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Complete List of Authors:	Yoon, Dong Hyun; Waseda University, Wakui, Daisuke; Waseda University, Nakahara, Asahi; Waseda University, Sekiguchi, Tetsushi; Waseda University, Shoji, Shuichi; Waseda University, Department of Electronics, Information and Communication Engineering

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PAPER

Selective droplet sampling using a minimum number of horizontal pneumatic actuators in a high aspect ratio and highly flexible PDMS device

Dong Hyun Yoon,^{*a} Daisuke Wakui^a, Asahi Nakahara^a, Tetsushi Sekiguchi^b and Shuichi Shoji^a

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This paper presents a droplet sampling device driven by the horizontal pneumatic actuators. High aspect ratio and highly flexible PDMS (polydimethylsiloxane) structure was proposed for the larger number of sampling than the number of actuators. Large deformation of the actuators occurred domino-deformation of parallel walls, and the deformations allowed for selective collection of target droplets. Dimensions of the PDMS structure, and ratio of resin and curing agent were optimized for efficient sampling under low pressure applied to the actuators. Five sampling modes were achieved in the simple one-layer structure consisted of one inlet, four walls, one drain channel, and two pneumatic actuators, formed by single-step soft lithography process.

15 Introduction

Sampling or sorting of target samples is one of key technology for researches in biology or biochemistry. In particular, the technologies have been rapidly progressed with development of micro- and nano-fluidics researches in recent years.¹⁻⁴ As a pre-treatment, the sample sorting has allowed highly efficient treatment with sample purification or sample enrichment.^{5,6} Furthermore, sampling after the main treatment enables selective collection and analysis of the targets.⁷

The target's sampling has been performed by various methods according to detection and operation principle. Representatively, the electric or magnetic methods to control samples show fast switching performance and high throughput.^{8,9} These methods are suitable to treat a large number of samples, but electric field used for the sorting may cause damage to the samples such as living cells.^{10,11} In contrast, indirect sample sorting or handling using fluids can reduce the damage that occurred by external force for the sample control. The fluidic handling is performed by different control schemes; mechanical^{12,13}, thermal^{14,15}, ultrasonic^{16,17}, or hydrodynamic¹⁸⁻²⁰ flow controls.

In particular, the mechanical control using microactuators or microvalves with flexible membranes is a well-known method for the active flow control. The flexible membranes control the flow resistances corresponding to the channel dimensions deformed. In fields of microfluidics, chemistry, and biochemistry, polydimethylsiloxane (PDMS) is a most frequently exploited material in recent years.^{21,22} Its flexibility has provided functions as pneumatic actuators, which are classified as a horizontal and a vertical type. Horizontal pneumatic actuators are generally known that the structure and fabrication are simple²³⁻²⁹, but the each actuator should be controlled one by one or all together with low

efficiency. In contrast, vertical types show better performance with addressed control of actuators formed on multi-layer pneumatic lines.³⁰⁻³² However, the type requires complex and precise structures and fabrication processes.

Accordingly, the use of horizontal pneumatic actuators has been restricted to small scale sample handling due to their inherent limit, even though those operation and fabrication are relatively simple compared to the vertical types. However, if the low efficiency of the horizontal type actuators will be improved, sample handling devices, which utilize the actuators will be also developed and widely used.

Based on this background, we challenged development of a sampling device utilizing improved horizontal pneumatic actuators, which is operated by simple pneumatic control in a single-layer PDMS structure. In particular, this research focused more on an increase in selectivity of the sampling device, while the number of actuators is minimized. For the performance improvement, this research presents a high aspect structure formed by highly flexible PDMS, and domino-deformation of the actuator-wall structure.

Principle

The improved selectivity for the sampling is obtained by the specific actuator structure and operation, as shown in Figure 1. The structure consists of highly flexible actuator membranes located next to parallel walls. The four walls separate a main channel into five sampling channels, and the introduced samples are sorted by actuator deformation. Especially, domino-deformation of the parallel walls caused by large deformation of the actuators allows for a large number of sample selections using a minimum number of actuators.

Moreover, combining the structure with sampling chambers and a

drain channel affords target sampling from continuous sample flow, as shown in Figure 2. When the flow resistance of the drain channel is lower than that of the sampling channels, samples are thrown out into the drain channel. Then, the flow resistance of each channel, the drain channel and sampling channels, is simultaneously controlled by the actuator operation. With closing the drain channel, the samples are collected in the sampling chambers, and the samples are classified according to the flow resistance of the sampling channels controlled by the actuators. Especially, connected pneumatic lines to control the sampling modes and drain allow for efficient target sampling with minimized actuator operation. Finally, returning to the sample waste undesirable is simply performed by releasing pressure, which is used for drain channel closing.

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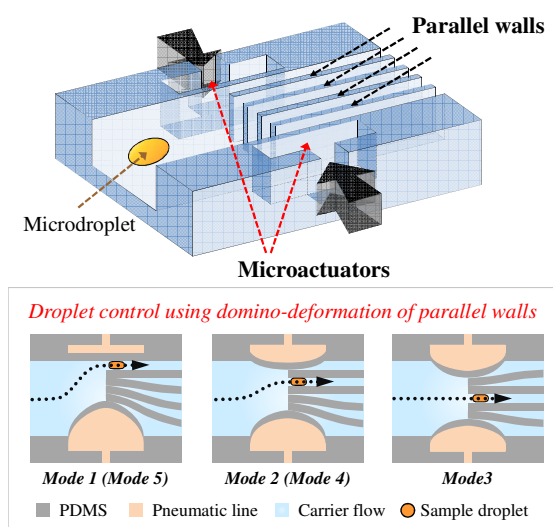


Fig. 1 Schematic view of the proposed microactuators-wall structure, and droplet control via domino-deformation of parallel walls corresponding to the combined actuator deformation.

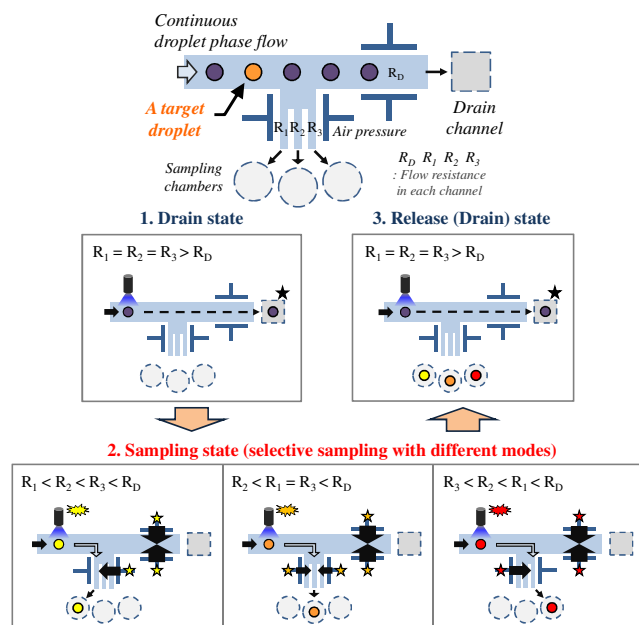


Fig. 2 Selective sampling of target droplets from continuous flow using the actuator structure and sample drain channel.

Device design and fabrication

Design of the sampling device is shown in Figure 3. The device consists of droplet generation part and sampling part which consists of fluidic channels and pneumatic lines. Especially, two pneumatic lines were designed for five different sampling, and the two lines control domino-deformation of parallel walls and closing of a drain channel simultaneously.

The width of the five sampling channels and four parallel walls are $30\mu\text{m}$ and $40\mu\text{m}$, respectively. Thickness of the actuator membrane is $40\mu\text{m}$ and its length is $500\mu\text{m}$. The introduction channel is divided into sampling channels and a drain channel diagonally. All structure, fluidic channels and pneumatic line, is formed on a single-layer and the structure is about $200\mu\text{m}$ tall.

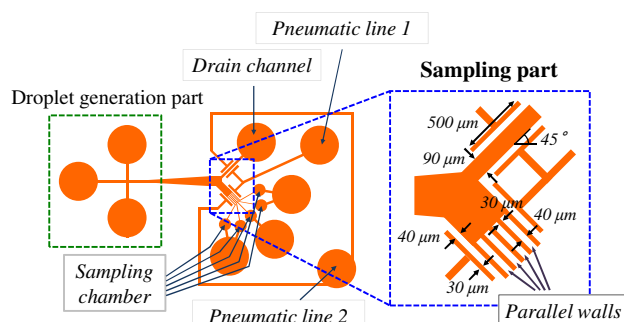
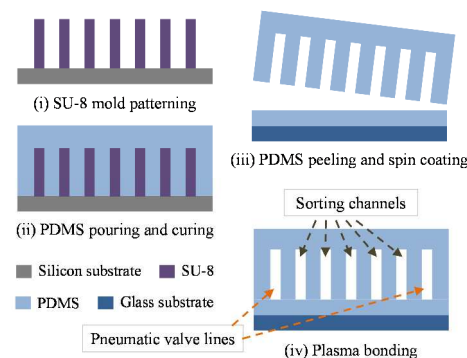
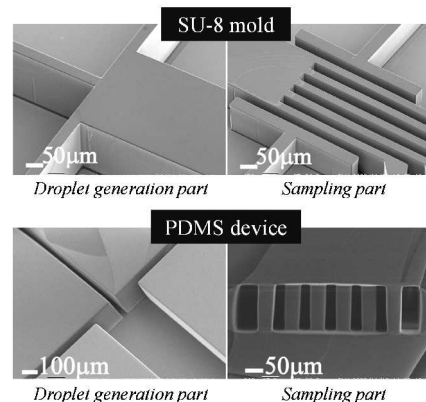


Fig. 3 Design of the microdroplet sampling device, and dimensions of actuators and walls.

(a) Fabrication process



(b) SEM images of mold and device



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Fig. 4 Fabrication process of the device (a), and SEM image of each part (b).

The device was fabricated by soft lithography process, as shown in Figure 4 (a). Negative photoresist, SU-8, was patterned on a silicon substrate, and PDMS base resin and curing agent (SILPOT 184, Dow Corning) were mixed in 15:1 ratio for a highly flexible device. After pouring on the SU-8 mold and degassing process, the PDMS was cured at a temperature 75 °C for 1 hour. The PDMS structure was bonded with a PDMS-coated glass substrate using O₂ plasma treatment (Aiplasma, Matsushita electric works). Figure 4 (b) shows SEM images of the droplet generation part and the sampling part. The SU-8 mold and PDMS structure of high aspect ratio were successfully fabricated.

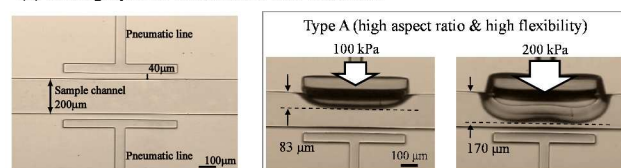
Experiments

Pneumatic pressure was generated by the pressure compressor (PC3-5 5T, YAEZAKI KUATSU) and the pressure was controlled by the pressure regulator (IR2020-02BG, SMC). To generate water in oil droplets, 5 % surfactant (Extran MA02) was added to the 5 mM methylene blue aqueous solution as a sample phase, and canola oil was used as a continuous phase. The sample and continuous phase fluid were introduced into the microchannels by syringe pumps (KDS 210, KD Scientific), and images were captured by a CCD camera (JK-TU53H, Toshiba).

Evaluation of horizontal PDMS actuator

Actuator performance of high aspect ratio and highly flexible PDMS structure was evaluated, as shown in Figure 5. The width of the channel and the thickness of the actuator membrane were 200 μm and 40 μm, respectively. The horizontal deformation corresponded to pressure applied to the actuators, and the deformation amount via different aspect ratios of the membrane and mixing ratios of PDMS resin to curing agent is shown in Figure 5 (b). Especially, the deformation of flexible high aspect ratio PDMS, Type A, was two times larger than that of the others. Furthermore, the PDMS structure was not damaged and plastic deformation was not observed, even after 100 time actuations.

(a) Photographs of the actuator and actuation



(b) Deformation of the actuators via aspect ratio and flexibility

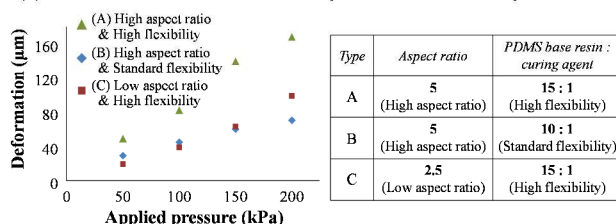


Fig. 5 Evaluation of high aspect and highly flexible microactuator (a) visualization of the actuator deformation, and (b) deformation amount via applied pressure under different aspect ratios and mixing ratios of PDMS.

Domino-deformation of parallel walls with the actuators

The actuator was applied to the simple and efficient flow control by combining with parallel walls. Figure 6 shows five different flow control modes using domino-deformation of the walls with actuation. The combination of pressure applied to two actuators allowed five control modes, which is larger than the number of actuators. The maximum pressure applied to the actuator was 250 kPa, and sampling to the farthest channel from the actuator was achieved in the condition. The deformation did not perfectly close the sampling channels, but flow resistance control by the deformation was sufficient to control a flow and droplet sampling. Furthermore, the indirect and gentle sample control in the flow and droplet is comparatively free from electrical or mechanical damages discussed in the conventional methods. The flexible droplets were deformed, and larger droplets, maximally 300 μm in diameter, than the channel width was successfully sorted in the device.

Five different droplet sampling using two actuators

Finally, it was developed that sampling of target droplets from continuous flow (total flow rate was 4.8 μl/min) by combining the actuator and wall structure with a drain channel, as shown in Figure 7 and ESI† Movie 1. Because the initial flow resistance of drain channel is smaller than that of sampling channels, the droplets were thrown out into the drain channel. Then, target droplets were collected one by one via actuator operation. The selection of targets to five different chambers is performed by the principle evaluated in section 4.2. In addition, pneumatic lines for the different collection modes were connected to drain channel actuators, thus the actuators at the drain channel were simultaneously operated with the sample collection without additional actuator control. Deformation of each actuator and closing ratio of the drain channel depends on the pressures for sample selection, but combined deformation of the two actuators switched over from drain state to sampling state successfully. Moreover, suitable pressure charge and release allowed for efficient sampling of a single target from continuous sample flow, and five different sampling modes by handling only two actuators were achieved in the simple structure. Furthermore, the PDMS structure endured more than 100 times actuation and longer than 1 hour continuous operation.

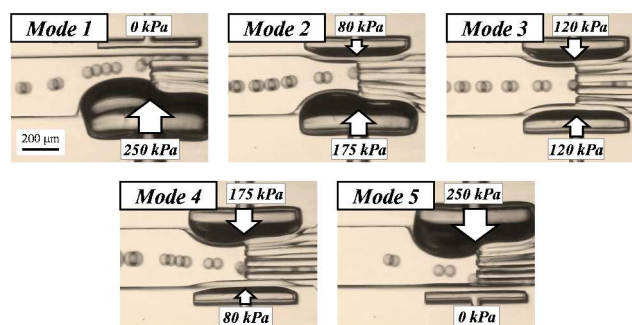


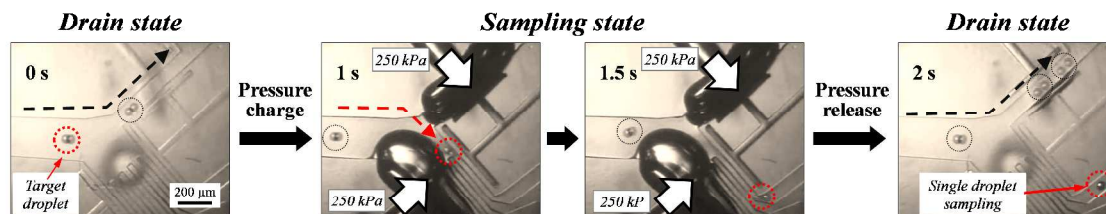
Fig. 6 Selective droplet control into five different channels by domino-deformation under combined pressures of two actuators.

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(a) State switching by actuator operation



(b) Selective sampling via applied pressure to the actuators

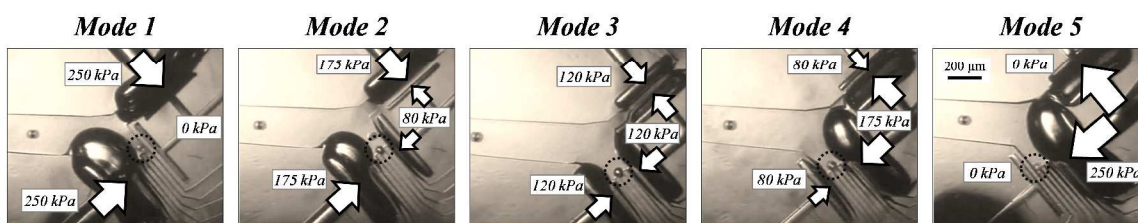


Fig. 7 Five different droplet sampling modes (a) using state switching by actuator operation in a drain channel and (b) pressure combination applied to the actuators.

Discussion

The proposed device successfully performed the selective sampling by improved structure and working principle. The critical and main issue in the device is large and excessive deformation of the actuators and walls. Therefore, it is expected that the actuator-wall structure and their domino-deformation presented in this research can be applied to various devices formed by flexible materials for handling of various samples. In contrast, the performance is highly dependent on material's mechanical property, elasticity. The PDMS used in this research is known as an elastomer, however its elasticity depends not only on mixing ratio, baking time and temperature, but also on time after device completion. Furthermore, humidity and temperature in the room make more complex results related to the property empirically. Hence, a change in the mechanical properties of the device should be carefully considered for long term use. Furthermore, the selection mode can be expanded by considering mechanical deformation of the actuators and walls.

Detection and systemization are other critical issues for the sampling. In this research, the sampling device evaluated independently without detection or control systems. Manual control of pneumatic line is employed for the samplings, thus the shortest response time for on-off switching from detection was about 0.5 s, even though the actuators responded within 0.1 s mechanically. This slow response by the manual operation limited conditions of the sample flow and purity of the sampling, for instance, some samples too close to the target were collected simultaneously. Therefore, introduction rate and distance between

the droplets are also carefully considered for the selective sampling based on the switching time. Moreover, integration with detection system or automation of the system is required to improve its performance, and the systemization is under investigation including chemical or biological applications.

Conclusions

The presented device performed the multimode sampling using the minimum number of actuators successfully. To achieve simple and efficient sampling, this research developed and investigated a horizontal PDMS actuator and parallel wall structure as follows. First, it was achieved that the large deformation using highly flexible and high aspect ratio PDMS structure. Second, the large deformation applied to the domino-deformation of parallel walls for the droplet sampling, and the combination of actuators and walls allows for the improvement of selectivity under the small number of actuators. Finally, sampling of target samples using fully integrated device, which consists of the actuators, parallel walls, and drain channel, was successfully demonstrated.

The whole structure is formed by simple fluidic channels and pneumatic lines on a single-layer PDMS, and the device was fabricated by single-step soft lithography process. Accordingly, the structure and fabrication are remarkably simple compared with general active sampling devices and systems. Moreover, the sample control of low damage with the soft material and fluids are also another advantage of this device and scheme. Thus, the device can be applied to cell sorting of low damage, as well as flexible droplets.

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Notes and references

^aFaculty of Science and Engineering, Waseda University, 3-4-1, Okubo,

10 Shinjuku, Tokyo 169-8555, Japan. Tel: +81 3 5286 3384; E-mail:

yoan@shoji.comm.waseda.ac.jp

^bInstitute for Nanoscience and Nanoengineering, Waseda University, 513, Tsurumaki-cho, Waseda, Shinjuku, Tokyo 162-0041, Japan

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