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Electric Symphony: Unveiling the Potential of Reduced-Temperature Cured Conductive Ink

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ABSTRACT

This study evaluates the performance of low-temperature-cured graphene nanoplatelets (GNP) and silver nanoparticles (Ag) hybrid conductive ink. The ink's electrical conductivity characteristics are assessed for applications in flexible and printed electronics. Results show that the GNP/Ag hybrid conductive ink achieves high electrical conductivity as compared to traditional high-temperature-cured inks, with robust adhesion to diverse substrates. The low-temperature curing process mitigates thermal damage to temperature-sensitive substrates, expanding the ink's potential applications. These findings contribute to the development of efficient and versatile manufacturing processes for advanced electronic devices, positioning the GNP/Ag hybrid conductive ink as a promising material for emerging technologies.

1. Introduction

Conductive paste is being used in mass manufacturing to meet the ongoing need for electronic applications such as flexible electronics and power electronics, which are widely used in high-speed trains, and light-emitting diodes (LED). However, there are currently some problems with the conductive ink that is on the market, including poor printing quality, excessive electrical resistance, and weak mechanical strength [1]. This phenomenon prompts in-depth investigation into the production of conductive paste, especially in regard to increasing thermal conductivity and mechanical performance [2]. Conductive paste, as the name suggests, has a wide variety of usage in electronics industries. The unique characteristics make it an interesting field to be further explored as it has attracted many researchers.

The manufacture of internal components for a variety of electronic devices requires the use of die-attach adhesives. The difficulties with connecting semiconductors and circuit boards are growing as electronic device sizes are shrinking. High electrical and thermal conductivities, suitable

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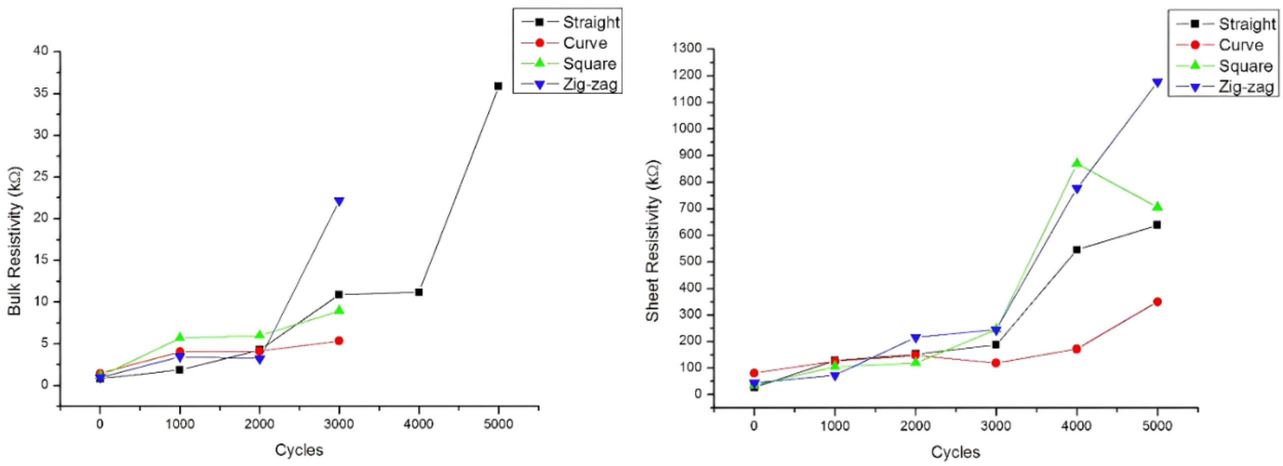
mechanical properties to avoid and support thermo-mechanical stresses, sufficient adhesion between the die and the substrate (to prevent the die from detaching from the substrate), and a high melting point to ensure reliability in harsh environments are required in order to ensure proper operation and wear resistance [3].

Due to graphene's high thermal conductivity, (5300 W/ mK), sintered hybrid conductive pastes of metal-based materials like silver-graphene or copper-graphene have become very popular. It has outstanding mechanical rigidity and electrical transport characteristics [2-4]

The majority of the pure conductive materials on the market today are unable to achieve high thermal conductivity [5]. Thus, researchers developed a new formulation that consists of graphene nano-platelet (GNP) which is a carbon-based material combined with metal-based materials which in this study is silver (Ag). The results of combining these two materials produced an enhanced ink property, both electrically and mechanically. The hybrid paste between GNP and Ag has greater properties as compared to its predecessors, the carbon-based conductive ink.

Low-temperature curing for GNP/Ag conductive ink is a ground-breaking technique that enables the creation of flexible and versatile electronics. By combining graphene nanoplatelets (GNP) and silver (Ag) particles, this method allows for conductivity at lower temperatures and expanding the possibilities for printed electronics on various substrates. It offers advantages such as reduced thermal damage, energy efficiency, and scalability in manufacturing, revolutionizing industries like wearables and IoT devices. Steps in fabricating a conductive ink require it to be cured thermally. A silver particle ink that exhibits a cure temperature as low as 80 °C was prepared and characterized [6,7]. In this study, a few parameters were set to determine which curing temperature is the best to obtain the optimum parameters for the ink to be as efficient as possible in terms of its electrical resistivity and mechanical properties.

In a study conducted by Norhisham, the behaviour of graphene nanoplatelets under stress testing was investigated to observe how conductive ink behaves under fatigue tests. The objective of the study was to investigate the behaviour of a conductive ink thin film when subjected to cyclic bending, specifically up to 5000 cycles. The study aimed to gather data for the development of electrical packaging. It focused on analysing the changes in surface roughness, sheet resistivity, and bulk resistivity of the thin films as the cyclic bending cycles increase. Additionally, the paper aimed to determine the durability of the graphene nanoplatelets (GNP) thin film under high cycle stress by observing its performance. According to the study, as printed ink cracks over time, it loses its adhesion thus increasing the resistance [8]. Figure 1 shows the results of resistance increases over time when subjected to cyclic stress.



(a) (b)

Fig. 1. Resistivity over cyclic bending (a) bulk resistivity (b) sheet resistivity

2. Methodology

The hybrid ink consists of Graphene Nanoplatelets (GNP) with particle sizes of 25 µm with a surface area of 120 to 150 m^2/g, silver flakes (10µm, ≥99.9 % trace metals basis), and silver acetate (99.99% trace metals basis). Ethanol (denatured ethanol 9 9%), 1-Butanol (99.9 %), and also Terpineol (~65 %α, ~20 %γ, ~10 %β) was also used as the organic solvent. An ultrasonic bath was utilized as the main instrument to mix the whole materials (Table 1).

Table 1
List of materials/ equipment used in the process of fabricating conductive ink

Name	Materials/ equipment specifications
Graphene nano-platelet (GNP)	25µm with a surface area of 120 to 150 m2/g
Silver Flakes (SF)	10µm, ≥99.9% trace metals basis
Silver Acetate (SA)	99.99% trace metals basis
Ethanol	Denatured ethanol 99%
Butanol	1-Butanol (99.9%)
Terpineol	~65%α, ~20%γ, ~10%β

2.1 Preparation of Gnp/Ag Powder in Ethanol

The 0.005 g of GNP was mixed with 0.429 g of silver flakes (SF) and 0.042 g of silver acetate (SA) in 5 ml of ethanol. Before mixing SF and SA, GNP powder was sonicated in ethanol for 10 minutes and the sonication process was continued for another hour after putting SF into the solution. SA was put into the mixture and sonicated for an additional hour. The mixture was heated at 70 °C and constant stirring was applied using a magnetic stirrer at 200 rpm. This process was continued until the ethanol was evaporated. The dried solution was cured in the oven for 1 hour at 250 °C and the cured solution was pounded until a fine powder texture was achieved (Figure 2).

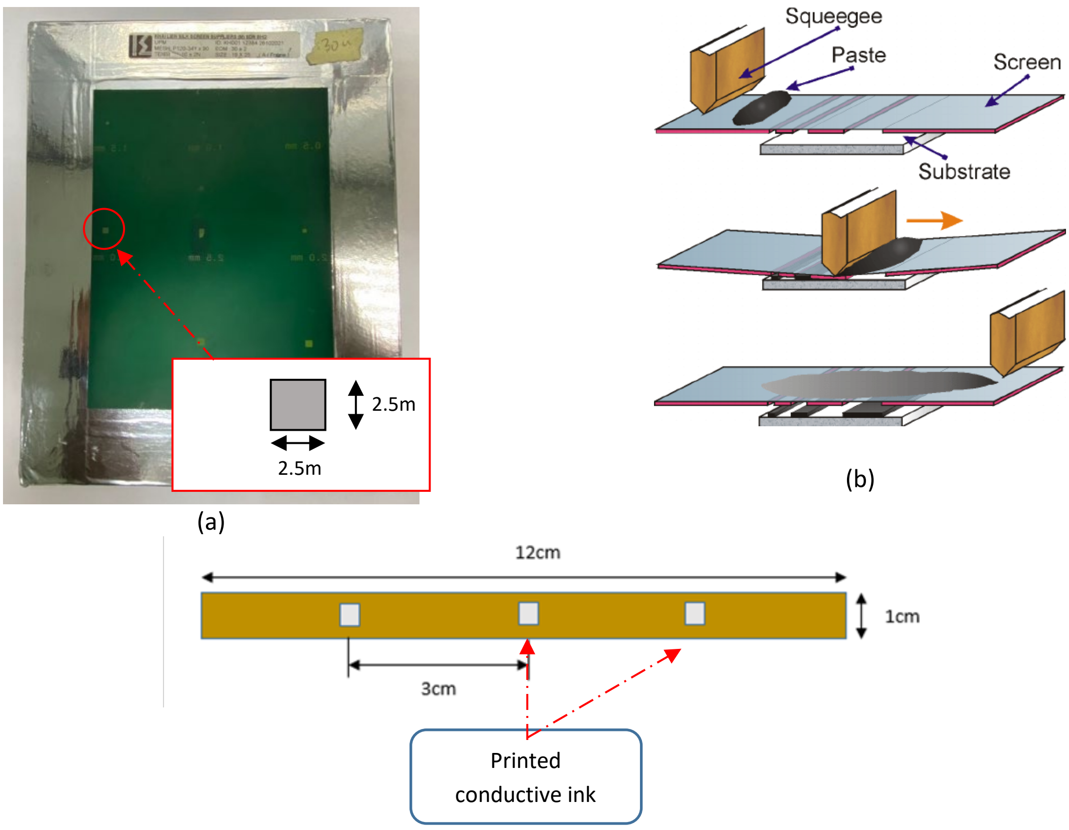
Fig. 2. powdered texture achieves after being treated in UF55 universal oven for 1 hour at 250 °C

2.2 Conductive Paste Fabrication with Optimal Formulation

The hybrid powder was weighed to determine the ratio of terpineol and butanol that need to be added to form a paste. Every 0.52 g of powder required 0.058 g of butanol (approximately 3 drops) and 0.084 g of terpineol (approximately 3 drops). The mixture was blended using a thinky mixer for 3 minutes. This produced consistent pastes, which were printed on the substrate.

2.3 Test Sample Preparation using Different Thermal Curing Temperature

The ink was printed onto a copper substrate with a dimension of 12 cm x 1 cm. Screen printing technique was used using a silkscreen with printed patterns and were traced using a squeegee. The dimension of the printed ink was 2.5 mm x 2.5 mm and 3 cm distance apart for each point as illustrated in Figure 3 below.



(c)

Fig. 3. Printing procedure of conductive ink onto copper substrate using silkscreen and squeegee (a) silkscreen with printed images to print conductive ink onto copper substrate (b) Printing technique using silkscreen and squeegee to print conductive ink on copper substrate (c) Printed conductive ink on 12cm x 1cm copper substrate with 3cm distance apart from each other

2.4 Testing Procedure
2.4.1 Curing method for stretchable conductive ink

The test samples were then thermally cured inside a UF55 industrial oven with a variation of temperature. 100 °C, 120 °C, and 140 °C were the selected temperatures and the samples were cured for 30 minutes to study the effects of thermal curing temperature on electrical resistivity. The structure of the paste was observed using a SEM and light microscope. The electrical performance of each paste was also measured using a multi-meter in terms of resistance measurement.

The UF55 Universal Oven is a highly reliable and versatile oven used in laboratory and industrial settings. Its robust stainless-steel construction ensures durability and easy maintenance. With precise temperature control capabilities, it offers a wide temperature range and maintains temperature accuracy and stability within the oven chamber. The UF55 Universal Oven is equipped with a highly efficient heating system, distributing heat evenly for consistent temperature conditions. Safety features such as over-temperature protection and door safety switches enhance user safety. The oven's user-friendly control interface allows for easy programming and monitoring of temperature parameters. Widely used in research, pharmaceuticals, food processing, and other industries, the UF55 Universal Oven serves various applications including drying, sterilization, curing, and heat treatment processes, making it a reliable choice for diverse thermal applications. Test samples are cured individually inside UF55 as per the parameters set.

2.4.2 Sample scanning using Scanning Electron Microscope

The cross-section of the printed sample was examined and evaluated using SEM. To prevent charge accumulation, the printed sample was coated using the auto fine machine [9]. In order to extract information from a material at the nanoscale, SEM uses electron beams. Backscattered (BSE) and secondary electron (SE), which produce a grayscale image of the material at every high magnification, are the major signals that are detected. It uses a beam of electrons to create high-resolution images of a sample's surface. It scans the sample by moving the beam in a raster pattern and collects signals such as secondary electrons and backscattered electrons. These signals provide information about the sample's topography and composition. SEM imaging is widely used in various scientific fields for detailed surface analysis. A JEOL JSM-5050PLUS/LV emission scanning electron microscope (SEM) at 20 kV of accelerating voltage was used in this process.

2.4.3 Resistance measurement using a multimeter

Another instrument used was a multimeter to measure numerous electrical properties. It is a useful tool for both professionals and amateurs because it incorporates multiple measurement capabilities into a single instrument. A multimeter allows users to measure voltage (AC and DC), current, resistance, continuity, capacitance, and occasionally frequency thanks to its digital display and selection dial or buttons. Resistance of conductive ink was taken at the initial state before the cyclic test starts at 1000 cycles, 3000 cycles, and 5000 cycles of bending test.

2.4.4 Cyclic bending test using a bending test rig

The performance of the conductive ink was evaluated through a fatigue test, which was conducted using a bending test rig to further assess its electrical capacity (Figure 4). The test rig adheres to the flexibility and stretchability testing guidelines for printed electronics detailed in IPC 9204 of the Association Connecting Electronics Industries (ACEI)[10]. A test sample was affixed to both ends of the rig, one moving and one stationary. The rig is equipped with a counter that records the number of cycles completed. The test utilized a DC power supply of approximately 7VDC. The rig was set to 55 rpm, and a total of 5000 cycles were performed. The resistance of the sample was measured at the initial run, 1000 cycles, 3000 cycles, and 5000 cycles. Basically, the loading rate is approximate to 05 mm/min and the displacement is around 0.5N, respectively.

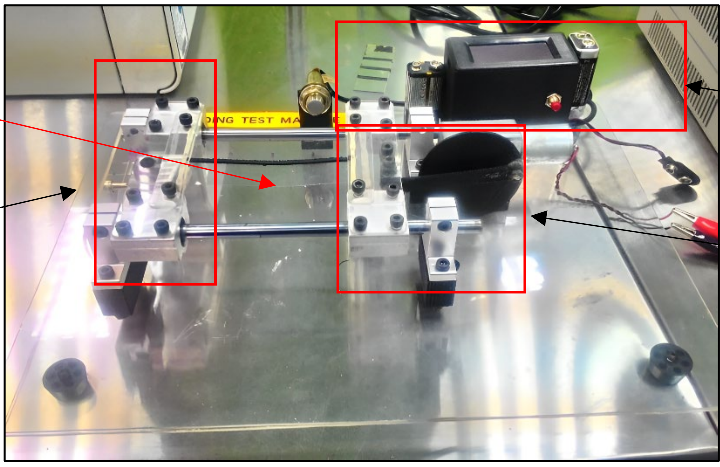


Fig. 4. Bending test rig

3. Results and Discussions

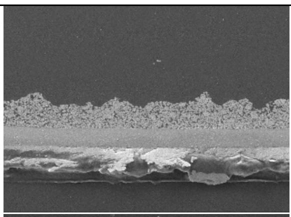
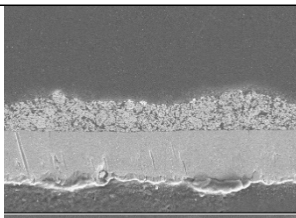
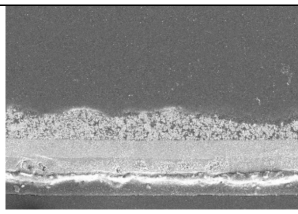
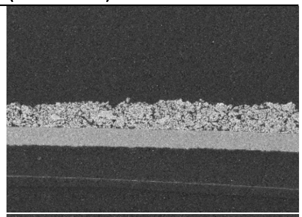
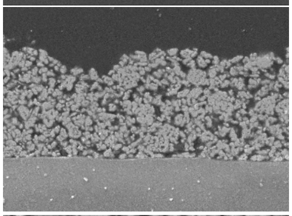
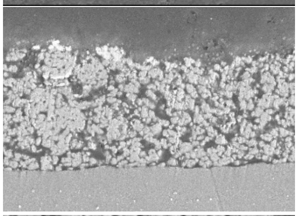
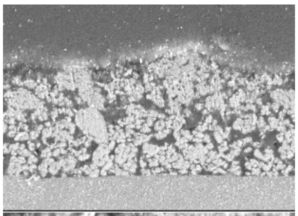
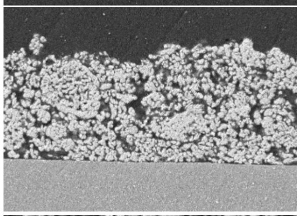
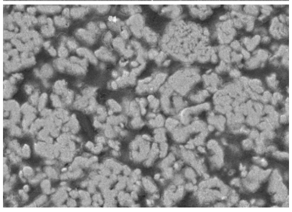
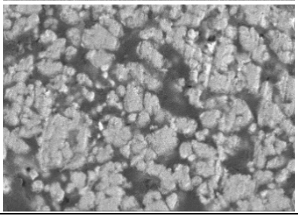
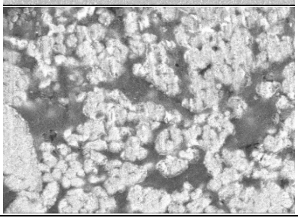
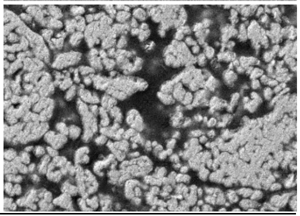
3.1 Morphological Analysis

The structure of the conductive ink reveals variations in surface shapes and characters among the samples. The finest surface details were observed in the sample cured at 140 °C as compared to the others. However, this distinction is purely aesthetic, as the particles in each sample were assessed using a SEM to obtain a more detailed view of the morphological structure. The sample cured at 120 °C displayed a rough and bubbly surface, as shown in Table 2 below, due to the boiling temperature of butanol being at 117 °C. This boiling action leads to alterations in the physical and structural properties of the paste after curing [2].

The curing process is necessary to enhance the bonding between the particles [11]. The resistivity of GNP/Ag conductive ink can be lowered when cured at a lower temperature, such as 100 °C, compared to a higher temperature of 140 °C. This phenomenon can be attributed to several factors. At lower curing temperatures, the conductive particles in the ink have more time to redistribute, allowing them to form a more uniform and interconnected conductive network. This improved interparticle contact leads to lower resistivity. Additionally, higher curing temperatures can promote particle agglomeration, where the conductive particles cluster together, hindering effective electrical conductivity and resulting in higher resistivity [11,12]. Moreover, excessive heat during curing can cause ink degradation, altering the properties of the conductive particles or binder material, which can further increase resistivity. The specific behaviour can depend on factors such as ink composition, formulation, curing time, and substrate effects.

The SEM was utilized to observe the cross-section of each sample in depth. Scanning electron microscopy (SEM) examined the surface morphology of silver patterns printed with butanol and terpineol-based silver hybrid inks. The findings provided insight into the effects of thermal temperature selection on the quality, homogeneity, and surface characteristics of the printed structures. The samples were observed under 200x, 800x, and 2000x of magnifications.

Table 1
SEM comparison between samples cured at 100 ˚C, 120 ˚C, 140 ˚C and 250 ˚C

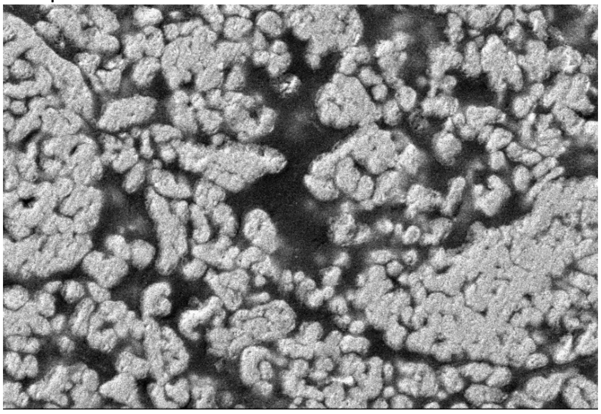
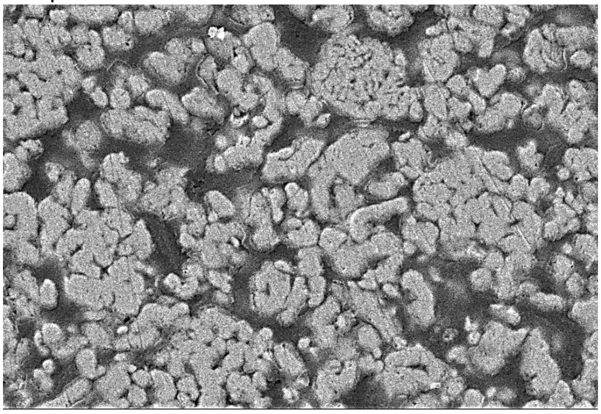
	Sample cured at 100˚C	Sample cured at 120˚C	Sample cured at 140˚C	Sample cured at 250˚C (baseline)
200x zoom				
800x zoom				
2000x zoom				

At 2000x magnification, the particles of the sample cured at 100 °C showed a better bond between each other, as suggested by Table 3 below. This result shows a well-interconnected structure that produces effective conductivity. However, it is worth noting that the surface of the printed ink appears to be rough at 200x magnification, which may contribute to how the GNPs disperse [13,14].

In the case of the sample cured at 120 °C, remnants of the solvent can be observed on the top layer of the printed ink. This suggests that the solvent is still present in the conductive ink even after the curing process. Inadequate binding between the Graphene and Silver resulted in poor electrical conductivity. The presence of even small traces of solvent in GNP/Ag conductive ink can lead to an increase in resistivity in the cured ink. Several factors can contribute to this occurrence. Firstly, solvents (butanol and terpineol) used in the ink formulation can act as insulators, impeding the electrical conductivity of the conductive particles.

When comparing the sample cured at 100 °C to that cured at 250 °C, GNP and Ag particles are still scattered and do not form a uniform bond. The sample cured at 100 °C produces relatively good conductivity when compared to the samples cured at 120 °C and 140 °C. For the sample cured at 250 °C, it is revealed that the ink exhibits a well-dispersed distribution of metallic particles within the paste pattern. The particles appear to maintain a uniform morphology, which is crucial for achieving desirable electrical conductivity. The image depicts finer morphological characteristics of the printed ink at 2000x magnification, providing a comprehensive analysis of particle distribution and interactions between specific particles. Metallic atoms self-organize into an intricate web of interconnected structures. A high degree of particle dispersion indicates that the composition of the base material allows the particles to maintain an ideal shape, promoting effective electrical conduction.

Table 3
Comparison between a sample cured at 250 °C (baseline) and a sample cured at 100 °C

Sample cured at 250°C	Sample cured at 100°C
	

3.2 Electrical Characterization

To comprehensively understand the effects of temperature on electrical conductivity, the study employed a range of temperatures in relation to electrical resistance [15]. The initial thermal curing temperature was set at 250 °C, and various additional curing temperatures were selected and investigated to observe their impact on the electrical conductivity of the samples. The results were meticulously recorded and compiled for further analysis. Interestingly, each curing temperature parameter exhibited distinct behaviour when compared to the baseline. The electrical conductivity of the samples displayed variations at different temperatures, indicating that the relationship between temperature and electrical resistance is not linear. This finding underscores the significance of exploring a wider range of temperatures to gain a comprehensive understanding of the intricate effects on the material's electrical properties. Figure 5 in the research is essential for illustrating the observed tendencies. It effectively demonstrates how electrical conductivity varies across the range of curing temperatures, enabling the identification of trends and critical points or transitions in the material's behaviour. The experimental data opens new avenues for inquiry and analysis, as it reveals a variety of trends for different curing temperatures. In-depth research into the mechanisms underlying these peculiar behaviours may lead to the development of innovative materials with tailored electrical characteristics for specific applications.

Resistance measurements were taken using a multi-meter to assess the electrical conductivity of the hybrid ink cured at low thermal temperatures. Readings were recorded in the initial state and compared to the baseline. The resistance at 250 °C is measured at 2.03 Ω, and after calculating the resistivity using a bulk resistance formula, the value is $0.83\text{x}10^{-4}$ Ω/sq, which is significantly lower than the other readings[16]. This phenomenon is attributed to the sintering, wherein the reduction of total surface energy drives the solid-state material process[5]. During this process, solvents were decomposed and removed from the die-attach, and small particles like nano-sized ones can provide an advantage due to their high surface energy.

The significant drop in resistivity from 140 °C to 250 °C in Figure 5 can be related to Table 4, which illustrates the cross-section of the conductive ink. In the image of the sample cured at 140 °C, it is noticeable that the particles are more widely spaced compared to the sample cured at 250 °C. This difference is attributed to the boiling factor, which occurs when the solvent reaches its boiling temperature. The boiling action agitates the particles, reducing interaction between them and resulting in higher resistivity. Conversely, in the sample cured at 250 °C, the sintering effects take place and promoting strong bonding between the particles and resulting in much lower resistivity.

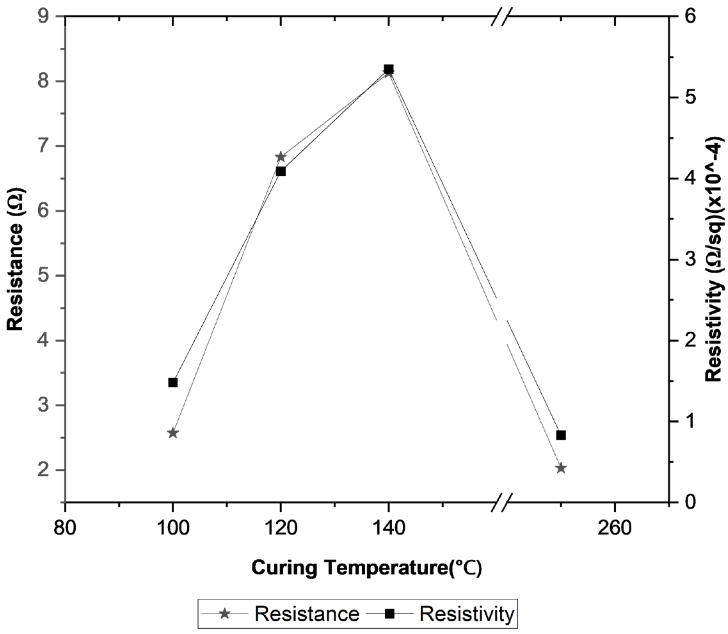


Fig. 5. The resistivity increases gradually as the curing temperature increases, except for the sample cured at 250 °C, where sintering occurs, resulting in a significantly lower resistivity

Table 4
Sample cured at 100 ˚C, 120 ˚C, 140 ˚C and 250 ˚C (baseline) with their respective resistivity

Sample	Resistance (Ω)	Resistivity (Ω/sq)
Cured at 100°	2.57	1.48×10^{-4}
Cured at 120°	6.83	4.09×10^{-4}
Cured at 140°	8.13	5.35×10^{-4}
Cured at 250°	1.30	0.50×10^{-4}

The sintering process is observed in the sample cured at 250 °C. Conductive ink, widely used in electronic applications such as printed electronics, flexible circuits, and sensors, is produced through a crucial process known as sintering [15]. Conductive ink consists of metallic particles dispersed in a liquid medium to form a thick paste. Sintering is essential for the printed conductive ink to establish a continuous conductive pathway, enhance electrical conductivity, and improve adhesion to the substrate. This process involves heating the substrate with the ink deposited on it to high temperatures, typically ranging from several hundred degrees Celsius. As the metal nanoparticles melt, they coalesce and form necks, creating a fused, continuous conducting network. Simultaneously, the liquid medium evaporates, leaving behind a layer of interconnected metallic particles [16,17]. Precise control of sintering factors such as temperature, duration, and environment is crucial to achieving the desired conductivity and protecting the substrate. Successful completion of the sintering process enables the production of functional electronic devices on various surfaces, facilitating innovative and versatile electronic applications.

The impact of reducing the curing temperature on the performance of the conductive ink can be observed as the sintering process may not fully meet the criteria. The SEM analysis of surface morphology offers a visual representation of this process. The temperature required for effective sintering primarily depends on the boiling point of the solvent, which, in this case, is terpineol (219 °C).

Due to the presence of conductive metal particles (Ag) in the hybrid paste, the resistivity increases proportionally with higher thermal temperatures [18]. The sintering process occurs when all solvent components have been eliminated at elevated temperatures [19], such as 250 °C in this case, since terpineol boils at 219 °C and butanol boils at 117 °C. This process fosters strong bonding between Ag and GNP, contributing to enhanced electrical conductivity.

Effective bonding between conductive particles in a material and low resistivity are closely linked to the curing temperature. Lower temperature curing of GNP/Ag conductive ink promotes effective bonding and results in lower resistivity. Curing at lower temperatures allows for extended heat exposure, facilitating the redistribution and interconnection of conductive particles. This prolonged exposure encourages robust bonding between particles, leading to a continuous and well-connected conductive network with minimal gaps. As a result, the resistivity of the cured material is reduced [13]. Moreover, low-temperature curing facilitates a more uniform and controlled bonding process. The gradual drying and solidification of the ink at lower temperatures promote cohesive and consistent bonds between the conductive particles and binder material. This uniform bonding enhances the continuity of conductive pathways, minimizing resistance and contributing to low resistivity.

Curing a conductive material, such as GNP/Ag conductive ink, at temperatures close to the solvent's boiling point, can lead to poor bonding between conductive particles, resulting in higher resistivity. Rapid heating at higher temperatures might not provide adequate time for particles to redistribute and establish strong interconnections. This insufficient bonding creates weak connections and gaps within the conductive network, hindering electron flow and increasing resistivity.

Furthermore, curing at elevated temperatures can potentially cause ink degradation [20], altering the properties of the binder or conductive particles. Higher resistivity can arise from this degradation, which can also degrade the bonding between particles and reduce overall material conductivity. Higher curing temperatures, such as 120 °C and 140 °C, might adversely affect the interaction of the cured material with the substrate, leading to challenges in adhesion and electrical contact [7]. These substrate-related changes can contribute to increased resistance in the cured material. It is crucial to optimize curing conditions, either by employing lower temperatures or controlled heating techniques, to ensure proper bonding and reduce resistance in the dried GNP/Ag conductive ink, thus mitigating these concerns.

In addition to further testing the conductive ink's performance, a cyclic bending test was applied to each test sample. The resistance of the conductive ink samples was measured in their initial state, at 1000 cycles, 3000 cycles, and 5000 cycles. As indicated in the table 5 below, the characteristics of each sample demonstrate a gradual increase in resistance and resistivity (Figure 6).

Table 5
Sample cured at 100˚C, 120˚C, 140˚C, and 250˚C (baseline) resistivity after 1000, 3000 and 5000 cyclic bending test

Sample (°C)	Resistivity at 0 cycle (Ω/sq)	Resistivity at 1000 cycle (Ω/sq)	Resistivity at 3000 cycle (Ω/sq)	Resistivity at 5000 cycle (Ω/sq)
100	$1.48x10^{-4}$	$4.41x10^{-4}$	$5.20x10^{-4}$	$5.85x10^{-4}$
120	$4.09x10^{-4}$	$5.04x10^{-4}$	$6.64x10^{-4}$	$6.88x10^{-4}$
140	$5.35x10^{-4}$	$7.56x10^{-4}$	$8.30x10^{-4}$	$8.70x10^{-4}$
250	$0.50x10^{-4}$	$0.57x10^{-4}$	$0.65x10^{-4}$	$0.71x10^{-4}$

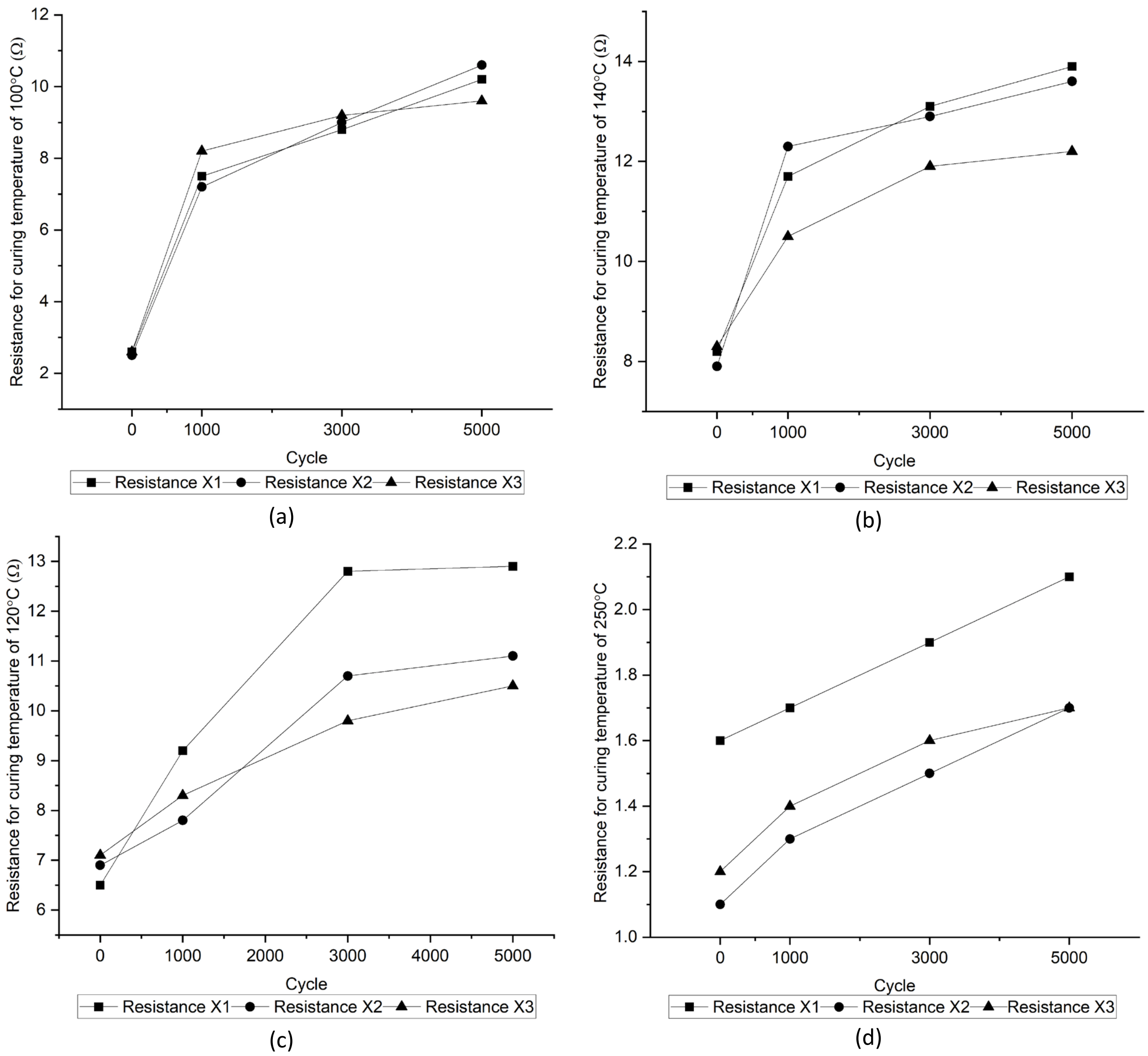


Fig. 6. Resistance over cyclic bending test (a) sample cured at 100 °C (b) sample cured at 120 °C (c) sample cured at 140 °C (d) sample cured at 250 °C

The resistance of a conductive ink tends to increase as it undergoes multiple bending cycles. This phenomenon is primarily influenced by several factors that impact the structural stability and electrical conductivity of the ink. During bending, mechanical stress can induce microstructural changes in the ink, resulting in cracks or discontinuities that disrupt electron flow and elevate resistance. Additionally, ink particles might experience misalignment, aggregation, or detachment as the ink layer ages due to stretching, compression, and shear forces. These alterations compromise the conductivity of the ink layer, consequently leading to increased resistance. The fatigue and deformation induced by the substrate on which the ink is printed also affect the ink-substrate contact, contributing further to increased resistance [8].

Furthermore, the oxidation of metal particles within the ink, coupled with air and moisture exposure during bending, can diminish conductivity and escalate resistivity. This phenomenon is attributed to tunnelling resistance, which can eliminate the electrical conductivity threshold. Tunnelling resistance allows current flow between untouched particles or between particles coated with any insulating layer, creating specific gaps like those generated by the oxidation process on the particle surfaces [8]. Lastly, inadequate alignment of the ink's conductive fillers can undermine the effectiveness of the conductive network and elevate resistance.

Collectively, these factors emphasize the importance of considering the mechanical durability of conductive ink when designing flexible electronic systems.

4. Conclusion

Resistivity plays a critical role in assessing the performance of conductive ink. Lowering the curing temperature leads to a corresponding decrease in overall performance compared to the baseline, as evidenced by the analysis of electrical conductivity. The sample cured at 100 °C demonstrated promising characteristics, boasting the lowest resistivity (1.48 x 10^{-4} Ω/sq) compared to the samples cured at 120 °C (4.09 x 10^{-4} Ω/sq) and 14 0°C (5.35 x 10^{-4} Ω/sq). As the curing temperature was elevated, the resistivity gradually increases (1.48 x 10^{-4} Ω/sq, 4.09 x 10^{-4} Ω/sq, and 5.35 x 10^{-4} Ω/sq).

The SEM analysis revealed that, despite the low resistivity of the sample cured at 100 °C, Graphene Nanoplatelets (GNP) and Silver (Ag) particles remain dispersed and had not formed proper connections between them. This particle dispersion was observed in contrast to the samples cured at higher temperatures, where better interconnection was noticeable. Therefore, this research is very useful in the semiconductor industry because it will save time and costs in producing electronic hardware in the future. Additionally, this research can also replace the soldering process, thereby reducing pollution.

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