Three-Dimensional Analysis of Liquid Propagation at Microchannel Junction using ESEM

Natsumi Yagi and Akira Kawai

Department of Electrical Engineering, Nagaoka University of Technology
1603-1 Kamitomioka, Nagaoka, Niigata 940-2188 Japan
*kawai@nagaokaut.ac.jp

The three-dimensional wetting phenomena in a T-shaped microchannel made of a SU-8 photoresist accompanying with a pillar sub-pattern are analyzed by using an environmental scanning electron microscopy (ESEM). By lowering the sample temperature in the ESEM chamber at constant the H2O pressure, the water condensation occurs under H2O dew point and the liquid propagation in the microchannel can be observed. The experimental results show that the pinning angle of water flow at T-junction is 58 degrees in the T-shaped microchannel. In the case of the T-shaped microchannel with the sub-pattern, it is found that the pinning angle becomes low by adhering the water around the sub-pattern. These results can realize to the smooth flow at the channel junction without any water trapping for the three-dimensional microfluidic devices.

Keywords: ESEM, Microchannel, Pillar-pattern, Three-dimensional analysis

1. Introduction

In recent years, the three-dimensional microfluidic devices have been actively studied in varieties of fields such as electric, biological, and medical industries [1-6]. For extension to the three-dimensional, the micro channel and tube applications are recognized as the important key technologies. In the liquid propagation analysis in the microchannel, the volume-of-fluid (VOF) method the numerical simulation method is well known as one of the most typical methods [7-10]. The collaboration on the simulations and the experimental works are effective in order to analyze the liquid propagation. In this regard, an environmental scanning electron microscopy (ESEM) is an effective system in order to evaluate the three-dimensional wetting phenomena in a microstructure by the water condensation [11]. Meanwhile, in functional microchannel fabrication, it is required to design some corrugated surfaces or some pillars at an intersection among microchannels [12-15]. The authors have already discussed the guiding liquid propagation at the junction of the microchannel two-dimensionally [16]. The role of pinning effect around the sub-pattern was clarified. The effectiveness of ESEM observation in wetting phenomena was also confirmed.

In this paper, we discuss the three-dimensional liquid propagation in the microchannel by the in-situ ESEM observation. On the points of the three-dimensional observation and the pinning effect at the junction, we focus on the effectiveness of a sub-pattern of controlling the liquid flow in the microchannel network [17]. The in-situ ESEM observation contributes to the study of the microfluidic devices to extend the three-dimensional.

2. Experimental

2.1. Fabrication of microchannel

In this experiment, a T-shaped junction of microchannels were formed with a SU-8 photoresist (3050, Kayaku Chemical Corp.) by the typical photolithography process summarized in Table 1. A Si substrate was cleaned up with organic solvent by acetone, methanol and deionized water each for 5 min. For the high adhesion between the Si substrate and a photoresist film, a hydrophobic treatment with HMDS (hexamethyldisilxane) were performed on the substrate prior to the photoresist coating. The width and height of the channel were 100 and 40 μm, respectively.
2.2. Microchannel structure characteristics

Figure 1a shows the ESEM images of T-shaped microchannel structures in this experiment. Figure 1b is a T-shaped microchannel with a pillar shape sub-pattern at the right side of the junction center. The pillar shape was pinched toward a base. The top diameter and height of the pillar were about 50 and 40 μm, respectively.

2.3. In-situ observation using ESEM

The in-situ observations of water condensation and propagation in the T-shaped microchannels were carried out using the ESEM (XL30, Philips). A peltier cooling stage was equipped in order to control the sample temperature around the water dew point at the constant H₂O pressure (560 Pa). The T-shaped microchannel was cooled gradually under 10 °C as shown in Fig. 2a, at under 0 °C, the relative humidity on the sample surfaces reached to a saturation vapor condition. Likewise, the T-shaped microchannel with the sub-pattern was cooled as shown in Fig. 2b. In the first stage, the distilled water was introduced from the external system.

By lowering the temperature, the condensed water began to generate gradually at -1.2 °C. Then, the condensed water progressively covered on the whole area of the sample surface. Finally, by raising the stage temperature, the condensed water was evaporated and disappeared.

3. Results and discussion

3.1. Water propagation in the T-shaped microchannel

Figure 3 summarizes the time-lapse ESEM images of the water propagation in the T-shaped microchannel by tilting the sample stage at 30 degrees. In the beginning of the observation (Fig. 3a), the condensed water was filled out from a side-wall bottom of the channel and propagated along the side-wall. Then, the condensed water were propagated from both the front and left sides of the junction (Fig. 3b). In the front side, the right edge of condensed water relatively delays to reach the junction and to fill up to the height of the microchannel (Fig. 3c). And then, the condensed water both of front and left sides of the junction coalesce (Fig. 3d). The condensed water was held at the junction due to the liquid pinning effect at the corner of the right side (Fig. 3e). The condensed

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Table 1. SU-8 microchannel fabrication process.

<table>
<thead>
<tr>
<th>Processes</th>
<th>Parameter condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>HMDS vapor</td>
<td>10 min</td>
</tr>
<tr>
<td>heating treatment</td>
<td>120 °C / 60 s</td>
</tr>
<tr>
<td>spin coat</td>
<td>500 rpm / 5 s, 3000 rpm / 30 s</td>
</tr>
<tr>
<td>pre-bake</td>
<td>95 °C / 30 min</td>
</tr>
<tr>
<td>exposure</td>
<td>4 s</td>
</tr>
<tr>
<td>post exposure bake</td>
<td>65 °C / 1 min</td>
</tr>
<tr>
<td>development</td>
<td>95°C / 5 min</td>
</tr>
<tr>
<td>rinse</td>
<td>30 s</td>
</tr>
<tr>
<td>hard bake</td>
<td>130 °C / 10 min</td>
</tr>
</tbody>
</table>

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(a) T-shaped microchannel

(b) T-shaped microchannel with the sub-pattern

Fig. 1. ESEM images and schematics of T-shaped microchannels.

(a) T-shaped microchannel

(b) T-shaped microchannel with the sub-pattern

Fig. 2. Temperature changes of peltier stage in ESEM.
water was overflowed from left side of the junction (Fig. 3f). In Fig. 3f, In the right side bottom of the channel junction, a slight dry area can be confirmed due to the strong pinning effect. It will become a seed of bubble generation.

3.2. Basic property of water condensation

The time lapse number of dew water droplet size by size is shown in Fig. 4. By counting the number of dew water droplet size by size in the standard area as shown in Fig. 3b (dashed line area) in the each time-lapse ESEM images, the following basic property of water condensation can be obtained. Figure 4a is a time lapse number of water droplet in small size. Figure 4b is a time lapse number of water droplet of the “others” in the explanatory notes of Fig. 4a. In the explanatory notes, the items represented numbers are each surface area (square micrometer) of the water droplets. As a result, in Fig. 4a, at an early dew stage, the relatively small water droplets less than 10 µm² is obtained a majority. As the time passes, the number of water droplet decreased and the ratio of the large water droplets less than 100 µm² but the other area group increased. In Fig. 4b, as the time passes the quite large water droplets were condensed by coalescing water droplet each other. Finally, at 28.6 min passed, the area of a water droplet increased to be a half of the standard area. This phenomena can be divided into two stages, “condensation” and “coalescing”. The border between two stages occurs at 20.8 min.

3.3. Three dimensional analysis of water propagation

In order to analyze the three-dimensional water propagation, the height and position of the time-lapse ESEM images in the microchannel in propagating water are shown in Fig. 3. The height and the position in the microchannel were defined in Fig. 5. From these results, the position in the

![Fig. 3. Time-lapse ESEM images of the T-shaped microchannel.](image_url)

![Fig. 4. Time-lapse number of dew water droplet size by size.](image_url)
microchannel and the height of water the time lapse water propagation were shown in Fig. 6. In Figs. 6a, 6b, and 6c, the water propagated from the base to the top of the microchannel. Particularly, water propagation in Fig. 6b is explained by the following Lucas-Washburn equation [18-20]

\[ l = \left( \frac{d \gamma \cos \theta}{4 \eta} \right)^{\frac{1}{2}} \],  

(1)

where the symbols \( l \), \( d \) and \( t \) represent the length of the liquid column, the diameter of the capillary tube and passed time, respectively. The symbols \( \gamma \), \( \eta \) and \( \theta \) denote the surface energy of liquid, the viscosity and contact angle, respectively. In addition, the water at the center position delayed from both side edges as shown in Fig. 6b. From this result, the water at the center were pulled by the surface tension proceeding from the side walls made of SU-8 photoresist. In Fig. 6c, the tendency of propagation is a little different from other positions due to the wall contamination. Furthermore, from the results of Figs. 6a, 6b and 6c, the instant velocity were calculated as shown in Fig. 7. From the above results, the water propagated in microchannel can be discussed by using total energy \( \Delta G \). The change of total energy \( \Delta G \) can be expressed by the following equation.

\[ -\Delta G = \Delta S_1 \gamma / A + \Delta S_2 \gamma / R + \Delta S_3 \gamma / Si \],  

(2)

where the subscripts of surface energy, \( w \), \( A \), \( R \), \( Si \) represent water, air, SU-8 photoresist walls, Si substrate, respectively. In addition, \( \Delta S_1 \) (between water and air), \( \Delta S_2 \) (between water and SU-8) and \( \Delta S_3 \) (between water and Si) are the difference of surface area when the water propagates \( \Delta x \) as shown in Fig. 5b. If the water flow approaches the junction, the water flow is classified into two stages: "stay" and "pass" as follows.

stay : \( \Delta G < 0 \), \( \Delta S_1 \) : small
pass : \( \Delta G > 0 \), \( \Delta S_1 \) : large

In the case of the pass, the total energy \( \Delta G \) is greater than zero because the difference of surface area \( \Delta S_1 \) is larger than that of the case of the stay as shown in Fig. 8. In this regard, the both corners of the junction can be a significant factor between the two ways. The pinning effect is easy to cause at the corners.
3. Pinning angle measurement

In Fig. 3, the pinning effect occurs at the right side corner. In this case, the pinning angle is defined as the angle forming between the baseline at the junction and the contact line of water and Si substrate as shown in Fig. 9. The pinning angles were measured in the time lapse ESEM image shown in Fig. 3 accompany with other moments. As a result, the time lapse pinning angle is defined as shown in Fig. 10. From the result, at time passes, the pinning angle gradually increased. Noteworthy, the threshold angle of the water propagation from junction to right side of the microchannel was clarified to be 58 degrees. After the water propagation the pinning angle decreased rapidly.

3.5. Effect of sub-pattern

Figure 11 shows the time-lapse ESEM images of the water propagation in the T-shaped microchannel with sub-pattern by tilting the sample stage of 45 degrees. As shown in Fig. 11a, 11b, the condensed water propagation occur from the front to left sides of the junction. Then, the water propagated from right side of the microchannel. (Fig. 11a) In order to analyze these liquid condensation phenomena, we employ $\Delta P$ defined as a pressure difference across the interface between water and ditch interface as the following well known Young-Laplace equation,

$$\Delta P = \frac{2\gamma}{r}$$

where $r$ is radius of the a ditch. It is clearly stated that a pressure difference $\Delta P$ becomes large as the channel width becomes narrower. Around the sub-pattern at the right side of the junction, the water meniscuses are guided and formed due to the pressure difference $\Delta P$. Particularly, in the right

Fig. 8. Two kinds of water propagation at the junction.

Fig. 9. Definition of pinning angle.
channel bottom, water condensation is noticeable. In Fig. 11g, the left channel is almost filled up by coalescing the water around the sub-pattern due to the liquid pinning effect. On the contrary, in the left side bottom of the channel junction, a slight local dry area can be confirmed because the meniscus condensation around the left side sub-pattern is dominant. Noteworthy, at the right side corner, the water was not held. From this result, it is clarified that the sub-pattern is effective to smooth the liquid flow in the channel junction by decreasing the pinning angle.

4. Conclusion

In conclusion, by the in-situ three-dimensional ESEM observation, it is clarified that the pinning angle of water propagation is 58 degrees at the T-shaped microchannel junction. In the case of the T-shaped microchannel with the sub-pattern, it is found that the pinning angle becomes low by adhering the water around the sub-pattern. Therefore, it is clarified that the sub-pattern is effective to control the liquid flow at the channel junction by decreasing the pinning angle. These results can realize to the smooth flow at the channel junction without any water trapping for the three-dimensional microfluidic devices.

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