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PRINT-CONSISTENCY AND PROCESS-INTERACTION FOR INKJET-PRINTED COPPER ON FLEXIBLE SUBSTRATE

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ABSTRACT

Printed electronics is a fastest growing and emerging technology that have shown much potential in several industries including automotive, wearables, healthcare, and aerospace. Its applications can be found not only in flexible but also in large area electronics. The technology provides an effective and convenient method to additively deposit conductive and insulating materials on any type of substrate. Comparing with traditional manufacturing processes, which involves chemical etching, this technology also comes to be relatively environmental friendly. Despite its status, it is not without its challenges. Starting from the material being compatible in the printer equipment to the point of achieving fine resolutions, and with excellent properties are some of the challenges that printed electronics face. Among the myriad of printing technologies such as Aerosol Jet, micro-dispensing, gravure printing, screen printing, Inkjet printing, Inkjet has gained much attention due to its low-cost, low material consumption, and roll-to-roll capability for mass manufacturing. The technology has been widely used in home and office, but recently gained interest in printed electronics in a research and development setting. Conductive materials used in Inkjet printing generally comprises of metal Nanoparticles that need to be thermally sintered for it to be conductive. The preferred metal of choice has been mostly silver due to its excellent electrical properties and ease in sintering. However, silver comes to be expensive than its counterpart copper. Since copper is prone to oxidation, much focus has been given towards photonic sintering that involves sudden burst of pulsed light at certain energy to sinter the copper Nanoparticles. With this technique, only the printed material gets sintered in a matter of seconds without having a great impact on its substrate, due to which it is also preferred in low temperature applications. With all the knowledge, there is still a large gap in the process side with copper where it is important to look how the print process affects the resolution of the print along with the effect of post-print processes on electrical and mechanical properties. In this paper, a copper Inkjet ink is utilized for understanding the effect of Inkjet print parameters on the ejected droplet and its resolution. Post-print process is also quantified using a photonic sintering equipment for excellent electrical and mechanical properties. To demonstrate the complete process, commercial-off-the-shelf components will also be mounted on the additively printed pads via Inkjet. Statistically, control charting technique will be utilized to understand the capability of the Inkjet process.

Keywords: Inkjet, konica minolta, copper ink, photonic sintering, printed electronics, control chart

NOMENCLATURE

AM	Additive manufacturing
IJ	Inkjet
PZT	Lead Zirconate Titanate (piezoelectric ceramic material)
IPL	Intensed Pulse Light
SMD	Surface Mount Devices
CIJ	Continuous Inkjet
DOD	Drop-on-Demand Inkjet
TIJ	Thermal Inkjet
KM	Konica Minolta
LED	Light Emitting Diode
ECA	Electrically Conductive Adhesive

1. INTRODUCTION

Printed electronics have come a long way in recent years proving the fact that manufacturing of electronics can be done with just a click of a button. Conventionally, fabrication is mostly based on photolithography, that is a complex and time consuming process involving various hazardous chemicals. With myriad of additively printing platforms, this technology is

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involved not only in flexible electronics, but conformal as well, i.e. there is no restriction of substrate anymore. Considering the advantages and benefits of Additive Manufacturing (AM), the technology is still continuously evolving, however, it is not as prevalent in industry due to its high cost and requirements for large facilities. Due to this, there is increase in demand for high quality, low cost fabrication solutions that can be used in mass manufacturing facilities with a greater throughput than traditional methods. In this regard, direct-write printing techniques have emerged to be promising methods to fabricate various electronic devices, which include Aerosol Jet, Inkjet, micro-dispensing, gravure-offset, all of which have their own benefits and limitations. Among these, Inkjet appears to be getting more attention because of its much higher throughput, low-cost, low material consumption, and non-contact process. The technology is definitely not recent, and has been widely used in home and office, however, lately, it is being used as a low cost tool to manufacture various aspects of electronic systems in a research setting.

Much work is put in printed electronics using state-of-art direct-write technologies. These include Aerosol Jet, micro-dispensing, and Inkjet, that can accommodate various types of materials with wide range of viscosity. Detailed reviews and multiple applications including multi-layer substrates using these techniques is studied by many researchers [1-6], especially Inkjet due to its capability to utilize least material. In many of the studies, silver is been the preferred metal of choice in printed electronics due to its availability and ease in its post-print treatment [7-10]. Additionally, researchers have also explored the use of base temperature while printing for immediate curing of the printed sample instead of using a oven, that opens up another category of applications in low temperature electronics [11-13]. Different substrates have also been utilized with custom made silver nanoparticle ink on PET, glass, silicon [14]. Authors have also utilized combination of Inkjet and micro-dispensing direct-write for SMD interconnections using silver ink with conductive adhesive and low temperature solder [15,16]. Apart from silver, other metals including copper, nickel, carbon nanotubes, graphene are also studied [17-19]. In regards to copper ink synthesis, techniques have been developed using thermal decomposition, ultraviolet irradiation, reduction of copper salts, and chemically controlled process, to be able to effectively jet with no clogging [20-23]. These techniques, although efficient, are not economically suitable because of chemicals and equipment requirements. Due to this, a commercial ink is used in this study to avoid inclusion of those variables, and focus more on the process side of Inkjet printing the ink.

In terms of throughput, IJ comes to be dominant compared with others owing to the fact that there are hundreds, and even thousands of nozzles used during the printing. Due to multiple number of nozzles and with printing speeds in hundreds of mm/s, a complex print can be done in few minutes, which makes it a perfect technique for both, small and high volume production. However, in regards to material compatibility, IJ lacks behind since it requires the material to be in liquid form within a very narrow range of viscosity. In addition, clogging of the nozzles is also more frequent with little to no way of recovering them. Although the technique has its drawbacks, the benefits far outweighs its flaws. Being able to print with very little material consumption and with the properties as close to the bulk material brings a lot to the table of flexible hybrid electronics systems

1.1. Inkjet Technology

Inkjet printing is a deposition technique that additively builds up structures by generating droplets placed at a point with extreme precision and volume. The process can achieve high quality and reproducible structures, due to which it is recognized as an advanced and economical method for electronics manufacturing. The printing is further classified into two categories. Continuous inkjet (CIJ) and Drop-on-Demand (DOD). As the name suggests, in CIJ, a continuous stream of material is running through the print head that leads to a continuous flow of droplet production. As a consequence, some of the droplets gets unused and are re-circulated back into the reservoir resulting in potential contamination. To avoid this, DOD technique is introduced in which droplets are only generated when required i.e. more efficient. Although there is a drawback of jets clogging when idle, this technique is generally preferred over CIJ. Droplets generated through DOD are further classified into two types: Thermal inkjet (TIJ) and piezoelectric DOD. TIJ are low-cost printers in which a resistor inside the ink chamber is heated up and the bubble of ink vapor pushes the ink out the nozzle. This technique requires the ink to have a volatile component to form the vapor bubble. In piezoelectric DOD, a voltage pulse is supplied that actuates that piezoelectric transducer (PZT), creating pressure waves within the ink reservoir, thus generating the droplets. Among these two, the preference is given to piezoelectric inkjet printing due to the larger extent of control over the velocity and volume of the droplet. A flowchart explaining the different categories is shown in FIGURE 1.

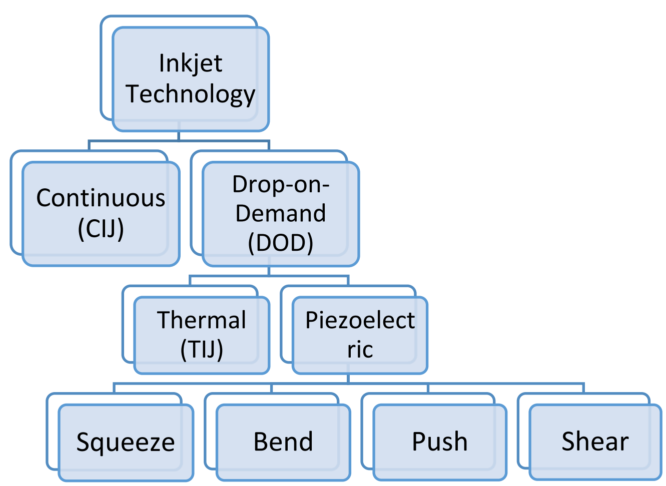


FIGURE 1: DIFFERENT CATEGORIES OF INKJET

The piezoelectric DOD technology can also be classified in four mode types: squeeze; bend; push; and shear. For the extent of this paper, the discussion will only focus on shear mode. FIGURE 2 shows the structure of shear-mode piezo actuator. The piezoelectric walls formed by piezoelectric ceramics covers the ink pressure chamber. A voltage pulse is applied to the walls that results in shear-mode deformation, due to which the ink droplets are ejected. The pulse consists of forward and reverse voltage which creates the corresponding electric field that increases and decreases the volume of the ink pressure chamber respectively. This process happens in a sequence resulting in a pressure wave inside the ink chamber that ejects the droplet out of the nozzle. The cross-section of the ink chamber is shown inset of FIGURE 2. Detailed process can found in literature [24].

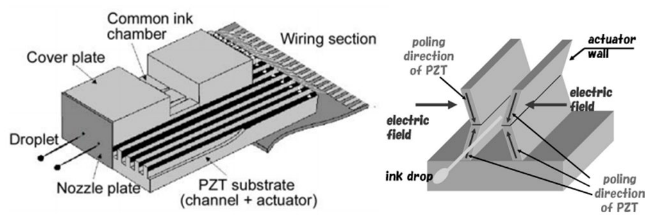


FIGURE 2: CROSS SECTION OF KONICA PRINT HEAD

1.1.1. Basics of Waveform

A waveform, in a piezo-based inkjet printer, defines how much and for how long the PZT element should deform to create a nice, clean, and round droplet. It consists of voltage that determines how much and pulse timings that determines how long. Both of these can have a great impact on the droplet shape, speed, stability, and the final print. The waveform starts with a ON voltage parameter that deforms the PZT and expands the ink channel to draw the ink followed by ON pulse width which specifies the time the ON voltage should be supplied. The waveform then moves to OFF voltage to contract the ink channel back to push the liquid and eject the droplet and OFF pulse width repeatedly for thousands of times in a second. Voltage parameter is responsible for the droplet volume and its speed, while pulse width is responsible for determining the maximum speed of the droplet. In general, ON voltage is set to be twice of OFF voltage, and OFF pulse width is set to be twice of ON pulse width. Apart from the droplet characteristics, these parameters also decide the jetting frequency of the print head that is different for every print head.

1.1.2. Equipment in-house

The inkjet printer used in the study is from SUSS MicroTec, PiXDRO LP50 desktop R&D Inkjet printer, shown in FIGURE 3. The printer is equipped with piezo-electric DOD Konica Minolta (KM) print head assembly. The print head, shown in FIGURE 4, consists of 512 jetting nozzles, 256 nozzles arranged in one line, with 42pL as nominal droplet volume. Other specifications for the head are listed in TABLE 1.

FIGURE 3: INKJET PRINTER IN-HOUSE

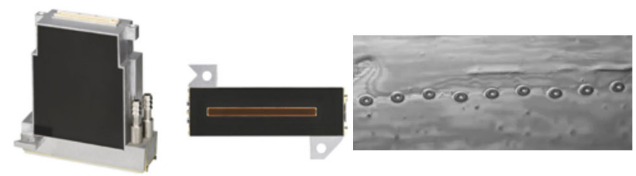


FIGURE 4: KONICA MINOLTA 512 PRINT HEAD

TABLE 1: KONICA MINOLTA 512 SPECIFICATIONS

KM512	
Technology	Piezo Drop-on-Demand (Shared wall 3 cycle)
Resolution	180dpi × 2lines = 360dpi
Number of Nozzles	256 nozzles × 2lines = 512 nozzles
Nozzle Pitch	70.5µm
Drop Size	42 pL
Max Frequency	7.6kHz
Printing Width	36.1mm

1.2. Photonic Sintering System

The photonic sintering system used in this study is from Xenon X-1100 high IPL model with linear stage interface, shown in FIGURE 5. The system delivers up to 9 Joules/cm^2 of radiant energy/pulse, with maximum lamp voltage range of 3000V. Some other specifications of the system is mentioned in TABLE 2.

FIGURE 5: PHOTONIC SINTERING SYSTEM IN-HOUSE

TABLE 2: XENON X-1100 SINTERING SYSTEM SPECIFICATIONS

Specifications of Xenon X-1100 Photonic Sintering System	
Max radiant energy (J/cm²)	9
Lamp Voltage Range (V)	1000-3000
Peak radiant power (kW/cm²)	8
Pulse length range (µs)	100-7000
Pulse modes	Single, Burst, Continuous

2. EXPERIMENTAL

2.1. Test Design

The aim of this article is to establish process conditions of pre-print, during the print, and post printing to obtain best physical, electrical, and mechanical properties. To this extent, the design consists of simple traces with rectangular pads at each end. The dimensions with the design are shown in FIGURE 6.

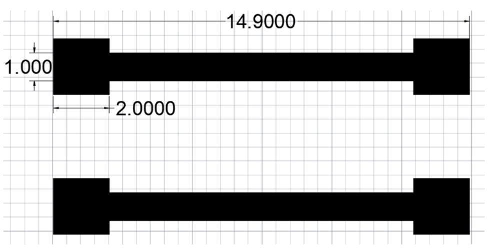


FIGURE 6: CAD FILE OF TRACE FEATURE

In addition to FIGURE 6, a design was also created that includes the pads for the mounting of SMD components, as shown in FIGURE 7. SMD includes standard size 2512 and 0805 resistors, capacitors, inductors, LED. To attach the SMDs, an additional step to Inkjet printing of pads is required for a strong interconnection, that involves printing of ECA. Since ECA is relative viscous compared with Inkjet inks, nScrypt direct-write technology is used to dispense the ECA. Process studies on ECA dispensing can be found in another paper___.

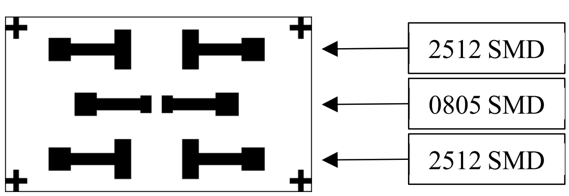


FIGURE 7: CAD FILE FOR SMD ATTACHMENT

2.2. Materials and Methods

Metallic Nanoparticles have received great attention in additively printing industry. They provide a very promising method for the fabrication of low-cost electronics. Various metals such as silver (Ag), palladium (Pd), nickel (Ni), gold (Au), copper (Cu), have been utilized for the fabrication of various electronic systems such as flat panel display, organic light-emitting diodes (OLEDs), sensors. The preferred metal of choice has been silver due to its availability and ease in post-print treatments, however, it comes to be expensive. Copper is seen to be the alternative due to its low-cost but requires additional photonic sintering instead of thermal due to oxide formation. To explore this further, copper is used as the material in this study. Material specifications for the material is provided in TABLE 3.

TABLE 3: COPPER INK SPECIFICATIONS

Material Specifications for Copper Inkjet ink	
Material	Copper
Metal Content (wt%)	20
Viscosity (cP)	30
Sintering	Photonic

In regards to the procedure, initially, the droplet was characterized at some of the important waveform parameters such as voltage, pulse width. In addition, droplet speed and volume was also quantified for multiple number of runs to understand the stability. With the waveform optimized, the test design is printed on a polyimide film that is wiped by iso-propanol to remove any impurities. During the print, the base temperature was kept to be at room temperature to avoid any pre-oxidation of the copper ink. The printed design was brought over to the photonic sintering system, where copper is sintered using IPL in a matter of few seconds. Effect of certain photonic sintering parameters such as voltage, energy, number of bursts was quantified by measuring the electrical resistivity of the printed sample. With the set of optimized process parameters, SMD components are mounted and frequency response is recorded along with sample-to-sample variation in each type of SMD. Detailed procedure is provided below in FIGURE 8.

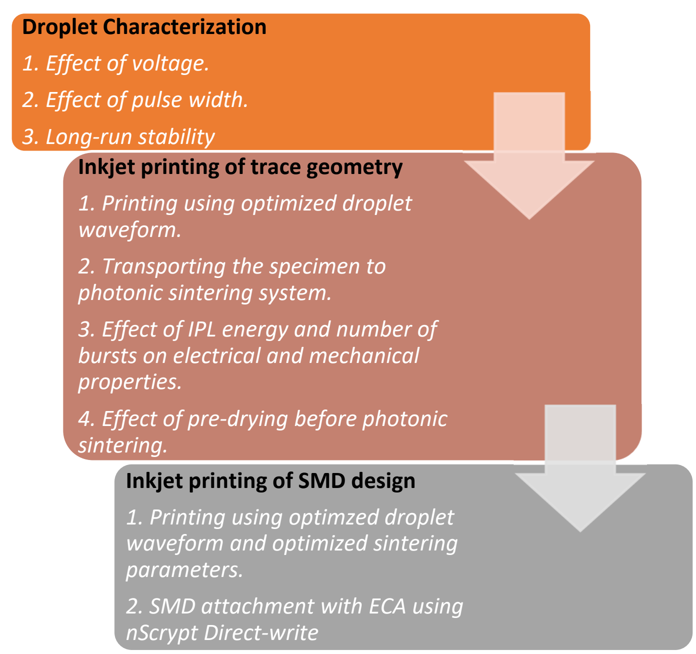


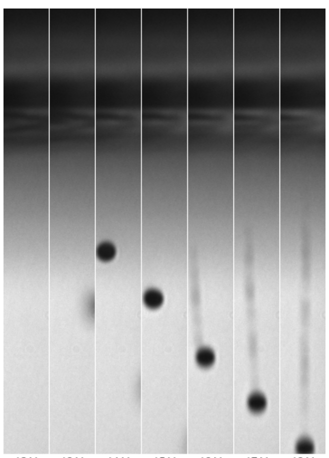
FIGURE 8: DETAILED PROCEDURE LAYOUT

3. RESULTS AND DISCUSSION

This section presents the results from multi-step process study of Inkjet printing with copper while focusing on achieving electrical properties close to bulk copper with maximum adhesion to the substrate.

3.1. Inkjet Printing of Copper

The process of printing copper starts with loading the ink into the reservoir and analyzing the jetted droplets. To get a nice, clean, and round jetted droplet, waveform needed to be optimized. Some of the important parameters that affect the shape, speed, drop volume, include voltage and pulse width. It is important to understand their effect on the jetted droplet to achieve a fine print. FIGURE 9 shows the effect of various voltages on the droplet at the same moment of time. Starting from 12V, there is little to no jetting initially because of very slow droplet speed and not within the camera view, with round droplet starting to jet around 14V and 15V, and satellites starting to form after 15V. Although the higher droplet speed is preferred and achieved on higher voltages, satellites or tails are not ideal since they can result in scatter of ink particles around the design, thus resulting in short-circuit.



- Higher droplet speed at higher voltages, however, with satellites (tails) formation.

FIGURE 9: EFFECT OF WAVEFORM VOLTAGE ON DROPLET

Long-run stability of the copper ink is also quantified. This parameter was selected so as to understand if there is any change in the droplet formation over multiple runs. Apart from droplet formation, change can also be represented by the droplet speed and droplet volume. FIGURE 10 shows the droplet stability over 30 runs. In long duration of prints, it becomes imperative to understand if the droplet formation is stable over that long duration. Looking at FIGURE 11, droplet speed is shown, that comes to be at an average of 1.53 m/s, with not much change over 30 runs. Similarly, droplet volume, shown in FIGURE 12, shows the average volume to be 38pL. Not much difference is seen over the duration of 30 runs. To look at the accuracy of the volume provided by the droplet analyzer software, manual calculation was also performed by measuring the radius of the droplet. Since pL (trillionth of a liter) is a very small volume unit, there is negligible difference between manually calculated (36pL) and software provided (38pL) values.

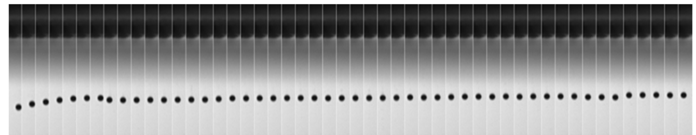


FIGURE 10: LONG-RUN STABILITY OF DROPLET

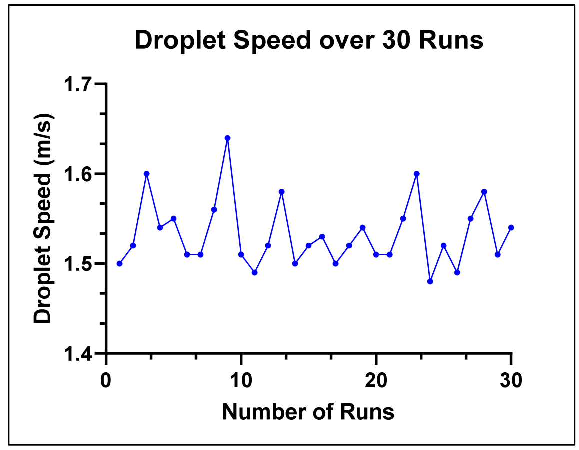


FIGURE 11: DROPLET SPEED OVER 30 RUNS

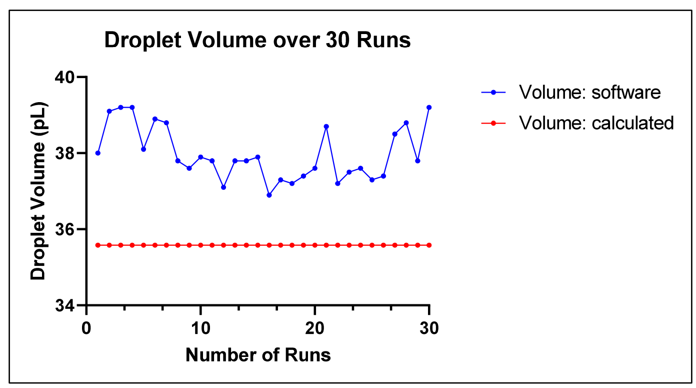


FIGURE 12: DROPLET VOLUME OVER 30 RUNS

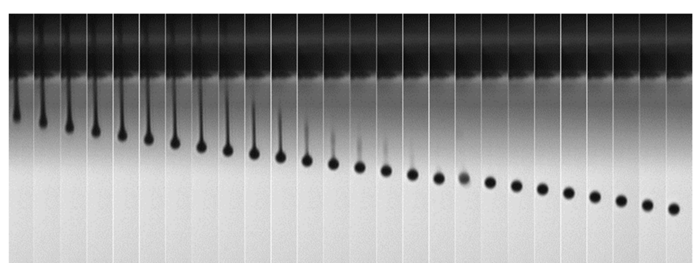


FIGURE 13: DROPLET FORMATION OF COPPER INK

Next, the droplet formation at the optimized waveform parameters is recorded. FIGURE 13 shows the frames at the same moment of time stitched together for a single droplet. Initially, droplet starts to come out the nozzle with a long tail, and in later moment of time, it forms into a nice and round droplet. The final waveform parameters are listed in **Error! Not a valid bookmark self-reference.**.

TABLE 4: WAVEFORM PARAMETERS FOR DROPLET FORMATION

ON Voltage (V)	15
OFF Voltage (V)	7.5
ON Pulse Width (µs)	11
OFF Pulse Width (µs)	22
Phase Width (µs)	43

3.2. Photonic Sintering of Printed Copper

With the Inkjet print process parameters optimized, next step is to bring the printed sample over to the photonic sintering system. To get the best electrical and mechanical properties, various parameters of the system are studied. These parameters and their effect on electrical resistivity and shear load to failure are discussed next.

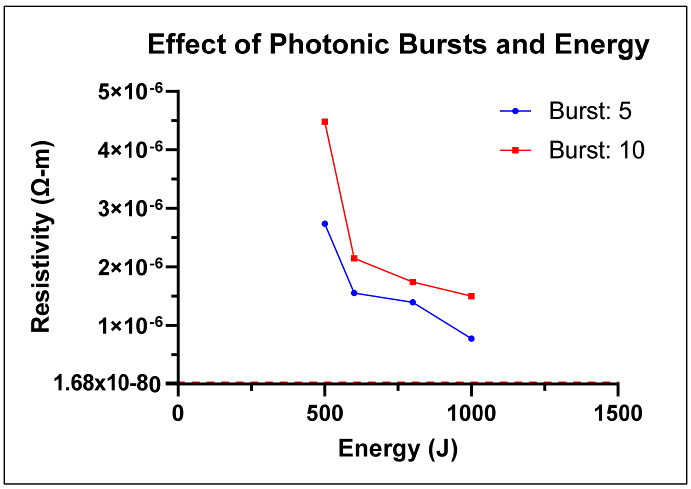


FIGURE 14: EFFECT OF PHOTONIC ENERGY AND NUMBER OF BURST ON RESISTIVITY – PART I

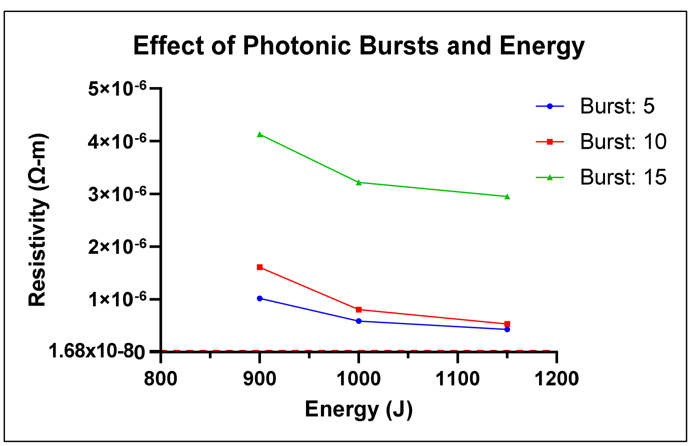


FIGURE 15: EFFECT OF PHOTONIC ENERGY AND NUMBER OF BURST ON RESISTIVITY – PART II

FIGURE 14 shows the effect of photonic energy on electrical resistivity of printed copper. With increase in energy from 500 J to 1000 J, there is clear decrease in resistivity as one would expect. However, the resistivity degrades when the number of bursts is increased from 5 to 10. Here, number of bursts is referred to the pulse sequence that is repeated specified number of times and stops. In regards to the lamp voltage used, it was noticed that going above than 2000 V, the printed copper started to burn. Hence, in most part of the study, voltage is fixed to 2000 V. Optical pictures are included later to show the burned samples.

Going in-depth to the effect of energy and number of bursts, FIGURE 15 shows the resistivity when the energy is increased to 1150 J with bursts varying from 5 to 10. The trend further proves that resisitivity gets better at higher energy levels and degrades at higher bursts. With a fixed voltage of 2000 V, maximum energy the system can generate is 1150 J, hence the energy to sinter copper is fixed to 1150 J.

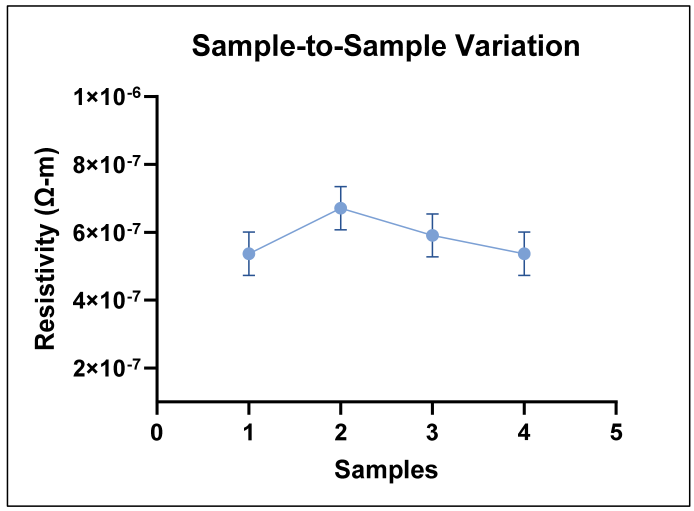


FIGURE 16: SAMPLE-TO-SAMPLE VARIATION IN RESISTIVITY

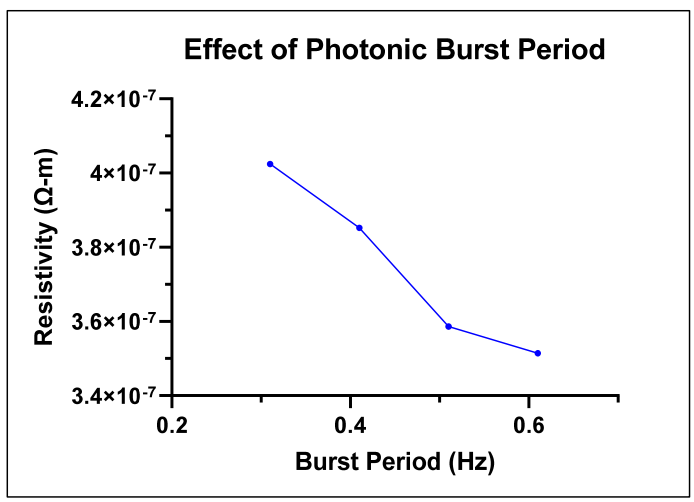


FIGURE 17: EFFECT OF BURST FREQUENCY ON RESISTIVITY

Using some of the optimized sintering parameters, sample-to-sample variation is studied, shown in FIGURE 16. With the average resistivity still very high of about 34 times the bulk copper between multiple samples, more optimization is to be done to reduce this factor. Since it was noticed that bursts of 5 gave the least resistivity, the period of those 5 bursts was varied from 0.3 Hz to 0.6 Hz, as shown in FIGURE 17. Least resistivity was found to be at 0.6 Hz frequency, and increases with decrease in the frequency. However, the resisitivy is still high compared with bulk copper.

To reduce the resistivity, another approach is implemented. After photonic sintering, it was noticed that the printed copper, although provided the connectivity, did not dry completely with the set sintering parameters. In addition, increasing the energy or voltage of the system resulted in burning of the sample and polyimide. Therefore, a pre-drying method was carried out. In this, after Inkjet printing of copper, the sample was put in standard convection oven for few minutes to dry partially and then photonic sintered under the set parameters discussed earlier. From FIGURE 18, changing the pre-drying time from 5 minutes to 30 minutes and photonic sintering after, there is drastic decrease in the resisitivity, reaching to 5.5 times the bulk copper for 15 minutes pre-drying. Although 30 minutes provided more lower resisitivity, it was not chosen due to oxide formation seen in optical pictures.

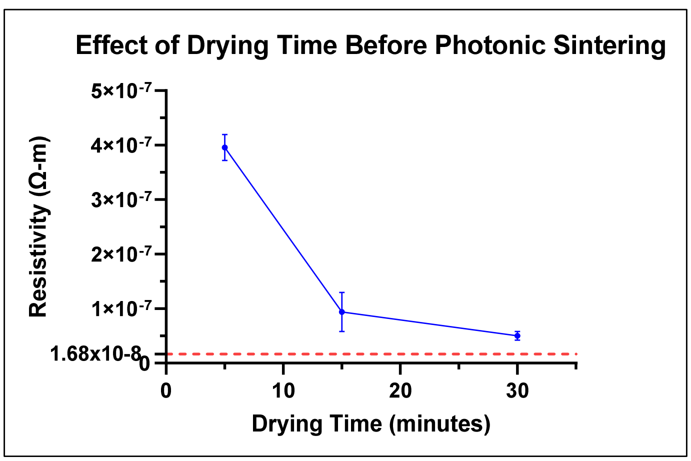
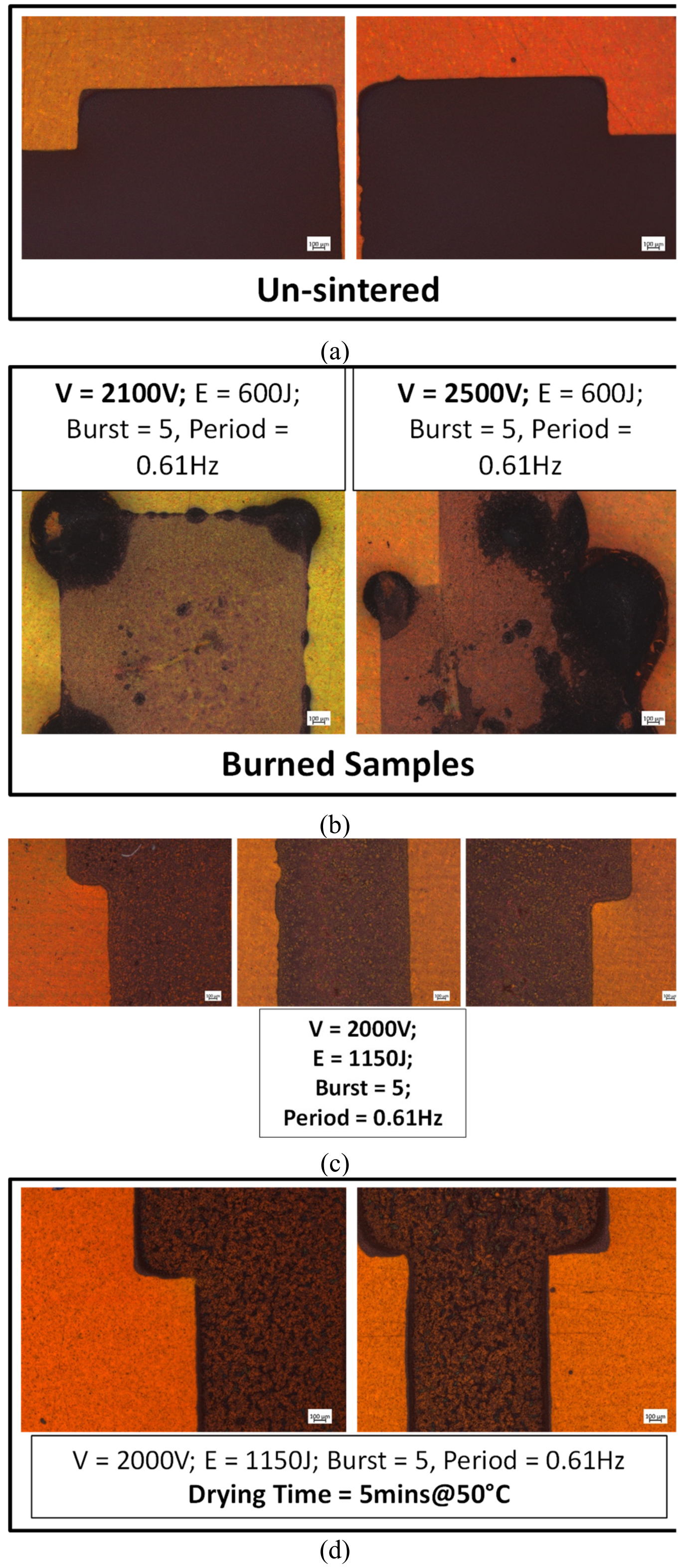
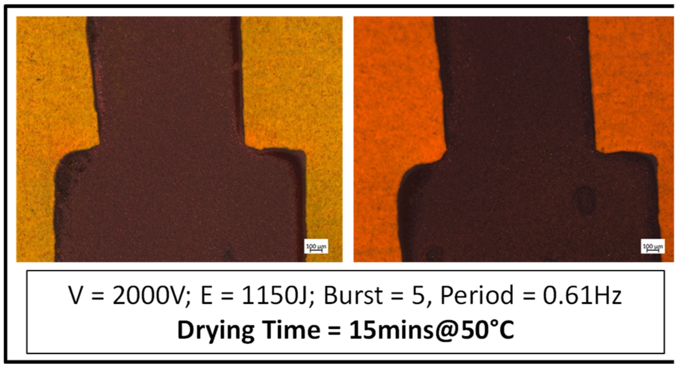


FIGURE 18: EFFECT OF PRE-DRYING TIME ON RESISTIVITY

FIGURE 19 shows some of the optical pictures of unsintered printed copper. Also, burned samples can be seen at voltages higher than 2000 V, with some of the other parameters mentioned in FIGURE 19. Pictures are also shown for the samples when pre-drying is not implemented. With pre-drying, sample definition and microstructure improved for 10 and 15 minutes, however for 30 minutes, oxide formation is seen in the copper, as shown in FIGURE 19.





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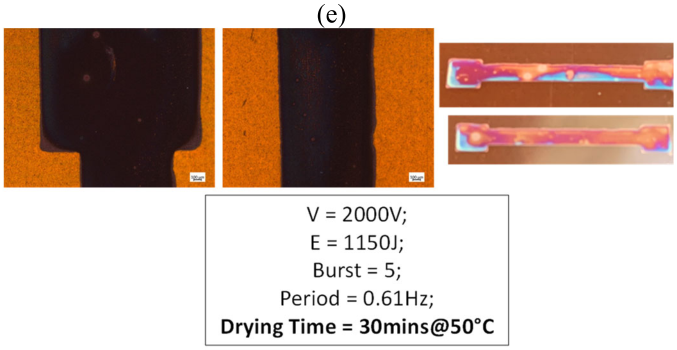

(f)

FIGURE 19: OPTICAL PICTURES OF PRINTED COPPER. (a) UN-SINTERED SAMPLE. (b) BURNED SAMPLES AT HIGH LAMP VOLTAGES. (c) COPPER SAMPLES WITH NO PRE-DRYING. (d) PRE-DRYING TIME OF 5 MINUTES. (e) PRE-DRYING TIME OF 15 MINUTES. (f) PRE-DRYING TIME OF 30 MINUTES, WITH OXIDATION SHOWN ON RIGHT

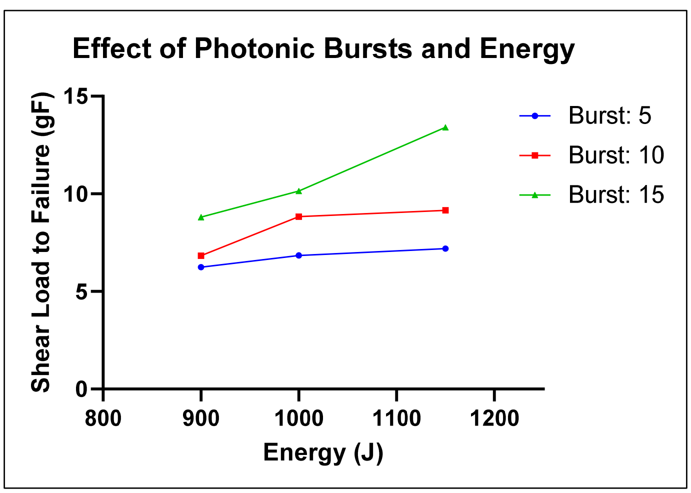

FIGURE 20: EFFECT OF PHOTONIC ENERGY AND NUMBER OF BURSTS ON SHEAR LOAD TO FAILURE (WITHOUT PRE-DRYING)

With electrical properties optimized, attention was given to mechanical shear load to failure, since adhesion is also an important factor in printed electronics. Previously, it was shown how increase in photonic energy had an impact on electrical resistivity. Following the same procedure, shear test is performed using Dage Shear Tester at multiple energy levels and number of bursts. Higher the shear load, higher the adhesion of the printed line to substrate. As expected, shear load to failure increases with increase in energy, but also with the number of bursts, as shown in FIGURE 20. This comes to be opposite as compared with the case of resistivity, where there is degradation with increase in number of bursts.

After the inclusion of pre-drying time, the adhesion increases with increase in time, as shown in FIGURE 21. Ideally, one would want the case where there is least resistivity with maximum shear load to failure. But, this is certainly not the case. Although 30 minutes of pre-drying provided with least resistivity and maximum shear load to failure, it came with a consequence of copper oxidation. Hence, 10 minutes of pre-drying time proved to be the next best case.

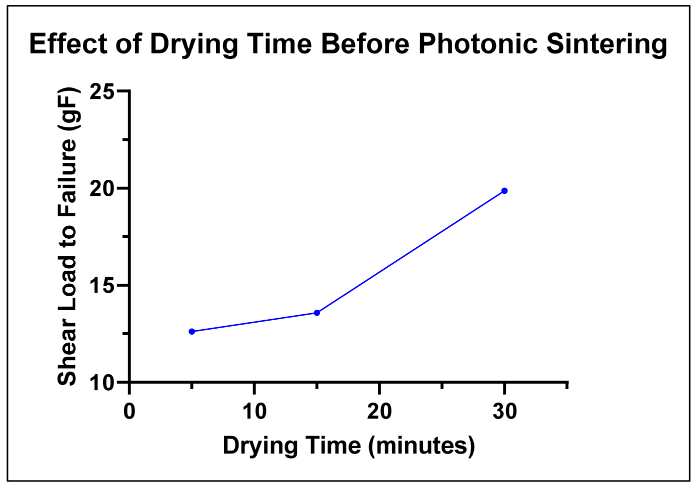

FIGURE 21: EFFECT OF PRE-DRYING TIME ON SHEAR LOAD TO FAILURE

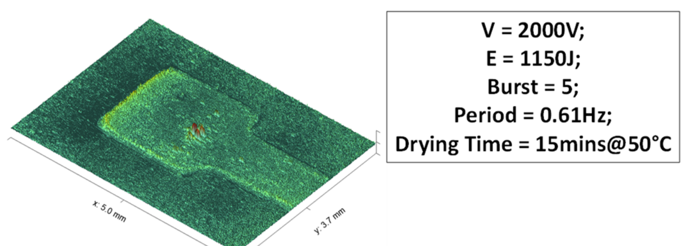

FIGURE 22: WHITE LIGHT INTERFEROMETRY SCAN OF PRINTED COPPER

Considering all the optimization in Inkjet printing and photonic sintering processes, physical characteristics are measured for the printed line. FIGURE 22 shows a White Light Interferometry scan for the line with the parameters listed in the inset. Surface profile along the trace is shown in FIGURE 23, with average height of about 400 nm with one pass of Inkjet printing.

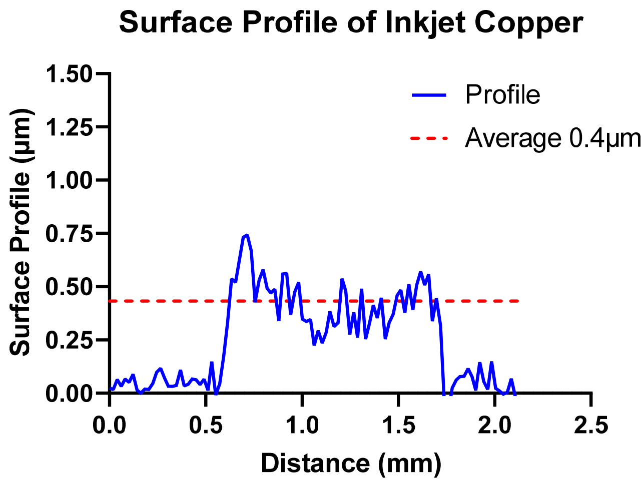

FIGURE 23: SURFACE PROFILE OF PRINTED COPPER

This study is still underway, and more data will be included in the final version of the paper. Some of the elements that are yet to be included are as following:

- *Effect of pulse width on the droplet.*
- *Minimum feature size.*
- *SMD component attachment, and frequency characteristics.*

4. SUMMARY AND CONCLUSIONS

In this article, Inkjet printing is utilized as an additive technology to print traces and mount SMD components on additive printed pads on flexible substrate. Inkjet, compared with other techniqiues, proves to be a very useful tool with much higher throughput and its roll-to-roll ability, In addition to its low-cost equipment setup, it also utilizes the least amount of material during the print. With number of nozzles ranging in hundreds and even thousands, allows a very complex print to be completed in a matter of few minutes. In terms of the material, copper conductive ink is used due to silver being an expensive material. However, with copper, there is an additional step of photonic sintering, that uses an IPL to sinter the material without having much effect on the substrate. Initially, to start-off, droplet characterization was performed with the copper ink to be able to generate clean and round droplets. The characterization involved understanding the effect of some waveform parameters on the jetted droplet. With the waveform selected, the droplet speed was achieved to be at an average of 1.5 m/s, with the drop volume of 38 pL. After the Inkjet printing, photonic sintering was used to study the effect of lamp voltage, energy, and the number of bursts on the electrical and mechanical properties. To further improve the properties, a pre-drying step was included to partially dry the ink before photonic sintering. Implementing these steps provided us with the electrical resistivity of around 5 times the bulk copper, with the shear load to failure in the range of 15-20 gF. This shear load to failure comes to be in general range with Inkjet printing of silver as well [15]. Using the complete optimized process, SMD attachment is peformed using the combination of Inkjet (utilized for traces) and nScrypt direct-write printing (utilized for conductive adhesive deposition for interconnection) for an all-additive manufacturing process of electronic systems. With this study, authors aim to benchmark the complete manufacturing all-additive process of printed features with best electrical and mechanical properties using Inkjet as the printing technology and copper as a conductive material.

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