

## MICROMACHINED TUBE-TYPE OF Si DROPLET GENERATOR

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### ABSTRACT

We developed the novel type of droplet generator for the bio-chemical reactions, and designed it by considering Reynolds and Weber numbers. The Si droplet generator was fabricated on a silicon-on-insulator wafer by using deep-reactive ion etching and was fixed in a piece of heat-shrinkable tubing by heating the tubing. We experimentally confirmed that the fabricated droplet generator formed droplets in 1 mm diameter with jetting.

### KEYWORDS

Droplet, Reynolds number, SOI, D-RIE process

### INTRODUCTION

Microelectromechanical systems (MEMS) technologies have been used to produce various types of miniaturized biochemical devices, micro-droplet generators fabricated by MEMS are now widely applied for various biochemical reactions. Conventional micro-droplet generators use sheath flow and sheared flow in microchannels fabricated on the flat surface of chip by photolithography and covered by a glass plate [1, 2].

Although the micro-droplet generators fabricated by MEMS can continuously produce small uniformly-sized droplets, they are hard to package because bonding the glass plate on the flow channel structures is not as easy forming flow channels because of the difficulty of forming piping joints on the glass plate to connect the flow channel in the chip to the outside pumping systems. We therefore developed a tube-type of Si droplet generator that does not have any piping joints or a cover plate.

### TUBE-TYPE OF Si DROPLET GENERATOR

#### Principle

Droplet formation in the conventional and proposed tube-type of Si droplet generators fabricated by MEMS is shown in Figs. 1(a) and 1(b). The proposed device consists of a Si droplet generator has lots of through-nozzles and is mounted in a tube, one end of is connected to a syringe pump and the other of which is immersed in a solution. Numerous droplets are formed by pushing the solution A into the Si generator. One advantage of the proposed generator is that one can easily produce the droplets by simply connecting the generator to a conventional syringe pump and push the solution into the suspension liquid, and another is that its fabrication does not require any complicated bonding process.

### Design

For producing the droplet configuration in our device, the jet liquid stream exhausted by the Si droplet generator must, as shown in Fig. 2, be in an unstable state. We therefore investigated the condition of the droplet formation. The continuity and the momentum equations for the fluid are

$$\nabla \cdot \mathbf{u} = 0,$$

$$\begin{aligned} \frac{\partial}{\partial t} \rho \mathbf{u} + \nabla \cdot \rho \mathbf{u} \mathbf{u} \\ = -\nabla P + Re^{-1} \nabla \cdot \mu (\nabla \mathbf{u} + \nabla \mathbf{u}^T) + Fr^{-1} \rho \mathbf{f} \\ + We^{-1} \int_f \kappa \mathbf{n}_f \delta(\mathbf{x} - \mathbf{x}_f) dA_f, \text{ and} \end{aligned}$$

$$\frac{D}{Dt} \rho = \frac{D}{Dt} \mu = 0,$$

where  $\mathbf{u}$  is the velocity vector,  $P$  is the pressure, and  $\mathbf{f}$  is the body force. The non-dimensional numbers  $Re$ ,  $Fr$ , and  $We$

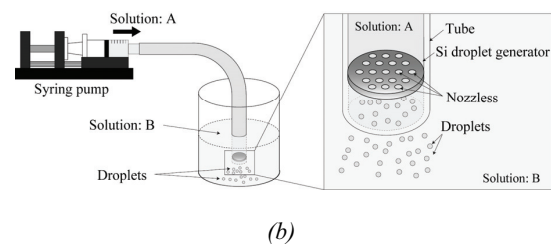
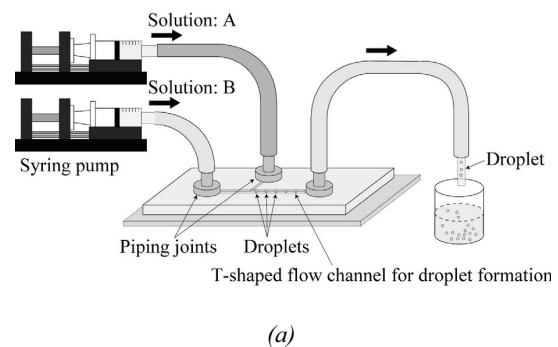


Fig. 1: Droplet generator fabricated by MEMS. (a) Conventional droplet formation using T-shaped flow channels. (b) Droplet generation by the proposed in-tube type of droplet generator.

Table 1: Solution and oil properties.

	Kinematic viscosity, $\nu$ [m <sup>2</sup> /s]	Density, $\rho$ [kg/m <sup>3</sup> ]	Viscosity, $\mu$ [Pa·s]	Surface tension, $\sigma$ [N/m]
Suspension fluid (oil)	$1.00 \times 10^{-5}$	932.23	$9.32 \times 10^{-3}$	-
Dispersed fluid (water)	$1.52 \times 10^{-5}$	997.04	$8.94 \times 10^{-4}$	$7.2 \times 10^{-2}$

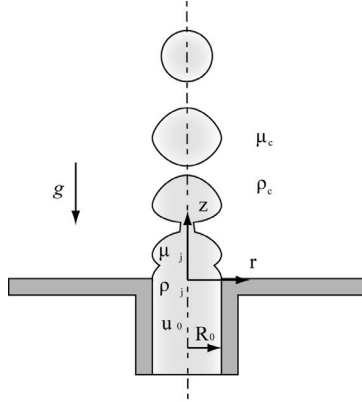


Fig. 2: Geometry of the droplet formation model.

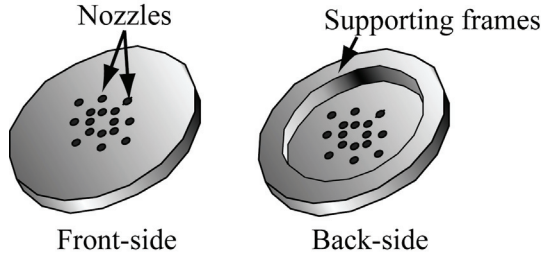


Fig. 3: Schematic view of the designed droplet generator.

in this equation are respectively the Reynolds number, Froude number, and Weber number and are given by

$$Re = 2R_0 u_0 \rho_j / \mu_j,$$

$$Fr = u_0^2 \rho_j / 2R_0 g (\rho_j - \rho_c), \text{ and}$$

$$We = 2R_0 u_0^2 \rho_j / \sigma$$

where  $g$  is gravitational acceleration,  $R_0$  is the nozzle hole diameter, and  $\sigma$  is the interfacial tension. As shown in ref. [3], for producing uniform droplets in the applied model the Reynolds has to be smaller than 200 and the Weber number has to be less than 1. We therefore determined the device specifications as follows.

**(Step 1)** We at first determined the types of the fluids. For producing water droplets in oil environment, we determined to use a water solution as the dispersed fluid and silicone oil (KF-96-10cS, Shin-Etsu Chemical Co., Ltd.) as the suspension liquid. The detailed specifications of the solution and the oil are listed in Table 1.

**(Step 2)** We then determined the nozzle diameter  $R_0$  needed to satisfy the Reynolds and Weber numbers conditions mentioned above.

We thus designed four Si droplet generators that were all 2400  $\mu\text{m}$  in diameter but had different nozzle diameter: 6, 24, 125, and 600  $\mu\text{m}$ . The basis structure of the designed generators is shown schematically in Fig. 3. The nozzles were formed on a diaphragm structure supported by a frame.

## FABRICATION

As shown in Fig. 4, the Si droplet generator was fabricated in two steps of deep reactive-ion etching (D-RIE). We used a 4-inch SOI wafer and the thickness of device, buried silicon dioxide, and substrate layer of the wafer were 10, 1, 300  $\mu\text{m}$ , respectively. We first applied a photoresist to the device layer of SOI wafer and patterned it by using an exposure system (Figs. 4(a) and 4(b)). We then formed the Si-based nozzle structures by a D-RIE process (Fig. 4(c)) and removed the photoresist used in that process (Fig. 4(d)). We then patterned the photoresist on the rear-side of the substrate layer, formed the nozzles on the diaphragm supported by the frame and the beam by D-RIE (Fig. 4(e)) and removed the photoresist used in that process (Fig. 4(f)). Finally, we fabricated the through-hole structure as a nozzle by using an HF wet etching process to remove the silicon dioxide layer (Fig. 4(g)). We easily separated the droplet generator from the SOI wafer by breaking the supporting beams (Fig. 4(h)).

The Si droplet generator fabricated on a SOI wafer is shown in Fig. 5. Each generator was supported by four beams to the Si substrate. A magnified SEM image of the generator is shown in Fig. 6. We fabricated the two types of Si droplet generator: a single-nozzle type for evaluating the condition of the droplet formation process (Fig. 6(a)), and a numerous-nozzle type for generating multiple droplets at the same time (Fig. 6(b)).

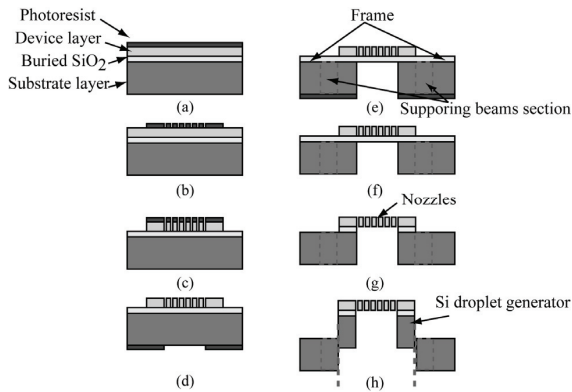


Fig. 4: Fabrication process of the Si droplet generator.

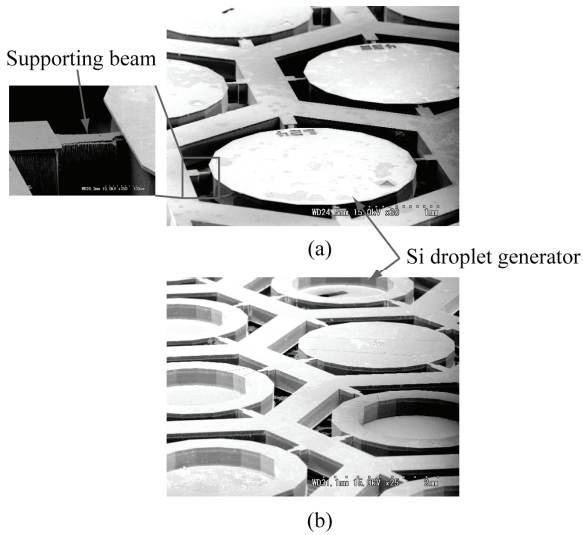


Fig. 5: (a) Front and (b) back views of a fabricated Si droplet generator.

## EXPERIMENT

### Assembling in-tube droplet generator

The most difficult problem in fabricating the in-tube type of droplet generator was attaching the generator to the tube. We solved this problem by using heat-shrinkable tubing (PTFE/FEP Dual-Shrink™ Tubing ZDS-L-190, HAGITEC Co., Ltd.). We mounted the droplet generator on the supporting jig and put it into a piece of tubing with an inner diameter of 4.8 mm (Fig. 7(a)) and reduced the inner diameter of the tubing to 1.78 mm by heating it (Fig. 7(b)). The shrunk tube held the generator tightly without glue.

### Droplet formation

The experimental setup we used to evaluate the performance of droplet generator is shown in Fig. 8. In the first trial used an adhesive to attach a single-nozzle type generator (nozzle diameter: 125  $\mu\text{m}$ ) to the end of a silicone rubber tube with inner and outer diameters of 4.0 and 2.0

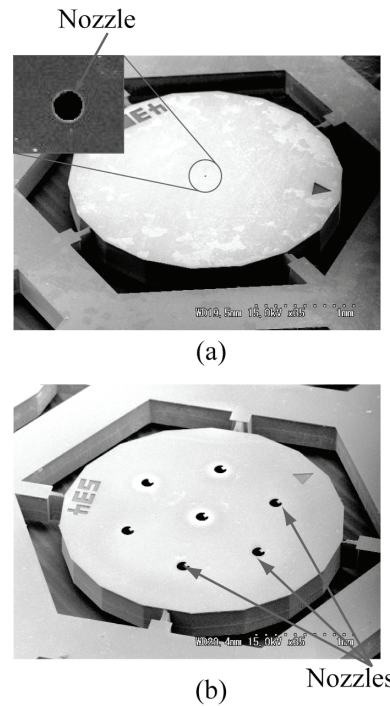


Fig. 6: Magnified view of Si droplet generators: (a) single-nozzle type, (b) numerous-nozzle type.

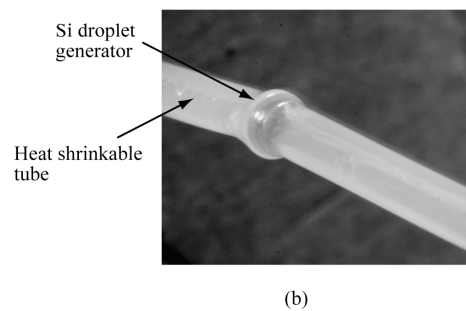
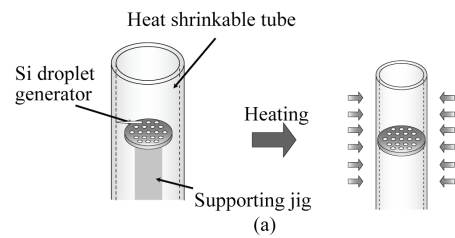


Fig. 7: Mounting of in-tube droplet generator in heat-shrinkable tubing: (a) before and (b) after heating.

mm, filled the tube with water (dispersed fluid), and pushed the water into the silicone oil (suspension liquid). The flow rate was controlled by using conventional syringe pump and pulse motor controller (D212 motor controller, Suruga Seiki Co., Ltd).

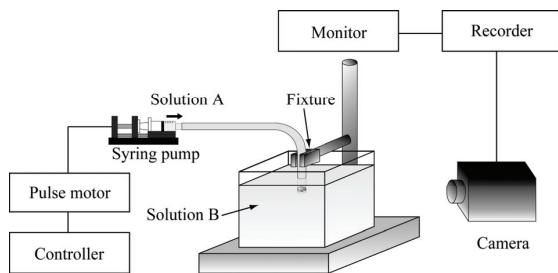
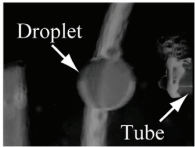
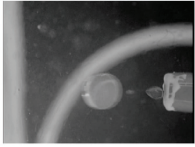

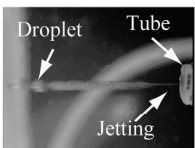
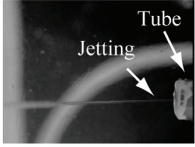


Fig. 8: Experimental setup for forming droplets.

Table 2: Relation between flow condition and droplet formation mode.

Reynolds number	Weber number	Observation result
3.57	$1.13 \times 10^{-3}$	
7.14	$4.54 \times 10^{-3}$	
8.21	$6.00 \times 10^{-3}$	
8.92	$7.09 \times 10^{-3}$	
35.7	$1.13 \times 10^{-1}$	

The values listed in Table 2 show the relation between the flow condition (Reynolds and Weber numbers) and the droplet formation. We observed three types of droplet/jetting formation modes at different ranges of the Reynolds number: uniform droplets ( $Re \leq 8.21$ ), droplets with jetting ( $8.21 < Re < 35.7$ ), and jetting without droplets ( $35.7 \leq Re$ ). The diameter of the formed droplet decreased with increasing Reynolds number, and we found that droplets as small as 1 mm in diameter could be obtained with jetting ( $Re = 10.7$ ).

## CONCLUSION

We developed a new kind of droplet generator for biochemical reactions and mounted it inside a piece of heat-shrinkable tubing by heating the tubing. Investigating the relation between Reynolds number and the mode of droplet formation, we found that droplets with diameters as small as 1 mm diameter could be obtained with jetting ( $Re = 10.7$ ).

In the future, we will try to form numerous droplets at the same time by using numerous-hole types of droplet generators and will investigate the relation between the Reynolds number and the mechanism by which uniform micro-scale droplets are formed.

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