diffusion distances, low Reynolds numbers, high interface-to-volume ratios (specific interface areas; solid/liquid or liquid/liquid) and small heat capacities. Exploiting these advantages, many researchers have developed novel methods to use microdevices for analysis, synthesis, and studies of cells [1–5]. Here, careful design of valves is extremely important in order to manipulate small volume samples.

Currently, the most widely used valves on a microchip are pneumatic. The concept of pneumatic valves that could exploit the flexible elastomeric property of polydimethylsiloxane (PDMS) was first described by Quake et al. [6,7]. Recently, large-scale integration has also been realized [8,9]. Although this type of valve is very sophisticated, it requires a compressor or vacuum pump to deliver air to the microdevice. This system is very large compared to a microchip and is also often noisy. Furthermore, air channels are required to send air to the valve. Such a channel system makes the microchip thick and complicated. Microvalves may also be designed using piezo electric technology [10,11]. By using a bimorph type piezo actuator, large displacement was achieved in these earlier studies. This makes the system compact and silent, but the actuator itself is still large compared to the microchips. These limitations make the design of more integrated devices such as three-dimensional integration or portable devices challenging.

Here, we propose application of an electroactive polymer as a valve component, despite the use of a piezo actuator in the microdevice. Electroactive polymers have a higher strain against the size of itself, compared with piezo electric actuator [12–14]. Moreover, valves can be controlled more directly using the electroactive polymer compared with other types of valves such as pneumatic, laser based [15], or thermo-responsive polymer based ones [16]. Although there have been some reports of microfluidic devices using this type of polymer, such as a tubular pump [17] or an on-chip pump [18], these devices do not include a stop valve function. In order to add this functionality, a device to close the channel is required and this may be difficult to fabricate compared with the pump structure.

The objective of this study was to develop a stop valve on a microchip using an electroactive polymer. First, a new structure

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for a stop valve was designed and then fabricated, and displacement of the diaphragm without fluid was measured. Second, the functionality of the stop valve installed on a glass microchip was demonstrated.

Fig. 1 shows the structure and actuation principle of the stop valve. As shown in Fig. 1A, the electroactive polymer becomes thin and elongated in the horizontal direction when a voltage is applied. The balance of applied stress (P) against the restoring force is represented by Eq. (1):

$$P = \varepsilon_0 \varepsilon_r \left(\frac{V}{d_0}\right)^2 \frac{1}{(1-\gamma)^2} = E\gamma \tag{1}$$

where ε_0 is permittivity in a vacuum, ε_r is relative permittivity in the polymer, V is the applied voltage, d_0 is the thickness of the polymer, E is Young's modulus of the polymer, and γ is the compressive strain of thickness direction. When the strain γ is small, P is approximately proportional to V^2. The valve exploits this property. The electroactive polymer was sandwiched between soft electrodes and placed on a dome-shaped diaphragm made of silicon rubber. Because the electroactive polymer cannot elongate in the horizontal direction on this structure, it elongates in a diagonal direction in order to close the penetrate hole in a chamber within a microchip when a voltage is applied as indicated in Fig. 1B. This actuation mechanism of the electroactive polymer membrane was verified previously [19]. The microchip is composed of two layers made of glass. The microchannel fabricated substrate (top layer), with penetrate holes for fluid flow, is fused to a plain glass slide (bottom layer). The jig of valves is composed of two layers fashioned from acrylic resin. The bottom layer (0.6 mm thickness) has a 5 mm diameter circular hole to fit the diaphragm elastomer, and a groove for a wire was also fabricated. The top layer (1.0 mm thickness) has a 3.5 mm diameter circular hole, and it was used to suppress the displacement of the electroactive polymer in the radial direction by holding down the polymer with the diaphragm. This layer is also required to protect the wire for safety reasons. To demonstrate the valve function, a linear microchannel with a valve structure in the center of the channel was designed as shown in Fig. 1C.

2. Experimental

2.1. Device fabrication

The microchip was made of glass and the microchannel was fabricated using a wet etching method [20]. Briefly, a mechanically polished Tempax glass substrate (bottom plate) was prepared and annealed before use. Next, Cr and Au layers were evaporatively deposited on the substrate under a vacuum, a positive photoresist was spin-coated on the metal, and UV light was exposed through a photomask. After that, the photoresist was developed and the metal layers were etched. The bare glass surface with the microchannel pattern was then etched with HF solution. After glass-etching, the remaining photoresist was removed.

After etching, holes for fluid flow were drilled on the microchannel fabricated glass substrate (700 μm thickness). For penetrate holes for valves, 100 μm diameter holes were made from the opposite side to the microchannel fabricated side, to a depth of 500 μm, using an end mill and a diamond coated drill (FSK). Next, 400 μm diameter holes were made from the microchannel fabricated side to a depth of 200 μm depth to avoid breaking the drill. For the microchip inlet and outlet holes, 400 μm diameter holes were made at the edge of the microchannels. Finally, the substrate was sealed with another plain Tempax glass plate (700 μm thickness), and the two plates were thermally bonded in a furnace. The fabricated microchip and the penetrate holes are shown in Fig. 1A–C. The width and depth of the microchannel were 300 μm and 150 μm, respectively.

The electroactive polymer film was produced from the raw materials [21]. At first, the hydrogenated nitrile rubber was dissolved in the solvent. Into the solution, a cross-linked agent was added to be mixed.

The mixture solution was applied on a plastic film with mold release properties, dried and heated at 150 °C for about 60 min to obtain the dielectric film. The dielectric film had a thickness of about 18 μm.

Electrodes were formed by a mixture of an acrylic rubber with carbon black (carbon) to each of the front and back surfaces of the dielectric films. The constitution and process for production of the electrode film are as described above. The electrode had a thickness of about 5 μm.

These films can be patterned by molds and punching dies as Fig. 2D. Then, they are stacked by laminating and the plastic film was removed sequentially. Further, the actuator was fabricated as a valve with a silicon rubber diaphragm (Young's modulus: 2 MPa) that is shown in Fig. 2E made by mold. The overall diameter of the actuator was 5 mm, the movable membrane diameter was 3.5 mm, and the thickness of the diaphragm was 300 μm. The holes and grooves of the acrylic resin jigs were fabricated by a milling machine.

2.2. Experimental set up

The fabricated microchip was set in the jig by screws and the system was fixed on a microscope stage (Fig. 2F). As shown in Fig. 1C, just one side of the microchannel was used. Fluid was controlled by a microfluidic flow control system (MFCS, Fluigent), and the pressure was set to constant. To visualize fluid flow, micro tracking particles were used, as described in previous reports [22–24]. Fluorescent spherical polystyrene particles (Fluoro Spheres, 2 μm diameter, Molecular Probes, Invitrogen) were dispersed into the fluid (100 times diluted by distilled water from the original solution) and flows at the outlet microchannels were observed *in situ* using a fluorescent microscope (IX-71, Olympus) with an objective lens (10×, 0.30-NA) and GFP filter set. The microscope was focused on the center of the microchannel and the image was recorded using interfaced software (cellSens, Olympus) through a CCD camera (DP72, Olympus). The used power source for electroactive polymer was a function generator (33220A, Agilent) and a high voltage amplifier (HEOPS-10B2-LC1, Matsusada Precision, maximum voltage: 10 kV). All experiments were carried out at room temperature.

3. Results and discussion

3.1. Diaphragm displacement measurement

In order to verify the deformation of the diaphragm, diagonal displacement of the diaphragm membrane was measured. A laser displacement measurement system (LK-G3000/LK-G10, KEYENCE) was used, and the center part of the displacement was plotted. Displacements recorded in 50 V/μm and 60 V/μm fields are summarized in Table 1. If the voltage was increased above 70 V/μm, the valve was sometimes broken. Therefore, a field of just under 60 V/μm is the maximum recommended for stable use. To confirm the attachment of the diaphragm to the microchip, the displacement was also measured when the valve was not incorporated into a microchip. When the valve was incorporated into the microchip, displacement was according to the applied voltage. On the other hand, the displacement approached a plateau in another condition around 55 μm. This was because the bottom of the diaphragm had already reached the microchip. Therefore,