Lab on a Chip

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COMMUNICATION



# Elastomeric membrane valves in a disc†

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**We present elastomeric membrane valves integrated into a centrifugal microfluidic platform for precise control of fluid on a disc. The amount of the fluid passing through the valves, which depends on the rotating speed of the disc and the membrane thickness, has been characterized, and could be precisely controlled by tuning the disc motion.**

Lab-on-a-disc, which applies centrifugal force to pump fluid, has been a powerful tool for biological and chemical analysis due to its capability of integrating and automating all the processes into a disc-shaped device with simple, size-reduced, and cost-efficient instrumentation.[1,2] For the ultimate goal of sample-to-answer system, it is essential to precisely control the movement and the position of fluid throughout the process.[3,4] For that purpose, valving is one of the most important functions, thus many types of valves have already been developed for centrifugal microfluidic platforms. There have been two large classes of valves for lab-on-a-disc: (*i*) passive types of valves based on the capillary effect and the surface properties of the disc materials,[5,6] and (*ii*) active types of valves based on the sacrificial materials.[7,8] The fluid transfer through the passive valves is simply controlled by the rotational speed, the channel design, and the surface properties of the disc, but the vapor migration or moisture absorption among the chambers containing liquid samples and reagents are unavoidable issues.[2] To deal with this problem, a sealing technique for liquid reagent storage has also been developed.[9] On the other hand, the active type sacrificial valves are gas-tight and the actuation is programmable independently of the location of the valves by selectively melting paraffin wax using laser irradiation for not only opening but also closing the individual valves. Although this elegant scheme allows us to carry out more complicated processes for practical assays,[10,11] it requires an additional light source for the actuation.

In this communication, we have introduced a novel passive type valve based on an elastomeric membrane integrated into a disc. A simple room temperature bonding technique was employed for irreversible bonding of a polydimethylsiloxane (PDMS) membrane to a thermoplastic plate.[12] We have investigated the flow rate through the membrane valve against the spin speed of the disc and the membrane thickness. Precise control of the fluid volume passing through the valves was demonstrated by controlling the spin program of the rotating disc. This study provides a unique method to integrate elastomeric membrane valves into a conventional centrifugal microfluidic platform, as well as a new concept of valving technique for centrifugal microfluidics.

Schematic diagram of the elastomeric membrane valves in a disc is shown in Fig. 1. At the closed state, two chambers—inner and outer—were physically separated by a wall which is in contact with an elastomeric membrane in the middle of the channel. The membrane effectively confined the transport of the fluid from the inner to the outer chamber at the normal state. When the disc was spun faster than a critical rotating speed, the elastomeric membrane was deflected due to the pressure increase, thus the valve was open and the fluid in the inner chamber flowed toward the outer chamber.

Fabrication process for the integration of the elastomeric normally-closed membrane valves into a disc is shown in Fig. 2. PDMS (Sylgard 184; Dow Corning, MI) and polycarbonate (PC) (I-Components Co., Ltd, Seongnam, Korea) were used as the top and the bottom layer of the disc, respectively. The top layer was fabricated by moulding PDMS on a PC master, which has been fabricated using a CNC milling machine (3D modeling machine; M&I CNC Lab, Osan, Korea). The elastomeric membrane was prepared by spin coating PDMS on a PC plate. The thickness of the membrane was adjusted by controlling the spin rate of the spin coater. After plasma treatment of the top PDMS layer and the spin-coated PDMS membrane, the top layer was bonded onto the membrane and peeled off. The holes for injecting fluids and ventilation were formed by punching through the PDMS assembly. The bottom layer of the disc was fabricated by milling a 5 mm-thick PC plate with a CNC milling

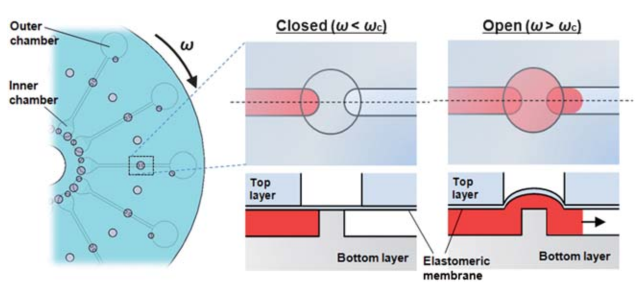


**Fig. 1** Schematic diagram of a centrifugal microfluidic platform integrated with elastomeric membrane valves. When the angular velocity of the disc, $\omega$, overcomes a critical value, $\omega_c$, the fluid in the inner chamber is transported to the outer chamber due to the deflection of the elastomeric membrane by increased pressure.

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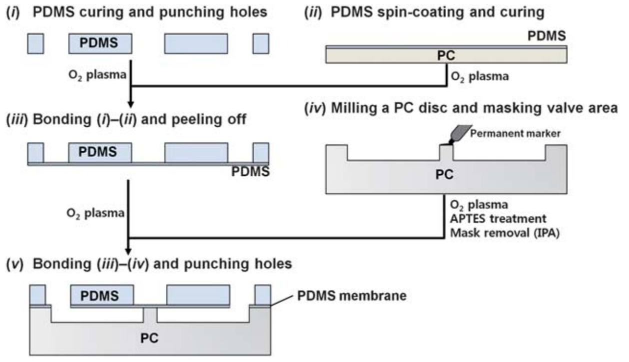


**Fig. 2** Fabrication process of elastomeric membrane valves integrated on a disc. A simple room temperature bonding technique based on APTES was utilized for irreversible bonding of a PDMS membrane and a thermoplastic plate.

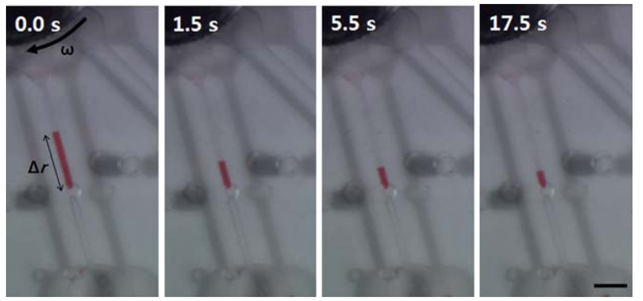


**Fig. 3** Captured images of the fluid passing through an elastomeric membrane valve, for which the thickness was 100 μm, integrated on a disc spinning at 700 rpm. Temporal change of the fluid volume remaining in the inner chamber was monitored by measuring the radial extent of the fluid, $\Delta r$, to analyze the flow rate. Scale bar = 5 mm.

machine. For irreversible bonding of the top PDMS and the bottom PC layers, a room temperature PDMS-thermoplastic bonding technique was utilized.[12] The 3 mm-diameter circular area for the valve, which should not be bonded but in contact with a membrane, was masked using a permanent marker (Namepen; Monami Co., Ltd, Yongin, Korea). The masked PC disc was treated with oxygen plasma, and placed in an aqueous solution of 1% v/v 3-aminopropyltriethoxysilane (APTES) (Sigma-Aldrich Corp., MO) for 20 min at room temperature. After the surface treatment, the ink mask was removed with isopropyl alcohol. Finally, the top PDMS layer with the membrane was treated with oxygen plasma, and bonded onto the APTES-treated bottom PC layer by keeping in conformal contact at room temperature for a few minutes. 10 μL of an aqueous red dye solution was injected into the inner chamber and the disc was centrifuged at 400 to 800 rpm using a spin-stand equipped with a programmable spinning motor (200 W Servo Motor; Yaskawa Electronic Corp., Kitakyusu, Japan). The movement of the fluid was recorded using a camera (Toshiba IK-TF5; Toshiba Corp., Tokyo, Japan) with a strobe light.

The flow rate through the elastomeric membrane valves in the disc against the rotating speed of the disc and the membrane thickness has been characterized. The volumetric flow rate through the membrane valve, $Q = \alpha\Delta P^4/\mu D^3$, where $\alpha$ is the geometric factor depending on the channel design; $\Delta P$ is the pressure which is defined as $\rho r\Delta r\omega^2$, where $\rho$ is the mass density of the fluid, $r$ is the average distance of the liquid element from the rotating centre, $\Delta r$ is the radial extent of the fluid, and $\omega$ is the angular velocity of the disc; $\mu$ is the dynamic viscosity of the fluid; and $D$ is the bending stiffness of the membrane, which is proportional to the cube of the membrane thickness, $h^3$.[13] In this study, the amount of fluid remaining in the inner chamber could be calculated according to the time by measuring $\Delta r$ in the straight channel shown in Fig. 3. Temporal change of the amount of fluid in the inner chamber against the spin rate of a disc and the membrane thickness was measured as shown in Fig. 4a and c, respectively. We calculated the average flow rate from 0 s to 0.5 s as the initial flow rate, and compared it with the values from the theoretical model. Here, more than 4 independent valves at different positions on a disc were selected for the measurement. The initial flow rate increased as the rotation speed of the disc increased from 400 rpm to 800 rpm (see Fig. 4b), while it decreased as the membrane thickness increased from 90 μm to 140 μm (see Fig. 4d). The initial flow rate was strongly dependent upon the rotating speed of the disc and the membrane thickness, and we could precisely control the initial flow rate by adjusting those parameters. These results were in good agreement with the theoretical model, in which the flow rate is proportional to $\omega^8/h^9$. As the rotating speed of the disc increased and the membrane thickness decreased, the volume of fluid in the inner chamber more rapidly decreased. The critical spin rate of a disc, at which the membrane valves start to open, also decreased as the membrane thickness decreased. Since the flow rate also depends on the fluid volume, or more precisely, the centrifugal pressure, the flow rate more rapidly decreased and reached the steady state, as the initial flow rate increased. At the conditions when the flow rate was very low, *i.e.* low rotating speed or thick membrane, the fluid was transported through the valves with almost constant flow rate for a long time. For example, when we rotated the disc integrated with 100 μm-thick membrane valves with a spin speed of 300 rpm, the fluid, of which the initial volume was 10 μL, was passed through the valves with a constant flow rate of about 8 nL $s^{-1}$ over 10 min.

When we stopped the disc, the valve was immediately closed and the flow was also stopped due to the stiffness of the membrane. Based on this fine open-to-closed actuation, we could precisely control the injection of the fluid from the inner chamber to the outer chamber as shown in Fig. 5. Here, the spin program was designed so that each spin step could release only 2 μL of sample. A disc containing 10 μL of liquid sample was spun for 40 s with a 40 s interval, repeated 5 times. For this purpose, different rotating speeds—420, 470, 550, 730, and 2000 rpm—were applied in each step, because the flow rate depends on the fluid volume remaining in the inner chamber. These results demonstrate a potential of the proposed elastomeric membrane for precise control of the liquid transfer by employing a feedback control system. In addition, mixing the released fluids was possible between each step at relatively low rotating speeds without additional inflow of the fluid.

The long term stability of this elastomeric membrane valve based on the PDMS and the thermoplastic materials could be a challenging issue. Even in the conventional capillary valves, the surface tension of the fluid significantly influences the burst pressure in practice. We characterized the membrane valves when the device surfaces are hydrophilic due to the APTES treatment during the PC–PDMS bonding process. After a longer period of storage time, however, the critical spin rate of a disc could be changed because the contact angle of the fluids on the surface becomes larger and the narrow channels for liquid transfer work like the capillary valves. To deal with this problem, we treated the surface with oxygen plasma to make it

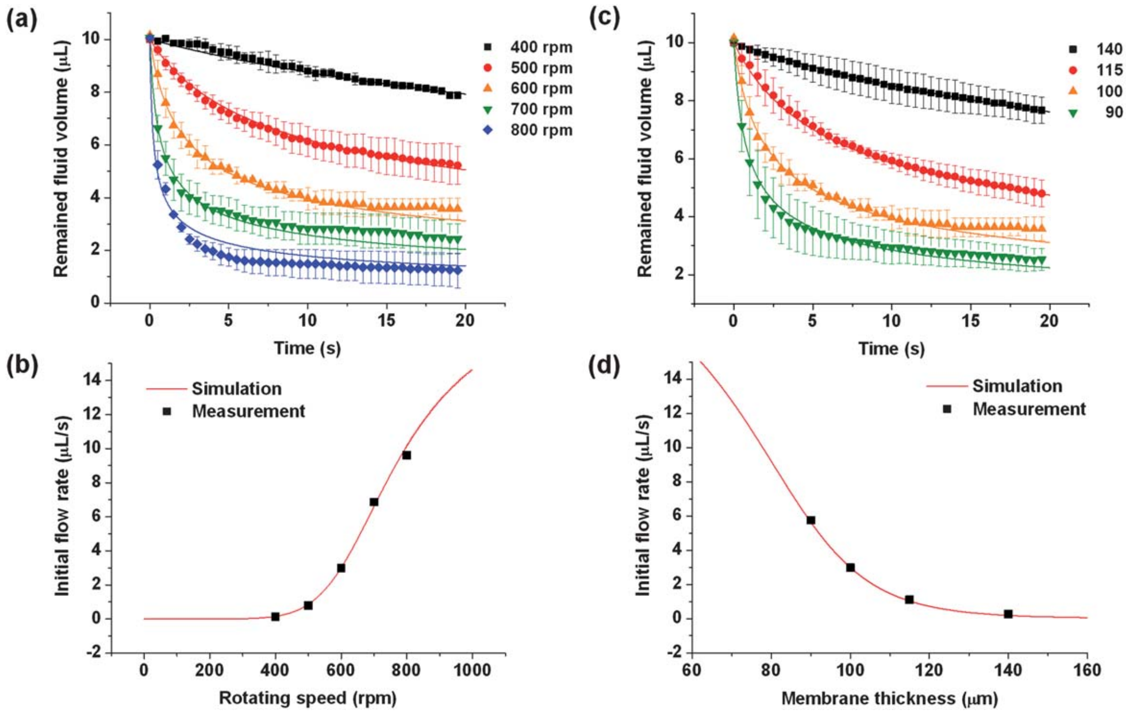


**Fig. 4** Characteristics of the elastomeric membrane valves. **(a)** Temporal change of the fluid volume confined by 100 μm-thick membrane valves against different rotating speed of a disc. **(b)** Initial flow rate – average flow rate from 0 s to 0.5 s according to the rotating speed. **(c)** Temporal change of the fluid volume remained in the inner chamber against the membrane thickness at 600 rpm. **(d)** Initial flow rate as a function of the membrane thickness.

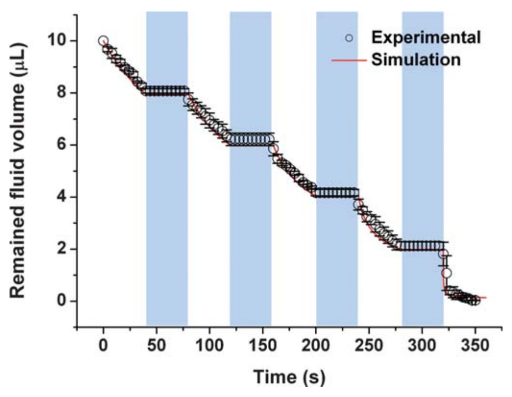


**Fig. 5** Precise control of the fluid volume passing through the valves. The disc was stopped for 40 s (colored regions) every 40 s. In each step, different rotating speed, 420, 470, 550, 730, and 2000 rpm, were employed. The membrane thickness was assumed to be 113 μm in the simulation model.

hydrophilic again when we performed the experiments after several days of storage. One of the approaches often used in a commercial product for the long term stability is to keep the device in a gas tight pouch. We can also modify the disc design for minimizing the effect of fluidic interaction with the channel walls.

In summary, the proposed novel valving method based on the elastomeric membrane valves would open a new way to control the fluids in a lab-on-a-disc platform. In addition, this alternative concept for the confinement and the regulation of fluids in a centrifugal platform has a worthy potential to be evolved towards multi-layered programmable or self-regulating lab-on-a-disc systems, by following examples of conventional microfluidic chips based on the similar kinds of elastomeric membrane valves.

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