Confocal Micro-PIV Measurement of Droplet Formation in a T-shaped Micro-junction

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Abstract. This paper aims to investigate a mechanism of microdroplet formation using “multicolor confocal micro particle image velocimetry (PIV)” technique. The present system can measure dynamical behavior of multiphase flow separately and simultaneously. It also enables to identify the interactions between two immiscible fluids. We have applied this system to measure the water droplet formation at a micro T-shaped junction. We have also succeeded in dispersing fluorescent tracer particles into both phases. The interaction between the internal flow of to-be-dispersed water phase and of continuous oil phase is measured as a liquid-liquid multiphase flow. As a result of PIV measurement and interface scanning, the relationship between flow structure of each phase and interface shape is clarified. It indicates 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.

1. INTRODUCTION

For 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. Especially microdroplet is used as a smallest micro chamber to obtain fast reaction process and to make monodisperse microspheres or capsules.

There are some 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).

\[
Ca = \frac{\mu \cdot U}{\gamma} \tag{1}
\]

\( Ca \) means a ratio of shear stress and interfacial tension. The shear stress acts to tear off the tip of to-be-dispersed phase and 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 its border of change as \( Ca \sim 1 \), 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 each phase as a parameter to control $Ca$ number, because viscosity and interfacial tension affects each other.

2. MEASUREMENT METHOD

2.1 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. 1. 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.), high-speed camera (Phantom v7.1, Vision Research Inc.) and an inverted microscope (DMIRE2, Leica Microsystems).

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 two illumination lasers (543-BS-A03, Melles Griot Inc. and Excel 1500, Laser Quantum Ltd.) are combined using a dichroic mirror in the laser combiner and led to the laser port of the confocal scanner using optical fiber. Lasers illuminate the target flow, and Stokes' shifted fluorescent light from the fluorescent particles dispersed in the flow are emitted back through confocal scanner. Then, the multi-wavelength emitted light is separated and filtered at the separation unit, which is shown in Fig.2, and recorded by two high-speed cameras. The present system can collect a sequence of images at a speed of up to 2000 frames per second (fps).

![Fig. 1 Schematic diagram of the multicolor confocal micro PIV System](image-url)
2.2 Target Microchannel

Figure 3 is a schematic illustration of a droplet formation device with a T-shaped junction which width of 100 µm and depth of 80 µm. The patterned microchannel is placed and sealed off on a cover glass where PDMS is spin-coated at a thickness of 10-20 µm 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 µL 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 oil phase flows in the straight main channel at a constant flow rate from inlet A, and the water phase flows from inlet B to the T-shaped junction at a constant flow rate too. Syringe pumps control both flow rates.

The oil phase flow intersects with the water phase flow at the T-shaped junction, where the water flow 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 oil–water interface and the viscosities of the working fluids.

3. RESULTS AND DISCUSSIONS

3.1 Preliminary Experiment

As a preliminary experiment, we searched critical Ca number which indicates the changing point of the droplet formation mechanism. We choose two immiscible fluids 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 and interfacial characteristics.

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. 4 and 5. Relatively slow continuous phase flow rate, 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 Characteristics of two immiscible fluids

| Working fluid  | Continuous phase (inlet A) | To-be-dispersed phase (inlet B) |
|----------------|----------------------------|---------------------------------|
| Silicone Oil   | 0.984                      | 1.162                           |
| Viscosity [mPa•s] | 68.6                      | 10.58                           |
| Flow rate [ml/hr] | 2.4 ~ 100.0               | 2.0, 8.0                        |
| Average flow velocity [mm/sec] | 0.83 ~ 3.47         | 0.69, 0.28                      |
| Interfacial tension [mN/m] | 11.7                     |                                  |
| Capillary number | \( 4.89 \times 10^{-4} \sim 2.04 \times 10^{-2} \) |

As a result of preliminary measurement, fig 6 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 points for each \( Q_d \) plot are about \( Ca = 3.0 \times 10^3 \). It is thought to be causally related to the three-dimensional gap between tip of to-be-dispersed phase and channel wall.
3.2 PIV Measurement

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 $Ca = 3.0 \times 10^{-3}$ for our experimental condition. So we chose two condition of $Ca = 1.63 \times 10^{-3}$ and $8.14 \times 10^{-3}$. Other conditions of PIV measurement is written in Tab. 2. The measurement region is 229 x 172 µm with the in-plane spatial resolution of 0.286 µm/pixel, and measurement height is at its center height.

The refractive indices of the working fluids and channel material needed to match exactly to minimize the refraction and reflection of light at the interface. In this experiment, two immiscible liquids were used as working fluids for refractive index matching: silicone oil (KF-6002, Shin-Etsu Chemical Co. Ltd., Japan) as the oil phase and a mixture of dilute water and glycerol (Wako Pure Chemical Industries, Ltd., Japan) as the water phase. Silicone oil was chosen because it has a refractive index of 1.4122, which is almost equal to that of the PDMS material of the microchannel (refractive index: 1.412). Also, the mixture of dilute water and glycerol has a refractive index of 1.412 when mixed in the proportion of 43% dilute water and 57% glycerol.

To visualize the oil 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 0.9 to 1.4 µm. The $\phi$ 1 µm diameter red fluorescent particles (FluoSpheres F8821, 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 3µm.
Table 2  Conditions of PIV Measurement

|                  | Continuous phase (inlet A) | To-be-dispersed phase (inlet B) |
|------------------|-----------------------------|---------------------------------|
| Working fluid    | Silicone Oil                | Glycerol solution               |
| Flow rate [µl/hr] | 8.0                         | 40.0                            |
| Average flow velocity [mm/sec] | 0.278                        | 1.390                           |
| Capillary number | 1.63E-03 (Squeezing)        | 8.14E-03 (Dripping)             |
| Tracer particle  | φ0.9 – 1.4µm Green Fluorescent silica | φ1.0µm Red fluorescent polystyrene |
| Measurement Depth [µm] | 3.10 – 3.84                  | 3.34                            |
| Refractive index  |                             | 1.412                           |
| Interfacial tension [mN/m] |                             | 11.7                            |

(Experiment temperature : 25°C)

The results of multicolor PIV measurement at each phase are shown in Fig. 7. We compared each three periods, Period 1: after generating droplet, the remaining tip of to-be-dispersed phase reaches to the lower edge of the junction. Period 2: just before detachment of to-be-dispersed phase. Period 3: just after detachment. The left images are the results of low Ca condition and the right images are that of high Ca condition.

At the period 1, the tip of to-be-dispersed phase of low Ca condition is sharper than that of high Ca condition. It derives from difference of shear force affected by the continuous flow near the interface. Strong shear force of high Ca condition drugs and elongates the tip surface of to-be-dispersed phase. This shear force also makes flow circulation inside the tip of to-be-dispersed phase. Only high Ca condition makes strong vortex which has vortex core.

At the period 2, the continuous phase is clogged up by to-be-dispersed phase at low Ca condition. 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 3, the continuous flow follows after the droplet at low Ca condition. In contrary, the continuous flow pushes the droplet at high Ca condition.
3.2 Three-dimensional Reconstruction of PIV Results

The piezoelectric device can control z-position of objective lens precisely. Each plane are measured every 3µm pitch in z-direction, and we can make three-dimensional velocity distribution map by piling up velocity datas 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 8 shows three-dimensional flow structures of each phase at squeezing condition. The timing period is about 0.45t from droplet generation.

About to-be-dispersed phase, flow path goes straight to its tip and there are 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 four 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 are less interaction between two phases in the case of queezing condition and time period, it may change depends on $Ca$ number and another parameters. We have to speed up the confocal scanning speed for further investigation.
4. CONCLUSIONS
From these results, it seems that the important parameter is a gap between the tip of to-be-dispersed phase and channel wall. If the gap is large, surrounding continuous fluid gives strong shear stress on the surface of to-be-dispersed phase and finally it tears off (“dripping”) its tip. On the contrary, smaller gap can’t put through the continuous fluid flow, and it leads to low shear stress, which is not enough to tear off small droplet, resulting in “squeezing”.

In conclusion, the droplet formation mechanism depends on the $Ca$ number and gap region that is determined by flow rate of to-be-dispersed phase and channel size. Since both parameters are dependent on time, it is not enough to decide the critical $Ca$ number, which identifies change of formation mechanism from “squeezing” to “dripping”. The present measurement also clarifies mechanism of the transient behavior.

We also constructed a multicolor confocal micro-PIV system to measure behavior of multiphase flow. The selection of lasers, optical components and fluorescent materials for ideal multicolor separation is a key point of this system. It is also important that the optical arrangement in the multicolor separation unit. Using the present system, we can remove unnecessary scattering light and obtain only particle fluorescent images in different phases. The present system was applied to measure the liquid-liquid multiphase flow in a microchannel. It clarifies the strong interaction between phases in detail.

NOMENCLATURE

\begin{align*}
W & \quad \text{channel width} \quad [\text{m}] \\
D & \quad \text{channel depth} \quad [\text{m}] \\
A & \quad \text{area of bubble surface} \quad [\text{m}^2] \\
Ca & \quad \text{Capillary number} \quad [-] \\
Q & \quad \text{flow rate} \quad [\text{m}^3/\text{sec}] \\
u & \quad \text{flow velocity} \quad [\text{m/sec}] \\
D_{b} & \quad \text{length of droplet} \quad [\text{m}] \\
\alpha & \quad \text{thermal diffusivity} \quad [\text{m}^2/\text{s}] \\
\mu & \quad \text{viscosity} \quad [\text{mPa sec}] \\
\gamma & \quad \text{interfacial tension} \quad [\text{mN/m}] 
\end{align*}
Subscripts

\(c\) fluid of continuous flow
\(d\) fluid of to-be-dispersed flow

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