공학석사학위논문

Deposition pattern of colloidal particles
in electrohydrodynamic (EHD) jet printing

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패턴 형성에 관한 연구

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이 논문을 공학석사학위논문으로 제출함
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Abstract

Deposition pattern of colloidal particles in electrohydrodynamic (EHD) jet printing

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In this study, the colloidal suspensions containing silica nanoparticles were deposited by electrohydrodynamic (EHD) jet prninting and the line-patterns were systematically observed. EHD jet printing is a method to eject the fluid and form line-patterns by applying high DC voltage similar to ink-jet printing that applies pressure to generate droplets.

To make the line-pattern with a uniform shape, the stability of EHD jetting should be guaranteed. It depends on the operating conditions (DC voltage, flow rate, and the distance between the nozzle and the substrate) and the properties of the suspension (viscosity, surface tension, dielectric constant, and conductivity). Among the material properties, we focused on the effect of viscosity on the stability of jetting by controlling the concentration of poly(vinyl alcohol) (PVA).

For the wide range of concentration, we observed the effect of operating conditions and the concentration of PVA on the surface profile of line-patterns by using optical microscope, scanning electron microscope (SEM), and profilometer. From these observations, we could find a change of the microstructure of silica/PVA and its effect on the surface profile of line-patterns. Consequently, the uniformity of

the surface profile was decreased with the increase of PVA concentration. Conclusively, we could control the uniformity of the deposited pattern shape by changing the polymer concentration.

Key words: electrohydrodynamic (EHD) jet printing, the stability of jetting, deposition pattern of colloidal particles, surface profile of line-patterns

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Chapter 1 Introduction

There has been growing interested in direct-write technologies which are any technique or process capable of depositing various types of materials on the substrate[1]. Also, it has several advantages of deposition due to low cost, no mask for patterns, and a free choice of substrates, and a variety of applications[2,3,4,5,6]. Among them, the ink-jet printing which is a widely used method for the direct-write applies pressure to generate droplets through a nozzle. When the fine resolution patterns are need, the ink-jet printing should require the nozzle with the smaller diameter than the width of pattern because the diameter of droplet which is ejected by the ink-jet printing is about twice as big. Also, when the ink-jet printing deposits colloidal suspensions with using a small diameter, there is the probability of the nozzle clogging due to the flow-induced aggregation which is often happened in the ink-jet printing[7]. In contrast to the ink-jet printing, in case of the EHD jet printing which is a method to eject droplets or jet by applying the high DC voltage between the nozzle and the counter electrode to create an electrostatic force at the interface of fluid, it doesn't need a small diameter of the nozzle for high resolution printing because an electrostatic force induces the formation of a cone-jet shape whose jet diameter is typically smaller than the size of the nozzle by more than 1 order of magnitude[8,9,10]. By obtaining a cone-jet shape, it is possible to deposit and pattern materials as required even for the smaller size than the diameter of the nozzle. However, when the high DC voltage is applied, it is not that the drop which is hanging on the tip of the nozzle instantly changes into a cone-jet shape. While the drop is changed into a cone-jet shape, there are several jetting modes (Dripping, micro-dripping, and cone-jet) depending on the applied DC voltage[11]. Each mode is

determined by the operating conditions (DC voltage, flow rate, and the distance between the nozzle and the substrate) and the properties of ink (viscosity, surface tension, dielectric constant, conductivity). There are many variables to have an effect on the EHD jetting. It is difficult to control each variable for a cone-jet. So, many researchers have focused on the systematic study about the cone-jet mode with the dimensionless number which is based on the properties of solution and the operating conditions[10,11]. In this study, we have experimentally observed the effect of viscosity of solution on the stability of jetting by controlling the concentration of Poly(vinyl alchol) (PVA). When the concentration of PVA is greater than 3wt%, the stable cone-jet mode doesn't exist anymore. Then, we could expect that the stability of jetting be related to the dimensionless group which is nondimensionalized by the viscosity of solution.

Previous study mainly has observed the surface profile of line-patterns of colloidal particles and the effect of operating condition on the width of line-patterns for the deposited patterns. In this study, we have observed even the stability of jetting is achieved and the width of deposited patterns is similar, the surface profile of line-patterns and the microstructure of the surface could be different due to the concentration of PVA. Therefore, we have investigated the effect of the concentration of PVA on the microstructure of line-patterns and found that the microstructure of line-patterns is related to the surface profile.

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Chapter 2 Experimental

2.1. Materials

Silica nanoparticles (Ludox HS-40) were purchased from Sigma Aldrich. It is dispersed in water at 40wt% and electrically stabilized with the negative charged at pH 9 according to the supplier. Silica particle has an averaged diameter of 21 nm, a specific surface area of 220 m^2/g, and a density of 2.37×10^3 kg/m^3. Poly(vinyl alcohol) (PVA) with a molecular weight of $(31\text{-}50) \times 10^3$ mol/g, a degree of hydrolysis of 87-88%, and a density of 1.27×10^3 kg/m^3 was purchased from Sigma Aldrich. A 20wt% PVA solution was prepared by dissolving PVA in deionized water at 353K for 3h. The weight fraction the silica/PVA solution is represented in Table 2-1. All experiments were conducted with the samples stirred at 200rpm for 24h because there is the time dependence[12].

2.2. Characterization

The sample was characterized by measuring the viscosity, the surface tension, permittivity, and conductivity. The viscosity was measured with a stress-controlled rheometer (DHR, TA Instrument, USA). The surface tension was measured with the force tensionmeter (K100 SF, Germany). The conductivity was measured with Cond 720 (inoLab, Germany). The permittivity was measured by dielectric analyzer (SI 1260 impedance/gain-phase analyzer and dielectric interface, Solartron, U.K.). All measurements were conducted at room temperature. The measured values of viscosity, surface tension , density, permittivity, and conductivity were presented in Table 1. The surface and the cross-section of the deposited pattern was were observed with scanning electron microscope (SEM, JSM-6700F, JEOL, USA). The surface profile of the deposited pattern was measured with the optical pofilometer (μSurf, Nanofocus, Germany).

Table 2-1 The composition and the property of Silica/PVA solution.

Silica [wt%]	PVA [wt%]	Conductivity [mS/m]	Dielectric constant	Density [Kg/m^3]	Surface tension [mN/m]	Viscosity [cP]
20	1	367	78	1125	45	3.4
20	2	395	78	1128	43	4.5
20	3	408	78	1131	42	7.6
20	4	413	78	1133	42	11
20	5	420	78	1136	41	35

2.3. Printing set-up

The size of nozzle which is stainless steel is OD 360 μ m and ID 180 μ m. The fluid was injected at a constant flow rate to the nozzle by using syringe pump (Longer Pump, Model LSP02-1B). The nozzle and the counter electrode which is the copper plate were connected to the high-voltage power supply (NanoNC). The substrate which was used as glass was laid on the counter electrode. By using the high-speed camera (Photron fastcam-ultima 512) with the light source (MORITEX, 250W Metal Halide lamp), the jetting was observed. The moving stage worked with the stepping motor (DS 102, Stepping Motor controller, SURUGA SEIKI). A schematic configuration of the printing set-up is shown in Figure 2-1.
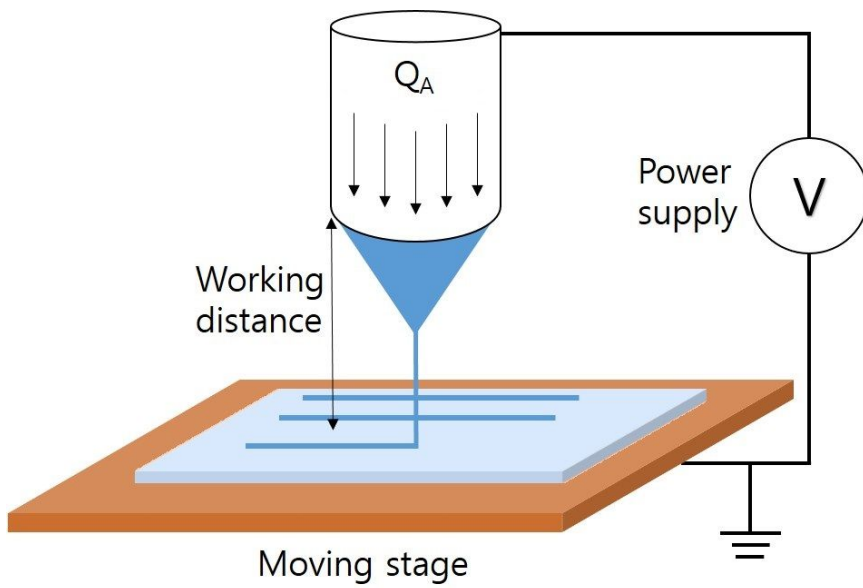
Figure 2-1 A schematic configuration of the printing set-up

Chapter 3 Result & discussion

To get the deposited patterns, the jetting should be conducted and certainly have an effect on the shape of the deposition[13]. So, at first, we will see the stability of jetting. Then, we will observe the deposited patterns.
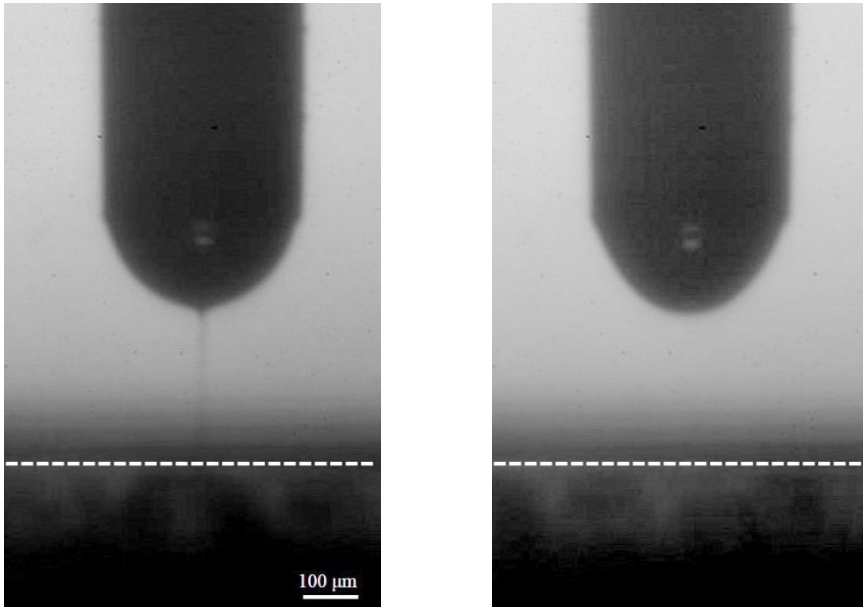
3.1. The stability of jetting

EHD process has many variables which consist of operating conditions and properties of the solution. The variables are as below.

Density (ρ), surface tension (γ), permittivity of free space (ε_0), permittivity of fluid (ε_r), conductivity (K), viscosity (η), diameter of nozzle (d), working distance (L), the applied flow rate (Q_A), and applied DC voltage (V_A).

So, though the dimensionless analysis based on the presented variables above, we can find six dimensionless groups which is shown in Table 3-1[11]. From previous study, we could know that there is the required conditions for a cone-jet shape. First, the electrical relaxation time T_q ($=\varepsilon_0\varepsilon_r/K$) is smaller than the hydrodynamic relaxation time T_h ($=d^2L/Q_A$). When this condition is satisfied, it means that the liquid inside is quasi-neutral and free charges only are at the liquid-gas interface. Then, the charge layer will be developed at the interface[9]. Second, α and β are greater than 1. It means the applied flow rate and voltage is greater than the minimum flow rate and voltage for a cone-jet. For the comparison, we have fixed the operating conditions as follows. The flow rate is 0.01 ml/h, the working distance is 400 μm, the diameter (OD) of nozzle is 360 μm. The calculated ratio of all samples is shown in Table 3-1 and the required conditions for a cone-jet are met.

From this result, we can expect that all samples show a cone-jet shape. However, actually, when the concentration of PVA is greater than 3wt%, the stable cone-jet mode doesn't exist anymore. The cone-jet periodically appears and disappears at PVA 4wt% in Figure 3-1. However, the concentration of PVA is lower than 4wt%, the stable cone-jet can be seen in Figure 3-2. As shown Table 2-1, there is no big difference except the viscosity. When we compare the dimensionless values in Table 3-1, we can see the increase of the value along with the viscosity because this is based on only the property of the solution. This dimensionless value is considered as the ratio of the characteristic velocities. The propagation velocity of a perturbation across the jet by molecular (viscous) diffusion ($\eta/\rho d_0$) and the characteristic velocity of the liquid bulk $(Q_m/d_0{}^2)$[11]. This ratio means how the fluid is accelerated. As the ratio of all samples are greater than 1 in Table 3-1, the fluid of samples is accelerated by the same way. However, previous studies don't link this ratio to the stability of jetting and there is not a theoretical approach. For now, it is difficult for us to explain this result in detail. Though these results, we could suggest that there be a range of the value to achieve the stability of jetting with the viscosity.

Figure 3-1 The instability of jetting. The cone-jet periodically appears (left) and disappears (right) at PVA 4wt%. The bottom line presents the substrate.
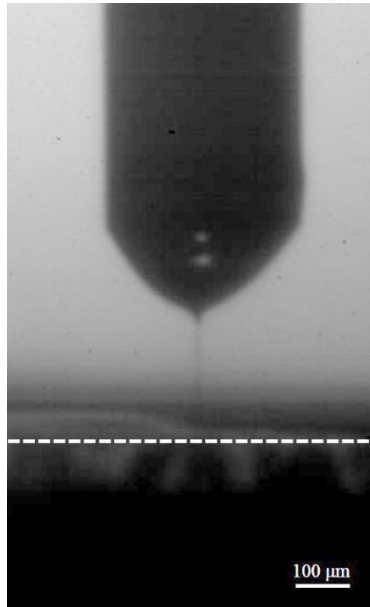
Figure 3-2 The stability of jetting. The cone-shape is maintained. The bottom line represents the substrate.

Table 3-1 The calculated value of dimensionless parameter.

PVA [wt%]	$\dfrac{T_q}{T_h}$ $\dfrac{\varepsilon_0 \varepsilon_r Q_A}{d^2 L K}$	χ $\dfrac{\eta}{\left(\gamma^2 \rho \frac{\varepsilon_0 \varepsilon_r}{K}\right)^{1/3}}$	ε_r ε_r	L/d $\dfrac{L}{d}$	α $\dfrac{\rho K Q_A}{\gamma \varepsilon_0 \varepsilon_r}$	β $\dfrac{\sqrt{\varepsilon_0} V_A}{\sqrt{\gamma d}}$
1	1.01×10^{-10}	2.09	78	1.11	13.54	1.77
2	9.37×10^{-11}	2.92	78	1.11	16.46	1.82
3	9.01×10^{-11}	4.67	78	1.11	18.03	1.84
4	8.96×10^{-11}	7.36	78	1.11	18.50	1.84
5	8.81×10^{-11}	23.9	78	1.11	19.66	1.86

3.2. Deposition pattern

From previous study, the effect of the shape of deposited patterns are shown[14]. Therefore, in this study, we have observed the width of line-patterns depending on the operating conditions and found the effect of the concentration of PVA on the shape of deposited patterns. Though the stability of the jetting, we could know a range of the concentration of PVA for a stable cone-jet. In this range (PVA 1, 2, 3wt%), we got the various width of line-patterns with using the speed of moving stage and the applied DC voltage in Figure 3-3 and 3-4.
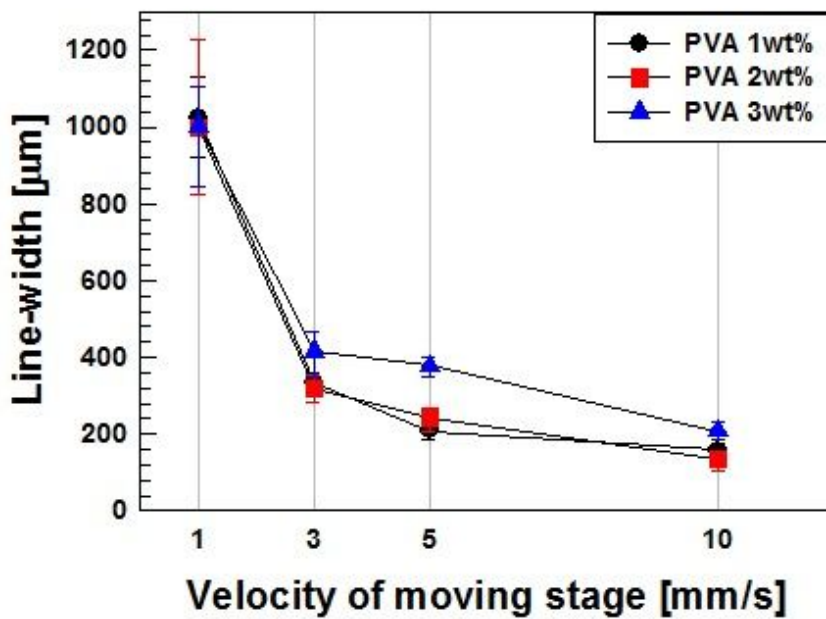
Figure 3-3 The width of line-patterns depending on the velocity of moving stage. We fixed the applied DC voltage at 2.4kV.
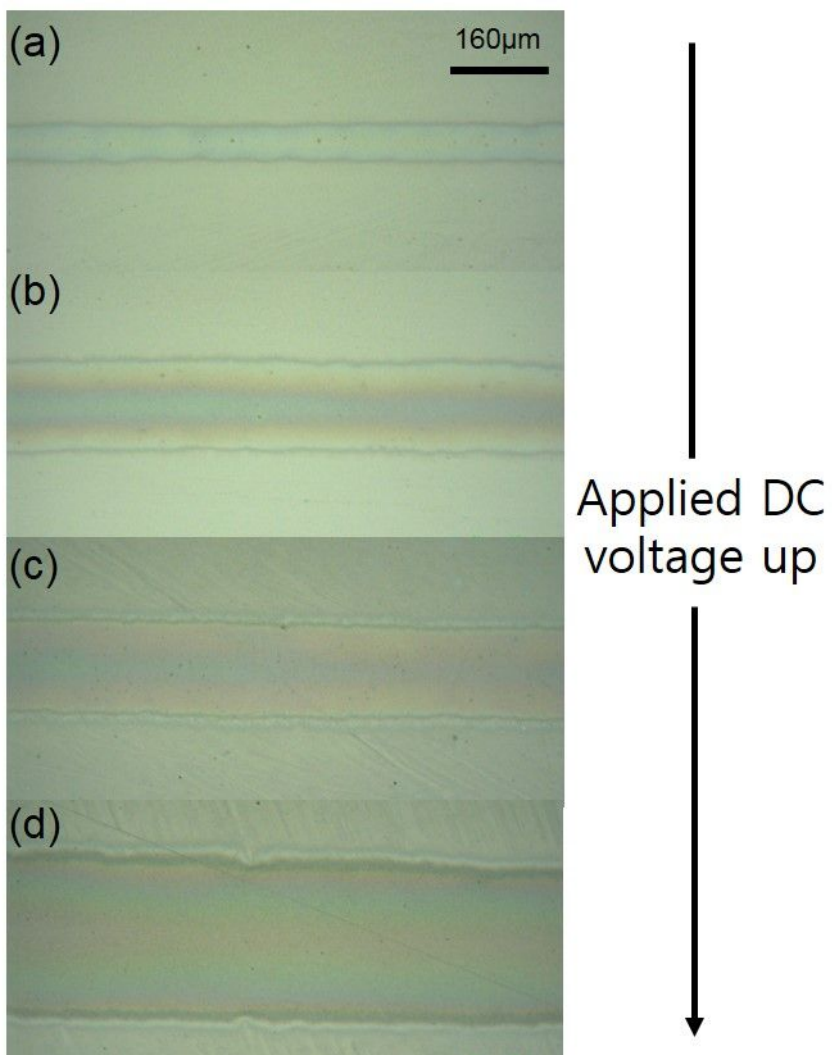
Figure 3-4 The width of line-patterns depending on the applied DC voltage. We fixed the velocity of moving stage at 5 mm/s. The used sample is PVA 1wt%. The applied DC voltage is (a) 2.0 kV (b) 2.2 kV (c) 2.4 kV (d) 2.6kV

The width of line-patterns decreases as the speed of the moving stage increases. As the ejected volume is piled up on the substrate, the width of line-patterns is increased. On the other hand, in case of DC voltage with the fixed velocity of moving stage, the width of line patterns is increased as the applied DC voltage increases. Because the applied DC voltage induces the electrical force at the interface, it can be considered as the external force. So, the flow rate is increased. As a result, the width of line-patterns is increased. From these result, we could see that it doesn't seem that the width of line-patterns is affected by the viscosity. It looks like being just determined by the operating conditions in this study.

By using the optical profilometer, we have measured the surface profile of line-patterns. It is presented in Figure 3-5 and 3-6. As the figures are shown, the surface profile between PVA 1wt% and 3wt% is similar when the width of line-pattern is about 200 μm. However, in case the width which is about 800μm, it looks different from the surface profile and the roughness of the surface. Then, it is obvious when the roughness of the surface was compared in Figure 3-7. The roughness is represented in the variation of height of the middle of line-pattern which is roughly 800μm.
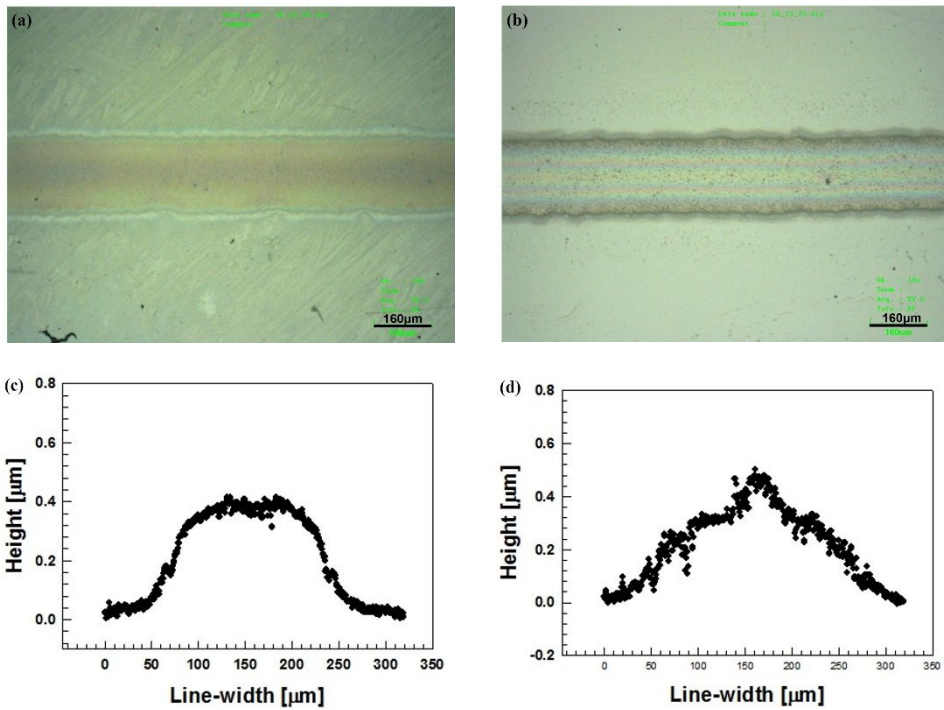
Figure 3-5 The surface profile of line-patterns. (a) and (c) are PVA 1wt%. (b) and (d) are PVA 3wt%. The velocity of moving stage is 10 mm/s and the applied DC voltage 2.4kV.
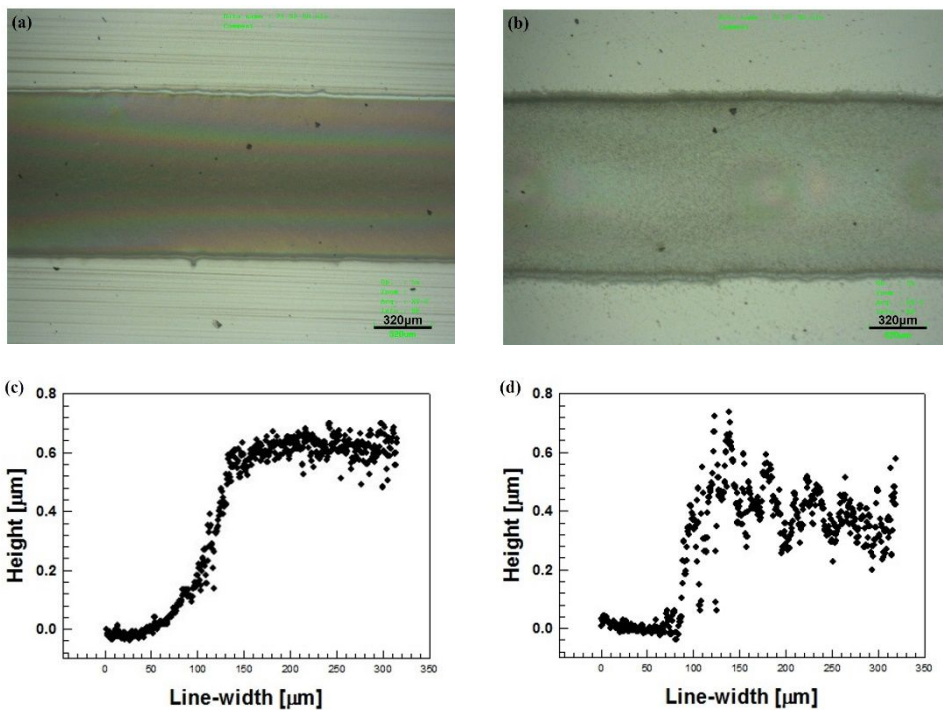
Figure 3-6 The surface profile of line-patterns. (a) and (c) are PVA 1wt%. (b) and (d) are PVA 3wt%. The velocity of moving stage is 1 mm/s and the applied DC voltage is 2.4kV.
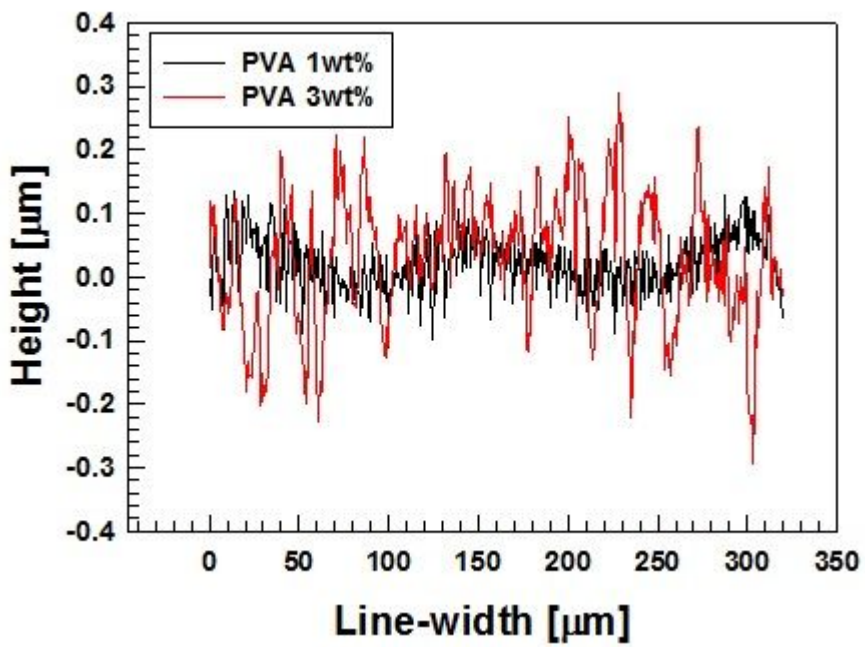
Figure 3-7 The roughness of the surface between PVA 1wt% and 3wt%. The roughness is represented in the variation of height of the middle of line-pattern which is roughly 800μm.

To find out the difference between them, we have observed the surface of line-pattern with scanning electron microscope (SEM) in Figure 3-8 and 3-9. SEM images show what makes the difference between them in the surface profile. As shown in figures, there is the different microstructure along with the concentration of PVA. Now, we could understand the difference of the surface profile between PVA 3wt% and PVA 1wt% by the microstructure. We will discuss the difference of the surface profile between PVA 1wt% and PVA 3wt% in microstructure of silica/PVA.
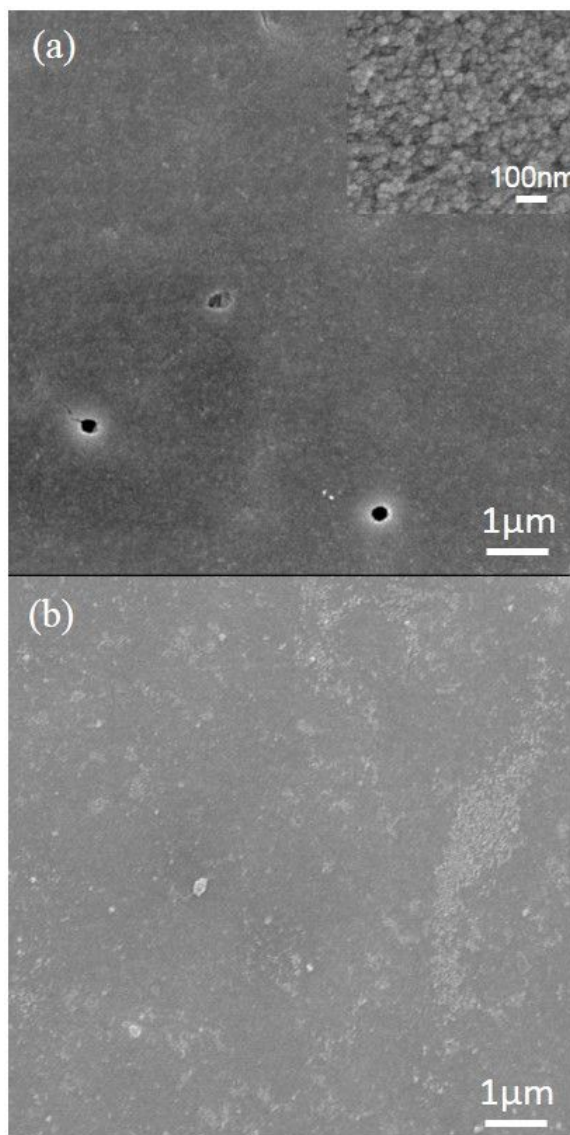
Figure 3-8 SEM images of line patterns of PVA 1wt%. The width of (a) and (b) is about 200μm and 800μm.
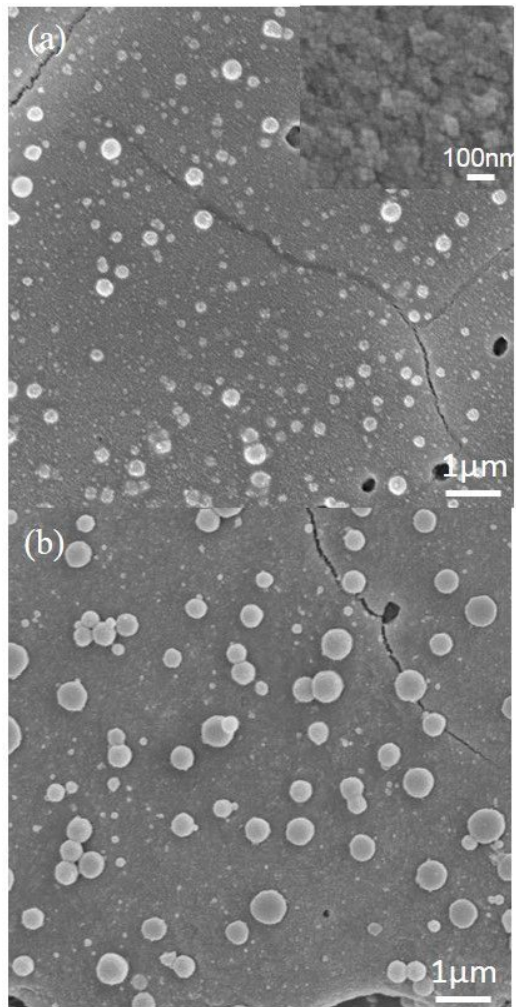
Figure 3-9 SEM images of line patterns of PVA 3wt%. The width of (a) and (b) is about 200μm and 800μm.

3.3. Microstructure of Silica/PVA

To figure out the difference of the microstructure, we have to consider the relationship between silica and PVA and the effect of the concentration of PVA on the microstructure. In case of silica, it has the silanol group (-SiOH) on the silica surface. Then, there is the hydrodgen bonding between the hydroxyl group (-OH) of PVA and silanol group of the silica[17,18]. Therefore, the amount of PVA adsorption is affected by the pH value because the number of silianol groups is increased as the pH value decreases. In this study, the pH of all samples is 9. At pH 9, it can presume that the amount of adsorbed PVA is very small[12,17,18]. In other words, the most PVA is non-adsorbed. Then, we could expect that the concentration of the free polymer in PVA 3wt% is higher than PVA 1wt%.

Due to the non-adsorbed polymer, there can be the interaction between the colloidal particle and the free polymer. In case of a mixture of colloid and polymer, there is the attractive force between colloidal particles when the free polymer is expelled from the space between colloidal particles. As the polymer is expelled, it induce an unbalanced osmotic pressure which makes colloidal particles together. Then, if this force is enough, there will be the flocculated colloidal particles. In generally, it is called depletion flocculation. This depletion force is a function of the concentration of the free polymer[19,20,21,22]. At this point of view, we could suggest that there be the stronger depletion force in PVA 3wt% than PVA 1wt%. Due to the difference of depletion force, the occurrence of depletion flocculation is only in PVA 3wt%.

However, until now, we didn't consider the interaction between colloidal particles. In case of silica, it has the negative charged on the surface of the silica particle at

pH 9 and is electrically stabilized[18]. It means if silica/PVA is in medium without evaporation, the suspension will be stable. Therefore, the flocculation is created during drying. We thought about the process of drying to figure out what happened. Because of evaporation of the solvent, there is the increase of the concentration of everything in suspension except the solvent. Then, silica suspension contains not only silica particle but also ions such as Chlorides (NaCl) and Sulfates (Na_2SO_4). So, the increase of the concentration of ions lead to decrease of debye length and the surface charge of the silica particle. Then, it was observed by the vertical small angle x-ray scattering[23]. So, we can expect that the effect of the interaction between colloidal particles be small during drying.

Then, if we consider the effect of concentration of PVA during drying, the increase of concentration at beginning will enhance depletion force because depletion is affected by the concentration of the free polymer. However, if it continues drying, the concentration of PVA will be higher than the specific value. As a result, depletion force doesn't increase anymore along with the concentration of PVA. Then, at higher concentration, the effect of concentration inversely reduces the depletion force[19].

Now, we consider the effect of the rate of evaporation on the flocculation. We assumed that the flocculation be induced by the depletion force. Then, the size of the flocculation will determined by the kinetics. The rate of evaporation is related to the kinetics. To find it out, we have controlled the rate of evaporation by the humidity. The controlled humidity is 20% and 80%. We have deposited the solution on glass by a pipette. Because the deposited patterns are dried for a few seconds by using EHD jet printing. The ejected volume by EHD jet printing is too small to observe the effect of the rate of evaporation. The results are shown in Figure 3-10 and 3-11.
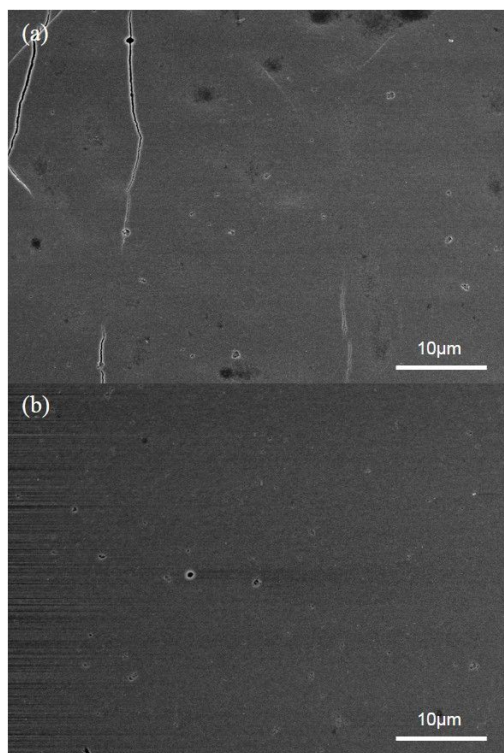
Figure 3-10 The microstructure of PVA 1wt%. The deposition was made up of a pipette on the glass. The humidity is (a) 20% and (b) 80%.
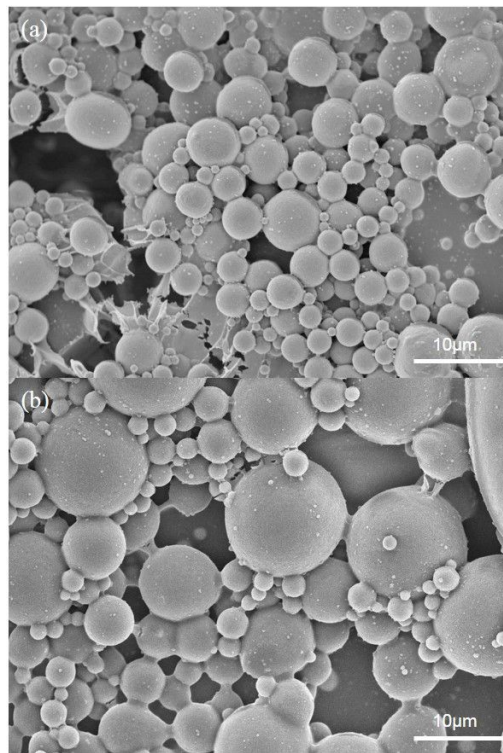
Figure 3-11 The microstructure of PVA 3wt%. The deposition was made up of a pipette on the glass. The humidity is (a) 20% and (b) 80%.

As shown in Figure 3-10, in case of PVA 1wt%, there is not a flocculated particles regardless of the humidity. On the other hand, there is flocculated particles for PVA 3wt% and the size of the flocculation is different depending on the humidity. Even if we didn't know the exact rate of evaporation, we would see the effect. Then, it is understood in this way. The difference of the rate of evaporation is the difference of time to the concentration being varied. So, the humidity 80% has more the time for the flocculation than 20%. The difference results in the difference of the size. So, from these result, we can speculate the similarity and the difference of the surface profile between PVA 1wt% and 3wt% when the width of line-patterns is about 200 and 800μm.

Chapter 4 Conclusion

In this study, we have deposited the suspension containing silica nanoparticles and made line-patterns with using EHD jet printing. To make the uniform line-patterns, it needs the stability of jetting. So, we have observed the effect of viscosity on the stability of jetting by controlling the concentration of PVA. Then, we could see a range of the viscosity of the solution for a stable cone-jet.

The width and the surface profile of deposited patterns are observed by the optical microscope, SEM, and profilometer. We have investigated the effect of operating conditions on the width of line-patterns. Also, we have found the link between the microstructure and the surface profile of line-patterns.

From these result, we would determine a suitable concentration of PVA for a stable cone-jet and the surface profile of line-patterns which we want and operating conditions to make the proper width of line-patterns.

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국문요약

본 연구에선, 나노입자를 포함한 현탁 콜로이드를 이용하여 분사 및 선형 패턴 형성을 전기수력학적 젯 프린팅을 이용하여 수행하였다. 전기수력학적 프린팅은 잉크젯 프린팅에서 액적을 분사하기 위해 압력을 가하는 것과 같이 전기장을 가하여 액적을 분사 시키는 것이다. 이러한 전기수력학적 젯 프린팅을 이용하여, 일정한 선폭을 가지는 선형 패턴을 형성하기 위해서는, 분사 안정성이 매우 중요하다. 전기수력학적 젯 프린팅의 분사 안정성에 영향을 미치는 요소는 공정 조건 (전압, 유량, 그리고 노즐과 기판과의 거리) 및 사용되는 용액의 물성 (점도, 표면장력, 유전상수, 그리고 전기전도도)이다. 다양한 변수 중에서, 고분자 PVA의 농도를 조절하여 용액의 점도를 제어함으로써, 점도가 분사 안정성에 미치는 영향을 관찰하였다. 광학 현미경, 주사 전자 현미경, 그리고 조면계를 이용하여 공정 조건이 선형 패턴의 선 폭에 미치는 영향을 조사하고, 고분자 농도에 따른 미세구조의 차이가 선형 패턴의 표면 조도에 미치는 영향을 조사하였다.