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Characterization of Inkjet-Printed Silver Patterns for Application to Printed Circuit Board (PCB)

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Abstract – In this paper, we describe the analysis of inkjet-printed silver (Ag) patterns on epoxy-coated substrates according to several reliability evaluation test method guidelines for conventional printed circuit boards (PCB). To prepare patterns for the reliability analysis, various regular test patterns were created by Ag inkjet printing on flame retardant 4 (FR4) and polyimide (PI) substrates coated with epoxy for each test method. We coated the substrates with an epoxy primer layer to control the surface energy during printing of the patterns. The contact angle of the ink to the coated epoxy primer was 69^o, and its surface energy was 18.6 mJ/m^2. Also, the substrate temperature was set at 70^{o}C. We were able to obtain continuous line patterns by inkjet printing with a droplet spacing of 60 ㎛. The reliability evaluation tests included the dielectric withstanding voltage, adhesive strength, thermal shock, pressure cooker, bending, uniformity of line-width and spacing, and high-frequency transmission loss tests.

Keywords: Inkjet, Reliability, Silver, Printed circuit board, Epoxy

1. Introduction

Photo-lithography is generally used for the fabrication of micro-structures. It requires many process steps, including photo-resist coating, exposing, developing and etching processes, to generate patterns after thin film deposition [1-4]. Due to the demand for low-cost manufacturing and shorter product development times, an alternative simple direct writing method has been proposed especially for printed electronics applications. Recently, inkjet printing, a direct writing technology, has been attracting growing interest for the production of micro-patterns. Its advantages include low-cost manufacturing and low-temperature micro-fabrication on flexible substrates, which are especially attractive for paper electronics, identification tags, and disposable electronic devices. Moreover, the ink consumption of this technique can be reduced because inkjet printing techniques use a drop-on-demand process that allows for the delivery of precise amount of various solution-based materials, such as nano-particle colloids, polymers, organo-semiconductors, and organo-metallics.

In recent years, various issues regarding the use of inkjet technology in industrial manufacturing have been reported. First, control of the waveform and voltage for driving the piezo actuator is critical to ensuring the stable jetting of droplets from the nozzles because printed patterns are formed by the deposition and connecting of individual liquid droplets ejected from a nozzle onto a substrate. Also, inkjet-printed patterns often produce a coffee ring effect or line bulges due to the differential evaporation rates between the center and edges, droplet spacing, surface wettability, and hydrodynamic instability. To obtain uniform and reliable patterns without the coffee ring effect and line bulges, many researchers have proposed various methods, including controlling nano-particle size, stabilizing nano-particle dispersion, controlling ink solvent properties, changing droplet spacing, heating substrates, altering surface wettabilities and mixing several solvents having different properties [5-8].

Recently, several studies on the reliability of inkjet-printed patterns have been conducted to evaluate inkjet printing technology as a fabrication process for the printed circuit board (PCB) industry. However, the methods and standards for evaluating the reliability of inkjet-printed patterns have not yet been entirely established [9]. For this study, we selected seven different items and conditions for reliability evaluation that have been commonly used in the conventional PCB industry according to the standard of the Institute for Interconnecting and Packaging Electronic Circuits (IPC), TM650. We intensively characterized the inkjet-printed Ag patterns according to these selected methods and standards. The test patterns for these tests were fabricated by inkjet printing of an Ag nano-particle ink on a FR4 rigid substrate and a polyimide (PI) film. The surface wettability was controlled by coating each substrate with an epoxy layer with a thickness of 5 ㎛. During the jetting process, each substrate was heated to promote the

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evaporation of the solvent. Substrate heating enhances the evaporation rate of liquid droplets, thereby enabling the avoidance of line bulges and leading to fine and uniform pattern sizes.

2. Experimental methods

2.1 Experiment apparatus and materials

A piezoelectric print-head system with a 50 ㎛ nozzle diameter (MicroFab Co., USA) was used for printing the Ag ink. The printing system was composed of a print-head, a motorized X-Y stage with positioning accuracy of 2 ㎛, a heatable working table, and an alignment system. Before printing the patterns, we first optimized the droplets ejected from the nozzle by controlling the voltage and waveform of the piezo actuator drive to ensure stable single droplet deposition during all the experiments [10]. We used Ag ink (Advanced Nano Products Co., Korea) in which ~20 nm Ag nano-particles were dispersed in a triethylene glycol monomethyl ether (TGME) solution with a viscosity of 12.8 cP, a vapor pressure of 0.001 kPa at 20 ^oC, and a surface tension of 35.9 mN/m, respectively. Rigid FR4 substrates were used to fabricate the specimens for the withstanding voltage, adhesive strength, thermal shock, pressure cooker, line-width uniformity, and high-frequency transmission loss tests. Flexible PI (Doosan Electronic Corp.) films were used to fabricate the specimens for the bending test which was carried out using a Toyoseiki MIT-SA bending taster. Viscosities and surface tensions were measured with a Brookfield DV-II + Pro viscometer and a Kruss K9 tension-meter, respectively. A UNION DZ2 microscope was used to evaluate the line-width and spacing uniformity of the patterns. The American Society for Testing and Materials (ASTM) D3359 adhesive strength test rating system was used along with 3M 610 Scotch tape to measure the adhesion of the Ag patterns. The dielectric withstanding voltage, thermal shock, pressure cooker, and high frequency transmission loss test were measured with CHROMA 6330 high-speed electric load, ESPEC high temperature tester, ESPEC pressure cooker tester, and AGILENT E8362B/C Series network analyzer, respectively.

2.2 Substrate preparation and inkjet printing

The cleaned FR4 substrates and PI films were coated with an epoxy primer solution, which was prepared by mixing bisphenol-A-epoxy resin, sulfone hardener, and ketone solvents. The substrates and films were coated with a primer layer to control the contact angle of the ink as patterns were formed on the substrate during inkjet printing. The primer layer, with a thickness of 5 ㎛, was spin-coated onto the FR4 and PI surfaces at 4000 rpm for 1 minute. Then, to attach the epoxy film onto the FR4 and PI surfaces, the samples were immediately loaded into a convection oven and baked at 170 ^oC for 30 minutes. Sequentially, the inkjet printing was completed within 10 minutes after completion of the surface treatment of the substrate and films to prevent any change in their surface wettability. The reliability evaluation patterns were printed onto the substrates with a droplet spacing of 60 ㎛ and a surface temperature of 70 ^oC.

3. Results and discussion

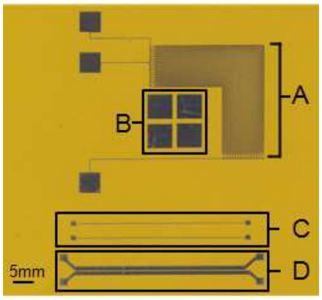
In this study, we selected seven different test items and conditions for reliability evaluation used in the conventional PCB industry according to the standard of the Institute for Interconnecting and Packaging Electronic Circuits (IPC) TM650. We intensively characterized the inkjet-printed Ag patterns according to the selected methods and standards. Table 1 shows the test conditions, substrate type and evaluation results for the reliability test items, including the withstanding voltage, ASTM D3359 adhesive strength, thermal shock, pressure cooker, bending, line-width uniformity, and high-frequency transmission loss tests.

Fig. 1 shows the test patterns made on the FR4 substrate for the reliability analysis items. The withstanding voltage test was conducted by transmitting 1000 V DC for 30 seconds through both ends of 5 cm line pattern and examining it for any evidence of spark discharge, short circuiting, or dielectric breakdown (Table 1). The test patterns were printed with a line-width and spacing of 120 ㎛ and a thickness of ~1 ㎛ as shown in “A” in Fig. 1. All inkjet-printed Ag patterns passed the withstanding voltage

Table 1. Reliability test items, conditions, substrate type and evaluation results

Test items	Conditions	Substrate type	Evaluation results
Withstanding voltage	No spark discharge, short circuiting, or dielectric breakdown at 1000 V DC, 1 mA, 30 seconds	FR4	Pass
Adhesive strength	No peel off in 1mm cross-cut tape test	FR4	Pass
Thermal shock	Variation of resistance within ± 10% at –55 ^oC, 15 minutes and 125 ^oC, 15 minutes, 100 cycles	FR4	Pass
Pressure cooker test	Insulation resistance above 100 ㏁ at 121 ^oC, 2 atm, 97% RH ; 24 hr, 48 hr	FR4	Pass
Bending	The angle of rotation: 135 ^{o}, velocity of rotation: 175 times/min above 10,000 times for a copper film	PI	Pass
Line-width uniformity	Uniformity variation within ±10%	FR4	Pass
High-frequency transmission loss	less than 1 dB/cm at 10 GHz	FR4	NG

(FR4: Flame Retardant 4, PI: Polyimide, RH: Relative Humidity)



Test patterns	Reliability analysis items
A	Withstanding voltage and line width uniformity
B	Adhesive strength
C	Thermal shock
D	Pressure cooker test

Fig. 1. Standard test patterns formed by Ag inkjet printing on the FR4 substrate for reliability analysis items

test without showing any sign of spark discharge, short circuiting, or dielectric breakdown phenomena.

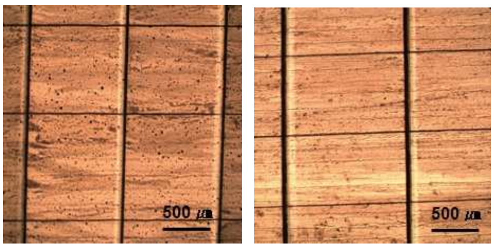
The adhesive strength test was performed using the ASTM D3359 rating method for the cross-cut tape test. 5 mm x 5 mm square patterns were prepared for ASTM D3359 ("B" in Fig. 1) [11]. The ASTM D3359 rating method is measured on a scale from 0B to 5B (Table 2) and the rating is calculated by observing the ratio of the pattern test area removed by a strip of 3M 610 Scotch tape.

The tape is removed with a rapid perpendicular pull force after it is first attached to the cross-cut pattern. The ASTM D3359 adhesive strength rating achieved by the test pattern was 5B with no observable peeling of the cross-cut patterns, as demonstrated by the 'before and after' micro-scopic images of the pattern shown in Fig. 2. The test result shows that inkjet-printed patterns have high adhesive strength to the epoxy-coated FR4 substrate.

The thermal shock test was conducted by observing the change in insulation resistance after 100 cycles of temperature variation from –55 °C to 125 °C for 30 minutes.

Table 2. ASTM D3359 adhesive strength rating

Rating	Ratio of removed area
5B	0%
4B	Less than 5%
3B	5 ~ 15%
2B	15 ~ 35%
1B	35 ~ 65%
0B	More than 65%



(a) (b)

Fig. 2. Microscopic images: (a) before adhesive strength test (initial status) and; (b) after adhesive strength test

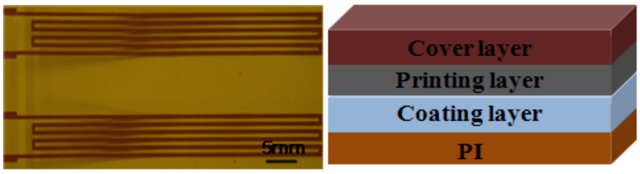
The variation in resistance after the thermal shock test was found to be within ± 4.5% for the five samples with no signs of spark discharge or short circuiting. The narrow variation of resistance less than ± 10% (pass condition) demonstrates that the inkjet-printed Ag patterns have good resistance characteristics to external thermal shock.

The pressure cooker test was conducted by measuring the insulation resistance ("D" in Fig. 1) through both ends between two independent Ag lines at 121 °C, 2 atm, and 97% RH after 24 hours and 48 hours, after the test patterns were printed ("D" in Fig. 1). The pressure cooker test is a standard for determining the occurrence of leakage current. In the pressure cooker test, the average insulation resistance values and standard deviation after 24 hours and 48 hours for five test samples were 7.7×10^5 ±1.7 MΩ and 7.5×10^4 ±1.6 MΩ, respectively. There was no remarkable difference in the resistance for five samples. These results show that inkjet-printed pattern has good stability of insulation resistance at high temperature, pressure and humidity conditions.

Fig. 3(a) shows a photographic image of the bending test pattern and Fig. 3(b) shows a cross-sectional scheme of the experimental section of the test sample. The serpentine-shape patterns were printed with a line-width and spacing of 1 mm and length of 110 mm on the epoxy-coated PI film. Lamination process was performed at 170 °C and 25kgf/cm^2 for 90 minutes after covering the inkjet- printed Ag patterns with a cover layer of 5 ㎛ in thickness.

The bending test was conducted by measuring the rotation number of the printed Ag patterns could be bent or folded over before failure. The samples were folded through an angle of 135° from their initial position at rotation velocity of 175 cycles/min.

As show in Table 3, the inkjet-printed Ag patterns passed the test criterion above 10,000 times with showing 10,900 ± 1226 times. The variation rate of the rotation number was ~ 11.2%. In general, nanoparticles have a strong tendency to agglomerate, which causes inter- and intra-agglomerate pores during thermal sintering. Generation of pores and the lack of densification may result in weak mechanical properties. Also, it is very difficult to control the size and number of the pores during thermal sintering [12]. Therefore, the irregular generation of the pores inside Ag patterns causes the instability of mechanical properties,



(a) (b)

Fig. 3. Bending test Ag patterns: (a) photographic image and; (b) cross sectional scheme of the fabricated specimen

Table 3. Bending test results of Ag patterns

Sample number	Rotation number	Average ± standard deviation
1	10,524	10,900 ± 1226
2	9,872	
3	12,952	
4	11,023	
5	10,128	

which may results in the variation of the rotation number in the bending test because we used thermal sintering process and the ink suspension of nanoparticles of 10~20 nm.

The uniformity test of the line patterns was analyzed with a microscope after printing thirty six Ag lines in '⌐' shape with the line-width and spacing of 120 ㎛, respectively ("A" in Fig. 1). We found the inkjet printing conditions that would produce patterns without any bulges or merging by controlling the surface wettability and substrate temperature as reported in previous researches [13]. We coated the substrates with an epoxy primer layer to control the surface energy during printing. The contact angle of the ink to the epoxy primer was 69 ^{o} and the surface energy of the epoxy primer was 18.6 mJ/m^2. The substrate temperature was set at 70 ^{o}C. We obtained continuous line patterns by inkjet printing with a droplet spacing of 60 ㎛. Once printed, the line uniformity test pattern was left in open air at room temperature to dry for 10 minutes and sintered at 170 ^{o}C for 30 minutes. Fig. 4 shows a microscopic image of the line uniformity test pattern with an inset showing a magnified image. Measured width and spacing of the printed Ag lines were 126 ± 3㎛ and 114 ± 3㎛, respectively. The line uniformity test pattern thus showed a uniformity of line-width and spacing of 120 ㎛ ± 10%.

The transmission loss of the inkjet-printed microstrip line was estimated using three dimensional-full electro-magnetic (EM) simulation, as shown in Fig. 5. The schematic view for the High Frequency Structural Simulator (HFSS) simulation and the simulated result for RF transmission are shown in the Fig. 5 (a) and (b), respectively. The microstrip line for the high-frequency transmission loss test was printed on a 5 ㎛-thick primer covering a 0.8 mm-thick FR4 substrate, which was 1.7 mm wide, 50 mm long, and 5 ㎛ thick. The simulated results of the high-frequency transmission loss simulation showed an insertion loss of –1.8 dB at 10 GHz and reflection loss of more than –30 dB at 10 GHz. The ground of the microstrip line was formed on the backside of the FR4 substrate. For the transmission loss test at high frequencies, the RF characteristics of the inkjet-printed microstrip line were measured using a vector network analyzer (VNA) over a range from 1 GHz to 10 GHz. As shown in Fig. 6, the 50 mm-long micro strip line shows an insertion loss of – 5.0 dB at 10 GHz and a reflection loss of –15 dB at 10 GHz. The large discrepancy between the simulation and the measurement could be explained by the non-uniformity of the silver nanoparticles on the substrate. The resistivity value was 5.3 $\mu\Omega$·cm in the simulation. Hollow holes with a diameter of around 1~2 ㎛ and a depth of 50~150 nm were observed all over the inkjet-printed Ag surface through the surface analysis using a surface profiler, an atomic force microscope and a scanning electron microscope (data not shown). The hollow holes took around 20% area of the whole printed Ag surface. Therefore, in comparison with the simulated results using a smooth surface without

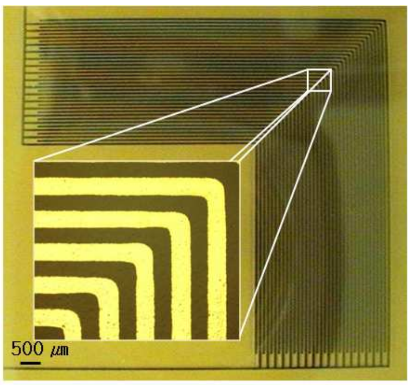
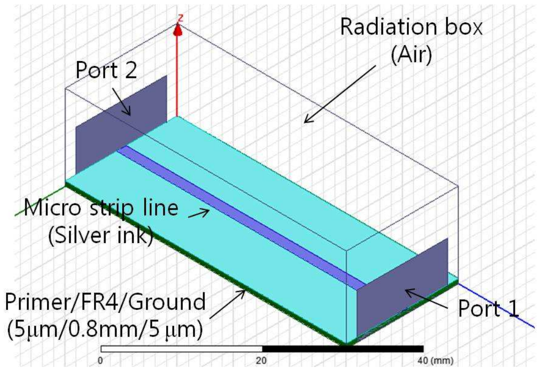
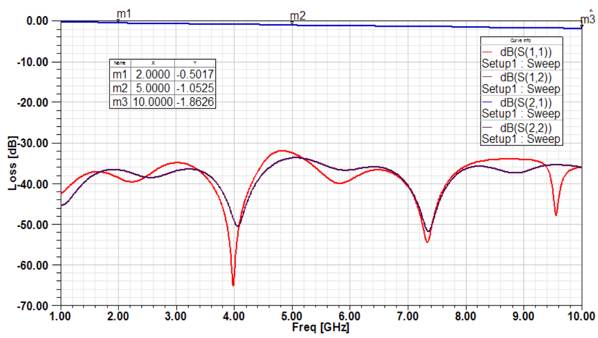


Fig. 4. Microscopic image of line width uniformity test pattern with line and spacing of 120 ㎛ ± 10%



(a)



(b)

Fig. 5. (a) HFSS simulation layout for high-frequency transmission loss test and; (b) simulated results of high frequency transmission loss simulation

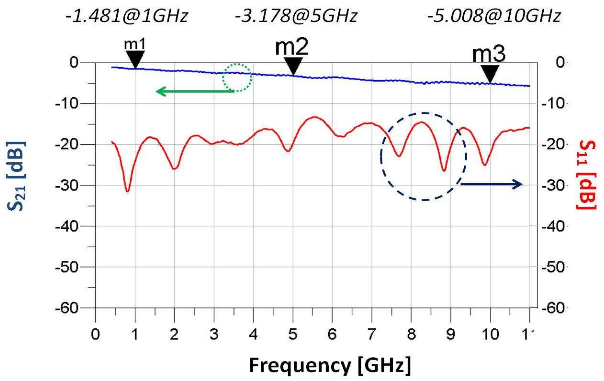


Fig. 6. Measured results of high-frequency transmission loss test

hollow holes, RF insertion loss and return loss might deteriorate due to the magnetic field generated by surface inductance near the hollow holes [14]. The non-uniformity of the silver nanoparticles could be a critical loss and main reason of the degradation.

In addition, the electrical resistivity ρ of the fully sintered lines was estimated and compared according to the relationship $\rho = RA/L$ (R: resistance, A: cross-sectional area, and L: length). The resistivity of the line made from the TGME-based ink was 5.3 $\mu\Omega\cdot$cm which corresponds to approximately 3.3 times the resistivity of bulk silver (1.6 $\mu\Omega\cdot$cm).

4. Conclusions

To characterize inkjet-printed Ag patterns to test their suitability for application in the PCB industry, we established seven test items and conditions for reliability evaluation following the Institute for Interconnecting and Packaging Electronic Circuits

(IPC) TM650 test method guidelines. The Ag patterns tested in the withstanding voltage test did not show any spark discharge, short circuiting, or dielectric breakdown phenomena after transmitting 1000 V DC for 30 seconds through both ends of a 5 cm line. The ASTM D3359 adhesive strength rating achieved by the tested sample was 5B, showing no loss of adhesion of the Ag pattern. The thermal shock test result showed resistance changed within ± 10% after testing without any spark discharge or short circuiting and the insulation resistance value for pressure cooker test was far greater than the pass condition of 100 MΩ at 121 °C, 2 atm, and 97% relative humidity after 48 hours. The bending test passed the test criterion above 10,000 times with showing 10,900 ± 1226 times. The line width uniformity test showing ±10% variation for the test samples, reached the required standard. The results of the high-frequency transmission loss test showed an insertion loss of – 5.0 dB/cm at 10 GHz for the micro-strip line and ground formed with silver ink.

In summary, this analysis confirmed that inkjet-printed Ag patterns are suitable for PCB applications insofar as the test results of the withstanding voltage, adhesive strength, thermal shock, pressure cooker, bending, and line-width uniformity are concerned. However, the inkjet-printed Ag patterns did not meet the required standards necessary to "pass" the high-frequency transmission loss test.

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