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Article

Active and Precise Control of Microdroplet Division Using Horizontal Pneumatic Valves in Bifurcating Microchannel

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Abstract: This paper presents a microfluidic system for the active and precise control of microdroplet division in a micro device. Using two horizontal pneumatic valves formed at downstream of bifurcating microchannel, flow resistances of downstream channels were variably controlled. With the resistance control, volumetric ratio of downstream flows was changed and water-in-oil microdroplets were divided into two daughter droplets of different volume corresponding to the ratio. The microfluidic channels and pneumatic valves were fabricated by single-step soft lithography process of PDMS (polydimethylsiloxane) using SU-8 mold. A wide range control of the daughter droplets' volume ratio was achieved by the simple channel structure. Volumetric ratio between large and small daughter droplets are ranged from 1 to 70, and the smallest droplet volume of 14 pL was obtained. The proposed microfluidic device is applicable for precise and high throughput droplet based digital synthesis.

Keywords: microdroplet; division; bifurcating microchannel; horizontal pneumatic valve; PDMS

1. Introduction

In recent years, microdroplet technologies have been applied to wide research fields as effective methods to handle small samples [1]. Quantitative volume control, protection and transportation of the small samples are realized in a miniaturized platform. Chemical reactions and biological encapsulations are those representative examples [2–8]. In use of the technologies, volume control of the droplets is an important issue to maximize the microdroplets' advantages.

In order to achieve volume controlled droplet generation, design optimization of channel structure is required [9–11]. An automated flow rate change of immiscible fluids is also useful to control the droplet volume [12]. Furthermore, electrical and chemical methods are helpful to control the droplets' size [13]. However, a wide range of volume control with simple structure and simple operation has been shown to be difficult in previous reports.

Microdroplet division technologies using simple hydrodynamic methods have been developed by many research groups [14–16] as well as our group. High throughput droplet generation of 3800 droplets/sec was achieved by using multi-stage droplet division [17,18]. The divided volume ratio from 1:1.5 to 1:35 was also obtained by optimizing the channel structure. However, since the volumetric ratios of divided microdroplets depend on flow rates, the volume and ratio control were difficult under fixed flow condition. Already, one research group challenged to an active droplet division [19]. They hydrodynamically controlled ratio of divided microdroplets using controlled flow rates of downstream by additional tuning flow. The controllable volume ratio was up to 5.

To control flow rate in microchannels, we developed horizontal pneumatic valves with a flexible material PDMS (polydimethylsiloxane) [20]. The valve system enables five different modes of droplet sorting with only two valves, and provides precise flow rate control with the simple structure. Thus, we applied the horizontal pneumatic valves to the microdroplet division. By integrating bifurcating microchannel with the valves, flow rates are effectively controlled, and precise size control with wide range is realized.

2. Concept and Principle

Principle of the controllable division of microdroplets with bifurcating channel using flow rate control of downstream is shown in Figure 1. When introduced microdroplets are mechanically divided at a bifurcation point, their volumetric ratio depends on flow rates of downstream channels by different flow resistance. If the flow resistance of two downstream channels could be controlled, it is possible to divide microdroplets in any volume ratio at the same time.

In order to control the flow rates of microchannels, two horizontal pneumatic valves are located at downstream of the bifurcation point (Figure 2). Original microdroplets are continuously generated at the cross junction and flow into the bifurcation point. Each pneumatic line is independently operated, so that the flow rate in each channel changed with deformation of the two valves. By changing applied pressure to the two valves, droplets are divided into two daughter droplets of different volumes.

Figure 1. Controllable microdroplet division in bifurcating microchannel using a change in flow resistance of downstream.

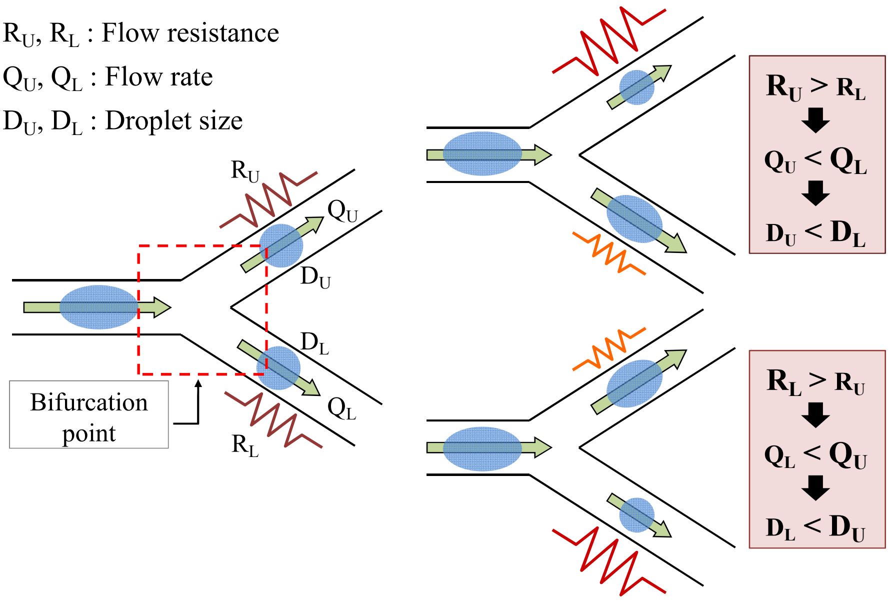
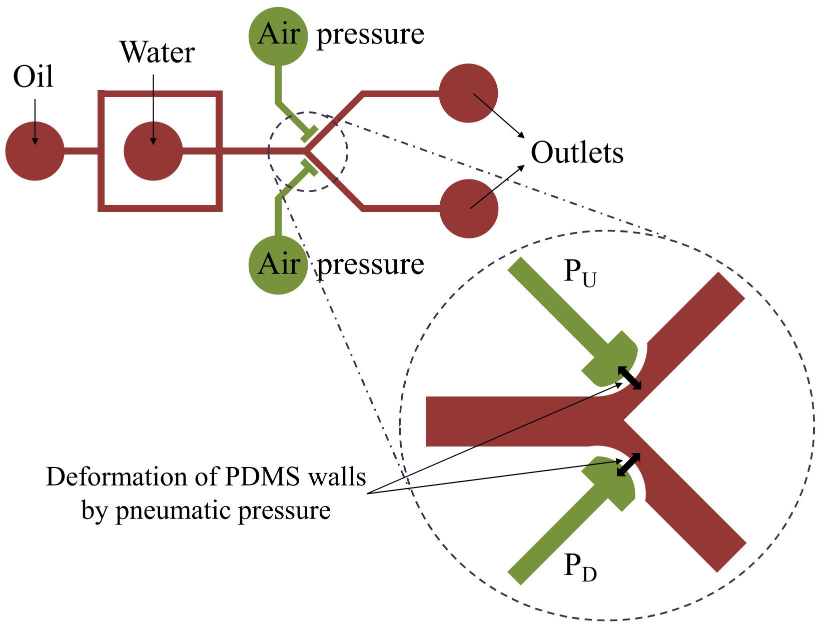


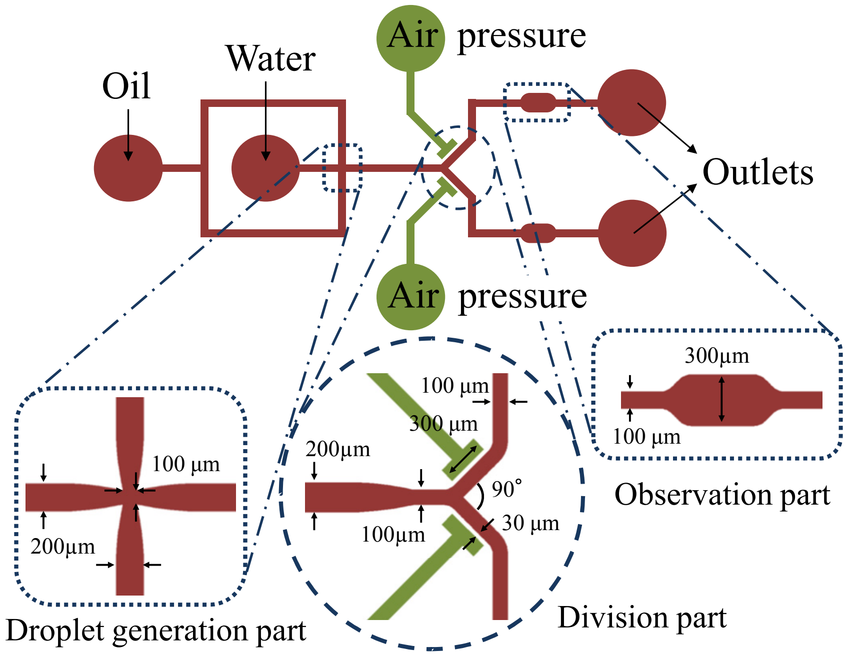
Figure 2. Schematic view of a total device and division part for droplet division utilizing horizontal pneumatic valves.



3. Design and Fabrication

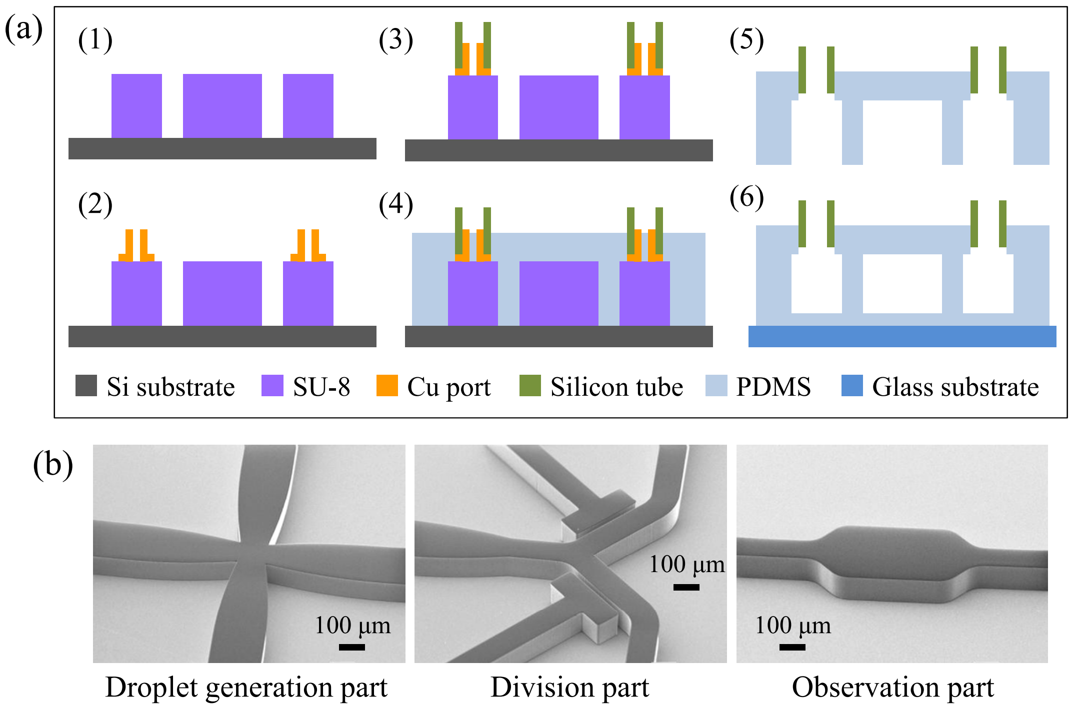
The microdroplet division device consists of droplet generation part, division part and observation part. Its structure and detailed size are shown in Figure 3. In the division part, nozzle like structure enables to locate original droplets in center of bifurcation point, which is useful to achieve stable droplet division. The width and depth of the downstream channel are 100 μm and 100 μm. The membrane thickness of two valves beside bifurcating channel is 30 μm.

Figure 3. Total design of the droplet division device and detailed sizes of each part.



PDMS device was fabricated by single step soft lithography process as shown in Figure 4a. After PDMS replicating from the SU-8 mold, the structure of PDMS is bonded with PDMS coated glass substrate after O_2 plasma pre-treatment. To obtain more flexible PDMS structure, resin and curing agent were mixed in 15:1 ratio. SEM (scanning electron measurement) images of each part of fabricated SU-8 mold are shown in Figure 4b.

Figure 4. (**a**) Fabrication process of the droplet division device. (**b**) SEM images of the SU-8 mold of each part.



4. Experiments and Discussions

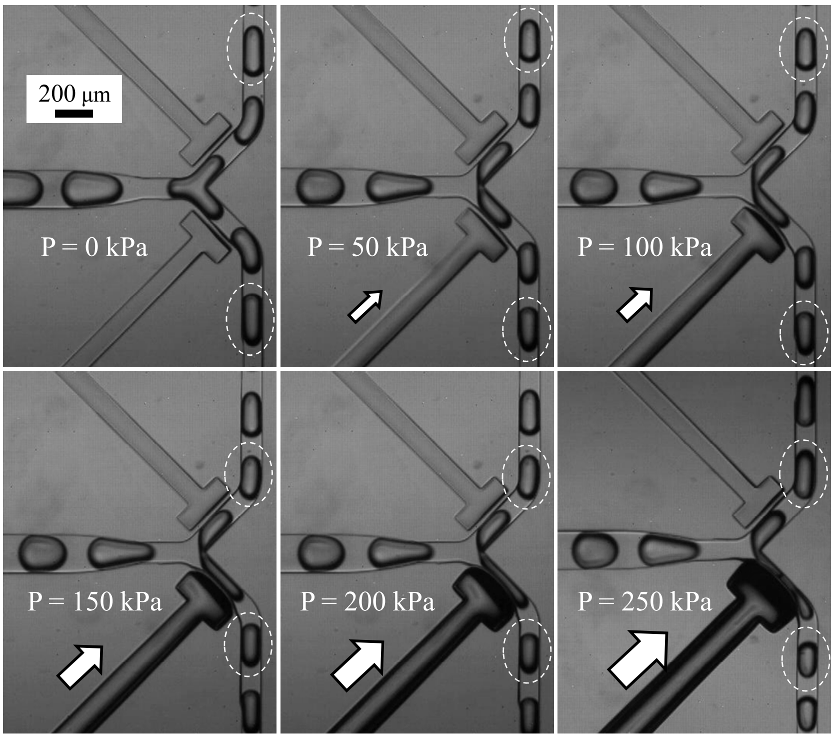
4.1. Experimental Set-Up

Deionized water and mineral oil (8042-47-5) were used for generation of original microdroplets, and they were introduced into the device by syringe pumps (KDS210, kdScientific) with syringes (1750CX, Hamilton). Pneumatic pressure to the valves was controlled by pressure regulator (2657 pneumatic pressure standard, Yokogawa). Experimental results were captured by high speed camera (FASTCAM-NEO, Photoron) and sizes of microdroplets were calculated by pixel counting. After syringe pump operation, waiting time about one minute was necessary for more stable flow, but droplet division was stably performed with valve switching of 1 Hz.

4.2. Microdroplet Division Results

Visualized microdroplet division results with different applied pressures to the valve of lower channel are shown in Figure 5. Original microdroplets, about 5 nL in volume, were generated by water and oil of 2 μL/min volumetric flow rate. When the applied pressure was 0 kPa, introduced water microdroplets was divided into almost half and half. With increase in pneumatic pressure to the lower valve, size of divided microdroplets in the lower branch channel decreased.

Figure 5. Visualization of droplet division with operation of pneumatic valves ($Q_{water} = Q_{oil} = 2$ μL/min).



Controlled ratios of droplets volume in upper and lower branch channels *versus* applied pneumatic pressure to lower valve are shown in Figure 6. By applying pressure from 0 kPa to 250 kPa, volumetric ratio change from 1 to 1.5 was achieved. The ratio of divided droplet sizes increased with a flow rate increase in a lower branch channel. Larger volumetric ratio was obtained with higher applied pressure of 300 kPa. However, the droplet division was not stable, and further design optimization was required.

Figure 6. Ratios of divided droplet volume with applied pressure to lower valve.

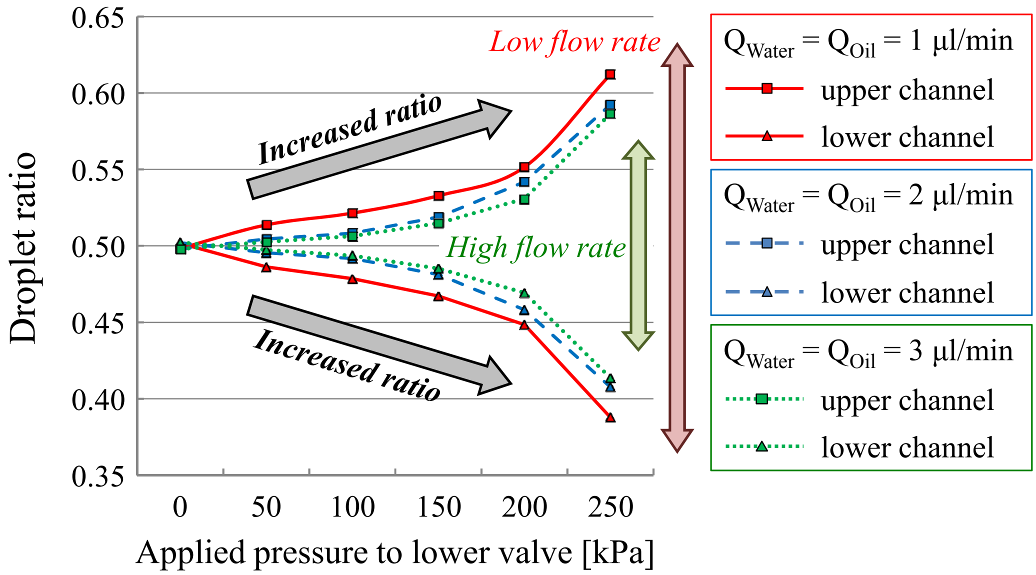
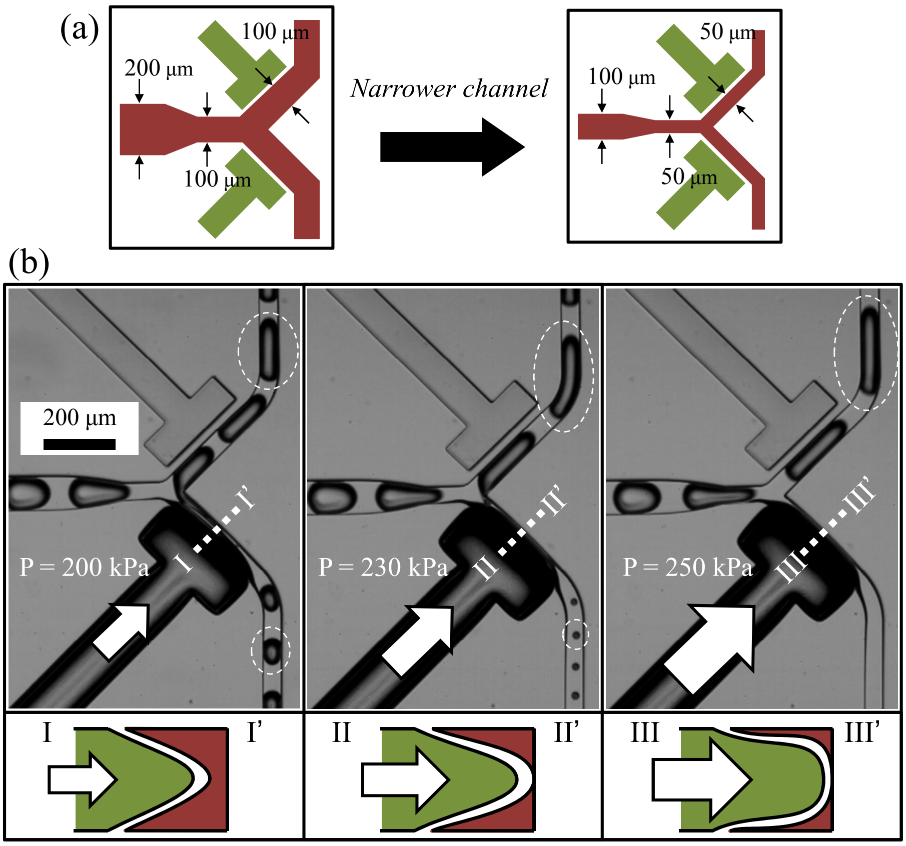


Figure 7. (**a**) Size change of division part for an effective use of the valve deformation. (**b**) Division results of large volume ratio with applied high pressure and cross section views.



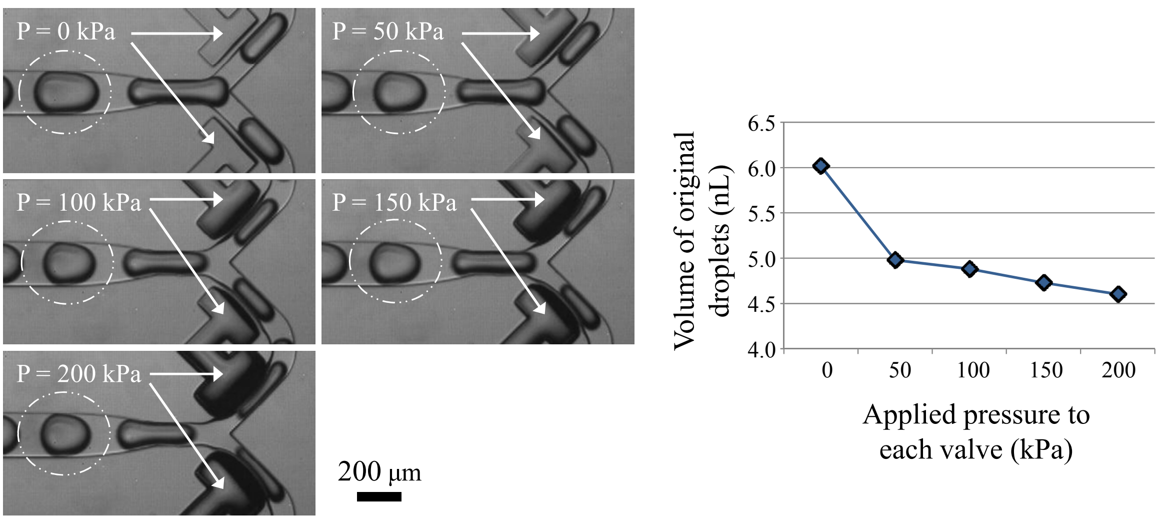
To achieve wide volumetric ratio of droplet division, a new device was designed and all channel width changed to half except pneumatic valve line as shown in Figure 7a. Narrow downstream channels realized large flow rate change under low applied pressure. As shown in Figure 7b, the new design makes a large channel blockage under same pressure compared with the previous device. With the applied pressure of 230 kPa to lower valve, the small droplets were about 14 pL in volume, and volumetric ratio between large and small droplets was larger than 70. Also, higher applied pressure of 250 kPa stopped the droplet division. Essentially, this horizontal pneumatic valve can't perfectly block the microchannel, but all droplets flowed into upper branch channel without division, since the droplet's surface tension was larger than hydrodynamic force in this case. Quantitative evaluation in a wide range of flow conditions with valve operation is under investigation. Additionally, since valve position is close to the bifurcation point, deformed valve membrane affect droplet division and probably enhanced an increment of the division ratio. To understand more precise correlation between applied pressure and droplet size, a new design by which the valves are located sufficiently far from the bifurcation point is under discussion.

4.3. Discussions

From experimental results, our investigations are focused on not only controllable division, but also the following points: effect of droplet speed into the bifurcation point and effect of pressure in the fluidic channel.

In Figure 6, when total flow rate is increased, volume ratio of divided droplet is decreased. The ratio decreases under high flow rate is related with dynamic droplet deformation. In the case of high droplet speed, the droplets are divided earlier at the bifurcation point, before deformation corresponding to the downstream flow rates. Since the droplets were located in center of channel and the bifurcation point, the ratio was closer to half and half than in case of slowly introduced cases. This result informs us that the droplet speed is an important factor for mechanical division of microdroplets.

Figure 8. A change in original microdroplets' volume in upstream due to valve deformation with applied pressure to each valve.



Volumes of generated original microdroplets with different pressure conditions are also shown in Figure 8. When water and oil were introduced as fixed flow rate of 2 μL/min, the size of generated droplets changed according to valve deformation. Since the deformed valves change flow resistance in micro channel, pressure at generation point of cross channel also changes. This result indicates that additional operations in downstream, which can change pressure of fluidics channels, influence droplet generation at upstream. Thus, it is necessary to consider this effect for more precise droplet generation result, in case of integration with other fluidic components as well as droplet division in this research.

Furthermore, properties of fluids such as viscosity and surface tension are also important issue in use of this droplet division method. These properties are closely related with dynamic formation and behaviors of the droplets. Thus, these properties should be carefully considered for more precise handing, as well as for fine size control.

5. Conclusions

Active and precise droplet division control in bifurcating microchannel was achieved by horizontal pneumatic valves. A wide range of the volume ratio of divided daughter droplets were realized with flow rate control of downstream channels by the simple structure.

In a future work, multi step division to generate droplets of large number with different volume, and integration with a droplet merging system are under investigation for high functional digitized droplet control. Also, many applications to not only microcapsule generation, but also droplet based chemical reaction are expected.

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