AC-Pulse Modulated Electrohydrodynamic (EHD) Direct Printing of Conductive Micro Silver Tracks for Micro-Manufacturing

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ABSTRACT

This paper presents a direct fabrication of highly conductive silver tracks with sub-20 µm microstructures on glass substrates using electrohydrodynamic jet printing (EHD) based on alternative current (AC) voltage. A new AC-modulated EHD technique is presented and used in directly printing by generating a fine jet through a large electrical potential between the nozzle and substrate. In the presented technique of AC-modulated EHD, when charge accumulates on the ink meniscus at the nozzle, a fine jet down to nano scale can be generated. The variables of fabrication process, like plotting speeds, curing temperature and number of layers, were investigated to achieve reliable jet printing of conductive silver tracks. Topography and electrical property of printed tracks were characterized and verified. By using modulated AC-pulsed voltage, we are capable of printing high resolution continuous patterns on insulating substrates. In the study, we successfully applied EHD for fabrication of highly conductive silver tracks on glass substrate. It was the first time that sub-20 µm silver tracks were demonstrated with resistivity about 3.16 times than bulk silver. The presented technique can be used for direct printing of micro scale electronic circuits and devices.

1. INTRODUCTION

In the last few years, there is a significant interest in finding feasible direct printing techniques for fabrication of micro-scale electronic devices with increasing applications in electronics, biotechnology and micro systems. Directly printing approaches, especially those based on ink jet printing in high-resolution fabrication situation, demonstrate attractive features in their application. The advantages include: (i) the ability to directly pattern on substrate, regardless of the mechanical property of substrate comparing with other pattern method like photolithography, (ii) rapid control of printing trajectory with programming based print control systems [1], (iii) simplified fabrication processes without mask or other additional steps, (iv) ability to print on substrates with multiple tracks and large areas, and (v) potential for mass production and low cost operation [2].

Conventional ink jet printing approaches are based on thermal or acoustic formation and ejection of liquid droplets through nozzles [3]. The fabrication processes are successfully applied in electronics [4-7], drug delivery systems [7], micromechanical devices [8, 9] and other areas. The maximum resolution using thermal or acoustic theory is 20-30 µm, resulted from the fact that the diameters of the droplets are typically bigger than 10 µm [10]. Even though with assistant technique, e.g., by combing lithography into these ink jet printing to confine the liquid flow [11], it remains a challenge to for electronics research and industry community to achieve the sub-micrometer level in fabrication processes.

Electrohydrodynamic jet printing (EHD) is a pattern method using electric fields to generate fluid flows to deliver inks to a substrate. When a liquid is supplied to a sufficiently high electrical potential, the liquid would form a stable cone and emit a jet on its summit. The technique that uses electric fields instead of thermal or acoustic offers some advantages of patterning compared with other direct write technologies. First, the droplets dimension could be reduced dramatically (40 nm - 1.8 µm) using electrohydrodynamic printing to optimize physical properties of the liquid droplet

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by controlling parameters [12]. Second, the diameter of the nozzle used could be larger than nozzles of other jet printing techniques. This would avoid blockages of particles of liquid.

However, before EHD can be adopted to manufacturing printing processes, there are still plenty of obstacles remain to be overcome. First, EHD is a patterning method that uses a fine jet generated at the apex of ink cone of an electrospray in cone-jet mode [13], which are determined by experimental parameter, such as electric field, conductivity and viscosity of fluids and flow rate [14]. Numerical analysis of formulations and relationships are hard to develop among many parameters. Lee et al. (2012) have tried to propose a formulation between droplet size, surface tension and applied voltage, but it was not suitable for jet formation situations [15]. More research about conditions and parameters for EHD needs to be conducted. Second, fabrication of filaments in micro and nano scale is attractive for applications of EHD in circuit fabrication as well as biomedical fields. However, highly conductive silver tracks with such high resolution are tremendously difficult to print. Since single printed layer would be only 50 nm thick, it is necessary to print multi-layers for better conductivity. On the other hand, multi-layers would play a negative role on resolution. Smaller diameter of nozzle is another key factor for high resolution, while blockage of the nozzle is unavoidable and conductivity of the silver line would be poor with thinner thickness[16].

Conductive microtracks have been obtained by direct printing of metallic organic ink with nanoparticles and subsequent thermal decomposition [17-19]. Wang had reported metallic pads and conducting tracks with 60 μm line width printed on Si substrates, showing excellent resistivity [20]. Youn et al. put emphasis to make high resolution micro patterning using EHD and succeeded in fabricating silver line with width down to 6 μm on silicon substrate using tilted-outlet nozzle instead of conventional nozzle [21]. Lee et. al. reported depositing silver tracks onto polyimide film by EHD, obtaining conductor lines as fine as 62 μm in width and 0.3-5 μm in thickness. These lines exhibited about eight times higher resistivity (4.8 μΩ·cm) than that of bulk silver after thermal curing process [12, 22]. However, no one have reported conductive silver tracks printed on highly insulating materials such as Ajinomoto build-up films (ABF).

In this paper, a new AC-modulated EHD technique is presented and used in directly printing by generating a fine jet through a large electrical potential between the nozzle and substrate. The use of electrohydrodynamically induced fluid flows through microcapillary nozzles for jet printing of highly conductive silver tracks based on AC voltage with sub-20 μm resolution was presented in the paper. High resolution printed metallic silver tracks with resistivity about 3.16 times bulk silver showed great potential applications in printed electronics. It was the first time that AC voltage was applied in EHD to print highly conductive silver filaments with dimension down to sub-20 μm level. The fabrication process proposed in the paper is able to on demand print metal tracks on highly insulating materials that do not transmit or neutralize electrons. Details of the proposed new method are presented in the following sections.

2. FABRICATION METHODS AND EXPERIMENTAL DETAILS

The EHD system proposed in this research consists of a three-axis (XYZ) stage, a dispensing system with pressure regulator, a nozzle, substrates, and a high AC voltage power supply, as shown in Figure 1. The nozzle is functioned as electrode, and used to produce a jet containing organic silver nanoparticles, which are supplied by a syringe to the nozzle. Desired voltage is generated, amplified and applied between nozzle and electrode. Waveform of the AC voltage is fixed while amplitude and frequency can be adjusted. The stage could provide a displacement for substrate to control trajectory of tracks by move 3-Axis stage in X-Y directions. This fabrication process is capable of on demand printing multi-layers on substrate by programming the movement of 3-axis stage.

In the last few years, several research works were presented on patterning of metal nanoparticles through electrohydrodynamic jet printing on glass and silicon substrate [22-26] and droplet analysis using different forms of voltage [27-30]. In the study, AC voltage was adapted because direct printing of highly conductive silver tracks on insulating materials in electronic industry would be a major application in the future. Situations are different printing on insulating substrate using DC voltage. Since the substrate will never transmit or neutralize any charges of droplets and inks. The charged ink accumulates on the substrate and then affected the whole electric field between nozzle and substrate. Two scenarios will happen: (1) the jet is affected by net charge of previously printed ink, resulting in deflection; (2) the jet is completely stopped, resulting in discontinuous tracks.

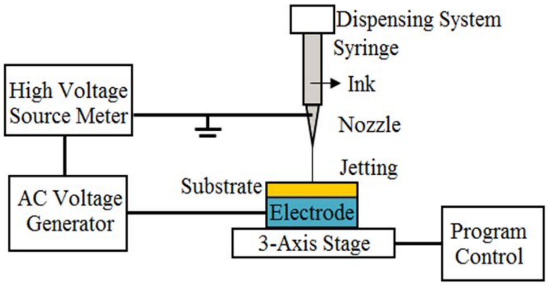

Figure 1. Schematic of electrohydrodynamic jet printing setups.

A solution to address this problem is to use AC voltage instead of DC, so that it is possible that silver tracks can not only be printed on glass substrate but highly insulating materials. In our earlier works presented in [31-32], a lab set up on EHD printing with precision table control was reported. The mechanism of fabrication process of EHD using AC voltage printing is shown in Figure 2. Due to the AC voltage signals, positive and negative charges are induced on the meniscus of the ink at the tip of the nozzle sequentially, forming positive and negative jet sequentially. Figure 2 (a) shows a circle of AC signal. In Figure 2 (c)-(II), negative charged jet would print negative ink on Ajinomoto build-up films (ABF) substrate first, resulting in negative ink printed on ABF in Figure 2 (c)-(III). As electrode polarity changes, a positive charged jet is generated (Figure 2 (c)-(IV)) and printed along the tracks. Since the duty ratio of the signal is one, the negative printed tracks would be neutralized by these positive charges. The printed tracks return to an electrically neural state that is ready for another period of ejections. An electronic pattern with a 100 µm width pad and a 2.3 µm silver track was obtained on ABF using AC voltage with nozzle outer diameter of 7 µm and inner diameter of 5 µm, as shown in Figure 2 (b). To acquire highly conductive micro tracks, multilayered printing was attempted by direct placing the deposition on top of each other in the study.

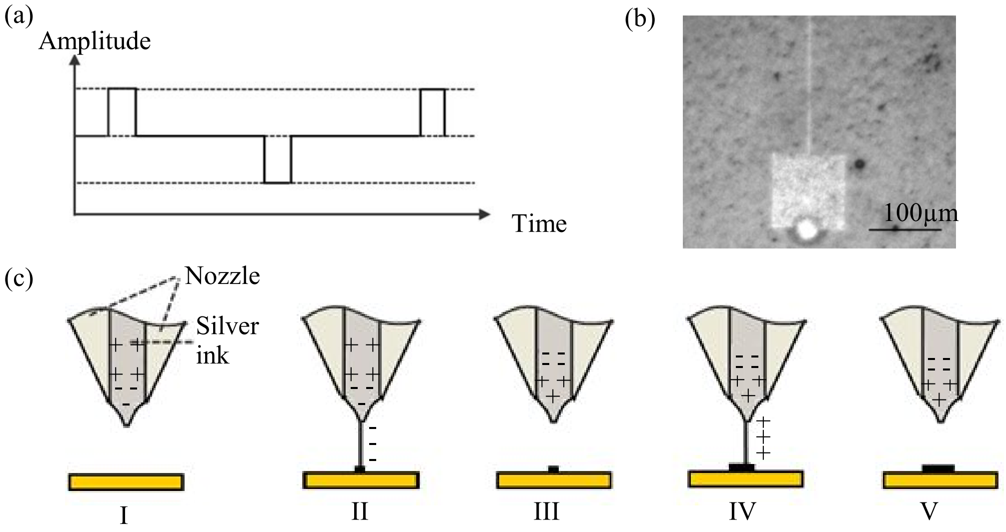

Figure 2. (a) AC voltage signal with duty ratio of one, (b) 30-layer straight silver track with line width of 19.53 µm printed on ABF, (c) mechanism of EHD using AC voltage printing on highly insulating substrate.

Silver nanoparticles (Silverjet DGP-40LT-15C) were purchased from Advanced Nano Products Co., Ltd. with 20-35 wt.% silver nanoparticles and about 65-80% triethylene glycol in weight with small amounts of surfactants to prevent agglomeration between silver nanoparticles. Nano silver particles are uniformly dispersed in the solvent as high solid content with low viscosity for ink jet applications. The geometric diameter of the silver nanoparticles is below 50 nm. Density of the dispersion nanoparticles is 1.45 g/mL +/- 0.05 g/mL at 25 ºC. The viscosity is 10-18 cP. General curing temperature recommended by the company is 120-200 ºC. The ac voltage was programmed with a waveform like alternative rectangular wave, as demonstrated in Figure 2 (a). The morphology of the printed tracks was investigated by an atomic force microscope (AFM) (Park Systems, XE-70). The electrical resistance was characterized by a two point probe measurement method.

In this paper, the experiments were conducted and details are presented as follows. First, a jet was obtained by applying high AC voltage after silver particles was supplied to the nozzle through the syringe. The tracks were formed by continuously moving the substrate controlled by 3-Axis stage. An AC voltage with amplitude of 820 volts and frequency of 130 Hz was provided by the generator. During the depositing process, the substrate was scanned over a distance of 50 mm at a speed of 7.5 mm/s. Multi layered printing was attempted by direct placing the deposition on top of each other. After the tracks formed, curing process was conducted by heating at ramped temperature with atmospheric pressure on a heating machine. During the experiment, a short nozzle-substrate distance of 20 µm was chosen for stable purpose. The effects of process parameters such as number of layers, curing temperature on resistivity of silver tracks were investigated to obtain the optimal conditions for printing high-conductive silver tracks.

3. RESULTS AND DISCUSSION

3.1. EFFECTS OF PLOTTING SPEED AND CALCULATION OF DROPLET SIZES

Based on the measurements of printed silver droplets, we were capable of calculating and analyzing the mechanism of printing process. We assumed the falling droplets are perfect sphere and printed dots are cylinder shape with average height and diameter measured from AFM image. Another assumption is that the triethylene glycol in the droplets would be volatilized during falling. As shown in Figure 3 (b), in order to print connective silver tracks instead of separate dots on substrates, two adjacent droplets has to be at least tangent to each other. The time between two adjacent droplets is half of cycle time of AC voltage. The maximum plotting speed can be calculated using equations (1). As shown in Figure 3 (a), once a droplet is formed, it will fall off and flatten on the substrate to form a dot. The volume of droplets and dots are equal. Once we measure the volume of dot on the substrate, the actual size of falling droplet can be calculated using equations (2).

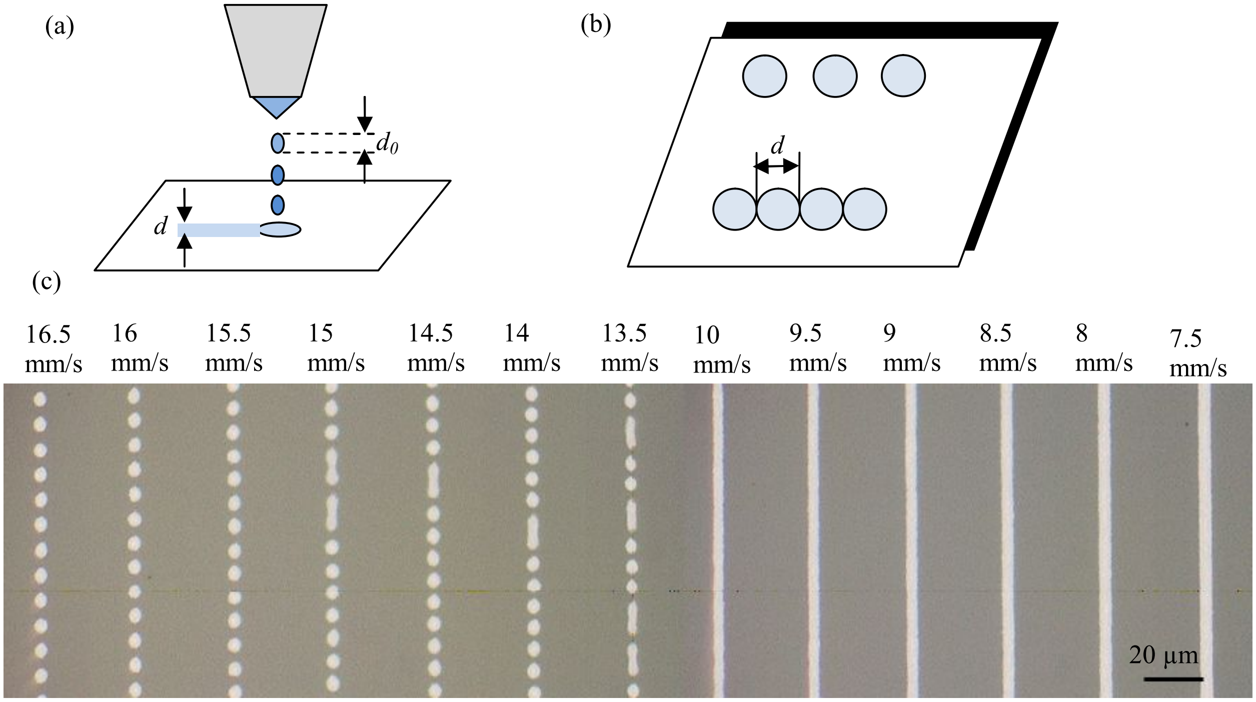

Figure 3. Sketches on a microscopic of (a) calculation of diameter of falling droplets; (b) requirements to obtain connective silver tracks; (c) Effect of plotting speeds: plotting speed increased from 7.5 mm/s to 16.5 mm/s with reduced line width and discontinuous pattern.

$$T = \frac{1}{f}, \qquad T_d = \frac{T}{2}, \qquad v_{\max} = \frac{d}{T_d} = \frac{2d}{T} = 2df \tag{1}$$

where, T = cycle time of AC voltage, f = frequency of AC voltage, T_d = time between two adjacent droplets, d = diameter of printed droplets, and v_{max} = plotting speed.

$$V_1 = \pi \times \left(\frac{d}{2}\right)^2 \times h, \qquad V_0 = \frac{4}{3} \times \pi \times \left(\frac{d_0}{2}\right)^3, \qquad V_1 = V_0, \qquad d_0 = 2 \times \sqrt[3]{\frac{3V_0}{4\pi}} = 2 \times \sqrt[3]{\frac{3V_1}{4\pi}} = \sqrt[3]{\frac{3 \times d^2 \times h}{4}} \tag{2}$$

where, V_1 = volume of printed droplets, h = average height of printed droplets, V_0 = actual volume of falling droplets, and d_0 = actual diameter of falling droplets.

The cross section of printed silver dots has an average height of 30.73 nm and an average line width of 6.06 μm. The maximum speed for connective silver tracks was 12.12 mm/s and the actual diameter of falling droplets was 0.92 μm. The ratio of diameter of nozzle and diameter of falling droplets was about 7.6. During the printing process, the accumulated charges in the meniscus will result in formation of droplets. Assuming the current in meniscus is stable and proportion to voltage, the total amount of charges in a droplet can be estimated by integral of electrical voltage and time. It turned out to be greater line width with increased amplitude of voltage and decreased frequency (increased cycle time), which means more charges accumulate during the time interval.

$$f = 1000Hz, \qquad d = 6.06\mu m, \qquad v_{\max} = 2df = 12.12mm/s$$

$$h = 30.73nm, \qquad d_0 = \sqrt[3]{\frac{3 \times d^2 \times h}{16}} = 0.92\mu m$$

We directly investigated the effect of plotting speed on printing process and silver tracks while the jetting speed and ink flow were kept constant by using fixed voltage. A 7 μm Nozzle was chosen with a fixed AC voltage of 400V, 1000Hz and duty rate of 10%. The distance between the nozzle and substrate was 20um.

When plotting at a small plotting speed, we were able to acquire stable silver tracks. As the plotting speed increased, the width of silver tracks reduced gradually, as shown in Figure 3 (c) with plotting speed increased from 7.5 mm/s to 10 mm/s. After the plotting speed was further increased, there were dots printed instead of continuous lines. Some of dots might be able to connected with adjacent ones, but the tracks are not interrupted. As plotting speed increased further, only silver dots were printed. Figure 3 (c) showed discontinuous silver tracks and dots tracks with plotting speed increased from 13.5 mm/s to 16.5 mm/s. The diameter of dots reflects the actual capability of printing system since they are formed by droplets.

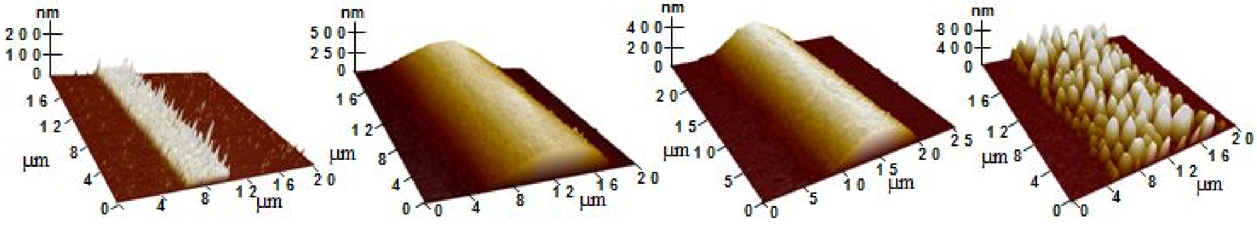

Figure 4. (a) 3D AFM image of single layer pattern, (b) 3D AFM image of 20 layers pattern, (c) 3D AFM image of 20 layers pattern cured with ramped temperature to 220 ºC, (d) 3D AFM image of 20 layers pattern cured instantly to 220 ºC.

3.2. EFFECTS OF CONNECTIVITY ON CONDUCTIVITY

The silver ink consists of triethylene glycol, surfactants and silver nanoparticles. Nano particles are dispersed in the solvent, which would be evaporated during printing process. However, with smaller diameter of droplets to achieve better resolution, when they fall onto the substrate, they will splash out and transform into single particle bunches, as shown in Figure 4(a). Since most of bunches are not interconnected, there is no electric conductivity of single layer silver tracks. By depositing multi layers on the same position, we can print 20 layers silver tracks with fine semi-ellipse cross section, as shown in Figure 4(b). The cross sectional measured maximum height of 60 nm for the single layer pattern with an average thickness of 27 nm and line width of 5.3 µm. Because the geometric diameter of silver nanoparticles is below 50nm, it was believed that there was only single nano particle printed on the substrate without any overlay. That was the reason why the inter connectivity is so poor that single layer silver tracks could not carry a current flow. The cross sectional measured maximum height of 310 nm for the 20 layers pattern with an average thickness of 167 nm and line width of 15.1 µm. By depositing the silver ink on the same position to acquire multi layer pattern, line width, line thickness and connectivity were increased with sacrifice of resolution. After curing process, the 20 layers silver track showed good electric conductivity.

3.3. EFFECTS OF CURING TEMPERATURE ON CONDUCTIVITY

To evaluate the effect of curing temperature on resistivity of micro silver tracks, a ramped curing temperature and instant curing temperature were first compared, as shown in Figure 4 (c) and (d). Figure 4 (c) was a 20 layers pattern which was gradually cured with ramped temperature from 40 ºC to 220 ºC for 30 minutes and kept at 220 ºC for 15 minutes. The sintering process resulted in a fine recomposition of silver nanoparticles. However, if the temperature was instantly raised to 220 ºC for 30 minutes, the high temperature caused drastic crystallization. Figure 4 (d) demonstrated adjacent nanoparticles formed their own grains into larger particles instead of smoothly formed continuous silver tracks. These independent hillocks were not connected with each other, resulting in poor connectivity and insulation.

The curing process involves heat transport to create the necks between nanoparticles and transform them into grain boundaries. Diffusion is the principal mechanism. Lubricants and surfactant polymers are burned off first and nanoparticles are contacted with each other with a point bonding. As time goes on, necks are generated and then transformed into grain boundaries, a denser structure with a remarkable reduction in pores and interstices.

Shrinkage occurs during curing process as results of pore size reduction, lubricants and surfactants evaporation. This largely depends on the composition of silver ink, temperature and time. Silver tracks were printed with 10 lays and 20 lays using the same nozzle with glass tip outer diameter of 7 µm, using an AC voltage with 130 Hz frequency and 820 volts amplitude. The silver tracks were cured under temperatures of 140 ºC, 160 ºC, 180 ºC, 200 ºC and 220 ºC using ramped pattern. There was a reduction in both line width and mean height (similar to maximum height) due to shrinkage. As maximum curing temperature went high, the shrinkage were more obvious. A maximal 14.2% reduction of line width and 17.8% reduction of mean height were observed in 10 layers sample. A maximal 10.2% reduction of line width and 14.5% reduction of mean height were observed in 20 layers sample.

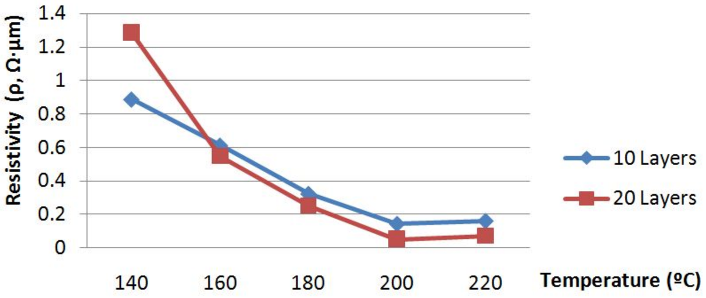

Figure 5. Resistivity of different printed tracks with different curing temperatures.

The resistance was measured with the help of an ohmmeter (Fluke 115) by contacting two probes on sintered silver tracks. Five measurements were taken at each sample at five different locations and then averaged. The electrical resistivity ρ of the printed silver patterns was calculated by using the formula "$\rho = R \cdot A/L$", where "R" is the resistance of the pattern measured, "A" is cross section area of the printed pattern and "L" is the length between the two measuring points. The cross section A of the silver tracks was calculated using equation "$A = w \times t$", where "w" is the width of silver tracks and "t" is the average thickness of the tracks, measured through AFM analysis. The electrical resistivity of printed patterns with different curing temperature is shown in Figure 5. The electric resistivity of cured tracks decreases with increasing curing temperature and tended to be a stable value. The cured silver tracks have a minimum resistivity of 0.051 Ω·µm under a curing temperature at 200 ºC, which is about 3.16 times that of bulk silver. The result shows a good conductive property for silver patterns by EHD. The resistivity was higher than bulk silver that is due to the calculation using the average thickness of the patterns and the development of void and cracks resulting from formation of coarse network.

4. ELECTRONIC COMPONENTS PRINTING

Figure 6(a) showed high resolution printed metal pads, interconnects for representative circuit patterns with critical dimensions as small as 18 µm showed great potential applications in microelectronic components fabrication. Figure 6(b) demonstrated silver tracks with line width of 12 µm printed on the ABF, which was a highly insulating material. These can be applied for micro capacitor or inductor fabrication.

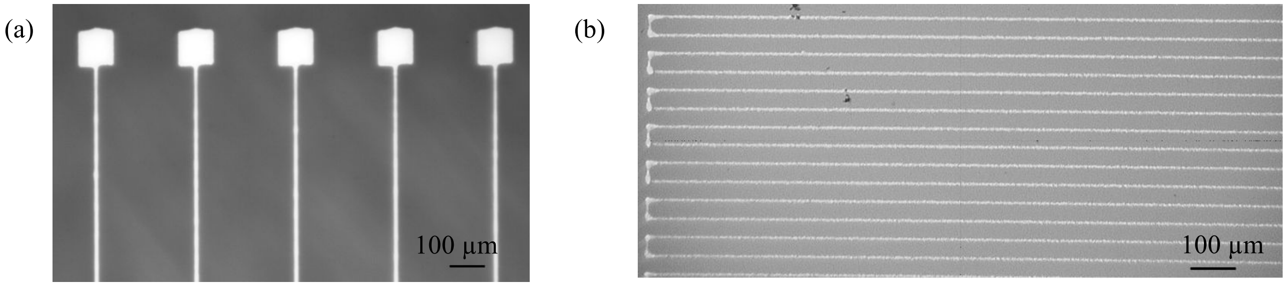

Figure 6. Printed electronic components: (a) printed metal pads and interconnects (b) an inductor pattern.

5. CONCLUSIONS

In this paper, a detailed method of AC modulated EHD direct printing was presented for direct printing of silver tracks with sub-20 µm resolution and high conductivity. Fabrication process and the detailed mechanism of EHD using AC voltage were discussed in the paper. The presented method can be used for the deposition of micro-scale functional conductive materials. Curing process was investigated to achieve desired resistivity of silver tracks. It was the first time that high resolution and conductivity printed silver patterns with sub-20 µm line width were fabricated by EHD, which showed great potential in PCB fabrication and biomedical applications. The minimum resistivity of printed track is 0.051 Ω·µm, which is about 3.16 times that of bulk silver (0.016 Ω·µm). The presented AC modulated EHD technique is capable to fabricate resistors, inductors and micro wires, which offer a simple and versatile method to on demand direct fabricate conductive patterns in micro/nano electronic manufacturing. The presented technique can be used for direct printing of micro scale electronic circuits and devices.

ACKNOWLEDGEMENTS

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