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Induction Sintering of Silver Nanoparticle Inks on Polyimide Substrates

*Hong Wei Tan**, Nitipon Saengchairat, Guo Liang Goh, Jia An, Chee Kai Chua, and Tuan Tran[^]

H.W. Tan, Prof. C.K. Chua

Engineering Product Development Pillar, Singapore University of Technology and Design, 8 Somapah Road, Singapore 487372, Singapore

*E-mail: hongwei_tan@sutd.edu.sg

N. Saengchairat, G.L. Goh, Dr J. An, and Prof. Tuan Tran

Singapore Centre for 3D Printing, School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Ave Singapore 639798, Singapore

[^]E-mail: ttran@ntu.edu.sg

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Abstract:

Additive manufacturing for electronics is one of the world's fastest growing technologies in recent years. This new and upcoming technology has facilitated many innovative applications and high commercialization of metallic nanoparticle inks. A robust and novel induction sintering technique is proposed to solve the major challenges faced by the current existing sintering techniques in sintering metallic nanoparticle inks. The induction sintering process is a non-contact selective sintering process, in which only electrically conductive pre-sintered printed patterns are heated up by alternating electromagnetic field through eddy current losses. It is also more energy efficient and effective as compared to thermal sintering since the induction sintering process only heats the electrically conductive printed patterns rather than the entire substrate. The electrical resistivity of the induction sintered printed pattern was measured to be $1.98 \times 10^{-8} \Omega \cdot \text{m}$, which is approximately 81% bulk conductivity of silver. This research breakthrough hence has the potential to overcome the challenges faced in additive manufacturing for electronics, in which printed patterns with high electrical conductivity can be fabricated on flexible substrates.

Metallic nanoparticle inks [1] are widely used for fabricating conductive tracks in additive manufacturing [2-6] for electronics [7-10], and they have significantly better electrical conductivity as compared to other types of conductive inks such as metallic-organic decomposition (MOD) [11] and conductive polymer inks [12-13]. These metallic nanoparticles are typically coated with a thermally decomposable, non-conductive organic outer layer, and hence the printed continuous path has limited conductivity. Therefore, an essential post-processing process, also known as the sintering process, is required to improve the electrical conductance within the printed patterns. The sintering process is a critical process as it can determine the final electrical conductivity of the printed patterns. During the sintering process, energy is introduced to the printed patterns via various means to first decompose the encapsulating organic additives and stabilizing agents around the metallic nanoparticles and then aggregate the neighbouring metallic nanoparticles into a single continuous, electrically conductive entity [7].

The thermal sintering technique is still widely used for sintering metallic nanoparticle inks due to its ease of implementation, low costs and higher reliability [14]. However, thermal sintering technique is very time consuming [15-17] and typically requires sintering temperatures between 100°C to 300°C [15, 18] for sintering these metallic nanoparticle inks effectively. Hence, lengthening the fabrication time in additive manufacturing for electronics and limiting the use of temperature-sensitive substrates like polyethylene terephthalate (PET) and polycarbonate (PC). Although there are many other types of sintering techniques (for example, intense pulse light (IPL) sintering [19], laser sintering [20-22] and microwave sintering [23]) to solve the time and temperature issues in thermal sintering, they are technically sophisticated and expensive. These sintering techniques each have their disadvantages too. For instance, the IPL sintering technique may cause undesirable defects in the sintered patterns, deteriorates the materials' properties of some substrates and requires many optimizations [24-25]. The laser

sintering process is also not ideal for sintering large areas with low writing speed, and the laser beam is only able to induce its energy onto the materials with a limited penetration depth [26]. Although the microwave sintering technique can sinter metallic particles in a relatively short time [27-28], the microwave energy only also has a limited penetration depth of a few microns on the printed patterns [23, 29-30] and may not be ideal for sintering thick printed patterns. Therefore, there is a need to bridge the research gap in a search for a novel contactless sintering technique which can allow the sintering process to be done in a low working temperature environment. Also, this novel sintering technique should have fast sintering speed, the ability to produce sintered patterns with high electrical conductivity and minimizing damages to both substrates and printed patterns during the sintering process.

A robust and novel induction sintering technique is proposed to solve the major challenges faced by current existing sintering techniques. In order to utilise the induction heating mechanism for sintering metallic nanoparticle inks, the printed patterns must be pre-sintered first to establish some initial electrical conductivity. Although the thermal sintering process is very time-consuming, it is one of the most reliable and stable sintering processes for sintering metallic nanoparticle inks. Therefore, the thermal sintering process was selected to be used as a pre-sintering process before induction sintering to allow an in-depth understanding of the fundamentals of the induction sintering process.

10 mm diameter circular patterns were printed onto polyimide substrates with one layer of silver nanoparticles at room temperature via the Optomec Aerosol Jet 5x system's pneumatic atomizer. The thermal pre-sintering temperature should ideally be above the silver nanoparticle ink's organic additives' decomposition temperatures if the substrates can allow, to have better electrical conductivity after the sintering process. The electrical conductivity of the thermally pre-sintered printed patterns is highly dependent on the sintering time and sintering temperature in the thermal pre-sintering process. Longer sintering time is required for the printed patterns

to achieve good electrical conductivity at the low sintering temperature, whereas shorter sintering time at high sintering temperature. The printed patterns were first thermally pre-sintered in a convection oven at 120°C for 60 minutes to introduce an initial electrical conductivity within the printed patterns and ensure the bulk of the organic additives present in the printed patterns were thermally decomposed. The electrical resistivity of the pre-sintered printed patterns is measured to be $1.352 \times 10^{-7} \Omega\cdot\text{m}$ (approximately 11.76% bulk conductivity of silver).

The induction sintering process can be done by simply placing the thermally pre-sintered printed patterns over the induction coil, and the induction heater is activated to induce eddy currents within the printed patterns (Figure S1, Supporting Information). It should be noted that the printed patterns should be placed directly on top and close to the induction coil during the induction heating process for effective heating where the magnetic field is most constant and concentrated. The induction heating mechanism is non-contact, fast and selective, and it is widely used for direct heating of electrically conductive materials [31-34]. It is also extremely advantageous as the induction heating mechanism can differentiate conductive materials from non-conductive materials to heat, in which only electrically conductive materials are heated up by the alternating electromagnetic field [31, 33-34]. The alternating electromagnetic field interacts with the conductive workpiece and induces eddy currents within the workpiece. The induced eddy currents hence generate a heating effect within the conductive workpiece through the Joule heating effects (also known as eddy current losses).

The power dissipation of the eddy currents in the circular printed patterns [35], P (W) can be expressed as

$$P = \frac{\pi^2 f^2 B_p^2 r^2}{4\rho} (\pi r^2 t_c), \quad (1)$$

where f is the operating frequency of the induction heater (Hz), B_p is the peak magnetic field (T), t_c is the thickness of the printed pattern (m), r is the radius, and ρ is the resistivity of the material ($\Omega \cdot m$). This equation assumes that the electromagnetic wave fully penetrates the material and there is no skin effect during the induction heating process at the frequency of magnetization. It should be noted that the printed pattern is placed within the induction coil during the induction sintering process for effective heating where the magnetic field is concentrated. According to equation 1, the power dissipation of the eddy currents in the printed patterns is inversely proportional to the electrical resistivity of the pre-sintered samples. The printed patterns with lower electrical resistivity can generate higher power dissipations during the induction sintering process for sintering, and hence generating higher temperatures over the same induction sintering time. Therefore, it can be concluded that the electrical resistivity of the pre-sintered printed patterns does affect power dissipation within the printed patterns during the induction sintering process.

As the eddy currents flow through the electrically conductive pre-sintered printed patterns, the Joule's heating effect can sinter at least most (if not all) of the deposited silver nanoparticles. The generated heat will also be conducted to the polyimide substrate due to strong temperature gradients. Thus, the polyimide substrate acted as a heat sink to cool the printed pattern during the induction sintering process as the polyimide substrate was not directly heated by electromagnetic induction [19]. In order to restrict the heating effects only to the printed patterns during the induction sintering process rather than the substrate, the duration of the induction sintering process must be shorter than the time taken for the printed pattern and the substrate to reach thermal equilibrium. Therefore, there is a need to use a multiple short-pulse induction sintering technique rather than an extended continuous single pulse induction sintering technique to ensure minimal or no substrate damages during the induction sintering process. The exact values of the induction sintering parameters (e.g. pulse heating on-time,

pulse heating off-time, number of cycles, induction power) must be chosen carefully to achieve both fast sintering and excellent electrical properties after sintering, and at the same time to reduce thermal stress and prevent substrate damages. Hence, the operating parameters of the induction sintering process are optimized with a pulse heating system in this section to achieve uniform induction sintering and good electrical conductivity within the entire printed patterns.

A combination of low induction power and short heating pulses were chosen to prevent overheating and substrate damages. A higher number of cycles of pulse heating can be utilized to further improve the final electrical properties of the induction sintered printed patterns while maintaining the temperature of the printed patterns under the glass transition temperature of the substrate during the induction sintering process to prevent substrate damage. The induction sintering process was optimised with a 50 cycles short-pulse induction sintering routine at 10.5% induction power, in which one cycle consists of 0.75s on-time and 2.25s off-time. An infrared camera was used to capture the thermograms and maximum temperatures attained within the printed patterns during the entire induction sintering process. However, the infrared camera can only be calibrated accurately within the temperature range from 125 – 300 °C. Therefore, any information obtained outside the calibrated temperature range were ignored due to inaccuracies.

Figure 1a shows the thermograms of the pre-sintered printed pattern during a pulsed induction sintering process. The printed pattern tends to get heated up at the rims first during the induction sintering process due to the higher concentrations of magnetic flux around the circumference of the induction coil. The heat generated from the eddy current losses at the rims of printed patterns was then conducted towards the center of the printed patterns. The entire printed pattern was heated uniformly by thermal conductance during the induction sintering process as silver is an excellent thermal conductor. The induction sintering process approximately took 5 cycles ($t = 12.75s$) to uniformly heat the entire printed pattern. From **Figure 1b**, it can be observed

that the maximum temperature of the printed pattern was maintained under 300°C during the induction sintering process to prevent deformation and deterioration of the polyimide substrate (glass transition temperature, $T_g = 385^\circ\text{C}$ [30]). No visible defect and damages were also observed on both printed patterns and substrates after the induction sintering process. Although electromagnetic induction does not directly heat the electrically non-conductive polyimide substrate, heat can be still conducted from the heated printed patterns and cause severe damages to the substrate. A multiple short-pulse induction sintering technique is proved to be more effective than long continuous single pulse induction sintering technique as it can allow the substrate to cool down between each heating interval and thus, significantly reduce temperature load of the substrate to prevent damage. At the same time, the multiple short-pulse induction sintering technique can also help to prevent excessive vaporization of the organic additives entrapped within the printed patterns and allow enough time for these vapors to escape to prevent surface morphologies defects like swellings and ruptures within the printed patterns.

From **Figure 1b**, it also can be observed that the maximum temperature of the printed pattern was going on an upward trend as the number of pulses increase. This is because the electrical resistivity of the printed pattern is decreasing with every pulse-heating cycle, and thus power dissipation within the printed patterns due to eddy current losses is increasing with every pulse-heating cycle. However, the rate of increase slows down as the number of cycles increases, due to the electrical resistivity of the printed patterns is approaching closer to the bulk resistivity of silver. The bulk resistivity of silver is the lowest electrical resistivity ideally that printed patterns can obtain. The average electrical resistivity of the induction sintered printed patterns were measured to be $1.98 \times 10^{-8} \Omega\cdot\text{m}$, which is approximately 81% bulk conductivity of silver. Printed patterns were also thermally sintered at 200°C for 240 minutes for better comparison, and their electrical resistivity was measured to be $3.705 \times 10^{-8} \Omega\cdot\text{m}$ (approximately 42.91% bulk

conductivity of silver). The induction sintering technique can achieve approximately twice better electrical conductivity than the thermal sintering technique at a fraction of the time taken.

Figure 1. a) Thermograms of the printed pattern during induction sintering at different time; and b) temperature profile (maximum temperature within circle c1) of the printed pattern against time.

Field emission scanning electron microscopy (FESEM) images in **Figure 2** show the surface morphologies of the sintered printed patterns under the various sintering conditions. **Figure 2a** shows discrete unsintered silver nanoparticles within the printed patterns before any sintering process. The unsintered nanoparticles are electrically non-conductive as there is a layer of thermally decomposable organic shell enveloping each nanoparticle to prevent electron flow.

The induction sintering process can be summarised as follows. Thermally decomposable, non-conductive outer layers of the silver nanoparticles in the printed patterns are first decomposed thermally during the thermal pre-sintering process to form contacting points with neighboring particles (see **Figure 2b**). Triggered by diffusive Ostwald ripening phenomenon [36-37] and the need to reduce total interfacial energy [38], these contacting nanoparticles can form sinter necks among themselves at elevated temperature. Further absorption of thermal energy then drives the aggregation process of the nanoparticles to form sinter necks among neighboring particles by releasing high surface energies to achieve the state of thermodynamic equilibrium. At this stage, the microstructures are still highly porous, and the electrical conductivity is not optimised yet. The printed patterns are partially sintered with some initial electrical conductivity at the end of the pre-sintering step, and they now can be sintered by the induction sintering technique.

During the induction sintering process, eddy currents are induced and generated Joule heating effect within the printed patterns. The generated heat is the main driving force that promotes further grain growth and densification of the silver nanoparticles. Large distinct dense microstructure with well-defined grain boundaries was observed in the induction sintered printed pattern (see **Figure 2d**). Denser microstructures and larger grain sizes facilitate electrons movements by widening electrons flow paths and increasing the number of percolating pathways [39]. Hence, explaining the good electrical conductivity achieved in induction sintered printed patterns. Also, better mechanical properties are commonly associated

with denser microstructures. **Figure 2c** shows the surface morphology of the printed pattern that is thermally sintered at 200°C for 240 minutes. It can be observed that the thermally sintered printed pattern has high porosity and directly affects its electrical resistivity. **Figure 2e** and **Figure 2f** show the cross-sectional views of the printed patterns under different sintering conditions. The cross-sectional view of the induction sintered printed pattern was observed to be denser than the thermally sintered printed pattern.

Table 1. Electrical resistivity of the printed patterns under the induction and thermal sintering processes (three similar printed patterns were sintered under each unique sintering condition).

Sintering Technique	Sintering Parameters	Total Time Taken (minutes)	Average Electrical Resistivity ($\Omega\cdot\text{m}$)	Average Conductivity (% of bulk silver conductivity)	Average Percent Deviation (%)
Induction Sintering	Pre-Sintered at 120 °C for 60 minutes	60	1.98×10^{-8}	81.07	7.87
	50 cycles On time: 0.75s Off-time: 2.25s	2.5			
Thermal Sintering	200 °C 240 minutes	240	3.97×10^{-8}	40.37	7.23

Figure 2. FESEM images of the silver nanoparticle ink printed patterns on polyimide substrates under various conditions: a) unsintered, b) thermally pre-sintered at 120°C for 60 minutes, c) thermally sintered at 200°C for 240 minutes, d) induction sintered, e) cross-sectional view of thermally sintered at 200°C for 240 minutes, and f) cross-sectional view of induction sintered.

It is difficult to describe the silver nanoparticles quantitatively with only FESEM images as they are irregular and not spherical. These FESEM images were post-processed with an image analysis software, MIPAR to identify and measure the grain size and grain distributions. Equivalent circular area diameter was used to describe the grain size of the nanoparticles in FESEM images and used them as a comparison. Equivalent circular area diameter is defined as the circle with the equivalent circular area diameter that has the same projected area as the particle. **Figure 3a** and **Figure 3b** show the measured features and size distributions of silver nanoparticles for image processing in FESEM of the thermally sintered printed pattern at 200°C for 240 minutes and induction sintered printed pattern respectively. The mean grain sizes of silver nanoparticles under the thermal and induction sintering processes were 0.253 μm and 0.334 μm , respectively. Better surface morphology was observed in the induction sintering process as compared to the thermal sintering process, and bigger grain sizes can be achieved in a shorter sintering time. From the grain distributions, it can be concluded that the induction sintering process can produce larger grains as compared to thermal sintering.

The electrical resistivity of the 50 cycles short-pulse induction sintered printed pattern was measured to be approximately 81% bulk conductivity of silver. As compared to thermal sintering, the printed pattern sintered by induction sintering proves to have denser microstructures and higher electrical conductivity. It is also more effective and energy-efficient as compared to thermal sintering since the induction sintering process only heats the electrically conductive printed patterns rather than the entire substrate. From the above studies, it can be concluded that various combinations of induction power, pulse heating on-time, pulse heating off-time, the number of cycles are possible to allow different permutations for process optimizations. However, the parameters of the multiple short-pulse induction sintering technique ideally should be varied constantly for every pulse-heating cycle for optimal process optimization. This is because the material and geometries properties of the printed pattern

changes with every pulse-heating cycle and may lead to variations of thermal conduction and power dissipations within the printed pattern due to induction sintering. For instance, the densification of the silver nanoparticles is improving, and the organic additives content within the printed patterns are decreasing with every pulse-heating cycle, and hence leading to increasing thermal conductivity and electrical conductivity. The power dissipation within the printed patterns due to eddy current losses also increases as the electrical conductivity of the printed patterns increases.

Figure 3. Measured features and size distributions (equivalent diameter) of silver nanoparticles in FESEM images: a) thermally sintered printed pattern at 200°C for 240 minutes and b) induction sintered printed pattern.

In conclusion, the induction sintering technique proves to be a novel and effective sintering technique for sintering silver nanoparticle inks and this sintering technique can be extended for sintering other types of electrically conductive metallic nanoparticle inks. The induction sintering process was optimised with a 50 cycles pulse-heating routine to prevent the polyimide

substrate from damaging by maintaining the heating temperature under 300°C. The electrical resistivity of the induction sintered printed pattern was measured to be approximately 81% bulk conductivity of silver. As compared to thermal sintering, the printed pattern sintered by induction sintering proves to have denser microstructures and higher electrical conductivity. The induction sintering process also proves to be more effective and energy-efficient as compared to thermal sintering since it only heats the electrically conductive printed patterns rather than the entire substrate. However, heat still can be conducted from the heated printed patterns and cause serious damages to the substrate if the induction process is not optimised. Other sintering techniques such as IR and IPL sintering techniques may be used for pre-sintering of the printed patterns to significantly reduce the pre-sintering time needed and allow the use of temperature-sensitive substrates (Table S1, Supporting Information). Induction sintering may also be useful for sintering embedded electronics and thus promoting innovations in additive manufacturing for electronics. Induction heater with a higher power and higher operating frequency should also be considered for more effective sintering of smaller, narrower and thinner printed patterns. Further work includes induction sintering of metallic nanoparticle inks on temperature-sensitive substrates.

Experimental Section

Ink Preparation: Silver nanoparticle ink of 25 – 40% weight loading (UTDAg40TE, UT Dots Inc.), which has a recommended sintering temperature between 120 – 140 °C, was used in this experimental study [40]. The silver nanoparticle ink was well-shaken with a laboratory orbital shaker for 10 minutes to achieve homogeneity.

Ink Deposition: 75- μ m thick polyimide substrates are thoroughly cleaned to remove any undesirable organic substances and dust from their surfaces. Their surfaces were first wiped clean using ethanol, and after which they were sonicated in a distilled water bath for 15 minutes

to rinse the contaminated ethanol off their surfaces. The polyimide substrates were then air-dried before printing. 10 mm diameter circular patterns were printed onto polyimide substrates with one layer of silver nanoparticles (Figure S2, Supporting Information) at room temperature via the Optomec Aerosol Jet 5x system's pneumatic atomizer. These as-deposited printed patterns were dried in ambient conditions before sintering.

Sintering Processes: There were significant time intervals between the printing and sintering processes as these systems were not integrated into one machine. However, these long time intervals were not critical to the sintering process, and the sintering characteristics as only non-volatile materials remained in the dried printed patterns [24]. Three similar printed patterns were sintered under each unique sintering condition. Thermal pre-sintering was done in a convection oven (Venticell 55, MMM Group) at 120°C for 60 minutes. Thermal sintering was also done in the same convection oven at 200°C for 240 minutes. Induction sintering was carried out by placing the printed patterns above the induction coil (1 cm diameter induction coil of 4 turns). The induction heater (DW-UHK-6KW-I, OT-QD Shuimu Induction Equipment Co. Ltd.) has a maximum oscillating power of 6.6 kW at a frequency ranging from 1.1 MHz – 2.0 MHz. During the actual induction sintering process, the induction heater was measured to be operating at 825 kHz and at 10.5% of the maximum operating oscillating power of 6.6 kW. An infrared thermographic camera with 150 – 300°C calibration range (ImageIR 8300, InfraTec) was used to capture the thermograms of the printed pattern during the induction sintering process. The emissivity of the printed patterns was calibrated in the infrared camera accordingly.

Characterizations: Field emission scanning electron microscopy (FESEM) images were taken using the Schottky field emission scanning electron microscope (JSM-7600F, JEOL Ltd). These images were post-processed with an image analysis software, MIPAR to identify and measure the grain size distributions. The thickness of the printed patterns was also measured with the field emission scanning electron microscope. The electrical resistivity of the printed patterns

was measured by a four-point probe station (Pro4-440N resistivity test system, Signatone) at random positions within circle C1.

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