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# Resistivity of Graphene/Silver Hybridization Conductive Ink on Bending Test

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### ABSTRACT

In recent years, there has been a growing demand for stretchable electronic devices, such as wearable sensors and displays. Many researchers have been working on the development of stretchable conductive ink (SCI), which can maintain its electrical conductivity even when stretched or deformed. Graphene nanoplatelet (GNP) hybridization conductive ink is a promising material for stretchable electronics due to its high electrical conductivity and excellent mechanical properties. However, the resistivity of GNP ink on flexible substrates can be affected by various factors, such as the bending of the substrate. This paper aims to investigate the resistivity of hybridization conductive ink between GNP and silver (Ag) on a flexible substrate under different bending conditions. The study was carried out on the formulation and performance of GNP hybrids using GNP and silver flakes (Ag). The GNP hybrid ink was printed on a copper substrate using a mesh stencil method and cured at 250 °C for an hour. The resistivity was evaluated at room temperature before and after the bending tests in terms of electrical characteristics. The result of the resistivity value before performing the bending test was acceptable due to the lowest resistivity value in the range of  $0.963 \times 10^{-5}$  to  $1.293 \times 10^{-5} \Omega \cdot m$  at room temperature. The finding exposed that the resistivity values for each of the three samples of bending tests significantly changed after 1000 cycles. Overall, the results revealed that this hybrid conductive ink has good resistivity and performs with acceptable reliability. In future work, it is recommended that the conductive ink be printed on a more flexible substrate and the evaluation of temperature dependence can also be made more comprehensively.

## 1. Introduction

Stretchable conductive ink is a relatively new development in the field of electronics and has been the subject of extensive research in recent years. Stretchable conductive ink has a focus on flexibility and expandability while keeping excellent conductivity values. Conductive ink is simple and cost-

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effective thus ideal for flexible electronics [1-3]. The ink is designed to be both conductive and stretchable, making it ideal for use in a variety of applications that require flexible and durable electronic components. Future automotive, medical, and robotics applications, as well as wearable gadgets, will need the manufacturing of electronics that are extremely stretchable and conductive [4].

Conductive ink consists of a conductive filler, solvent, an adhesive component, and functional additives [5]. The metals Ag and Cu have been researched the most for printed electronics (PEs) out of all conductive fillers. According to [6], despite their high mechanical stability and conductivity, these materials are expensive to produce and are susceptible to oxidation in hostile environments. Other than metals like silver, gold, and copper, a range of organic and inorganic compounds, including conductive polymers and carbon-based materials like graphene, graphene oxide, and carbon nanotubes have been studied for use in the active layer [7,8]. The ink is usually printed onto a substrate using specialized printing techniques, such as screen printing or inkjet printing, to create electronic components with precise patterns and shapes. According to [9], screen printing is regarded as the most flexible and mature technology among the existing mass printing processes, as well as the simplest and quickest. Meanwhile, inkjet printing is advantageous due to its high resolution and adaptability. Since each microdroplet represents a pixel, drop-by-drop inkjet printing is applied to produce consistent pictures with great resolution [10].

Stretchable conductive ink has shown great potential for use in flexible and wearable electronic devices that are subject to deformation due to bending or stretching. The electrical conductivity and resistance of the ink under both conditions are important factors that determine its performance and reliability. Several studies have investigated the electrical and resistive properties of stretchable conductive ink under various conditions. According to [11], the polymer binder phase contributes significantly to the elasticity of stretchable conductive inks. The silver flakes are not elastic on their own, but they are trapped inside a binder structure that is stretchable. The stretchability and adhesion criteria were met by combining two amorphous binder types. This technique is referred described as "elastomeric chain polymerization." The outcome demonstrated the screen-printable silver conductive ink that can be stretched for at least 500 cycles at 20% strain without its resistance growing by more than 30 times of its initial value. Besides that, [12] developed a stretchable conductive ink based on polysiloxane matrix silver particles as the conductive filler under post-percolation threshold loading. Adding the vinyl-silane coupling agent and functionalized epoxy and acrylate additives increased the adhesion properties of both the conductive filler and the PDMS matrix. Stretching produces, a transient rise in conductivity, followed by a plateau. Meanwhile, [13] demonstrated the production of extremely conductive and flexible graphene ink using a high-throughput fluid dynamic technique. The electromechanical characteristics of a conductor were greatly improved by the incorporation of a serpentine topology. The 50-TPU/EGF conductor with a serpentine design displayed a small resistance variation of 9% at a tension of 300%. Mechanical bending and fatigue testing revealed little resistance variation.

Nowadays, the GNP hybridization conductive ink has been extensively studied for its potential use in flexible and stretchable electronics. Researchers have focused on the hybridization of GNP and silver (Ag) in conductive inks for several reasons. According to [14], to improve the conductivity of pure graphene inks, previous research has concentrated mostly on hybrid graphene and metal-based composite materials. Due to its superior oxidation resistance, electrical conductivity, and other desirable physical qualities that give it exceptional substrate adherence, silver nanoparticles (AgNP) conductive ink is the most popular nanoparticle-based conductive ink for use in printed electronics. By combining GNPs and silver, researchers can take advantage of the unique properties of both materials and overcome their individual limitations. Therefore, graphene and silver might be used to

increase the conductivity of graphene electrodes for large-area applications, such as extended interconnect lines, while retaining the benefits of graphene electrodes [15]. According to [16], it is promising to combine metal nanoparticles with graphene nanosheets to create a hybrid ink, therefore maximizing the benefits of these two materials to increase conductivity and decrease metal particle concentration. In recent years, researchers have investigated the effect of substrate deformation on the resistivity of GNP ink, particularly under bending conditions. In this context, a bending test can be conducted to investigate the effect of substrate deformation on the resistivity of GNP ink. Electrical conductivity measurements were used to assess the quality of the printed lines. These tests also gave insight into the line quality and integrity of flexible substrates exposed to controlled bending while monitoring electrical conductivity [17].

The purpose of this study is to investigate the resistivity of hybridization conductive ink between GNP and Ag on a flexible substrate under different bending conditions. By characterizing the resistivity of the ink as a function of the bending test, insights can be gained into the electrical performance and mechanical reliability of the material. The results of this study provide valuable information for the development of flexible and stretchable electronic devices based on GNP hybridization conductive ink.

## 2. Methodology

### 2.1 Material Preparation

In this research, a GNP hybrid conductive ink consisting of several conductive fillers was developed. The GNP hybrid formulation composed of GNP, Ag, and SA as conductive materials was synthesized with the organic solvents of ethanol, butanol, and terpineol. The sample preparation procedure for the hybrid GNP includes ink formulation, mixing process for powder and paste, ink printing, curing, and characterization of the samples.

The formulation of GNP hybrid used a specified percentage of filler loading and solvent, according to Table 1 below. The GNP hybrid formulation was based on the approach described by [18,19] for developing hybrid conductive ink. The powder or liquid substance to be weighed should be placed in a clean beaker and weighed using a digital scale. The mixing process of GNP hybrid is divided into two parts, which are powder and paste. Based on the formulation, the procedure was prepared for 10 sets, respectively.

**Table 1**  
Composition of GNP Hybrid conductive ink

Sample	GNP (g)	Ethanol (ml)	Silver flake, Ag (g)	Silver acetate, SA (g)	Ratio Butanol: Terpineol (drop)
1 set	0.05	5	0.4292	0.042	3:3

The 0.5 g of GNP was mixed with 50 ml ethanol in a small beaker. The beaker was covered with aluminum foil to prevent the ethanol from drying rapidly throughout the experiment. These two materials were combined using an ultrasonic bath for 10 minutes. The sonication method using an ultrasonic bath was used as the primary fabrication method during the whole experiment. The 4.292 g silver flakes, Ag were added to the GNP/ethanol mixture and continued sonication for 1 hour. Throughout the sonication procedure, the dispersibility of GNP in ethanol and Ag was observed.

The mixture was then added with 0.42 g of silver acetate, SA, and sonicated for an hour. Next, the solution was heated at 70 °C on a hotplate with 200 rpm of stirring until the remaining of the ethanol evaporated. After the stirring process, the mixture was transferred to a small white porcelain

beaker and cured in an oven at 250 °C for 1 hour. After the curing process, the cold mixture was mashed finely until it produced a powder. The powder was transferred to a container for the preparation of GNP hybrid paste. Figure 1 illustrates the mixture of GNP hybrid powder.



**Fig. 1.** The mixture of GNP hybrid powder

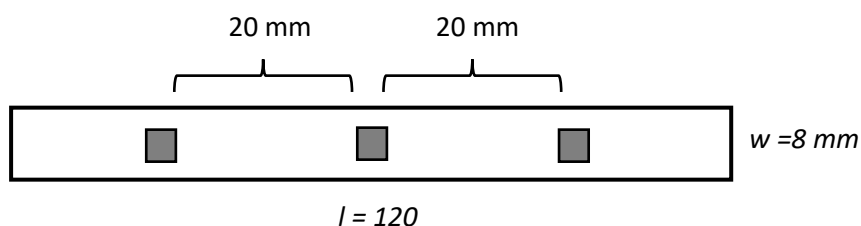
The powder was measured at 4.68 g in a small container. Then, 45 drops of butanol and 45 drops of terpineol were alternately added to the powder. The mixture was placed in the thinky mixer to obtain uniform blending. The ink paste consistency was finally obtained and appropriate for printing. Figure 2 depicts the GNP hybrid paste.



**Fig. 2.** The ink pastes

## 2.2 Printing Process

GNP hybrid paste was printed on copper substrates using a mesh stencil method. The used mesh stencil has a thickness of 10  $\mu\text{m}$ . The process started by placing the substrate below the mesh stencil, and then the paste was placed on the 3 mm x 3 mm grid. The paste was printed on the three selected points of the substrate strip, using a scraper until it was visible as illustrated in Figure 3. After printing, the mesh stencil was cleaned, and the procedure was repeated for all 9 samples.



**Fig. 3.** Schematic diagram of printing points

### 2.3 Curing Process

The curing procedure is a post-treatment performed after the printing of conductive ink. This procedure is required to strengthen the binding between the filler and binder particles. Curing is also used to strengthen the bond between the ink and the substrate. In this study, the universal oven UF55 is used for the curing process. After setting up the oven, the printed samples were placed on a tray and cured in the oven at 250 °C for an hour. Then, all the cured samples were fully soaked at room temperature.

### 2.4 Sample Characterization

The characterization for this study focuses on electrical properties. The samples were prepared specifically according to each relevant test standard. The resistance of the material used to form the circuit determines the conductivity of the circuit. Different weight percentages of conductive filler in the formulation provide various resistance levels.

### 2.5 Electrical Characteristic

The functionality of cured samples was determined by measuring the samples' electrical characteristics. In this experiment, a multimeter as in was used to measure the resistance of the conductive ink at room temperature. The scale of the multimeter was set at a range of 200 Ω before being applied to the samples. Each sample consists of three printed inks. The resistance of each ink was tested at three distinct points. The total resistance measurement for each sample is thus nine. Each sample reading will be averaged and monitored for further discussion.

By analysing the data, the volume of resistivity is utilised to assess the level of electrical resistance existing in the circuit and the efficacy of the established conductivity. The selected formulation criteria are based on the suitable volume of resistivity and will be applied realistically across all tested formulations. The resistivity formulation is shown in Eq. (1).

$$\rho = R \frac{A}{L} \quad (1)$$

where,  $\rho$  is resistivity in ohm meter (Ω·m), or micro-ohm-centimetre (μΩ·cm), R is the measured resistance of the film resulting from the ink (Ω), 'A' is the cross-sectional area, and L is the length.

### 2.6 Mechanical Characteristic

The reliability of conductive ink was determined by mechanical cyclic tests. A bending test was carried out to measure the durability of conductive ink before and after the experiment at room temperature (20.1 °C). The bending test was used to determine the resistance and resistivity of conductive ink while the process was operating at specific cycles. For each test, the number of cycles is 1000, 3000, and 5000. In this experiment, three samples were selected for respective bending tests.

### 2.7 Cyclic Bending Test

Three samples used for the bending test were labeled as S1, S2, and S3 respectively. Figure 4 shows the samples of conductive ink used in the bending test. The samples were gripped at both

ends of the substrate and only one side moved during the bending operation. The bending test apparatus rotates the wheel and connection sample holder using a 12 V DC motor, which bends the sample in accordance to the rail. Figure 5 shows the bending test machine used for this experiment. When the counter reached 1000, resistance measurements were obtained at specific points using a multimeter, as previously described. This process was repeated with the cycles of 3000 and 5000.



Fig. 4. Samples used for bending test

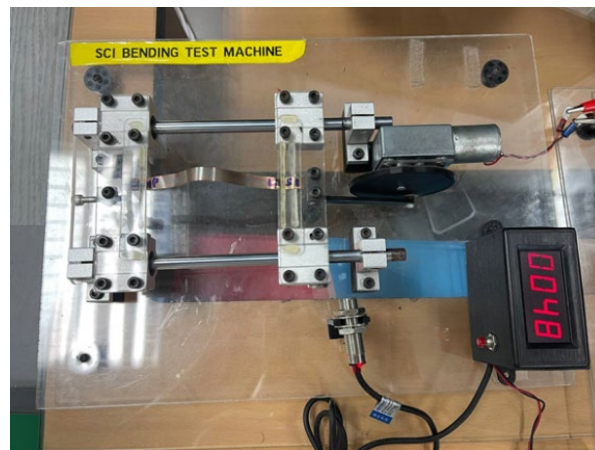


Fig. 5. Bending test machine

### 3. Results

#### 3.1 Resistance and Resistivity of GNP Hybrid at Room Temperature

The experiment was conducted to measure the value of resistance and resistivity of test samples at room temperature (RT). They were measured at three specified points per ink using a multimeter, yielding a total of nine data points per sample thus contributing to the resistance value. Resistivity was calculated using the average results of resistance at each point pattern number. Table 2 shows the resistivity measurement results of average resistance, average resistivity, and standard deviation values for all test samples.

**Table 2**

Measurement of resistivity of test samples

Sample	Average Resistance ( $\Omega$ )	Average Resistivity ( $\Omega.m$ )	Standard deviation ( $\Omega.m$ )
S1	1.293	1.293e-05	8.190e-07
S2	1.089	1.089e-05	6.939e-07
S3	1.063	1.063e-05	3.208e-07
S4	1.070	1.070e-05	1.096e-06
S5	1.089	1.089e-05	1.160e-06
S6	1.059	1.059e-05	2.796e-07
S7	0.963	0.963e-05	1.326 e-06
S8	1.081	1.081e-05	7.143 e-07
S9	1.215	1.215e-05	6.700 e-07

There is no standard benchmark for calculating standard deviation, however, the best standard deviation must have the lowest number. It shows how closely the data is clustered around the mean or average, as well as how far the data is dispersed from the mean or average [20]. From Table 2, sample S7 has a very large standard deviation as compared to other samples. It implies that the data is widely dispersed from the mean, indicating that their average resistivity is the greatest. Meanwhile, sample S6 has the smallest standard deviation indicating that the data are gathered close to the mean. This also demonstrates that the ink sample has the lowest and most constant average resistivity.

### 3.2 Measurement of Resistance and Resistivity of GNP on Bending Test

The experiment was conducted to measure the values of resistance and resistivity of test samples on a bending test at room temperature (RT). The purpose of this bending test is to investigate how the resistance or resistivity of conductive ink changes after bending. It was measured at three specified points per ink using a multimeter, yielding a total of nine data points per sample and thus contributing to the resistance value. The average resistance results at each point pattern number were used to calculate resistivity.

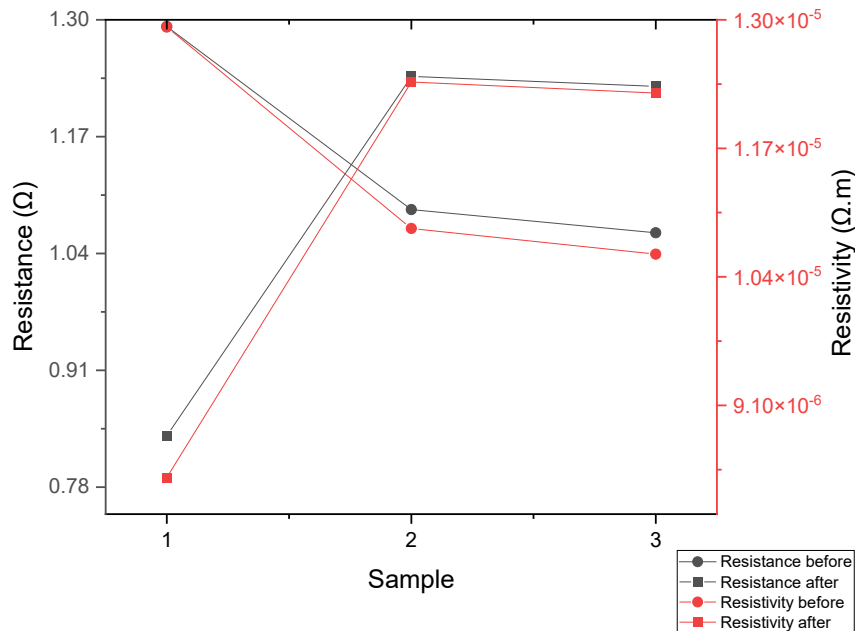
Table 3 shows the resistivity measurement data as the average resistance and average resistivity of three samples for 1000 cycles of bending testing. The average resistance for S2 and S3 samples before testing is lower than the average resistance after testing. This is comparable to the average resistivity value, whereas the value after testing increases. However, for sample S1, the resistance or resistivity decreases. Nevertheless, the resistivity will increase after the process of 1000 cycles because the ink on the substrate has been in an imperfect state. Therefore, the resistivity will increase and the conductive properties will decrease.

**Table 3**

Measurement of resistivity of the test samples for 1000 cycles of bending testing

Sample	Average Resistance ( $\Omega$ )		Average Resistivity ( $\Omega.m$ )	
	before	after	before	after
S1	1.293	0.837	1.293e-05	8.370e-06
S2	1.089	1.237	1.089e-05	1.237e-05
S3	1.063	1.226	1.063e-05	1.226e-05
Average	1.148	1.100	1.148e-05	1.100e-05

Figure 6 illustrates high average resistance as well as average resistivity for samples S2 and S3 after 1000 cycles of bending testing. After the bending test, S1 reduces the average resistivity. The resistivity rises after processing which is due to the considerable increase in dislocation density and dispersion produced by the high applied strain [21].



**Fig. 6.** Resistance and resistivity before and after bending test on test samples for 1000 cycles

Table 4 shows the resistivity measurement data as average resistance and average resistivity of samples S1, S2, and S3 for 3000 cycles of bending testing. The average resistance for S1 before the bending test is slightly lower than the average resistance after the test. Meanwhile, the average resistance and resistivity for samples S2 and S3 are obviously decreasing. It is possible that the ink is not dispersed properly over the stencil area during the screen-printing process. Due to the speed or viscosity of the ink during the screen-printing process, there are certain uneven gaps when the squeegee crosses the gap [22].

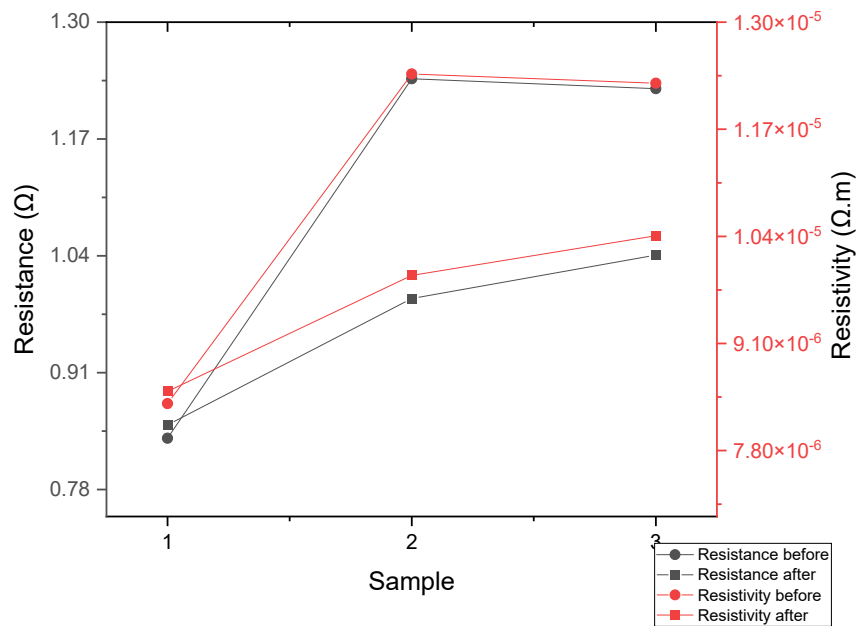
**Table 4**

Measurement of resistivity of the test samples for 3000 cycles of bending testing

Sample	Average Resistance (Ω)		Average Resistivity (Ω.m)	
	before	after	before	after
S1	0.837	0.852	8.370e-06	8.519e-06
S2	1.237	0.993	1.237e-05	9.926e-06
S3	1.226	1.041	1.226e-05	1.041e-05
Average	1.100	0.962	1.100e-05	9.617e-06

Figure 7 depicts the average resistance and resistivity for samples S2 and S3 after 3000 cycles of bending testing. In contrast to S1, the resistivity average increases after the bending test. The lowered resistivity is a consequence of the structure's extensive repair, which leads to the appearance of new dislocation-free grains [22].





**Fig. 7.** Resistance and resistivity before and after bending test on test samples for 3000 cycles

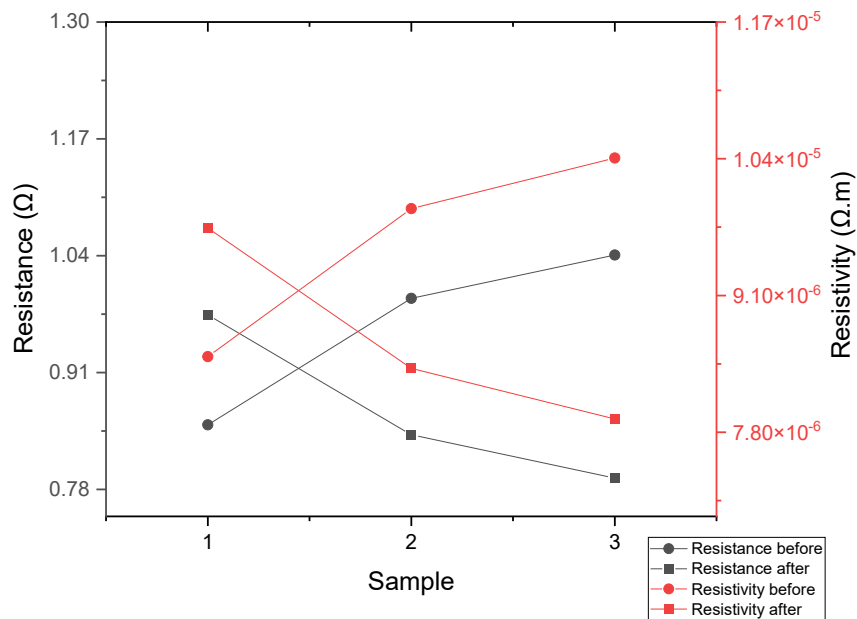
Table 5 shows the measured resistivity of the GNP hybrid before and after the 5000-cycle test. The average resistance and average resistivity for S1 increase after the testing. However, for samples S2 and S3, the resistance and resistivity decrease, respectively.

**Table 5**

Measurement of resistivity of the test samples for 5000 cycles of bending testing

Sample	Average Resistance (Ω)		Average Resistivity (Ω.m)	
	before	after	before	after
S1	0.852	0.974	8.519e-06	9.741e-06
S2	0.993	0.841	9.926e-06	8.407e-06
S3	1.041	0.793	1.041e-05	7.926e-06
Average	0.962	0.869	9.617e-06	8.691e-06

Figure 8 illustrates an increment in the average resistivity value for sample S1, notwithstanding the decrement in average resistivity for samples S2 and S3 after the bending test of 5000 cycles. The trend of the S1 resistance sample reading for these 5000 cycles is probably increasing due to the same reason as the previous cycle, where the ink on the sample decreases as the cycle increases.



**Fig. 8.** Resistance and resistivity before and after bending test on test samples for 5000 cycles

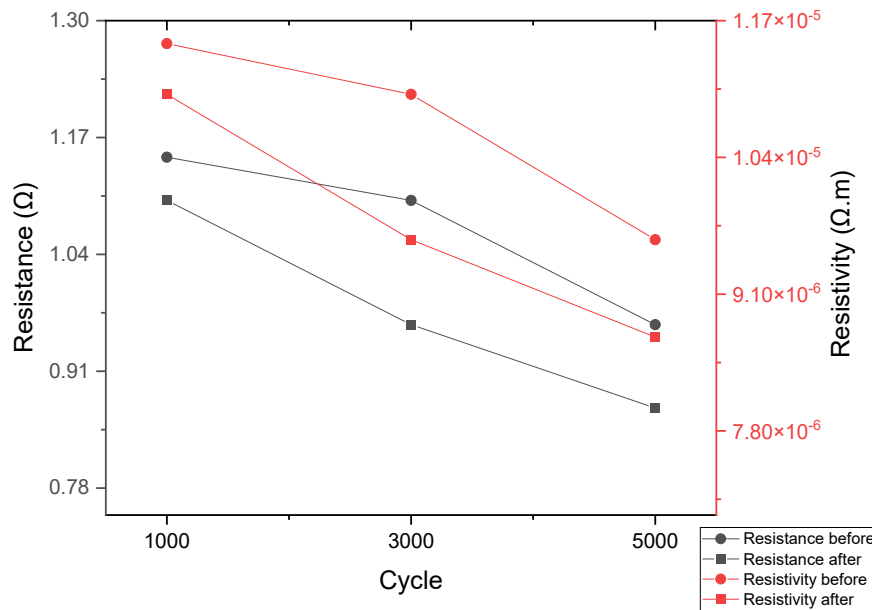
Table 6 displays the resistivity measurement data for all bending test cycles. The average resistance and resistivity for cycles 1000, 3000, and 5000 are higher than before testing. Each sample has a low standard deviation because graphene balances the pattern. The overall pattern of resistance remains fairly stable.

**Table 6**

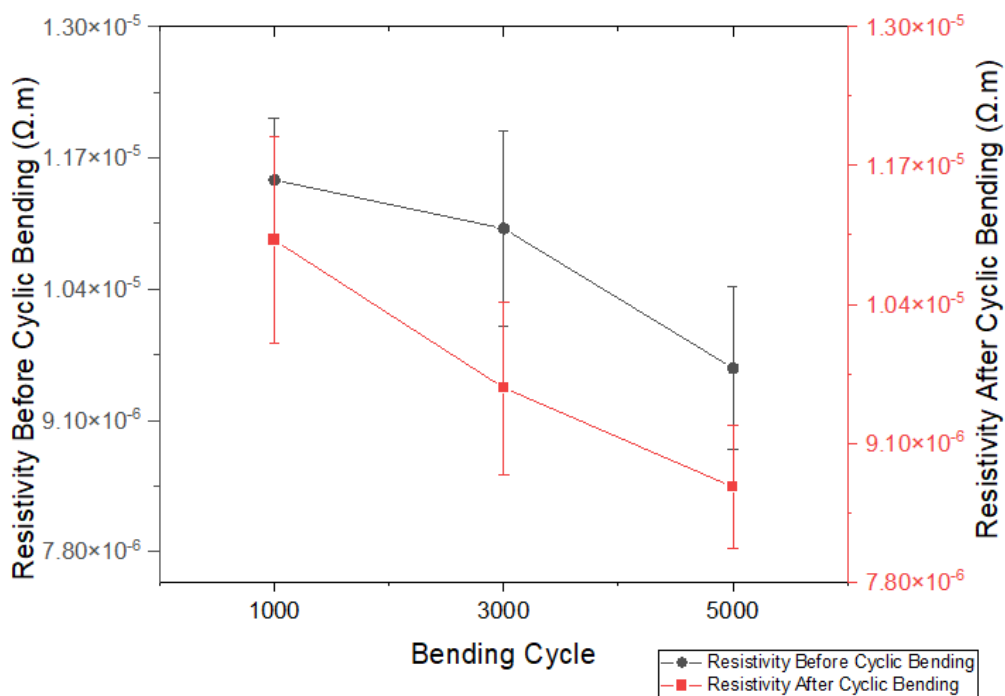
Measurement of resistivity of the bending test cycles

Cycle	Average Resistance (Ω)		Average Resistivity (Ω.m)		Standard deviation (Ω.m)
	before	after	before	after	
1000	1.148	1.100	$1.148 \times 10^{-5}$	$1.100 \times 10^{-5}$	$9.646 \times 10^{-7}$
3000	1.100	0.962	$1.100 \times 10^{-5}$	$9.617 \times 10^{-6}$	$8.0827 \times 10^{-7}$
5000	0.962	0.869	$9.617 \times 10^{-6}$	$8.691 \times 10^{-6}$	$5.734 \times 10^{-7}$

Figure 9 illustrates the pattern of the resistivity value before and after the bending test for the particular cycles. The pattern is presented along with the increment of the resistivity value and the gain number of cycles after the test. Meanwhile, Figure 10 shows the mean value of resistivity before and after bending at all cycles. The results shown in Figure 10 have the biggest standard deviation for resistivity after cyclic bending testing at 1000 cycles and for resistivity before cyclic bending testing at 3000 cycles as compared to the others.



**Fig. 9.** Resistance and resistivity before and after bending test on 1000, 3000, and 5000 cycles



**Fig. 10.** Mean value of resistivity before and after cyclic bending versus cycles

#### 4. Conclusions

The GNP are one of the active ingredients in producing conductive ink. The production of this conductive ink is expanded by mixing active materials such as metal-based inks, as an alternative for effective GNP development. In this study, GNP conductive ink was formulated using silver materials including silver flake (Ag) and silver acetate (SA), thus creating a hybrid conductive ink. The focus of this ink development is more on the preparation process and the investigation of resistivity and reliability. Comparisons were made by evaluating the resistance and resistivity values before and

after cyclic mechanical tests. Based on the study, three main conclusions were formed from the data of this research project:

- i. The GNP hybrid formulation at a curing temperature of 250 °C was an acceptable formulation due to the lowest resistivity value in the range of  $0.963 \times 10^{-5}$  to  $1.293 \times 10^{-5} \Omega.m$  at room temperature.
- ii. The behaviour of conductive ink was evaluated after fabrication using the electrical and mechanical tests for GNP hybrid conductive ink. The combination of graphene and silver conductive ink mixed with an organic solvent produced low resistivity and great flexibility. A new GNP hybrid composition minimized the amount of silver necessary to manufacture conductive inks.
- iii. Bending experiments on GNP hybrid conductive ink were undertaken to validate the resistivity relationship of the new conductive ink formulation. The resistivity values for each three samples of the bending test significantly change after 1000 cycles. The resistivity value on samples S1, S2, and S3 exhibited a reduction when applied to the bending test at 1000, 3000, and 5000 cycles.

In conclusion, the resistivity of GNP hybridization conductive ink on a flexible substrate is affected by various factors, such as bending and substrate composition. The bending test is a useful method to evaluate the mechanical and electrical properties of GNP ink and its suitability for flexible and stretchable electronic applications.

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