

# Temperature-Dependent Reliability of Inkjet-Printed Structures in Constant-Humidity Environment

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## Abstract

*Inkjet technology has shortened the processing time for current electronic components and minimizes material waste during their manufacture. Inkjet-printed nano silver ink IC interconnections are presented, but their reliability at varying temperatures as well as the evaluation of their reliability after humidity tests is not exhaustively explored. In this paper, we investigated the reliability of inkjet-printed interconnections from a variable environmental point of view. We tested nano silver inkjet-printed interconnections at 85°C in 85% RH for 1,000 hours for humidity evaluation and for 1,000 cycles from -40°C to 125°C to investigate temperature-cycling-based failures in combination with JEDEC Test Standards. Greek cross-structure, which is commonly used in the semiconductor industry, was used to evaluate fluctuation of the conductivity value after variable environment conditions. On the basis of the measurements obtained, we related electrical values before and after the tests using the Greek cross-structure. Also, the printing resolution of the inkjet-printed structures and the effect of these structures on the environmental reliability in the mentioned test runs were evaluated. The results of the temperature cycling test and of the humidity test show that the inkjet-printed silver structures have a good degree of reliability.*

## Introduction

Recent research has been demonstrating the formation of an electrically conductive interconnection by inkjet printing technology and discussing the potential of the technology [1-12]. Fast processing of conductive lines, which is more effective than screen printing, represents one of the benefits of transferring the technology to mass manufacture [4]. In addition, this innovative manufacturing method comes with several advantages such as maskless production, savings in material and energy, fewer process steps, hardly any waste liquid during manufacture, low-cost, and environmental impacts [1-12].

Inkjet technology uses nanotechnology to expedite manufacture and save energy in producing interconnections of electronic circuitry. To implement new specifications, the electronic packaging industry has been increasing the packaging density and functionality to meet customer requirements [1-12]. Currently, the tasks to be critically investigated are the evaluation of the printing process steps and of inkjet-printed interconnections.

In this paper, we investigated the reliability of inkjet-printed nano silver interconnections in various environments. Because of a gap in standardized reliability evaluation of inkjet-printed structures, we used several test standards that are well-known and widely applied in electronics. Similar select test conditions have been used by several other research groups in inkjet printing technology [7, 12]. In addition, using industrial standards enables

comparison of test results and improves competence and knowledge in inkjet technology for mass manufacture.

## Materials Selection

Nanoparticles are commonly being used in inkjet printing technology because of the advantages of lower sintering temperature and duration. This phenomenon is based on the “quantum size effect” [2, 13], which also helps meet particle size requirements for printhead nozzles. Another important requirement for suitable inkjet printing ink is nanoparticle stability in ink. The particle stability of the tested silver ink was controlled during formulation in a liquid vehicle consisting of ethylene glycol, glycerol, and ethanol (properties of the tested silver nanoparticle ink are shown in Table I).

Table I. Properties of nanoparticle ink

|   | Before sintering | After sintering |
|---|------------------|-----------------|
| Particle size (nm)                                | 30-50            | -               |
| Metal content (wt%)                               | 20               | -               |
| Viscosity (mPa.s)                                 | 12-14 mPa.s      | -               |
| Specific resistance ( $\mu\Omega\cdot\text{cm}$ ) | -                | 5               |
| Thickness ( $\mu\text{m}$ )                       | -                | ~2              |

Inkjet printing technology enables printing on flexible organic substrates and rigid substrates that are commonly used in electronic manufacture, e.g. flame retardant 4 (FR4) or low temperature co-fired ceramic (LTCC), or even on paper [1-12]. The flexibility of the substrate enables here several interesting applications, e.g., e-paper, organic radio frequency identification (RFID) tags, and organic light emitting displays (OLED). In our experiment, we selected and tested several flexible substrates, i.e., polyethylene naphthalate (PEN), liquid crystal polymer (LCP), and Kapton polyimide (PI). The substrates differ in their engineering properties, e.g., dielectric constant, moisture absorption, and coefficient of thermal expansion (CTE) [14] (several selected properties of the tested flexible substrates are shown in Table II).

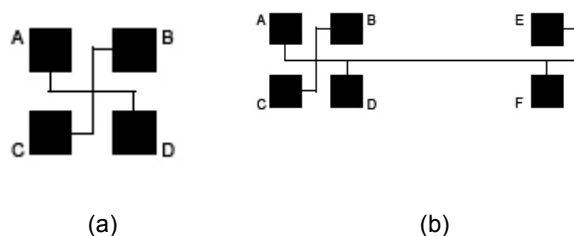
## Test Structure

Commonly used in the semiconductor industry because it does not need dimensional information for measurement of sheet resistance [15], the Greek cross structure was selected here to measure the resistivity of inkjet-printed structures. A Greek cross circuit has four pads connected to each other at equal lengths and line width. The symmetrical structure compensates for contact resistance and provides reliable results. The structure was used in [16] to measure the sheet resistance and resistivity of inkjet-printed traces. The test structure was multiplied, and resistivity was measured of pad D to pad F (Figure 1 (b)). The distance between

the two pads allowed us to measure electrical fluctuation in a long line of about 10.5 mm and secure reliable results in case changes occurred under varying conditions.

**Table II. Properties of substrates**

|                                 | Polyethylene Naphthalate (PEN) | Liquid Crystal Polymer (LCP) | Kapton Polyimide (PI) |
|---------------------------------|--------------------------------|------------------------------|-----------------------|
| Thermal expansion (CTE)(ppm/°C) | 18-20                          | 17                           | 20                    |
| Tensile strength (MPa)          | 250                            | 294                          | 139-231               |
| Melting point (°C)              | 270                            | 310                          | -                     |
| Dielectric constant             | 2.90                           | 2.85                         | 3.50                  |
| Moisture Absorption (%)         | 0.40                           | 0.04                         | 1.80                  |



**Figure 1.** (a) Greek cross test structure adopted from semiconductor industry. (b) Test structure of 4.5mm x 14.5 mm inkjet-printed on selected substrates; designed line width ~190  $\mu$ m.

A reliability test board consisting of 60 Greek cross structures in twelve rows for each substrate was inkjet-printed on PEN, LCP, and PI.

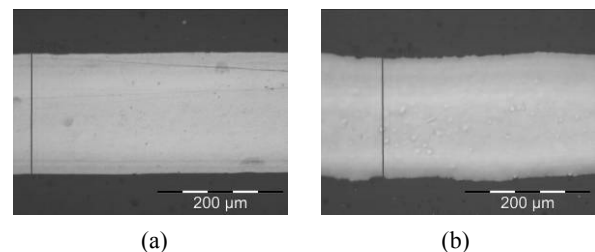
## Printing Process

The Greek cross structures were printed with a piezo printhead type inkjet printer. A digital printing file of the structure was created on a computer-aided design (CAD) platform. Before printing, the substrate surfaces were wiped with isopropanol to clear them of dust and contamination and were then subjected to UV/Ozone surface treatment for 5 minutes [17]. Cleaning and modifying the substrate surface help control the droplets over the substrate. In addition, an electronic coating allows also printing of highly aligned and narrow conductive lines or wide and thin patterns [1, 17, 18].

The cleaned substrates were placed on a metal substrate holder plate under the industrial inkjet printer. Because the printing file resolution affects the conductive interconnections, two different printing resolutions were evaluated, i.e., 600 dpi (dots-per-inch) and 1550 dpi. With an industrial-type inkjet printer, high resolution printing poses some challenges. A major problem we encountered during printing was bulking of droplets. Image masking, which also affects the long term reliability of printed structures, makes it possible to achieve adequate surface quality in

printing multi-layer structures [1]. Furthermore, high resolution, which determines the amount of silver ink deposited on the substrate, increases the amount of the ink and consequently the reliability of the structures. In high resolution printing, also electrical conductivity will be high.

On the other hand, it is important to evaluate the repetition of printed layers to understand the electrical performance and surface quality of inkjet-printed structures [19, 20]. We evaluated four-layer printing with 600 dpi and single-layer printing with 1550 dpi and printed similar pattern formations, which showed that the same amount of ink was printed on both substrates (Figure 2). From this experiment, we chose the four-layer printing with 600 dpi for the following reliability test.



**Figure 2.** (a) 600 dpi resolution, 4-layer printing (b) 1550 dpi resolution, single-layer printing. With both resolutions, interconnection line width measured ~190  $\mu$ m.

## Reliability Test Procedure

The inkjet-printed silver structure should be integrated to evaluate the reliability of such structures in varying temperature and environmental conditions [18]. Such an accelerated life test usually accelerates the failure mechanism, which again facilitates detection of failures much sooner than in regular service conditions [21]. High humidity and high temperature tests were run to identify possible material-related failures in harsh conditions.

In this study, we used the standardized test methods [22, 23] by JEDEC to determine the humidity-related and temperature-variation-related problems of our inkjet-printed silver structures. The conditions for each test were as follows:

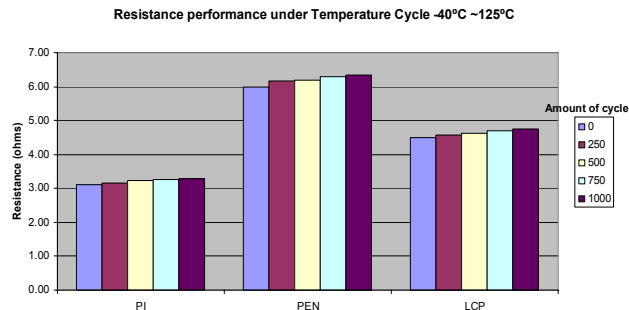
- In the humidity test, 85°C and 85% relative humidity (RH) was applied for 1,000 hours. According to the supplier, fluctuation in the humidity chamber was  $\pm 0.3^\circ\text{C}$  and  $\pm 2.5\%$  RH. The electrical performance of the samples was measured at every 250 hours.
- In the temperature cycling test,  $-40^\circ\text{C}$  to  $125^\circ\text{C}$  for 1,000 cycles was used with a cycle set at 30 minutes. The electrical performance of the samples was measured at every 250 cycles.

An important parameter that may directly affect reliability results is the selection of the sintering profile of inkjet- printed samples. In this experiment, test samples were sintered at  $220^\circ\text{C}$  for 60 minutes.

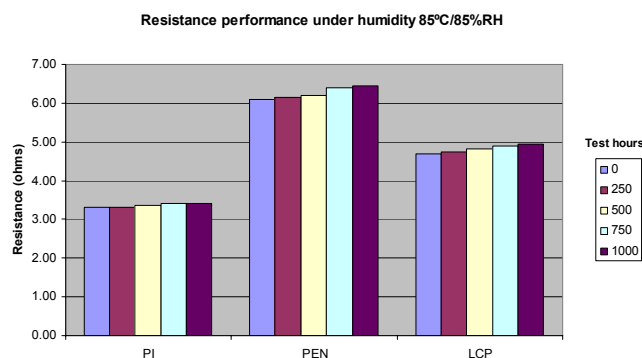
## Results

Silver nanoparticle ink with 600 dpi resolution was inkjet-printed in four layers on PEN, LCP, and PI substrates. The inkjet-printed samples were cured at  $220^\circ\text{C}$  for 60 minutes, and their electrical resistance was measured. The samples were then placed

in a humidity and a temperature cycling chamber for further tests (results shown in Figures 3 and 4).



**Figure 3.** Changes in resistance performance during temperature cycling at -40°C to 125 °C.

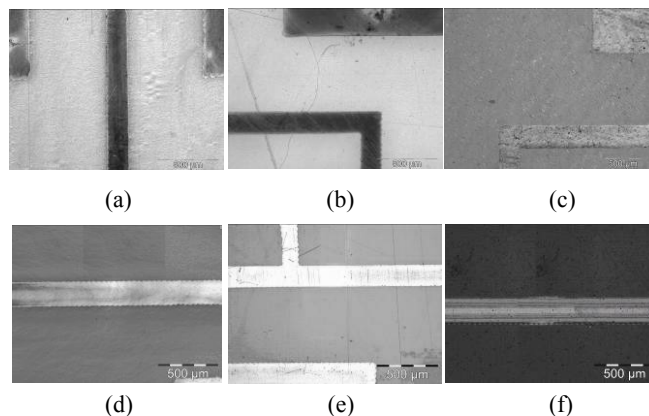


**Figure 4.** Changes in resistance performance in humidity test at 85°C and 85% RH.

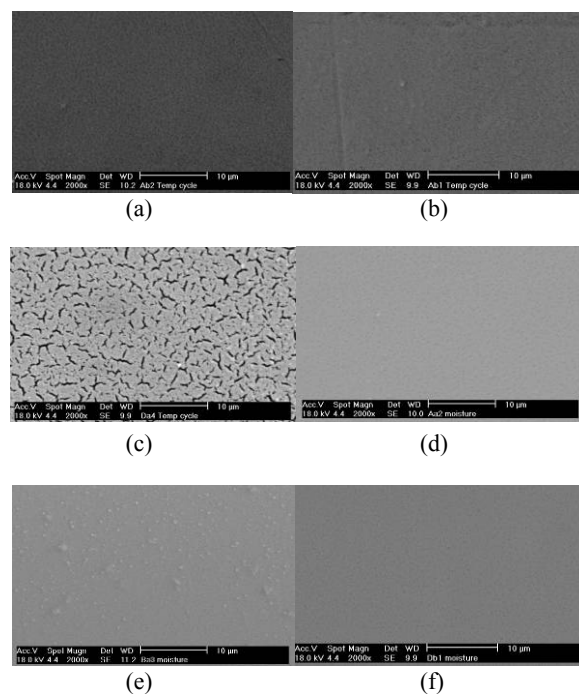
Our temperature cycling and humidity results showed similar trends in resistance. The inkjet-printed structures showed slight electrical fluctuation at varying temperatures and constant humidity. For temperature cycling, the resistance varied depending on the tested substrate from ~5-6% and for humidity ~3-6%. In the latter test, moisture affected the PEN and LCP substrates more than the PI substrate, which, though capable of absorbing more moisture than the others, may have been affected by the printing process. Silver droplets may have spread better on PEN and LCP than on PI during printing process. Hence the forming thinner and mechanically weakly bonded silver droplet structures on PI occurred against moisture absorption and some micro cracks (Figure 6) based on the thermal changes is noticed.

In temperature cycling, the substrates' coefficient of thermal expansion (CTE) was so close as to yield a similar change in resistance performance. Figure 5 shows several optical microscope pictures of inkjet-printed structures on PEN, LCP, and PI substrates.

Measured after the environmental tests, resistance showed no significant increase. In addition, the structures did not degrade or show any corrosion-related failure during the tests. Visual observation was done with a scanning electron microscope (SEM), and an energy dispersive x-ray spectrometry (EDS) was used for elemental analysis of the samples on selected sections. In x-ray spectrometry, the highest peak was registered from Ag L  $\alpha$  energy level. Results showed no oxidation during the tests on these inkjet-printed silver structures.



**Figure 5.** Microscope pictures of the selected samples. First row: temperature cycling samples after 1,000 cycles from -40°C to 125°C; (a) LCP, (b) PEN, and (c) PI. Second row: humidity test samples after 1,000 hours; (d) LCP, (e) PEN, and (f) PI.



**Figure 6.** SEM pictures of the samples. First row: temperature cycling samples after 1,000 cycles from -40°C to 125°C; (a) LCP, (b) PEN, and (c) PI. Second row: humidity test samples after 1,000 hours; (d) LCP, (e) PEN, and (f) PI.

## Conclusions

Nanoparticle-based silver ink was inkjet-printed on several flexible substrates, namely PEN, LCP, and PI, and their electrical performance was examined under several test conditions. Resistance measurements in a humid environment of 85°C/85% RH and in temperature cycling from -40°C to 125°C showed no significant fluctuation in the resistance of the tested samples. The resistance of the humidity and temperature cycling tested samples were measured and their fluctuation determined at ~3-6% and ~5-6%, respectively. Though the structures were created with low resolution in four-time printing, the higher resolution printing

deposited an equal amount of material. The reliability of the structures was evaluated based on the lower resolution printing.

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## References

- [1] M. Mäntysalo and P. Mansikkamäki, Inkjet Deposited Interconnections for Electronic Packaging, Proc. of IST Digital Fabrication, Alaska, USA, pg. 813-817, (2007).
- [2] H. Saito and Y. Matsuba, Liquid Wiring Technology by Ink-jet Printing Using NanoPaste®, Proc. 39<sup>th</sup> International Symposium on Microelectronics, California, USA, October 8-12, (2006).
- [3] H. Imai et al., Application of Inkjet Printing Technology to Electro Packaging, Proc. 39<sup>th</sup> International Symposium on Microelectronics, California, USA, October 8-12, (2006).
- [4] Y. Kawamura et al., LTCC Multilevel Interconnection Substrate with Ink-jet Printing and Thick Film Printing for High-Density Packaging, Proc. 39<sup>th</sup> International Symposium on Microelectronics, California, USA, October 8-12, (2006).
- [5] S. Nishi, Direct Metal Patterning for Printable Electronics by Inkjet Technology, Proc. 39<sup>th</sup> International Symposium on Microelectronics, California, USA, October 8-12, (2006).
- [6] K. Suganuma et al., Ink-jet Printing of Nano Materials and Processes for Electronics Applications, The 9th International IEEE CPMT Symposium on High Density Design, Packaging and Microsystem Integration (HDP'07), June 26-28, pg. 1-4, (2007).
- [7] S. Joo and D. F. Baldwin, Performance of Silver Nano Particles as an Electronics Packaging Interconnects Materials, Proc. 57<sup>th</sup> Electronic Components and Technology Conference, May 29-June 1, pg. 219-226, (2007).
- [8] C. Curtis et al., Metallizations by Direct-Write Inkjet Printing, NCPV Program Review Meeting, Colorado, USA, October 14-17, (2001).
- [9] J. G. Bai et al., "Inkjet Printable Nanosilver Suspensions for Enhanced Sintering Quality in Rapid Manufacturing," Jour. Nanotechnology, IOP Publishing Ltd., 18, 1-5, (2007).
- [10] D. J. Hayes et al., Low-Cost Display Assembly and Interconnect Using Ink-Jet Printing Technology, Proc., Display Works'99, San Jose, February, pg. 1-4, (1999).
- [11] D. Wallace et al., Think Additive: Ink-Jet Deposition of Materials for MEMS Packaging, 6<sup>th</sup> Topical Workshop on Packaging of MEMS and Related Micro-Nano-Bio Integrated Systems, November 18-20, California, USA, (2004).
- [12] R. N. Das et al., Printable Nanocomposites for Advanced Organic Packaging, Proc. 57<sup>th</sup> Electronic Components and Technology Conference, May 29-June 1, pg. 316-322, (2007).
- [13] P. Buffat and J. Borel, "Size Effect on the Melting Temperature of Gold Particles," Physical Review A, 13, 6, pg. 2287-2298, (1976).
- [14] J. J. Licari, Coating Materials for Electronic Applications: Polymers, Processes, Reliability, Testing (Noyes Publications, NY, USA, November 1, 2003) pg. 552.
- [15] S. Enderling et al., Design Rules to Minimize the Effect of Joule Heating in Greek Cross Test Structures, Jour. Semiconductor Manufacturing, 17, 2, pg. 84-90, (2004).
- [16] J. Pekkanen et al., Laser Sintering of Ink-jet Printed Ag Nano Ink Traces, Proc. 4<sup>th</sup> International Conference of on New Exploratory Technologies (NEXT) 2007, October 25-27, Seoul, Korea, pg. 111-114, (2007).
- [17] U. Caglar et al., Analysis of Mechanical Performance of Silver Inkjet-Printed Structures, Proc. 2<sup>nd</sup> IEEE International Nanoelectronics Conference, March 24-27, pg.6 (2008).
- [18] U. Caglar and P. Mansikkamäki, Evaluation of Usability of Inkjet-Printable Materials in Electronics Manufacturing, Nanotechnology, Materials and New Production (NMP), Helsinki, Finland, April 16-17, (2008).
- [19] D. Redinger et al., An Ink-Jet-Deposited Passive Component Process for RFID, IEEE Transactions on Electron Devices, 51, 12, December 2004, pg. 1978-1983, (2004).
- [20] V. Subramanian et al., All-printed RFID Tags: Materials, Devices, and Circuit Implications, Proc. of 19<sup>th</sup> International Conference on VLSI Design (VLSID'06), January 3-7, pg. 6- , (2006).
- [21] A. Seppälä, "Flip Chip Attachment on Rigid Substrates Using Anisotropic Conductive Adhesive Films," Ph.D. Thesis, (Dept. of Elec. Eng., Tampere University of Technology, Finland, November 14, 2003) pg 41.
- [22] Steady State Temperature Humidity Bias Life Test, EIA/JEDEC Standard Test Method A101-B.
- [23] Temperature Cycling Test, JESD22-A104C.

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