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Characterization of Silver Inkjet Overlap-printing through Cohesion and Adhesion

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Abstract – We introduce an understanding of silver (Ag) inkjet overlap-printing characteristics from the viewpoints of cohesion between ink droplets and adhesion between an ink droplet and a surface. The printing characteristics were closely monitored by changing the surface energy to elucidate the effect of adhesion and cohesion on printing instability, such as droplet merging and line bulging. The surface energy of the substrate was changed through the hydrophilization of a hydrophobic fluorocarbon-coated surface. The surface energy and ink wettability of the prepared surfaces were characterized using sessile drop contact angle analysis, and printing instability was observed using an optical microscope after drop-on-demand inkjet printing with a 50% overlap in diameter of deposited singlet patterns. We found that the surface energy is not an appropriate indicator based on the experimental results of Ag ink printing on a hydrofluoric-treated silicon surface. The analytical approach using adhesion and cohesion was helpful in understanding the instability of the inkjet overlap-printing, as adhesion and cohesion represent the direct interfacial relationship between the Ag inks used and the substrate.

Keywords: Inkjet printing, Line bulging, Droplet merging, Adhesion, Cohesion, Silver ink

1. Introduction

Inkjet printing is considered a promising alternative to conventional photolithography because it can reduce manufacturing costs markedly and simplify the manufacturing process steps by eliminating optical systems and photomasks. It has several other advantages. First, the pattern quality is not limited by the depth of focus of the optics, as droplets can travel relatively long distances. Second, patterning on nonplanar surfaces is possible with inkjet printing. Third, low-temperature microfabrication, which is especially attractive for electronic paper applications, identification tags, and other disposable electronic devices, can be performed on flexible substrates such as paper or plastics. Inkjet printing is applicable to various materials, including solution-based materials such as organometallics, organo-semiconductors, and polymers. It helps realize digitally driven direct productivity because it can transfer computer-aided designs directly onto device patterns and reduce material waste using a “drop-on-demand” material deposition [1-4].

The control of the wettability of ink on a surface is a useful method in reducing the size of a feature. A hydrophobic surface treatment can provide fine patterning from the large contact angle of a jetted droplet [2, 4, 8, 9]. If the liquid does not wet the surface, then a large contact angle will form, allowing the formation of small drop features. Therefore, the preference is to use a low-energy surface with a large contact angle to obtain fine patterns. However, these printed droplets may adhere poorly to a surface [2, 4]. In this case, unwanted phenomena, such as droplet merging and line bulging, in inkjet printing on a low-energy surface are often encountered. Droplet merging occurs when several droplets combine to form a single droplet because of surface tension and poor wettability of the substrate surface. Line bulging occurs when the printed pattern begins to swell out; it is particularly noticeable during continuous line printing [10-15].

In inkjet printing, the wetting of a jetted ink droplet on a substrate is regarded as adhesional wetting, in which liquid not originally in contact with the solid substrate makes contact and adheres to it. Inkjet printing makes the ink droplet come in contact with and adhere to a solid substrate by jetting an ink droplet generated from a nozzle. Fig. 1 shows a schematic drawing of a second droplet jetting from the nozzle near the substrate of a surface after the deposition of the first droplet during silver (Ag) inkjet printing by overlapping. The jetted droplets are overlapped by controlling the jetting space to obtain the printed pattern, as shown in Fig. 1. In this situation, two different phenomena can occur: (1) a cohesive force between the first deposited droplet and the jetted second droplet, and (2) an adhesive force between the deposited droplet and the solid surface. The cohesive force always competes with the adhesive force in the wettability of the surface. Therefore, understanding the printing characteristics from the viewpoints of cohesion between ink droplets and adhesion between an ink droplet and the surface is important.

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In the current study, we present a methodology to characterize Ag inkjet overlap-printing. It focuses on droplet merging and line bulging as problematic issues in inkjet printing based on adhesion and cohesion. The printing characteristics were closely monitored by changing the surface energy and properties of Ag inks to elucidate the effect of adhesion and cohesion during droplet merging and line bulging. The surface energy of the substrate was changed through the hydrophilization of a hydrophobic fluorocarbon (FC)-coated surface. Surface energy and ink wettability of the prepared surfaces were characterized using sessile drop contact angle analysis. Droplet merging and line bulging were observed using an optical microscope after drop-on-demand inkjet printing with a 50% overlap in the diameter of the deposited singlet patterns.

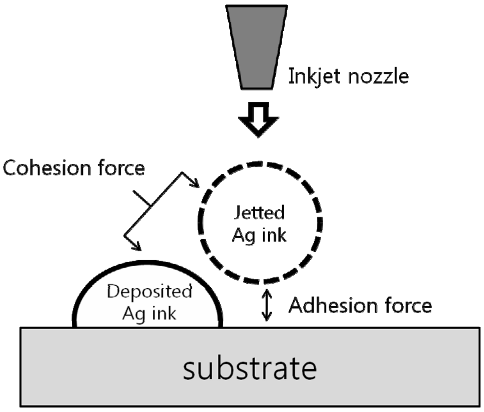


Fig. 1. A schematic drawing illustrating the overlapping when a second droplet is jetted from the nozzle near the substrate surface after the deposition of the first droplet during inkjet printing

2. Cohesion and adhesion

Cohesion occurs when intermolecular forces hold together particles of a substance. The work performed during cohesion, W_c, for a single liquid corresponds to the work required to pull apart a column of the liquid in a unit cross-sectional area. It is given by

$$W_c = 2\,\gamma_{LG} \tag{1}$$

where γ_{LG} is the surface tension of the liquid (i.e., ink).

Adhesion occurs when there is an attraction in the surfaces between two different bodies. In a system composed of a solid–liquid interface, the work of adhesion, W_a, is given by

$$W_a = \gamma_{LG}\,(\,1 + \cos\theta\,) \tag{2}$$

where W_a = work of adhesion, and γ_{LG} = the surface tension of the liquid (ink). In Eq. (2), $\cos\theta = 1$ for a contact angle = 0° and $W_a = 2\gamma_{LG} = W_c$. Therefore, a contact angle of zero results when adhesion between liquid and a solid is equal to or greater than the cohesion between liquid and liquid. An increase in the contact angle makes the liquid adhere to the solid less and the liquid cohere to itself more [16, 17]. If these two cases are applied to droplet merging and line bulging as undesirables encountered during overlap-inkjet printing, then the former case allows for easy, continuous line formation without any merging and bulging, whereas the latter case accelerates merging and bulging.

3. Experimental and materials

We used nonpolar Ag ink, DGH55HTG (ANP Co. Ltd., Seoul, Korea), in which Ag nanoparticles are dispersed in n-tetradecane (a nonpolar solvent). The physical and chemical properties of the used Ag ink are shown in Table 1. Drop-on-demand inkjet printing was performed using a Dimatix DMP-2831 printer (Dimatix-Fujifilm Inc., Santa Clara, USA) equipped with a 10 pl cartridge (DMC-11610) having 16 nozzles with a diameter of 19 μm. Ag ink was printed using a driving voltage of 20 V using only one nozzle; the jetting frequency was fixed at 5 kHz. The ejection velocity of the drops from the nozzle surface was about 5 m/s. The substrate temperature was set to 30 °C during the inkjet process.

The contact angle after hydrophobic coating and a UV/O_3 treatment was measured to characterize the changes on the surface. A DSA 100 contact angle analyzer (Krüss, Hamburg, Germany) was used to measure the contact angle. The contact angle of deionized (DI) water and diiodomethane (CH_2I_2) was measured on FC films to estimate the wettability and surface energy. Contact angle measurements were taken using the sessile drop method at least five times at different locations on the surface. The average values were used in the ensuing contact angle analysis. The measurements showed a standard deviation of < 2°. We employed the Owens-Wendt model [19] and the Girifalco [20] model to calculate the surface energy (γ_S) using the measured contact angles of DI water and CH_2I_2.

Table 1. Physical and chemical properties of Ag Ink

Ag ink product name	Surface tension (mN/m)	Dispersion solvent	Solid content (wt%)	Viscosity (mPa·s)
DGH55HTG	28.9	n-Tetradecane (nonpolar)	55.26	9.4

A previously reported surface treatment [10] was performed to control the wetting nature of the hydrophobic surface as follows. First, a hydrophobic FC film was spun on a bare silicon (Si) substrate at 2000 rpm using a fluoropolymer solution composed of a mixture of

Fluorad^{TM} FC 722 and FC 40 (3M, Seoul, Korea). Next, the FC-coated Si substrate was immediately loaded in a convection oven and was baked at 110 °C for 10 min. After baking, the FC film-coated Si substrate was treated using an AH-1700 UV/O_3 cleaner (Vision Semicon Co. Ltd., Bucheon, Korea) for a set period. A bare Si surface was prepared using hydrofluoric (HF) acid etch after chemical oxidation with a mixture of H_2SO_4 and H_2O_2. Finally, continuous line printing was performed using an overlapping rate of 50% of the diameter of a single printed droplet measured on each prepared surface. The space between the printed lines was the same as the diameter of the printed single droplet pattern. The printed Ag patterns were sintered at 250 °C for 30 min. We used the Tomoro ScopeEye image analysis software package v3.5 (Image Partnership Co. Ltd, Anyang, Korea) to measure the dimensions of the printed patterns in the obtained microscopic images.

4. Results and discussion

Hydrophilization of the hydrophobic FC film surface was carried out using a UV/O_3 treatment. This process was conducted to study the relationship between surface energy and the merging and bulging observed during inkjet printing. The film surface was characterized by measuring the static contact angle to investigate the effect of the UV/O_3 treatment on the hydrophobic FC films. The contact angle determines the wettability of an ink droplet on a surface, and it depends on the surface energy of the solid–liquid–air interface. A low contact angle implies high wettability (or a hydrophilic nature) and high surface energy. A large contact angle (i.e., > 90°) implies poor wettability and low surface energy [17, 18]. The FC film-coated Si substrates were soaked in the UV/O_3 cleaner for a given treatment time ($t_{UV/O3}$) up to 16 min with 2 min intervals.

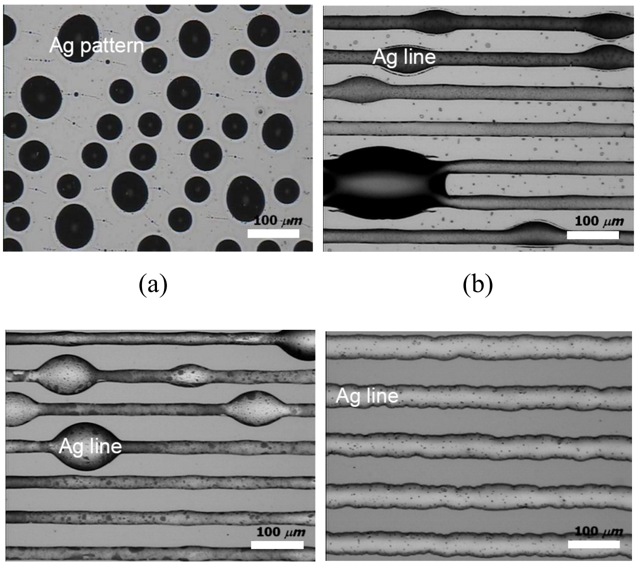
Table 2 shows the surface energy calculated using the Owens-Wendt model and the Girifalco model, as well as the resulting static contact angles of the probe liquids (i.e., DI water, CH_2I_2, and Ag ink) on the FC films as a function of $t_{UV/O3}$.

Table 2. Contact Angle and Surface Energy for Different Surface Conditions

Substrates		Surface energy (mN/m)	Contact angle of probe liquid (°)		
			DI water	CH_2I_2	Ag ink
FC film on Si		$\gamma_s^{FC\text{-}UV/O3}$			
UV/O_3 treatment time (min)	0	11.94	118.3	92.2	63.7
	2	12.82	114.5	90.8	62.4
	4	23.31	87.0	83.9	61.0
	6	42.11	58.6	79.1	58.3
	8	56.69	40.1	75.2	57.3
	10	70.34	15.0	75.4	55.4
	12	72.61	4.1	42.5	27.6
	14	72.70	3.0	36.1	15.7
	16	72.76	2.0	32.9	10.6
Bare Si		$\gamma_s^{silicon}$ 41.02	70.2	56.1	10.0

In Table 2, the term $\gamma_s^{FC\text{-}UV/O3}$ denotes the surface energy of a UV/O_3-treated FC film surface, whereas the term $\gamma_s^{silicon}$ denotes the surface energy of a bare Si surface. We used the Owens-Wendt model of over 87.0° and the Girifalco model of less than 58.6° for the contact angle of DI water, as the Owens-Wendt model is suitable for low-energy surfaces [19], whereas the Girifalco model is suitable for high-energy surfaces [20]. The contact angle of DI water began to decrease markedly after a UV/O_3 treatment period of 4 min. The contact angle of the nonpolar CH_2I_2 decreased from 92.2° to 75.4° up to a UV/O_3 treatment period of 10 min. It decreased markedly after a UV/O_3 treatment period of 12 min. Similarly, the contact angle of the nonpolar Ag ink followed the same trend as the nonpolar CH_2I_2. The calculated surface energy of the FC films increased slightly, from an initial value of 11.94 mN/m to 12.82 mN/m for a UV/O_3 treatment period of 2 min. However, after a UV/O_3 treatment period of 6 min, the surface energy increased markedly to 42.11 mN/m; it maintained this value in the range 70.34 to 72.76 mN/m after a UV/O_3 treatment period of 10 min.

Fig. 2 shows the results on inkjet printing of a continuous line with a 50% overlap in the diameter of the deposited singlet patterns using Ag ink by increasing the surface energy. Droplet merging occurred over the printing area on the FC film-coated Si substrates without UV/O_3 treatment -[Fig. 2(a)]; no line formed.



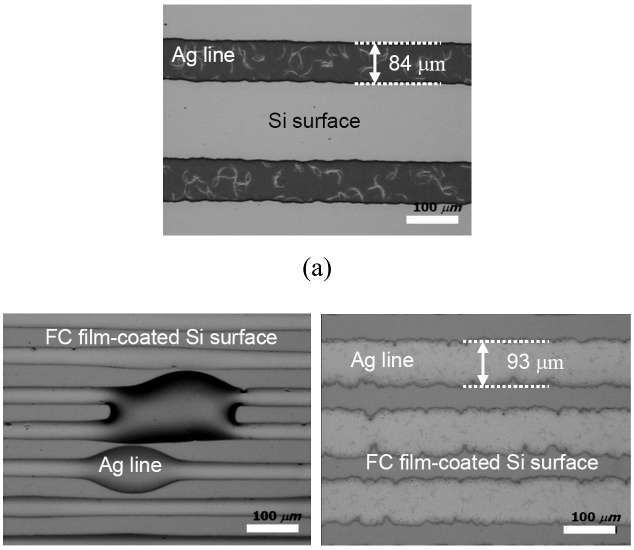
(a) (b)

(c) (d)

Fig. 2. Microscopic images of line printing of Ag ink on FC film-coated Si substrates: (a) before UV/O_3 treatment; (b) $t_{UV/O3}$ = 4 min; (c) $t_{UV/O3}$ = 8 min; (d) $t_{UV/O3}$ = 12 min (where $t_{UV/O3}$ = UV/O_3 treatment time). The white scale bar denotes a distance of 100 μm.

Droplet merging did not occur after a UV/O$_3$ treatment period of 4 min ($\gamma_s^{\text{FC-UV/O3}}$ = 23.31 mN/m), but line bulging was so serious that some parts of the printed lines were connected by beads, as shown in Fig. 2(b). This line bulging was continuously observed from $t_{\text{UV/O3}}$ = 4 min to $t_{\text{UV/O3}}$ = 8 min [Fig. 2(c)]. A continuous line without any droplet merging and line bulging after a UV/O$_3$ treatment period of 12 min ($\gamma_s^{\text{FC-UV/O3}}$ = 72.61 mN/m) was obtained, as shown in Fig. 2(d). The measured line width was about 48 μm.

The occurrence of droplet merging and line bulging on an untreated surface with the surface energy similar to that of the UV/O$_3$-treated FC film surface was investigated. The bare Si surface using HF acid etch after chemical oxidation using a mixture of H_2SO_4 and H_2O_2 was prepared. The solid surface energy was $\gamma_s^{\text{silicon}}$ = 41.02 mN/m, calculated using the Owens-Wendt model and the contact angles of DI water and CH_2I_2 (Table 2). The surface energy of the bare Si is similar to that of a UV/O$_3$-treated FC film surface for 6 min, $\gamma_s^{\text{FC-UV/O3}}$= 42.11 mN/m. We performed inkjet printing of Ag ink on the bare Si surface. Interestingly, a continuous Ag line without line bulging [Fig. 3(a)], unlike the UV/O$_3$-treated FC film surface for 6 min, was obtained as shown in Fig. 3(b). This experiment verified that the surface energy and contact angle of the probe liquid are convenient tools to characterize the wettability of a prepared surface before inkjet printing. However, using these techniques as indicators of whether droplet merging and line bulging will occur after inkjet printing of a continuous line is difficult. If we consider the surface energy only of the prepared surface, line bulging of a printed Ag line should occur on the bare Si surface with surface energy similar to a UV/O$_3$-treated FC film surface for 6 min.



(a)

(b) (c)

Fig. 3. Microscopic images of line printing of Ag ink on (a) a bare Si substrate; (b) an FC film-coated Si substrate treated using UV/O$_3$ for 6 min, (c) an FC film-coated Si substrate treated using UV/O$_3$ for 16 min. The white scale bar denotes a distance of 100 μm.

In the current study, we propose a methodology to characterize Ag inkjet overlap-printing, focusing on droplet merging and line bulging from W_a and W_c. The reasons for the proposal are as follows. (1) W_c and W_a are calculated from the surface tension of Ag ink and the contact angle on a prepared surface, which can be measured before inkjet printing. (2) The wettability of the ink can be quantified by normalization W_a to W_c. (3) W_a and W_c have a direct interfacial relationship with the formation of a line composed of overlapping jetted ink droplets, as shown in Fig. 1.

The cohesion on Ag ink (W_c^{Ag}) and the adhesion between Ag ink and the UV/O$_3$-treated FC film ($W_a^{\text{FC-UV/Ag}}$) were calculated from Eqs. (1) and (2) using the contact angle of Ag ink and its surface tension. As shown in Fig. 4, the value of W_c^{Ag} was constant according to the surface tension of Ag ink from Eq. (1). From Eq. (2), the value of $W_a^{\text{FC-UV/Ag}}$ changed according to the contact angle of Ag ink on the prepared surface. $W_a^{\text{FC-UV/Ag}}$ ranged from 41.70 to 45.31 mN/m up to a UV/O$_3$ treatment period of 10 min, increasing markedly to 54.51 mN/m after a UV/O$_3$ treatment period of 12 min.

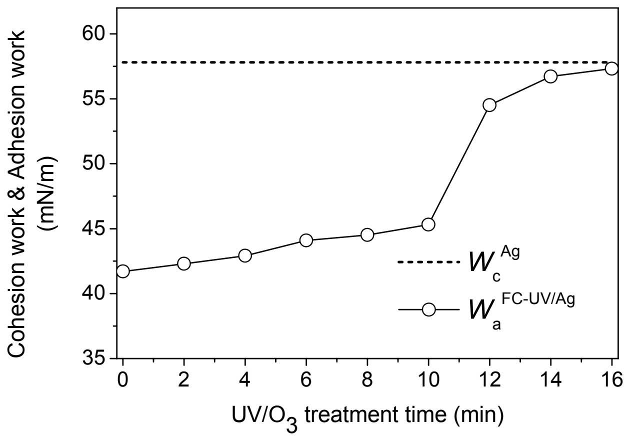


Fig. 4. Cohesion and adhesion on Ag ink used on the prepared surfaces (W_c^{Ag} = cohesion on Ag ink, and $W_a^{\text{FC-UV/Ag}}$ = adhesion between Ag ink and UV/O$_3$-treated FC film)

If $W_a^{\text{FC-UV/Ag}}$ was normalized to W_c^{Ag}, a value of $W_a^{\text{FC-UV/Ag}}$ = 42.91 mN/m corresponding to 74.24% of W_c^{Ag} = 57.80 mN/m would be required to avoid droplet merging. $W_a^{\text{FC-UV/Ag}}$ = 54.51 mN/m, which corresponds to 94.31% of W_c^{Ag} = 57.80 mN/m, was required to avoid line bulging. Adhesion between the bare Si and Ag ink ($W_a^{\text{Si/Ag}}$) was 57.36 mN/m, as calculated by Eq. 2 using contact angles and surface tension of Ag ink, which corresponds to 99.24% of W_c^{Ag} = 57.80 mN/m. In terms of adhesion, the bare Si can be regarded as a surface similar to a UV/O$_3$-

treated FC film surface for 16 min, with $W_a^{FC\text{-}UV/Ag} = 57.31$ mN/m. Both surfaces bear almost the same adhesion, as e the contact angles of Ag ink are almost the same in Table 2. As shown in Fig. 3(c), the UV/O_3-treated FC film for 16 min did not show any bulging in a printed Ag line. The width of the Ag line printed on the UV/O_3-treated FC film surface for 16 min was approximately 93 μm, which is 9 μm wider than that (approximately 84 μm) of the line printed on the bare Si surface. Therefore, expecting that the line bulging of Ag ink will not occur on the bare Si surface is reasonable based on the comparison W_a between the bare Si surface and the UV/O_3-treated FC film surface.

Analyzing the experimental results of this study from the viewpoint of adhesion and cohesion, we have the following results. (1) To prevent droplet merging, Ag ink requires a surface with $W_a^{FC\text{-}UV/Ag} > 42.91$ mN/m corresponding to 74.24% of $W_c^{Ag} = 57.80$ mN/m. (2) Ag ink requires a surface with a ratio of W_a/W_c in the range 94% to 99% to prevent any bulging of the printed line. The ratio of W_a/W_c can be an effective guide to prepare a solid substrate in predicting the quality of the inkjet-printed line.

5. Conclusions

Surface characterization was carried out using contact angle analysis and inkjet overlap-printing. Ag ink was employed to determine if W_c and W_a are effective indicators for the characterization of droplet merging and line bulging during inkjet printing. When performing the inkjet printing of a continuous line on bare Si and UV/O_3-treated FC film surfaces for 6 min with similar surface energies, we observed contrasting results. Line bulging did not occur on the bare Si surface but did on the UV/O_3-treated FC film surface for 6 min. This experimental result supports the notion that surface energy is not an appropriate indicator of whether droplet merging and line bulging will occur during inkjet printing of a continuous line by overlapping ink singlet. However, continuous lines without any bulging were identically printed in a comparative study involving bare Si and UV/O_3-treated FC film surfaces for 16 min with similar W_a. In addition, the range of wettability on the printing instability through the normalization of W_a to W_c was differentiated. To prevent droplet merging, Ag ink requires a surface with $W_a^{FC\text{-}UV/Ag} > 42.91$ mN/m, corresponding to 74.24% of $W_c^{Ag} = 57.80$ mN/m. To prevent bulging of a printed line, Ag ink requires a surface with W_a/W_c = 94%–99%.

In conclusion, the proposed analytical method using the values of W_a and W_c can be used as an effective indicator to characterize the merging and bulging phenomena in inkjet overlap-printing. The reason is that adhesion and cohesion represent the direct interfacial relationship between Ag ink and the substrate. Thus, they are practically related to the formation of a line by overlapping the jetted ink droplets.

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