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Cyclic Effect on Highly Thermal Hybrid Nano-Graphene Conductive Ink

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ABSTRACT

This research investigates the cyclic effect on highly thermal hybrid nano-graphene conductive ink. The goal is to develop and formulate a highly thermal graphene hybrid conductive ink by combining nano-graphene, silver flakes and silver acetate as conductive fillers which were mixed with chemical and organic solvents. The research evaluates the resistivity at room temperature before and after cyclic bending test in terms of electrical and mechanical properties. For this purpose, a new formulation of the conductive ink was developed using nano-graphene as the primary conductive fillers mixed with organic solvents. The mixture was sonicated and stirred to form a powder which was then dripped with 1-butanol and terpineol and mixed using a Thinky mixer to create a paste. This paste was printed onto copper substrates using a mesh stencil. The hybrid GNP paste was applied to a selected grid (3mm x 3mm) on five designated points of the substrate strip using a scraper. The samples were then cured at 250°C for 1 hour. Cyclic testing was performed using a cyclic bending test machine according to ASTM D7774-17. The formulation was characterised based on its electrical and mechanical behaviour. The resistivity of the hybrid GNP conductive ink at room temperature, without any cyclic loading, was set as the baseline. This baseline resistivity was then compared against measurements taken after 1000, 2000, 4000 and 8000 cycles. After the bending test, the reliability of the GNP hybrid formulation was assessed. Comparisons were made between the room temperature baseline and the post test samples in terms of electrical and mechanical properties. The result shows that the conductive ink exhibit initial improvements in conductivity under mechanical stress but extended cyclic bending leads to microstructural degradation with partial recovery observed after 8000 cycles indicating limited durability and potential self-healing properties. Future research should focus on enhancing the durability and self-healing properties of the hybrid conductive ink to ensure consistent performance under prolonged mechanical stress.

Keywords:
Graphene nanoplatelet (GNP); hybrid conductive ink; cyclic testing; cyclic bending test; temperature

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1. Introduction

The reliability of flexible and stretchable printed circuits using conductive ink has emerged because of the rapid advancement of technology and the increasing demand for flexible and stretchable electronic products [1]. Assessing the reliability of stretchable conductive ink is essential to ensure its high performance and durability throughout it intended operational lifespan. Conductive inks are mostly utilised in the printed electronics sector for the production of printed circuits, sensors, radio frequency devices, flexible displays, energy storage devices and battery test stripes [2-6]. The main benefits of these products include flexibility, simplified design and cost saving [7]. Conductive ink can be made from hybrid formulations that combine different material for enhanced performance. The main component is the conductive material can be derived from metal, carbon particles or conductive polymers. Ensuring the even distribution of these particles involves the use of resins. Resin also could enhance the mechanical strength and adhesion properties of the ink. Solvents are then incorporated to dissolve the resins and regulate the inks flow characteristics while additional additives are utilized to adjust the processing capabilities or enhance the functional attributes of the ink [8-10].

Recent advancement in the field of nanomaterials have highlighted the potential of nano-graphene based conductive ink for flexible electronics. Nano-graphene are valued for their exceptional electrical conductivity, thermal stability and mechanical strength making them ideal candidates for hybrid conductive ink [11,12]. According to Geim and Novoselov [13], the unique properties of nano-graphene arise from the two dimensional structure which provides high electron mobility and mechanical flexibility. Silver nanoparticles have also been extensively studied because of their high electrical conductivity and excellent oxidation resistance [14]. When combined with nano-graphene, these nanoparticles can enhance the overall conductivity and adhesion properties of the ink as reported by Gao *et al.,* [15]. The incorporation of silver nanoparticles into nano-graphene based ink has been shown to improve not only the electrical performance but also the mechanical stability of the material under stress.

Previous studies have shown that graphene and silver nanoparticle hybrid inks exhibit excellent initial conductivity but their performance can degrade over time because of microstructural changes induced by cyclic loading and environmental factors [16-18]. Ismail *et al.,* [17] observed that cyclic bending could lead to the formation of micro cracks within the conductive network which increase the resistivity of the ink. During cyclic loading the material experiences plastic deformation leading to the formation of dislocation, grain boundary sliding and other microstructural changes [16]. These changes can increase the scattering of electrons leading to an increase in electrical resistance and resistivity.

The development of flexible and stretchable printed circuits is crucial for advancing next generation electronics, yet ensuring their long term reliability remain a significant challenge. Current conductive inks often suffer from increased resistivity and inadequate adhesion under cyclic loading conditions, such as bending, stretching, and thermal stress, which can compromise the performance and durability of the circuits. For optimal performance, flexible and stretchable printed circuits with conductive ink must be free of defects and exhibit strong adhesion between the substrate, the ink and the mounted components particularly to withstand various loads such as bending, vibration, thermal stress, shock, and stretching during use [19]. During operation, components are subjected to repeated or cyclic loads such as cyclic bending. These stretching and cyclic load conditions require a thorough assessment of performance particularly in terms of reliability. Addressing the underlying challenges such as increasing resistivity and inadequate adhesion between the ink and substrate, studies employ cyclic bending and cyclic stretchability test to evaluate reliability [20-23]. Therefore, there is a need to formulate a new hybrid conductive ink that not only enhances electrical conductivity and adhesion but also withstands the demanding conditions of repeated mechanical and thermal stresses. This research aims to address these challenges by developing a hybrid conductive ink with improved performance characteristics designed to meet the demand of flexible and stretchable electronics. Additionally, the study will conduct reliability tests specifically cyclic bending tests to evaluate the performance of the formulated hybrid conductive ink.

2. Methodology

2.1 Material Preparation

The material used to prepare highly thermal nano-graphene hybrid conductive ink have been tabulated in Table 1. Nano-graphene, nano-silver, and silver acetate are three main materials that have shown great potential in the field of conductive inks because of their high conductivity, flexibility and ease of processing.

Table 1
Material of GNP hybrid conductive ink

Type	Material
Filler	Nano-Graphene with 25 µm nanoparticle size
	Nano-Silver with 10 µm particle size
	Silver Acetate
Chemical Solvent	Ethanol 99.99%
Organic Solvent	I-Butanol 99.9% butyl alcohol
	Terpineol ~65%α,~10%β,~20%γ substances

2.2 Formulation of GNP Hybrid Conductive Ink

For the preparation of the GNP hybrid conductive ink, 0.5 g of nano-graphene powder constituting 10% of the weight of 50 ml ethanol was mixed in a beaker. The nano-graphene was sonicated in ethanol for 10 minutes in room temperature, which resulted in a dispersion with a high defect content and reduced particle size. Following this, 4.292 g of silver flakes were added to the mixture and sonicated for 60 minutes. The silver acetate was weighed and added into the nano-graphene/ethanol mixture and sonicated for an additional 60 minutes. Extending the ultrasonic treatment to 60 minutes enhances the dispersion quality by reducing defects and increasing particle size. During the sonication process the dispersibility of nano-graphene in ethanol and silver was monitored.

Next, 0.42 g of silver acetate was added to the mixture followed by another hour of sonication. The specific weight of silver acetate used was determine based on the research conducted by Noor *et al.,* [16]. The solution then was heated on a hotplate at 70°C while being stirred at 200 rpm using a magnetic stirrer until the ethanol is fully evaporated. This equipment was used to ensure effective blending of the hybrid powder components. The stirring speed of 200 rpm was carefully chosen to ensure uniform mixing and to prevent any splashing that might occur at higher speeds.

After stirring, the mixture was transferred into a small white porcelain beaker and baked at 250°C for one hour to cure. Following the curing process, the mixture was allowed to cool to room temperature before being pound into fine powder.

For the preparation of the GNP hybrid paste, 30 drops of butanol and 30 drops of terpineol were added to the nano-graphene hybrid powder of 4.68 which is the total nett weight after the process of preparing the nano-graphene hybrid powder. The mixture of GNP hybrid powder with the organic solvent as binder were weighted to ensure the right setting then placed in the thinky mixer. Thinker mixer is a machine that demonstrate superior performance in dispersing nanoparticles in a variety of media while removing the air bubbles and mixed thoroughly.

The materials were blended for three minutes at a centrifugal speed of 2000 rpm. In order to prevent a reduction in the viscosity of the conductive paste which can occur because of extended mixing and increased shear rates the mixing process was intentionally restricted to a duration of three minutes. This is to achieve a balance between gaining optimal dispersion and preserving the desired viscosity of the paste. Adnan *et al.,* [24] observed that the morphological study indicates a shorter centrifugal mixing period facilitates an adequate dispersion of the micro nano filler resulting in superior interfacial bonding between the fillers at the optimum ratio. The increased velocity of the mixing procedure served an important part in the enhancement of both the strength and dispersion of the fillers.

2.3 Sample Preparation

In this research, copper was selected as the substrate material because of its high density, excellent thermal conductivity and significant specific heat capacity. Copper substrates are commonly utilized in high performance electronic applications such as high frequency circuit boards, power electronics and system requiring high temperature application. The copper substrates were used in their original form and were cut to the required dimensions of 120 mm in length, 7 mm in width and 0.1 mm in thickness. The substrates were etched using sandpaper to prevent oxidation. Additionally, each substrate was marked with a 20 mm distance from each end to guide the printing process as illustrated in Figure 1.

Fig. 1. Dimension of the cooper substrate

2.4 Printing Process

The nano-graphene hybrid paste was printed on copper substrates with a mesh stencil method. The nano-graphene hybrid paste was carefully applied to the designated grid using a scraper to ensure uniform distribution. The mesh stencil employed in this study was precisely measured and standardized to a grid size of 3.0 mm x 3.0 mm ensuring consistent and accurate deposition of the paste across the copper substrate. This standardization is important for achieving uniform thickness and alignment which directly impacts the electrical performance and adhesion properties of the printed conductive ink. Figure 2 illustrates the schematic printing process.

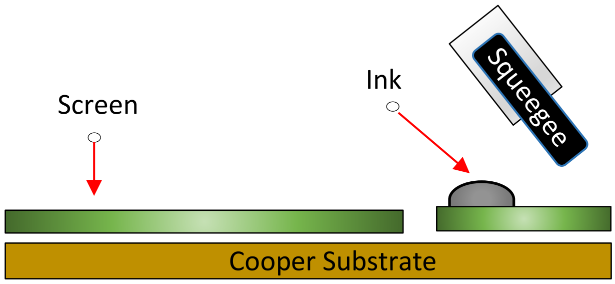

Fig. 2. Printing process using the mesh stencil

The paste was printed on the five selected points of the substrate strip. After the printing completed, the mesh stencil was cleaned, and the process was repeated on each sample. Figure 3 illustrates the paste printed on the cooper substrate ready for curing process.

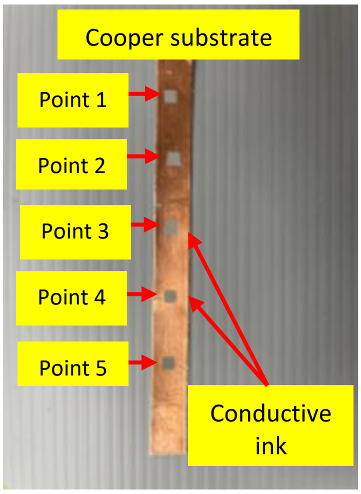

Fig. 3. Printing process using the mesh stencil

2.4 Curing Process

The curing procedure is a post treatment that is performed after the printing of the conductive ink. The curing step is essential because it assist strengthen the bond that exists between the filler particles and the binder. The printed conductive ink was cured at 250˚C for one hour inside an oven as shown in Figure 4. This curing temperature was selected because it effectively facilitates the bonding and stabilization of metal particles leading to the formation of a continuous conductive network which reduces electrical resistance and enhances conductivity. Furthermore, curing at this temperature for one hour ensures that solvents or binders in the ink are fully evaporated which minimizes the possibility of voids or defects in the cured ink layer. This duration also allows sufficient time for the cross thereby improving the overall mechanical strength and durability of the conductive ink.

(a) (b)

Fig. 4. Curing process; (a) laboratory oven (b) sample place inside the oven

2.5 Cyclic Bending Test

Cyclic testing has been conducted using a cyclic bending test machine. Cyclic test was performed according to ASTM D7774-17 where five-point loading system was used for this procedure. All testing is done in room temperature. Cyclic bending test were carried out to measure the durability of conductive ink before and after the experiment. The testing is conducted to verify the products capacity to endure various stresses and strains. In bending the samples, the substrate was held firmly at both ends and only one end was free to bend. A 12 V DC motor on the bending test equipment rotates the wheel and connection sample holder, bending the sample along the rail. With this cyclic bending test setup, the DC motor was set to run continuously horizontally with the speed resulting in a 4 second per cycle. As shown in Figure 5, a cyclic bending test machine is used to measure the durability of a sample.

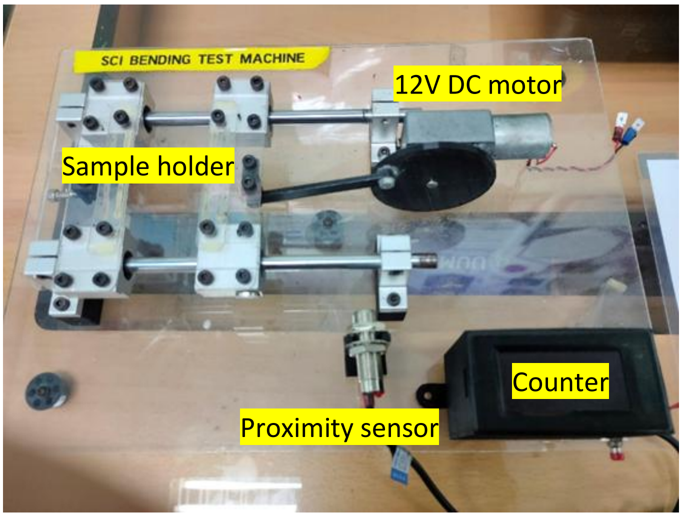

Fig. 5. Cyclic bending machine

As illustrated in schematically in Figure 6, the mechanical bending machine was made up of two holders connected on both sides of the substrate. One holder was joined to the DC motor, while the other was fixed. In the repeated movement of the bending, the outline of the substrate resulted in a bending radius, *r* of approximately 12.5 mm.

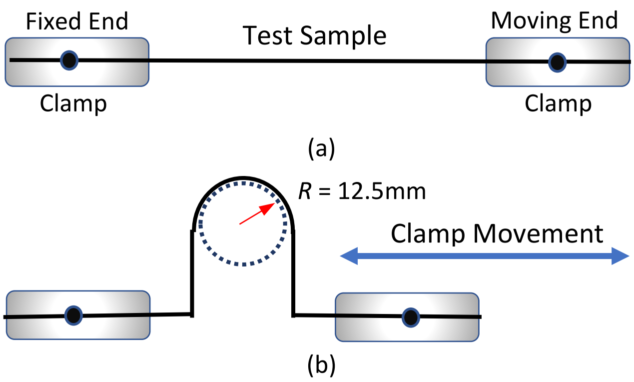

Fig. 6. Schematic of the test component in a cyclic bending machine with a constant bending radius (a) in a flat position and (b) in a bent position

In this research the cyclic bending test machine is done in room temperature. The test is done starting with 1000 cycle, 2000 cycle, 4000 cycle and 8000 cycle respectively. Once the counter hits 1000 cycle, resistance measurements are taken using a Two-Point probe. This procedure is repeated for 2000 cycle, 4000 cycle and 8000 cycle.

2.5 Electrical Characterization

Resistance is an important factor in determining the electrical properties of a circuit as it indicates how much current is restricted within a circuit trace, thus impacting its conductivity. The resistance measurements for all samples were taken at room temperature. Each sample was measured at five different points on a copper substrate strip using a two-point probe method to obtain the resistance values. This method involves applying of a voltage difference across the two ends of the material sample, followed by the measurement of the resulting electric current. Ohm's Law is applied to calculate the resistance. Figure 7 illustrates a setup for measuring resistance using Two-Point probe where voltage is applied to a resistor device. A pair of probes is used to measure the electric current passing through the sample or resistor.

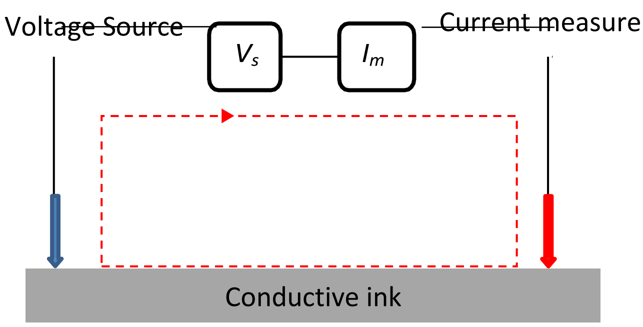

Fig. 7. Schematic diagram of Two-Point probe

Figure 8 shows the printed conductive ink pattern on the copper substrates for GNP hybrid was observed at five separate points with gap 20.0 mm between each point, namely point 1 (1.1, 1.2, 1.3), point 2 (2.1, 2.2, 2.3), point 3 (3.1, 3.2, 3.3), point 4 (4.1, 4.2, 4.3) and point 5 (5.1, 5.2, 5.3) of the 3 mm x 3 mm conductive pattern.

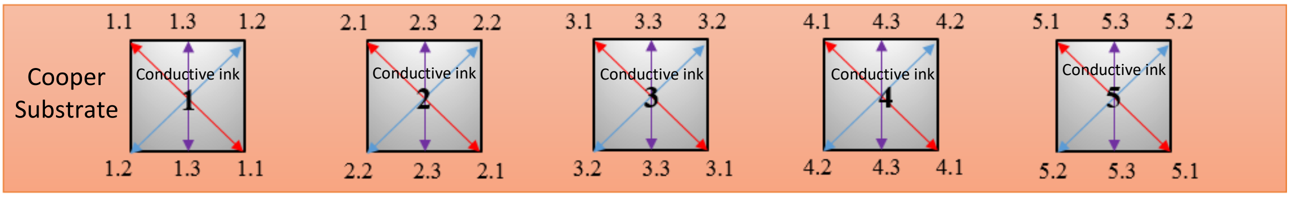

Fig. 8. Five points of printed GNP hybrid on copper substrates with observation positions

The data was used to calculate the resistance of all layer samples and then averaged. Then the resistivity of the point is calculated based on the formula stated in Eq. (1).

$$\rho=(R\times A)/L \quad (1)$$

When ρ represents resistivity, R denotes the resistance measured using the Two-Point probe, A is the cross-sectional area and L is the length. Understanding the relationship between resistance and resistivity is important in analysing printed conductors. Eq. (1) illustrates the connection between resistance and the trace cross-sectional area indicating that increasing the trace width leads to a decrease in resistance. This relationship between overall resistance and the shape and size of conductive paths is essential for assessing and comparing different material, width and design in printed conductors.

3. Results and Discussion

3.1 Cyclic Bending Test

In this research, the cyclic bending test was conducted in accordance with the ASTM D7774-17 Standard Test Method employed to assess the performance of these hybrid inks under repeated mechanical stress. The test was carried out at room temperature providing insights into how the nano-graphene hybrid inks withstand cyclic deformation over extended period. This section presents the results of the cyclic bending tests focusing on the changes in resistance and resistivity as well as the integrity of the conductive pathways in the inks after being subjected to various bending cycles.

Figure 9 present the baseline resistance and resistivity values of the conductive ink measured at room temperature before any cyclic testing was applied. This baseline serves as a reference point providing a standard for comparing how subsequent mechanical stress through cyclic bending impacts the electrical properties of the ink. At room temperature, the baseline measurement provides a reference point for both resistance and resistivity across different location on the same sample from P1 to P5. The resistance values at these points were relatively consistent ranging from 0.2 to 0.4 Ω with corresponding resistivity values between 6.0×10^{-6} Ω.m and 1.2×10^{-5} Ω.m. These values suggest that the nano-graphene hybrid conductive ink exhibits stable and uniform electrical properties across the sample without any applied mechanical stress. The consistency in both resistance and resistivity across the different points indicates a homogeneous distribution of conductive paths within the ink matrix. This uniformity can be attributed to effective curing and adequate dispersion of the filler material within the conductive paste [25,26]. At baseline, the resistivity values are relatively stable across the different points on the sample. This stability is because the material has not yet been subjected to any mechanical stress so the conductive pathways are intact and the material structure is undisturbed [27].

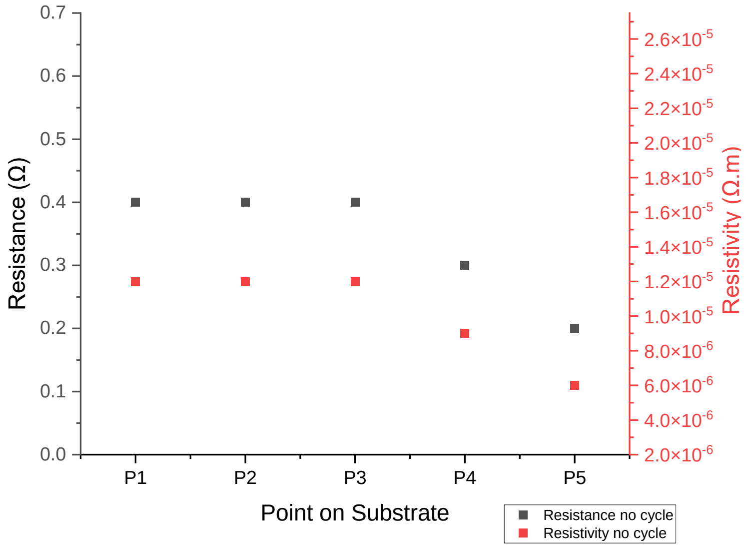

Fig. 9. Baseline resistance and resistivity

Figure 10 illustrates the resistance and resistivity measurements for the conductive ink after 1000 bending cycles. A significant reduction in resistance was observed across all points with values dropping to as low as 0.1 Ω at P2 and P3, and slightly higher at P1, P4 and P5 with 0.2 Ω with corresponding resistivity values of 6.0 $\times10^{-6}$ Ω.m. This decrease in resistance was accompanied by a corresponding reduction in resistivity indicating improved conductivity. The improvement can be attributed to the mechanical stress from bending which likely induced reorientation or compaction of the conductive fillers enhancing the electron pathways within the ink and thus lowering resistance [18,28]. The significant decrease in resistivity compared to the baseline suggests that the initial bending cycles helped compress the material potentially improving contact between the conductive fillers specifically nano-graphene and nano-silver thus, enhancing overall conductivity [29-31].

In this context, the mechanical force from bending pushes the nano-graphene and nano-silver particles closer together reducing the number of voids or gaps that could block the flow of electrons. This compression helps create more efficient and continuous conductive pathways ultimately enhancing the material overall electrical performance. This process may cause the particles to settle and align more effectively reducing the gaps between them and further lowering resistivity [32]. As the particles shift and realign under stress they create more optimal pathways for electron movement decreasing the overall resistance of the conductive network. This alignment may be particularly beneficial in enhancing the material long term performance under repeated mechanical stresses ensuring better reliability in practical applications.

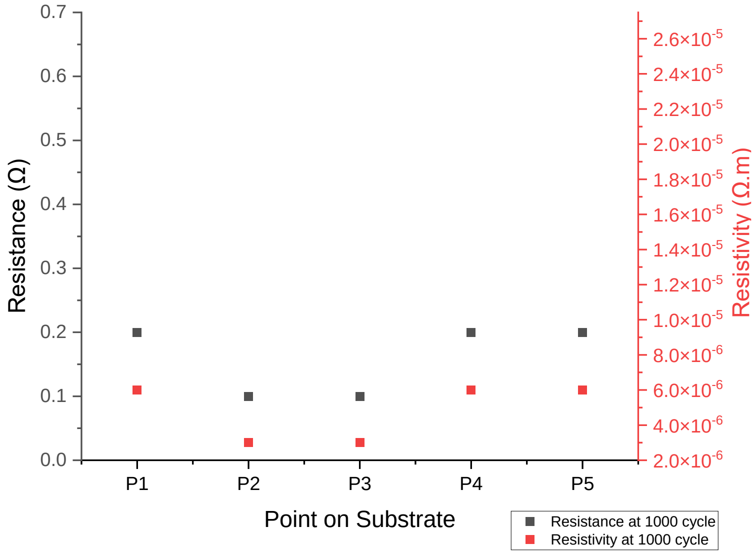

Fig. 10. Resistance and resistivity at 1000 cycle on bending test

Figure 11 illustrates the resistance and resistivity measurements for the conductive ink after 2000 bending cycles. After 2000 cycles, both resistance and resistivity remained relatively low with minimal changes compared to the 1000 cycle data. Point P1 recorded a resistance of 0.1 Ω and the resistivity decreased to 3.0×10^{-6} Ω.m. This stability suggests that the conductive network within the ink is resilient to mechanical deformation successfully maintaining its integrity and functionality despite the applied stress [33]. The resistivity values either remained stable or showed a slight decrease compared to the 1000 cycle results indicating that the material continues to benefit from the mechanical stress. This resilience suggests that the conductive network within the material remains intact or is even slightly improved [34]. It is possible that the material structure is still adjusting with conductive fillers continuing to align and settle, thereby maintaining or slightly enhancing conductivity [35].

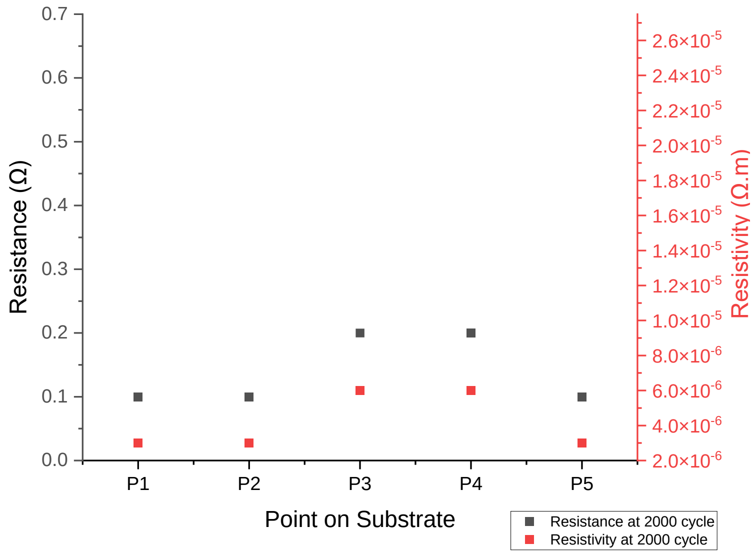

Fig. 11. Resistance and resistivity at 2000 cycle on bending test

Figure 12 present the resistance and resistivity measurements for the conductive ink after 4000 bending cycles. At this stage, signs of wear began to emerge particularly at P1 and P3, where resistance spiked to 0.6 Ω and 0.9 Ω, respectively. The corresponding increases in resistivity of

1.8×10^{-5} Ω.m at P1 and 2.7×10^{-5} Ω.m at P3 suggest that prolonged mechanical stress may have caused microstructural changes such as the formation of micro cracks or delamination within the conductive pathways leading to increased resistance [36]. In contrast, points P2, P4 and P5 remained relatively stable indicating that these locations were less affected by the cyclic stress.

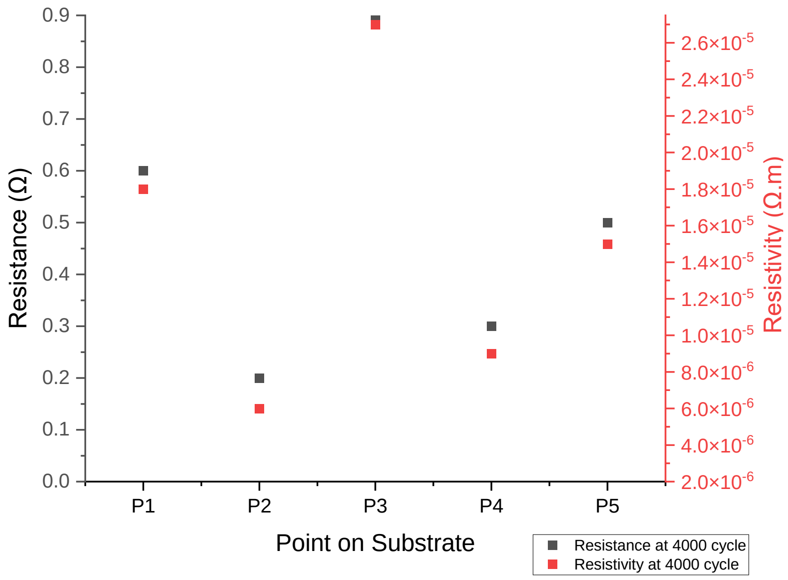

Fig. 12. Resistance and resistivity at 4000 cycle on bending test

The significant rise in resistivity at 4000 cycles, especially at P3 on the sample, suggests that the material is beginning to experience degradation in its conductive pathways [32]. Repeated bending of the substrate leads to stress concentration on the top surface. Fatigue cracks typically initiate in these stress-concentrated areas and propagate when the concentrated stress surpasses the material theoretical cohesive strength [12]. The theory is illustrated in Figure 13. The repeated bending likely causes micro cracks or delamination within the material disrupting the conductive network. These disruptions increase the distance between conductive particles or cause partial breaks in the pathways leading to higher resistivity [37].

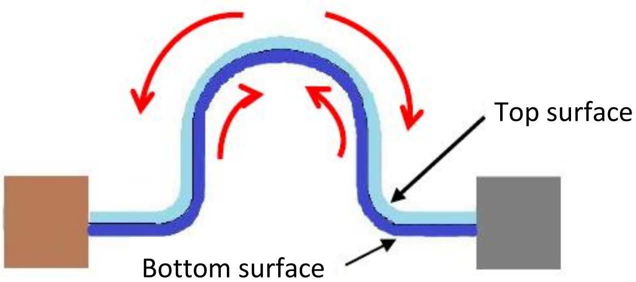

Fig. 13. The compression bending method induces tensile stress on the top surface and compressive stress on the bottom surface [12]

Figure 14 shows the resistance and resistivity measurements for the conductive ink after 8000 bending cycles. By 8000 cycles, resistance and resistivity values at most points exhibited some recovery with P1 and P4 returning to their baseline resistance of 0.4 Ω and 0.1 Ω respectively. The resistivity at these points also aligned closely with the baseline values. This recovery could be attributed to the reestablishment of conductive pathways as the material readjusts after extensive bending indicating a potential for self-healing under prolonged mechanical stress [34-36].

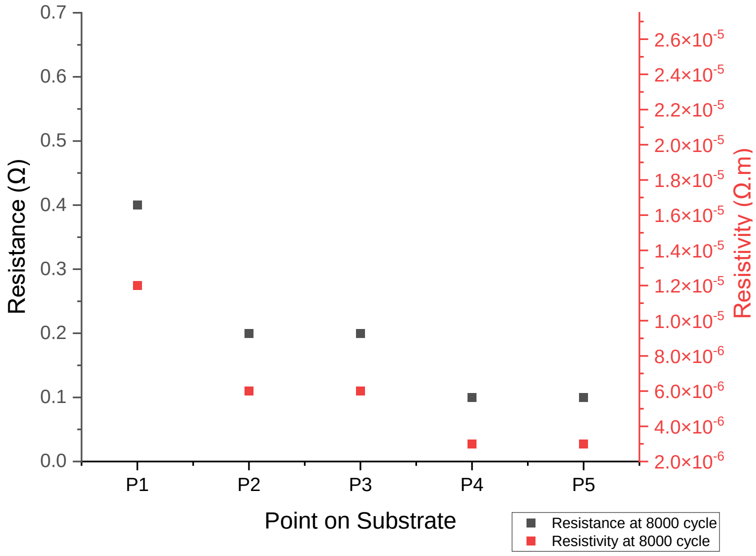

Fig. 14. Resistance and resistivity at 8000 cycle on bending test

After 8000 cycles, while some points demonstrated a partial recovery in resistivity, others remained elevated. This partial recovery suggests that the material may possess some capacity to self-heal. Partial recovery refers to reversible molecular processes, such as chain rearrangement or reorientation, while the specific mechanisms driving recovery and microstructural degradation has been stated previously [36,37]. Figure 15 shows the concept of self-healing under mechanical stresses. Over the extended bending cycles, the material may have reached a state where further stress prompted the conductive fillers to realign or redistribute partially restoring some of the conductive pathways [38]. However, area with persistently high resistivity likely indicate where the damage is too severe for the material to maintain low resistivity.

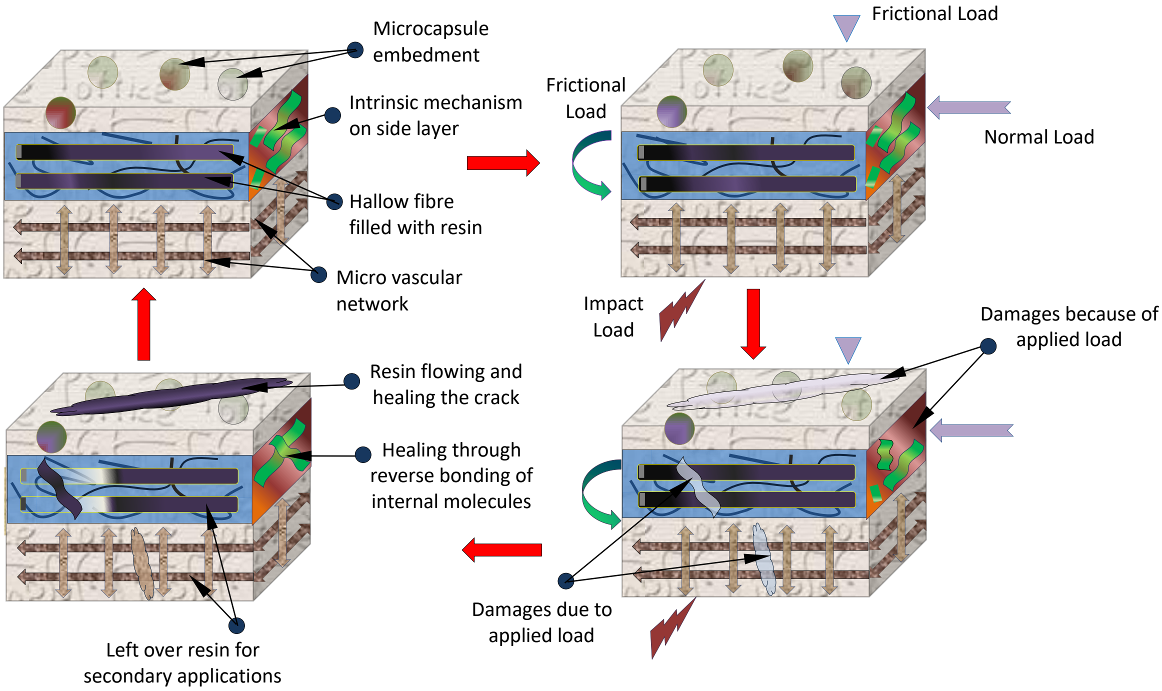

Fig. 15. Concept of self-healing [39]

The observed trend in resistance and resistivity across different cycles suggest that the microstructure of the nano-graphene hybrid conductive ink undergoes significant changes during cyclic bending. Initially, mechanical stress seems to compact the filler materials enhancing conductivity by reducing resistance and resistivity. However, with prolonged cycles the likelihood of micro crack formation and other structural deformations increases particularly at points that experienced higher resistance after 4000 cycles. This indicates the onset of degradation in the conductive network likely because of the breakdown of filler-matrix interactions or the initiation of cracks that interrupt electron flow [40].

As the nano-graphene hybrid conductive ink was subjected to cyclic bending, the resistance and resistivity initially decreased (indicating better conductivity) but then increased after 4000 cycles. This increase in resistance suggested that the material was starting to degrade due to the repeated bending. Micro cracks might have formed or the conductive pathways might have been disrupted leading to poorer electrical performance. By the time the material had been bent 8000 times, some of the resistance and resistivity values started to return closer to their original baseline levels (the levels measured before any bending cycles). This phenomenon is referred to as partial recovery [38,41]. The nano-graphene and silver particles within the ink might have moved or shifted during the bending process. Over time as the material is bent repeatedly these particles could realign or redistribute some of the broken or weakened conductive pathways. This suggests that the material has some inherent resilience. Despite the initial damage caused by bending like the formation of micro cracks, the material self-correct to some extent partially restoring the conductivity [42]. This partial recovery indicates that the material may be more durable than initially thought. Even after extensive mechanical stress it can still maintain some level of electrical performance which is important for applications where the material will undergo repeated mechanical deformation.

4. Conclusions

The results of the research demonstrate the analysis of resistance and resistivity measurements across different bending cycles reveals distinct trend in the performance of the conductive ink. Initially, the ink shows a decrease in resistance and resistivity after 1000 and 2000 cycles indicating improved conductivity because of mechanical stress induced realignment of conductive fillers. However, by 4000 cycles, signs of degradation become evident with increased resistivity at certain points because of microstructural damage such as cracks or delamination. Interestingly, after 8000 cycles, some recovery in resistance and resistivity is observed suggesting a potential self-healing or reorganization of the conductive pathways under prolonged stress. Despite this, the material begins to deteriorate after extended cycling highlighting the limits of its durability under continuous mechanical load. In this study, some error analysis addressed the conductive failures, such as such as ink degradation and conductive path disruptions, including formulation adjustments and stress-relief techniques, to enhance durability.

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