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## **INVESTIGATION OF DROPLET FORMATION MECHANISM IN MICRO T-SHAPED JUNCTION USING CONFOCAL MICRO-PIV MEASUREMENT**

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### **ABSTRACT**

This paper aims to investigate a mechanism of microdroplet formation at a micro T-shaped junction using a “Multicolor Confocal Micro Particle Image Velocimetry” technique. Our system can measure dynamic behavior of each phase of multiphase flow separately and simultaneously. The internal flows of two immiscible fluids are investigated as a liquid-liquid multiphase flow.

The droplet formation mechanism changes from “squeezing” to “dripping” at a critical Capillary number, which indicates the force balance between shear force and interfacial tension. We can obtain the interfacial geometry at the junction where the to-be-dispersed phase goes into the continuous one as well as their velocities from the results of PIV measurements and interface scanning. The interfacial geometry plays an important role in the flow structure and shear stress on the interface.

Small Capillary number means low flow rate of continuous phase, and it makes droplets larger than channel dimension. Because low continuous flow rate doesn't impose strong shear stress on the surface of the tip of to-be-dispersed phase, as a result, it becomes “squeezing” phenomena. On the other hand, strong shear stress tears off the tip of to-be-dispersed phase into smaller droplet in the condition of high Capillary number, resulting in “dripping” phenomena.

As a result of Micro-PIV measurement and interface scanning, it became clear that the gap between the tip of to-be-dispersed phase and capillary wall, and interface area play an important role in the flow structure and shear stress on the interface. In conclusion, droplet formation mechanism depends on not only the Capillary number but also a size of gap, which is determined by the flow rate of to-be-dispersed phase.

**Keywords: Confocal Micro-PIV, Capillary Number, Interfacial Tension, Multiphase Flow, Micro droplet**

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## INTRODUCTION

Over the last decade, a lot of microfluidic devices have been designed as micro total analysis systems (Micro-TAS) or lab-on-a-chip applications (Reyes *et al.*, 2002; Auroux *et al.*, 2002). These devices are used for many purposes such as mixing, separation, sensing, or chemical reactions and so on (Whitesides, 2006). Especially microdroplet is used as a smallest micro chamber to obtain fast reaction process and to make monodisperse microspheres or capsules (Song *et al.*, 2006).

These microchip devices usually handle more than two different materials, i.e. multiphase flow in order to achieve several purposes described above. These phases are liquid, solid, gas, gel, and so on. In order to clarify the phenomenon inside these devices, we have to measure the flow field or movement of each phase simultaneously.

However, velocity measurement of micro multiphase flow has yet to be accomplished. Although the conventional confocal micro PIV (Park *et al.*, 2004; Kinoshita *et al.*, 2005) is powerful flow analysis technique to measure the velocity distributions with high spatial and temporal resolution, they are not suitable for multiphase flow measurement. One of the main reasons is difficulty in separation of particle images of different phases due to difference in light intensity and its separation performance.

The purpose of this study is to develop a measurement system for micro multiphase flow to verify the interaction between different phases simultaneously. In this study, we constructed a “multicolor confocal micro PIV system”. The system applied to internal and external flow of a moving droplet, and the interaction between two liquid phases are investigated.

## DROPLET FORMATION MECHANISM

There are many previous studies about droplet formation mechanism using micro T-shaped junction. Most of all uses Capillary number ( $Ca$ ) in order to explain its phenomena.  $Ca$  is written in following equation (1).

$$Ca = \frac{\mu_c u_c}{\gamma} \quad (1)$$

$Ca$  means a ratio of interfacial tension  $\gamma$  and shear stress by the continuous flow viscosity  $\mu_c$  and velocity  $u_c$ . The shear stress acts to tear off the tip of to-be-dispersed phase. On the other hand, the interfacial tension acts to keep the surface of to-be-dispersed phase to minimize its surface area.

Garstecki *et al.* (2006) suggests that the mechanism changes from “squeezing” to “dripping” between  $Ca \sim 10^{-2}$ . Similarly, Tice *et al.* (2003) and Nishisako (2006) suggest that the bifurcation point of change is  $Ca \sim 1$ , and Graaf *et al.* (2005) suggests  $Ca \sim 0.1$ . This means it is inadequate to clarify this mechanism with only  $Ca$  number.

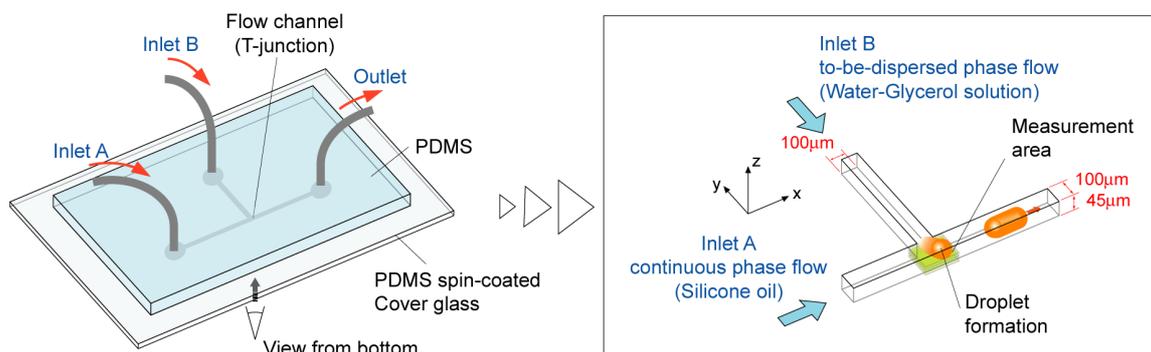
In this paper, we vary only flow rate of continuous phase as a parameter to control  $Ca$  number, because viscosity and interfacial tension affects each other.

## PRELIMINARY EXPERIMENT

### Target Microchannel

Figure 1 is a schematic illustration of a droplet formation device with a T-shaped junction which width  $w = 100 \mu\text{m}$  and depth  $h = 45 \mu\text{m}$ . The patterned microchannel is placed and sealed off on a cover glass where PDMS is spin-coated at a thickness of  $10 - 20 \mu\text{m}$  using the fabrication method of Hong *et al.* (2001). Two inlet ports and one outlet port are fabricated at the ends of the microchannel. The inlet ports are connected to separate  $250 \mu\text{L}$  glass syringes (Gastight Syringes 1725LT, Hamilton Company, USA) on syringe pumps (KDS100, KD Scientific Inc., USA) through Teflon tubes, and the outlet port is connected to the drain. The continuous phase flows in the straight main channel at a constant flow rate from inlet A, and the to-be-dispersed phase flows from inlet B to the T-shaped junction at a constant flow rate too. Syringe pumps control both flow rates.

The continuous phase flow is blocked with the to-be-dispersed phase at the T-shaped junction, where the to-be-dispersed phase is diverted and transformed into droplets. The droplets are generated sequentially at a constant frequency. The droplet size and the generation frequency are dependent on the inlet flow rates, the geometry of the channel, driving pressure, interfacial tension, wettability on the channel wall, shear force at the interface between two immiscible liquids and the viscosities of the working fluids.



**Fig. 1. T-shaped microchannel**

### Working Fluids

The refractive indices of the working fluids and channel material have to match exactly to minimize the refraction and reflection of light at the interface and channel wall. In this experiment, two immiscible liquids were used as working fluids for refractive index matching. One is the silicone oil (KF-6001, Shin-Etsu Chemical Co. Ltd., Japan) as the continuous phase, and the other is the water-glycerin mixture (Glycerin: Wako Pure Chemical Industries, Ltd., Japan) as the to-be-dispersed phase. Silicone oil was chosen because it has a refractive index of 1.4120, which is almost equal to that of the PDMS material of the microchannel.

## Droplet Size Control

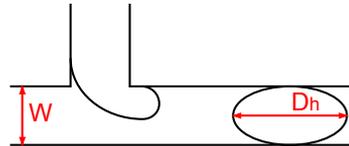
As a preliminary experiment, we searched critical  $Ca$  number, which indicates the changing point of the droplet formation mechanism. The characteristics of two immiscible working fluids and flow conditions are shown in Tab. 1. Although a small amount of fluorescein is dissolved into to-be-dispersed phase to visualize interface, other ingredients are same as for PIV measurement in order to keep liquid characteristics and interfacial tension.

We varied the flow rate of continuous phase  $Q_c$  and to-be-dispersed phase  $Q_d$ . The range of flow rate and  $Ca$  number are also shown in Tab. 1.

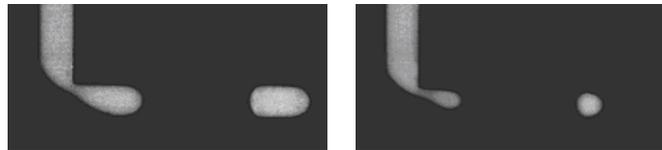
We choose droplet length  $D_h$  as a characteristic parameter of mechanism shown in Fig. 2 and 3. Relatively slow continuous phase flow, it means low  $Ca$  number, the diameter of droplet become larger than the channel size. Then it becomes a plug shape. On the other hand, for fast continuous phase flow, it means high  $Ca$  number, droplet is torn off from the tip of to-be-dispersed phase before attaching opposite wall. Then it becomes smaller droplet than channel size.

**Table 1. Flow conditions of preliminary experiment**

	Continuous phase	To-be-dispersed phase
Working fluid	Silicone oil	Water-Glycerin mixture
Specific gravity [g/cc]	0.98	1.158
Viscosity [mPa s]	44.1	7.46
Flow rate [ $\mu$ l/hr]	1.2 ~ 30.0	2.0
Average flow velocity [mm/sec]	0.095 ~ 3.175	0.159
Interfacial tension [mN/m]	11.1	
Capillary number	$3.79 \times 10^{-4} \sim 9.47 \times 10^{-3}$	



**Fig. 2. Size parameter of the droplet**

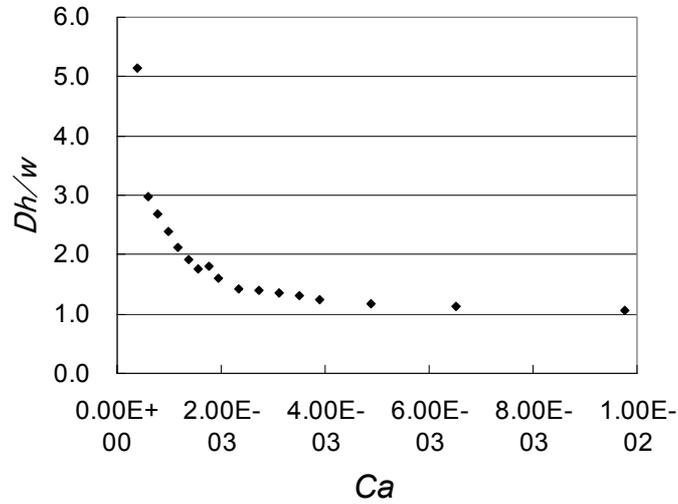


**Fig. 3. Droplet formation images**

**Left: Low  $Ca$  number condition (squeezing), Right: High  $Ca$  number condition (dripping)**

As a result of preliminary measurement, Fig. 4 shows the relation between  $Ca$  number and dimensionless droplet shape parameter, which is defined by dividing droplet length  $D_h$  by channel width  $w$ . This shape parameter indicates wall contact of droplet. When this parameter indicates over 1.0, the droplets attach to the channel wall and become bullet shape elongated streamwise direction. The

inflection point of plots is about  $Ca = 2.0 \times 10^{-3}$ .



**Fig. 4. Relation between Ca number and droplet shape**

## MEASUREMENT METHOD

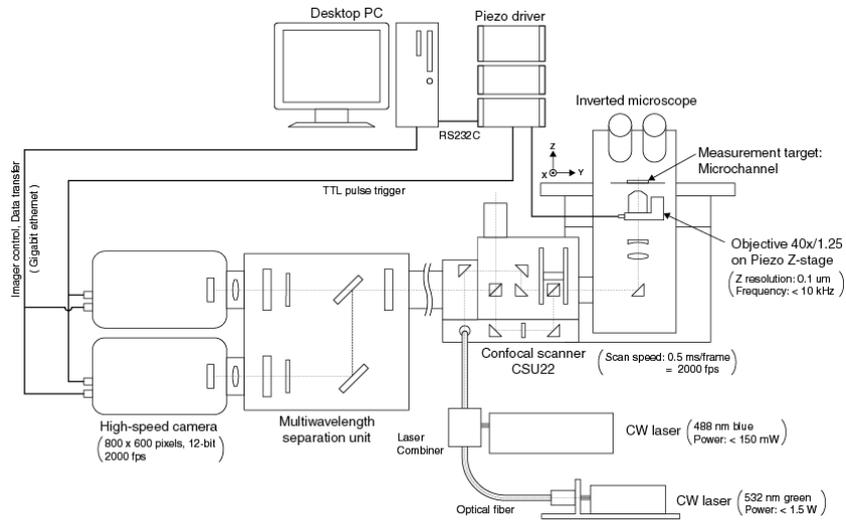
### Multicolor Confocal Micro PIV System

In order to measure multiphase flow characteristics simultaneously, we developed a multicolor confocal micro PIV system (Oishi *et al.*, 2006). Its configuration is shown in Fig. 5. Our system is based on a confocal micro PIV system, which was developed by Kinoshita *et al.* (2005), and consists of a high-speed confocal scanner (CSU22, Yokogawa Electric Corp., Japan), high-speed camera (Phantom v7.1, Vision Research Inc., USA) and an inverted microscope (DMIRE2, Leica Microsystems, Germany).

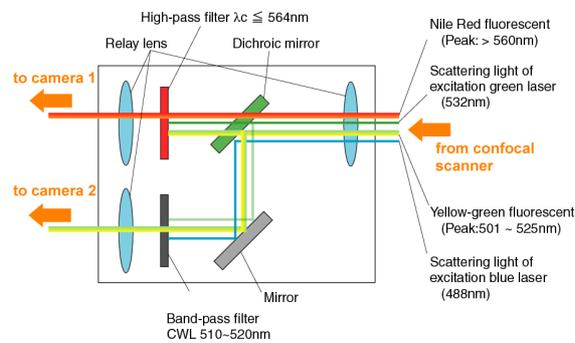
Our upgraded system allows multiphase flow measurement through additional devices and modules. To simultaneously measure multiphase flow phenomena, we added a second laser and camera, a laser combiner, and a multi-wavelength dividing unit to the system. The blue laser (543-BS-A03, Melles Griot Inc., USA) and the green laser (Excel 1500, Laser Quantum Ltd., UK) are combined using a dichroic mirror in the laser combiner and led to the laser port of the confocal scanner using optical fiber for illumination. Lasers illuminate the target flow, and Stokes' shifted fluorescent light from the fluorescent particles that dispersed in the flow is emitted back through confocal scanner.

Then, the multi-wavelength emitted light is separated and filtered at the separation unit, which is shown in Fig. 6, and recorded by two high-speed cameras. The optical design of the multicolor separation unit can separate and pass only emitted fluorescent light to the imaging device such as CCD or CMOS cameras. This unit consists of relay lenses, a dichroic mirror, high-pass and band-pass barrier filters. At first, light at four different peak wavelengths pass from the exit of the confocal scanner and travel toward the separation unit. These intensity peaks are found at 488 and 532 nm (scattering light of the two excitation lasers) and near 515 and 575 nm (emission from fluorescent particles). The barrier filters are long-pass filters (RG series, Melles Griot Inc., USA, E570ALP, Chroma Technology Corp., USA) for longer wavelength imaging, and the band-pass filters (F-10 series, CVI Laser Inc., USA and 50AF23, Chroma Technology Corp., USA, FF01-512/25, Semlock Inc., USA) for shorter wavelength imaging.

These optical filters were carefully chosen due to their fluorescent characteristics. A pair of relay lenses is placed to conjugate the focal planes of the confocal scanner and high-speed cameras. A wavelength separation dichroic mirror and optical barrier filters are placed within the collimating optics region between the relay lenses. The relay lenses also have different f-numbers to scale-up the confocal image twice as large.



**Fig. 5. Schematic diagram of the Multicolor Confocal Micro-PIV System**



**Fig. 6. Schematic diagram of the multicolor separation unit and its optical design**

### Micro-PIV Measurement Conditions

In order to clarify the droplet formation mechanism in more detail, we measured the velocity distribution of both phase using Multicolor Confocal Micro-PIV system. From preliminary experiment, the mechanism changes between  $Ca = 2.0 \times 10^{-3}$  for our experimental condition. So we chose two condition of  $Ca = 1.96 \times 10^{-3}$  and  $9.81 \times 10^{-3}$ . Other conditions of PIV measurement are written in Tab. 2. The measurement region is  $228.2 \times 171.1 \mu\text{m}$  with the in-plane spatial resolution of  $0.2852 \mu\text{m}/\text{pixel}$ , and measurement height is at its center height.

To visualize the continuous phase, we utilized oil-immersible microspheres made from porous silica particles (Godball E-2C, Suzuki Yushi Industrial Co. Ltd., Japan). These particles have high surface lipophilicity, and high traceability due to their low specific weight of porous structure. These particles were dyed using Fluorescein (Wako Pure Chemical Industries, Ltd., Japan) and dried in a vacuum desiccator. Clustered particles were separated using vibrating agitator. The distribution of particle diameter ranged between  $\phi$  0.9 to 1.4  $\mu\text{m}$ . The  $\phi$  1.0  $\mu\text{m}$  diameter Nile red fluorescent polystyrene particles (FluoSpheres F8819, Invitrogen-Molecular Probes Inc.) were dispersed in the water phase with a volume ratio of 0.4 %. The measurement depths of each particle are measured and it results in about 3  $\mu\text{m}$ .

**Table 2. Conditions of Micro-PIV Measurement**

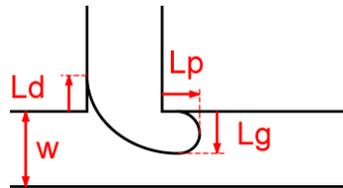
	Continuous phase (inlet A)		To-be-dispersed phase (inlet B)
Working fluid	Silicone oil KF-6001		Water / Glycerol solution
Flow rate [ $\mu\text{l/hr}$ ]	8.0	40.0	2.0
Average flow velocity [ $\text{mm/sec}$ ]	0.494	2.469	0.124
Capillary number	$1.63 \times 10^{-3}$ (Squeezing)	$9.81 \times 10^{-3}$ (Dripping)	-
Tracer particle	$\phi$ 0.9 ~ 1.4 $\mu\text{m}$ Green Fluorescent silica		$\phi$ 1.0 $\mu\text{m}$ Red fluorescent polystyrene
Measurement Depth [ $\mu\text{m}$ ]	3.10 ~ 3.84		3.34
Refractive index	1.412		
Interfacial tension [ $\text{mN/m}$ ]	11.1		

(Experiment temperature : 25  $^{\circ}\text{C}$ )

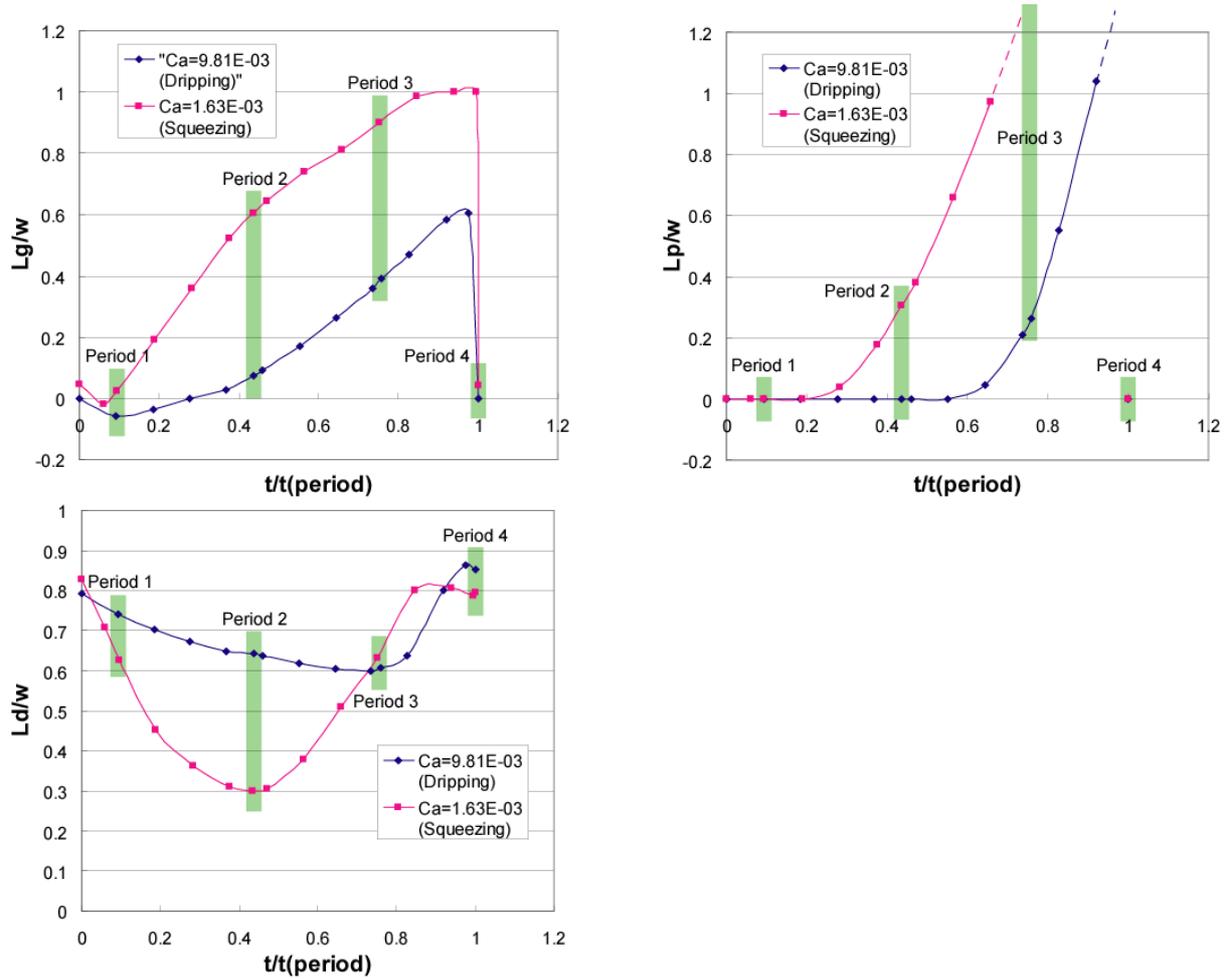
## RESULTS

### Transition of Interfacial Geometry

In order to clarify the difference between Squeezing and Dripping, time-series transition of interfacial geometry is verified. Figure 7 shows three geometry parameters of the tip of to-be-dispersed phase. These parameters are normalized by channels width and droplet formation period, and they are plotted in Fig. 8.



**Fig. 7. Geometry parameter of the tip of to-be-dispersed phase**



**Fig. 8. Time-series transition of interfacial geometry**

$Lg$  indicates the elongation of tip of to-be-dispersed phase to opposite channel wall. It elongates differently according to  $Ca$  number. In case of low  $Ca$  number, it elongates faster than in case of high  $Ca$  number because of smaller shear stress. And elongation speed becomes gentle and flat depending on increase of shear stress by narrowing continuous flow path. On the other hand, in case of high  $Ca$  number, the tip of to-be-dispersed phase is stretched by strong shear stress of continuous phase. As a result, elongation of  $Lg$  becomes shelving and it breaks before attaching to opposite channel wall.

$Lp$  indicates the elongation of tip of to-be-dispersed phase to downstream of main channel in analogy with  $Lg$ . Its developing timing in low  $Ca$  condition is faster than that in high  $Ca$  condition after breaking. It would appear that the tip of to-be-dispersed phase returns largely after breaking in case of high  $Ca$  condition because it is strongly elongated by strong shear stress.

Furthermore, time-series transition of  $Ld$ , which is separation point of to-be-dispersed phase from the upstream edge of merging section, fluctuates largely in case of small  $Ca$ . It indicates the phenomena that the continuous phase counterflows toward to-be-dispersed phase channel because of pressure increase of continuous phase by narrowing flow path.

### Confocal Micro-PIV Results

The results of multicolor PIV measurement at each phase are shown in Fig. 9. We compared each four periods shown in Fig. 8. Each timings are  $t/t_{period} = 0.093, 0.437, 0.755$  and  $1.000$ .

At the Period 1 that is after generating droplet, the new tip of to-be-dispersed phase appears to the main channel. The interfacial geometry of high  $Ca$  condition is sharper than that of low  $Ca$  condition, because of high shear stress of continuous phase affects to the interface. Its high shear stress also makes circulation motion inside the tip of to-be-dispersed phase. On the other hand, in case of low  $Ca$ , to-be-dispersed phase flows straight to its supplying channel.

Period 2 is the timing of tip growth. Different volume of tip of to-be-dispersed phase derives from the difference of the length of droplet formation period. In case of low  $Ca$ , continuous flow path narrowed by the tip of to-be-dispersed phase. It makes continuous flow faster, and to-be-dispersed phase is curved into main channel. In the to-be-dispersed phase of high  $Ca$  number, velocity gradient appears from upstream side to downstream side, but that of low  $Ca$  number is quite different.

Flow pattern of high  $Ca$  number of period 3 can be compared with that of low  $Ca$  number of period 2. The big difference is the curvature of interfacial geometry. The higher shear stress bends tip of to-be-dispersed phase and sharpens interfacial geometry. But after the elongation by shear stress and pressure gradient, mechanism turns into next phenomena. It is shown in the result of low  $Ca$  number at period 3. The continuous phase is clogged up by to-be-dispersed phase, and then, continuous phase squeezes the neck of to-be-dispersed phase. Furthermore, the velocity inside droplets is faster than that of near interface, it means this acceleration is not derived from shear stress by the continuous phase at the interface. On the other hand, at high  $Ca$  condition, the continuous flow is not clogged up and it accelerates flow near the interface of to-be-dispersed phase.

At the period 4, it is about to break the tip of to-be-dispersed phase into droplet. In case of low  $Ca$ , continuous flow aggregates to the breaking point because of pressure drop occurred behind the generated droplet. On the other hand, there is no aggregation at breaking point in case of high  $Ca$ . It can be explained by less continuous flow stagnation. It is the largest difference between two mechanisms.

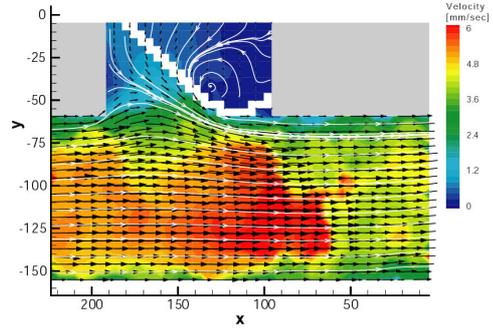
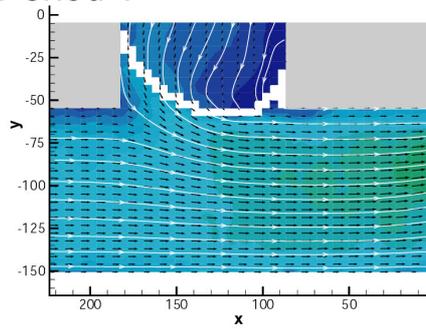
### Three-dimensional Reconstruction of PIV Measurement

The piezoelectric device can control z-position of objective lens precisely. Each plane are measured every  $3 \mu\text{m}$  pitch in z-direction, and we can make three-dimensional velocity distribution map by piling up velocity data of these planes. After that, in order to calculate vertical velocity component, continuity equation was used.

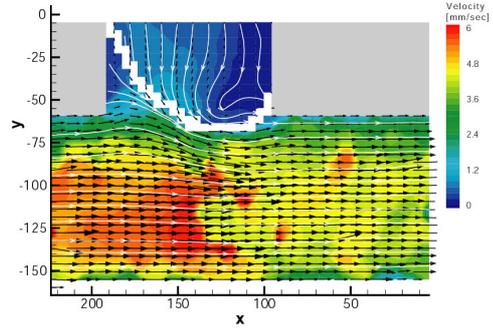
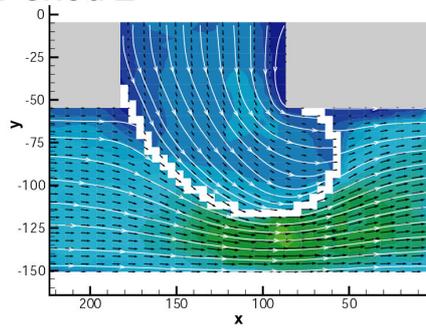
In this study, we considered flow structure symmetric about the mid plane (center height). Therefore, vertical velocity component becomes zero at the mid plane, and we use this assumption for the boundary condition. The calculation proceeds from the mid plane to the near wall plane.

Figure 10 shows three-dimensional flow structures of each phase at squeezing condition. The timing period is about  $t/t_{period} = 0.450$  from droplet generation.

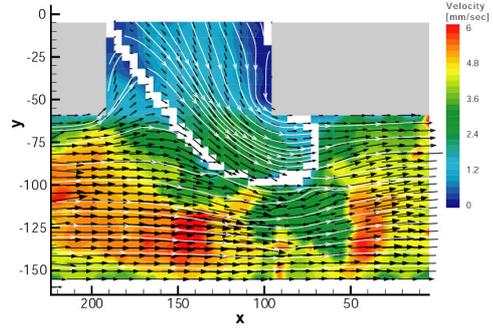
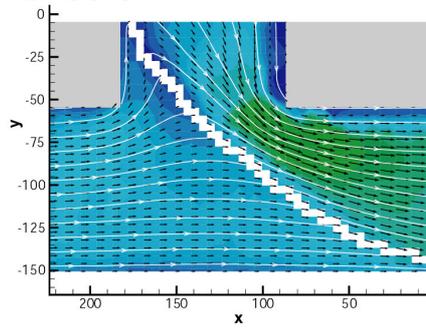
### Period 1



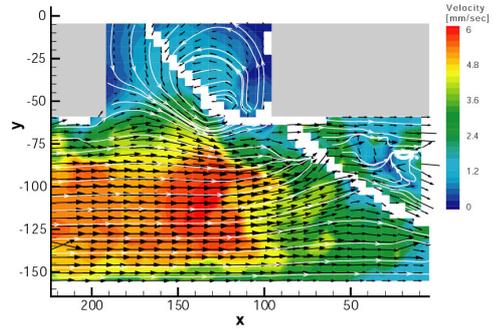
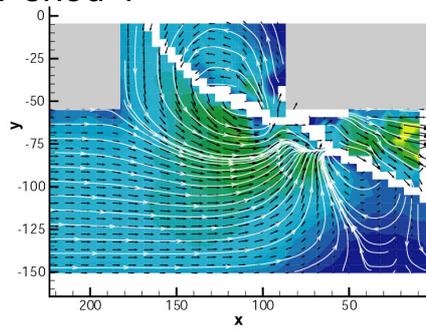
### Period 2



### Period 3



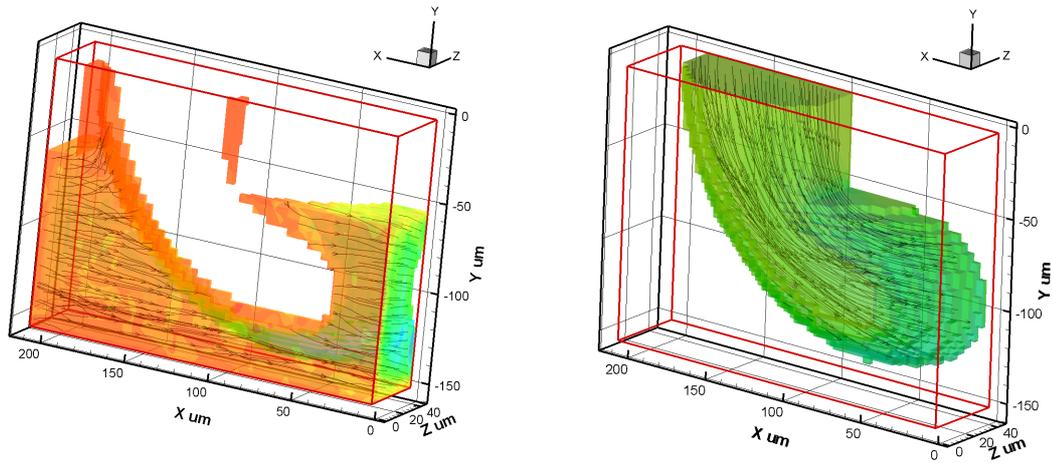
### Period 4



**Fig. 9. The results of PIV measurement at each phase**

**Left: Low Ca number condition, Right: High Ca number condition**

About to-be-dispersed phase, flow path goes straight to its tip and there is no vortex inside it. In contrast, continuous phase have three-dimensional flow structure. The faster flow at the centerline is divided to two direction, one is to the gap between tip of to-be-dispersed phase and channel wall, the other is to upstream of inlet channel of to-be-dispersed phase. Since these gaps near the channel corners are larger than that of mid plane, continuous flow moves to these corners, resulting in three-dimensional flow is generated. From these results, although there is less interaction between two phases in the case of squeezing condition at this time period, it may change depends on  $Ca$  number and another parameters.



**Fig. 10. Three-dimensional flow structures of each phase (half depth)**

**Left: Continuous phase, Right: To-be-dispersed phase**

## CONCLUSIONS

The droplet formation mechanism is a complex phenomenon which is resulted from the interaction of continuous and to-be-dispersed flows such as time-varying interfacial shape, pressure and shear stress induced by continuous flow. The tip of to-be-dispersed phase elongates into the junction with time, and it attempts to narrow the flow path of continuous phase. With this narrowing, velocity of continuous phase increases in inverse proportion to the narrowed path between the tip of to-be-dispersed phase and channel wall. However, when the main channel is clogged up by the to-be-dispersed phase, continuous phase flow rapidly decelerates.

In case of high  $Ca$  condition, the high shear stress derived from relatively fast continuous flow elongates the interface largely downstream of main channel before the main channel clogged up. And then shear stress surpasses the interfacial tension and breaks the tip of to-be-dispersed phase into small droplet, resulting in “dripping” mechanism.

In contrast, in the case of low  $Ca$  condition, the tip of to-be-dispersed phase reaches to the opposite channel wall and clogs up the main channel, because the low shear stress derived from relatively slow continuous phase cannot break to-be-dispersed phase before clogging up. The clogged continuous phase increases its pressure and it pushes interface. As a result, the flow path of to-be-dispersed phase becomes

narrow, and then the velocity of to-be-dispersed phase increases through there. According to the result of confocal micro-PIV measurement, it is clear that this acceleration doesn't derive from shear stress of continuous phase. And this phenomenon results in "Squeezing" mechanism.

In order to clarify this mechanism quantitatively, three-dimensional velocity field and interfacial shape should be measured. We succeeded it by piling up two-dimensional data from confocal micro-PIV measurement and equation of continuity. For further investigation, we need a repeatability of experiment and an improvement of temporal resolution to measure the local flow acceleration. Furthermore, in case of the transition condition between squeezing and dripping, the investigation of power balance between some parameters, which affects to each mechanism, is needed. And our system can provide techniques to clarify these mechanisms.

## ACKNOWLEDGMENTS

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