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Electrical Effects of GNP/Ag/SA Conductive Epoxy on Copper Flexible Substrate

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ABSTRACT

Various efforts to improve the performance of printed conductive ink have been conducted in order to obtain its full performance potential. This includes the investigation on the effect of conductive filler particle size towards the electrical conductivity performance. This study focuses on the product condition and electrical performance of graphene nanoplatelet (GNP), silved nano particle (Ag) and silver acetate (SA) as hybrid conductive filler components by varying the particle size of only one the filler component, which is GNP. The GNP/Ag/SA formulation with organic solvent was used by preparing three samples utilizing different GNP particle sizes of 5 µm and 25 µm. The purpose of this experiment is to collect data on the electrical conductivity of ink when employed in circuits and determine the best formulation of conductive ink. The obtained results revealed differences between the inks produced using 5 µm and 25 µm of GNP sizes. The resistivity of the 5 µm samples were was lower than 25 µm samples. The GNP of 5 µm recorded the lowest resistivity of 2.48 x 10-5 Ω.m as compared to 2.63 x 10-5 Ω.m recorded by 25 µm samples. The lower resistivity value indicates that the conductive ink formulated using 5 µm of GNP size has better electrical conductivity performance. The results signify that by using different particle sizes of only one component of hybrid conductive fillers produced different conductive ink performance, especially the electrical conductivity.

1. Introduction

In recent years, the utilization of electronic devices that employ electronic printing methods has become an important issue in developing future systems related to printing. Many researchers are constantly trying to understand and study the fundamentals regarding the fabrication of equipment using these printed electronics (PE) and the limitations of using mixed materials in terms of ink formulation in enhancing the capability in terms of electrical and mechanical aspects. The industry

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has also conducted development tasks in terms of application; however, the limitations in relation to the implementation of composite ink materials used have not yet been fully developed.

Printing electronic devices on various substrates is one of the printing technologies used to generate printed electronics (PE) [1,2]. According to current trends, advances in electronics production are rapidly driven by technology. New research is emerging and evolving in the direction of producing lighter and more flexible electronic hardware. Flexible print electronics (FPE) combines the benefits of both flexible and printed electronics [3-5]. Many countries throughout the world, including Malaysia, have extensively working on conductive ink in order to improve its properties so that it can be used and applied in a wider range of fields such as RFID, touch screens, electronic sensors, and printed circuit board (PCB) [6].

Conductive inks are becoming increasingly popular among electronic users. Printed electronics have emerged as a potential low-cost replacement for silicon-based electronics, and its complementary technology. Conductive ink (CI) can be distinguished by its flexibility and expandability while remaining resistively low. Low resistance changes the benefit of stretchability [7,8]. Printed electronics is also one of the fastest-growing technologies, with applications in healthcare, aerospace, advertising, and public transportation. Printed electronics was also effectively designed as a low-cost option for silicon-based electronics innovation and technology, [9,10].

In the past few years, more researchers have started to look into the comprehensive study and development of conductive ink technologies. The key objectives are to identify materials with extremely high electrical conductivity, particularly with strong mechanical resistance, environmental balancing, thermal resistance, and low corrosion [11,12]. One of the most important characteristics of conductive ink is its ability to have high conductivity while yet having good mechanical properties [13]. Conductive inks with good electrical and mechanical properties can be patterned using inkjet, screen, flexographic, direct writing, or gravure must be developed [14-16]. The printed graphene is shown in Figure 1.

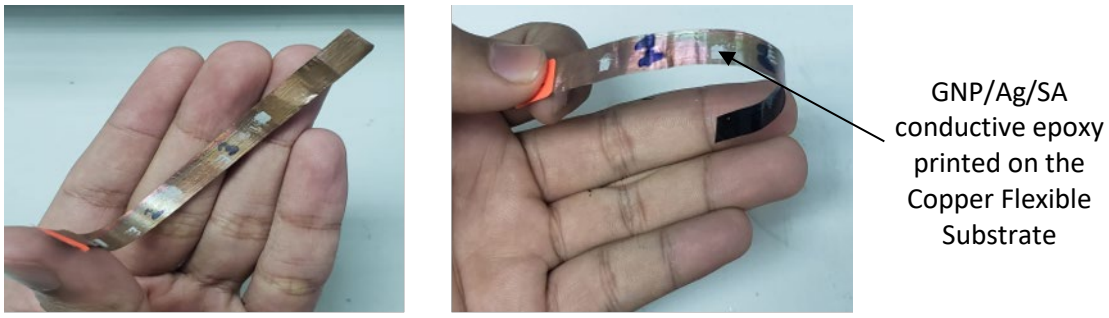


Fig. 1. Printed graphene at substrates

A conductive substance, a polymer binder, and a solvent are the three primary components involved in the production of an effective conductive ink. Conductive ink uses a variety of conductive materials, including metal-based compounds such as silver and copper, as well as carbon-based materials such as graphene and carbon nanotubes, and metal nanoparticles [17]. Furthermore, silver and copper are material options for varying conductivity in which copper oxidizes faster than silver.

Conductive inks based on graphene and carbon nanotubes, among other nanomaterials, are gaining appeal due to their high electrical conductivity and surface area [18]. Recently an increasing emphasis has been placed on the use of ecologically friendly conductive inks that use water as a solvent rather than organic solvents, which are hazardous to the environment. However, the high surface tension of water makes it unsuitable for use. Various natural and synthetic surfactants are

currently employed to lower water surface tension and ensure uniform dispersion of nanomaterials for smooth printing in a wide range of applications [19].

Furthermore, the epoxy based conductive ink from GNP/Ag/SA is most extensively used because of their high electrical and thermal conductivity, and also good in term of oxidation stability [3,11]. It has comparable performance from previous conductive ink such as silver flake and many more based on the zero-gap compound and it was being validated by the previous researchers [11]. The fillers been used in this study also has better chemical and mechanical stability comparing to other metallic materials. This study focuses on GNP/AgNP/SA because they are the main components of conductive ink, which function as conductive materials known as filler. It is excellent electrical properties, 5 µm and 25 µm graphene as fillers have good potential to improve the performance, functionality, and durability of various applications for next-generation electronic devices and energy storage devices. [20,21].

2. Methodology

Based on the results of the study, this experimental design was established to obtain the best formulation with GNP/AgNP/SA as a filler. The research started with formulation to create graphene powder and paste. It concentrated on GNP/AgNP/SA formulations with organic solvents or capping agents for application in conductive ink manufacturing procedures. This study focuses on certain organic solvent ratios with GNP formulation, silver flakes (Ag), and silver acetate (SA) paste.

In this study, silver flakes (Ag) were used as a filler loading in a hybrid formulation. When compared to other electrically conductive fillers, silver has the greatest potential for use as a conductive ink and adhesive due to its high electrical and thermal conductivities, chemical stability, low cost as compared to gold or graphene, and ability of its oxide form to conduct electricity. In hybrid formulations, silver acetate (SA) was used as a silver co-substrate to synthesize (Ag). (SA) is an appropriate precursor compound with a morphology that is controlled to produce Ag via thermal decomposition.

The 1-Butanol and terpineol were the primary organic solvents explored in this experiment, and the performance of the created pastes was evaluated in terms of product condition and electrical performance. Binders for GNP hybrid fillers were organic solvents that also operated as solvents. The goal of this study is to find out the resistivity properties of the GNP conductive ink composition. These qualities can be utilised as traits in the following studies. Finally, morphological observation was used to identify the microstructure of the conductive ink.

In this research, the following materials were used: GNP, Silver Flake, Silver Acetate, ethanol, 1-Butanol, and terpineol as organic solvents (see Table 1-7)

Table 1
The properties of 5 µm and 25 µm GNP powder

Specifications			
Grade	H	Grade	H
Form	Powder	Form	Powder
Colour	Black	Colour	Black
Surface area	50-80 m2/g	Surface area	50-80 m2/g
Average flake thickness	15 nm	Average flake thickness	15 nm
Average particle size	5 µm	Average particle size	25 µm
Density	0.03– 0.1 g/cm3	Density	0.03– 0.1 g/cm3

Table 2
The properties of silver flake

Specifications	
Assay	≥99.9% trace metals basis
Form	Flakes
Resistivity	1.59 μΩ.cm, 20 °C
Particle size	10 μm
Boiling point	2212 °C (lit.)
Melting point	960 °C (lit.)
Density	10.49 g/cm3 (lit.)

Table 3
The properties of silver acetate

Specifications	
Assay	≥99.9% trace metals basis
Form	Flakes
Reaction suitability	Core: Silver
	Reagent type: Catalyst
SMILES string	CC(O[Ag]) = O

Table 4
The specification of 1-Butanol

Specifications	
Colour	Colourless
Form	Form Liquid
Acidity as acetic	Acid ≤ 0.005%
Purity n-Butanol	99.9%

Table 5
The specification of terpineol

Specifications	
Mixture of isomers	65% α, 20 % γ, and 10 % β
Water content	<0.5%
Density	20 o/4° 0.934
Purity n-Butanol	99.9%

Table 6
The specification of Ethanol 99%

Specifications	
Colour	Clear colourless
Alcohol strength	99.5%
Acidity (as acetic acid)	50 mg/kg
Density at C/20	C 0.7883 – 0.7982 kg/l
Water content	0.3 wt.%

Table 7
The copper flexibility substrate

Specifications	
Density	8300 kg/m3
Thermal conductivity	401 W/m.K
Specific heat capacity	385 J/kg.K

The formulation of graphene nanoplatelets (GNP) was the study's initial step. The GNP/AgNP/SA formulation with an organic solvent or capping agent to be used in the manufacturing of conductive ink was subjected to the following inquiry. The study began with graphene nanoplatelets (GNP) formulation research focusing on the specific organic solvent ratio used in the paste formulation of GNP, silver flake (Ag), and silver acetate (SA).

The formulation of a powder GNP/AgNP/SA is important because it allows for the development of advanced materials with enhanced physical and chemical properties. This includes improved electrical and increased mechanical strength, and higher surface area for catalytic applications. The formulation also allows the production of GNP/AgNP/SA with tailored properties for specific applications. For example, a formulation of GNP/AgNP/SA can be tailored to achieve improved compatibility with a particular resin system. In addition, the tailored formulations can be designed to optimize the dispersion and orientation of the GNP/AgNP/SA in the composite matrix, leading to improved composite performance.

The formulation of the powder and paste graphene nanoparticles of GNP/AgNP/SA are shown in Table 8 and Table 9 below.

Table 8
Formulation of powder GNP/AgNP/SA

No	Item	Amount
1	Graphene Nanoplatelet (GNP)	0.05g
2	Ethanol	50ml
3	Silver Flakes	4.292g
4	Silver Acetate	0.42g

Table 9
Formulation of paste GNP/AgNP/SA

No	Item	Amount
1	Graphene Nanoplatelet (GNP)	4.56g
2	Butanol	26 drops
3	Terpineol	26 drops

The total value of the formulation had been decided before the process started. Graphene Nanoplatelets with the diameters of 5 µm and 25 µm were used as the main filler in this study. The dispersion of graphene was achieved by a simple method involving mixing, printing, and curing processes. Firstly, graphene with a diameter of 5 µm was weighed by using a digital analytical balance of about 0.05g. The same process was repeated for each case such as graphene diameter of 25 µm with different weight ratios depending on the mixture composition. The beaker was put at the center of the weighing pan and left for a few seconds for the unit to stabilize. In order to obtain accurate data when the substance was being weighed, the weight of the beaker was waived out by pressing the tare button until the display screen showed 0.0000 g.

The next step was adding 50 ml of Ethanol. The sample was protected with aluminum foil in order to run the sonication mixing using the ultrasonic bath. The process took around 10 minutes. The purpose was to disperse the GNP in a solution of ethanol and sonicate it to break up any agglomerates. After 10 minutes, silver flakes were mixed and sonicated using an ultrasonic bath and the process continued for one hour. After that, the same process was repeated for about one hour by mixing the silver acetate.

The next step to be taken was the stirring process by using magnetic stirring. The mixture was continuously stirred at 200 rpm until the mixture dried at a temperature of 70°C. After the whole mixture was dried, the mixture was put in the oven for one hour at a temperature of 250°C. Then,

the powder was dried at room temperature and allowed to cool. The last step was to use a mortar and pounded the mixture to become fine powder until fine. Figure 2 shows all the processes.

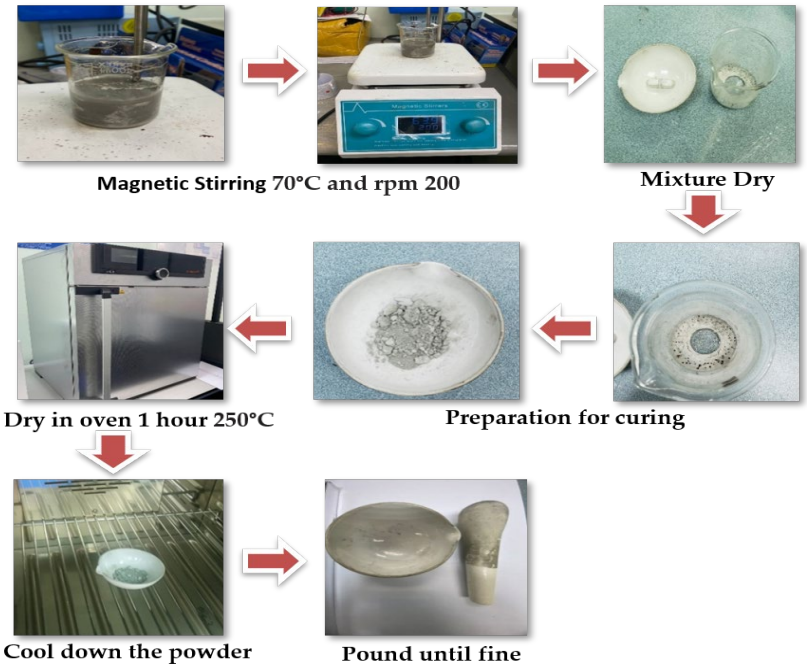


Fig. 2. Process for stirring, curing and pounding

Before the printing process begins, the process of creating ink paste in terms of preparing the formulation for the production of GNP/AgNP/SA must be completed successfully. After pounding the powder until became fine, it was weighed accurately to ensure that it was compatible with the formulation of mixing the butanol and terpineol solvents. By using a dropper, butanol and terpineol were added to the appropriate formulation, with the amount of 26 drops. The procedure was repeated by employing thinky mixer using the preset coordination. Figure 3 shows the entire GNP/AgNP/SA ink paste production process.

After completing all of the formulation processes for GNP/AgNP/SA powder and ink paste preparation, the printing process was completed by placing the conductive ink paste on the copper sheet using the mesh stencil method. The printing procedure began by applying the mixed paste to the chosen grid with a scraper. Once the paste was visible on the substrate, the sample was cured in the oven for 30 minutes at the set-up temperature of 250°C. Figure 4 shows the printing with a mesh stencil and Figure 5 shows the curing process. The analysis of particle shape, microstructure, and dispositions was part of the sample characterization. To determine the ink homogeneity, the surface microstructure of the printed samples was analysed using an image analyser. The resistivity values of samples were determined in an Ohms meter using a multimeter and mathematical equations. It is critical to understand the conductivity of the samples.

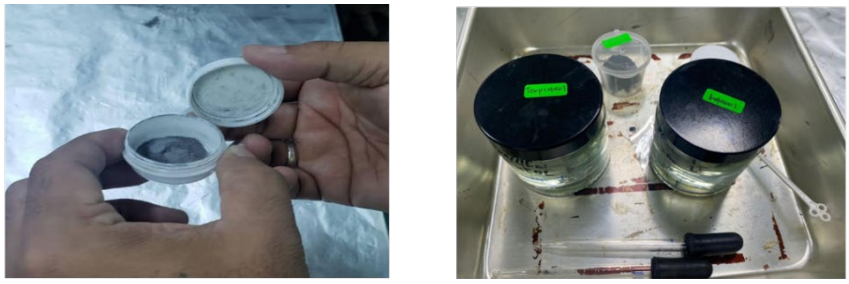


Fig. 3. GNP Hybrid ink paste production process

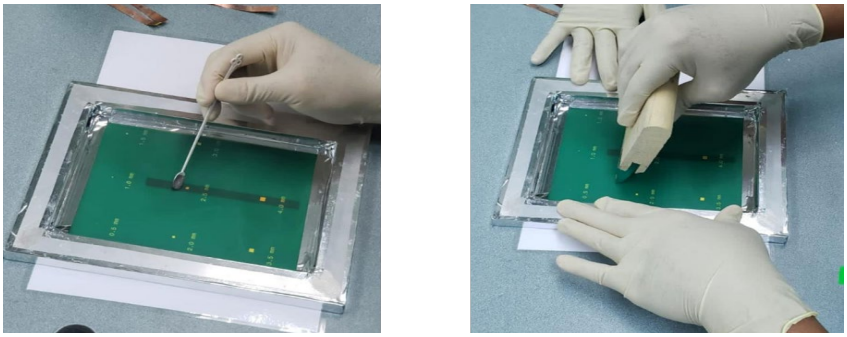


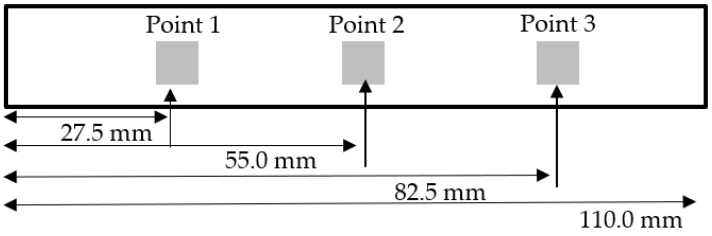
Fig. 4. Mesh stencil for printing method process

Fig. 5. Curing process

3. Results

Based on the results from the recorded data using a multimeter for resistance and the image from the microscope, the resistivity and microstructure of ink were discussed in order to determine the ideal ink formulation. Figures 6 (a) and (b) show the samples that already went through the initial process, from powder to paste and ending with the curing process. Three samples of 5 µm and 25 µm were used as test samples. Each sample has 3 points for the purpose of measuring the electrical properties as well as microstructure and behavior analysis.

(a)



(b)

Fig. 6. (a) Substrate stencil printing and (b) the schematic of printed sample ink on the substrate

The GNP/AgNP/SA initial values for the resistance were evaluated with a multimeter are shown in Tables 10 and 11. For the purpose of calculating the resistivity value, the average resistance value has been established. All of these sample images were also captured including the samples of 5 µm and 25 µm.

Resistance and resistivity are important parameters to be investigated because they are related to how electricity flows through materials. Resistivity is a measure of how easily an electric current flows through a material, whereas resistance is a measure of how much a material opposes the flow of an electric current [22]. When designing electrical circuits, the resistance and resistivity of a material are important because they determine how much current can flow through the circuit. Conductive ink is significant because it is a material that can conduct electricity. It is frequently used to create electrical circuits on surfaces such as printed circuit boards, which are found in a wide range of electronic devices. To ensure that the circuits work properly, the resistance and resistivity of the conductive ink must be carefully controlled. Furthermore, this experiment provides the electrical conductivity performance of GNP/Ag/SA conductive ink proportionally with all samples. It happened because conductivity depend of the electron mobility in the conducting materials with same dimension. The bigger electrical circuit will increase the possibility of the electron colliding with more ions and will increasing the resistance, and finally dropped the performance of conductivity.

By measuring electrical properties with a multimeter, the most important measurement that can be obtained is the resistance. Resistance measurements can be used to check the accuracy of a resistor or to ensure that it is functioning properly, but they can also be used in a variety of other situations and enable real-time monitoring and recording [23] It can also be used to measure the resistance of unknown conductors and to check the short circuits and open circuits. There are numerous situations in which the measurement of resistance is both interesting and important. In all these cases a multimeter is the appropriate test equipment to measure resistance.

Measuring resistance using all types of multimeters, whether analogue or digital follows the same principle. In fact, other types of resistance testing equipment follow the same basic principle. The basic idea is that the multimeter applies a voltage to the two probes, causing a current to flow in the item being measured for resistance. It is possible to determine the resistance between the two probes of a multimeter or other piece of test equipment by measuring the resistance. Due to

resistance-capacitance delay, a longer point is required to obtain the resistivity value for high resistivity samples [24,25]. The problem is that the current takes some time to reach the saturation level. Once the data has stabilised, only specific measurement points can be taken and the average value obtained. When time is spent inconsistently, it produces unstable resistivity values [17].

Table 10
Initial values for the resistance and resistivity of 5 µm

Sample 5 µm		Resistance (Ω)				Average Resistance (Ω)	Resistivity (Ω.m) x 10^{-5}
Sample 1	Point 1	0.8	1	1	1	0.95	2.85
	Point 2	0.8	0.9	0.9	0.8	0.85	2.55
	Point 3	0.8	0.8	0.9	0.9	0.85	2.55
Sample 2	Point 1	0.9	1.2	0.8	0.8	0.93	2.78
	Point 2	0.9	0.8	0.8	0.8	0.83	2.48
	Point 3	0.9	0.8	0.8	0.8	0.83	2.48
Sample 3	Point 1	0.9	0.8	0.8	0.9	0.85	2.55
	Point 2	0.8	0.9	0.8	0.8	0.83	2.48
	Point 3	0.8	0.8	0.8	0.9	0.83	2.48

Table 11
Initial values for the resistance and resistivity of 25 µm

Sample 25 µm		Resistance (Ω)				Average Resistance (Ω)	Resistivity (Ω.m) x 10^{-5}
Sample 1	Point 1	1	1.1	0.9	0.8	0.95	2.85
	Point 2	1	0.9	1.1	0.9	0.98	2.93
	Point 3	0.9	0.9	1	1	0.95	2.85
Sample 2	Point 1	1	1.1	0.9	0.9	0.98	2.93
	Point 2	1	0.9	0.9	0.9	0.93	2.78
	Point 3	1	1.1	0.9	0.9	0.98	2.93
Sample 3	Point 1	0.9	0.9	0.9	0.9	0.90	2.70
	Point 2	0.8	1	0.9	0.8	0.88	2.63
	Point 3	0.9	0.9	1	0.9	0.93	2.78

The measurement of resistivity value was based on data involving 5 µm and 25 µm. This means that each sample has its conductivity level. This is due to the agglomeration effect caused by a small amount of ink loading. The relatively thin screen-printing process on sample 1 point 1 at 25 µm produced the same result as the 5 µm sample. However, the resistivity of 5 µm sample increases because the printing process on the substrate is very good and fills the entire stencil space. It produces the highest value when compared to other values. Figure 7 shows the resistivity and resistance for GNP/AgNP/SA samples.

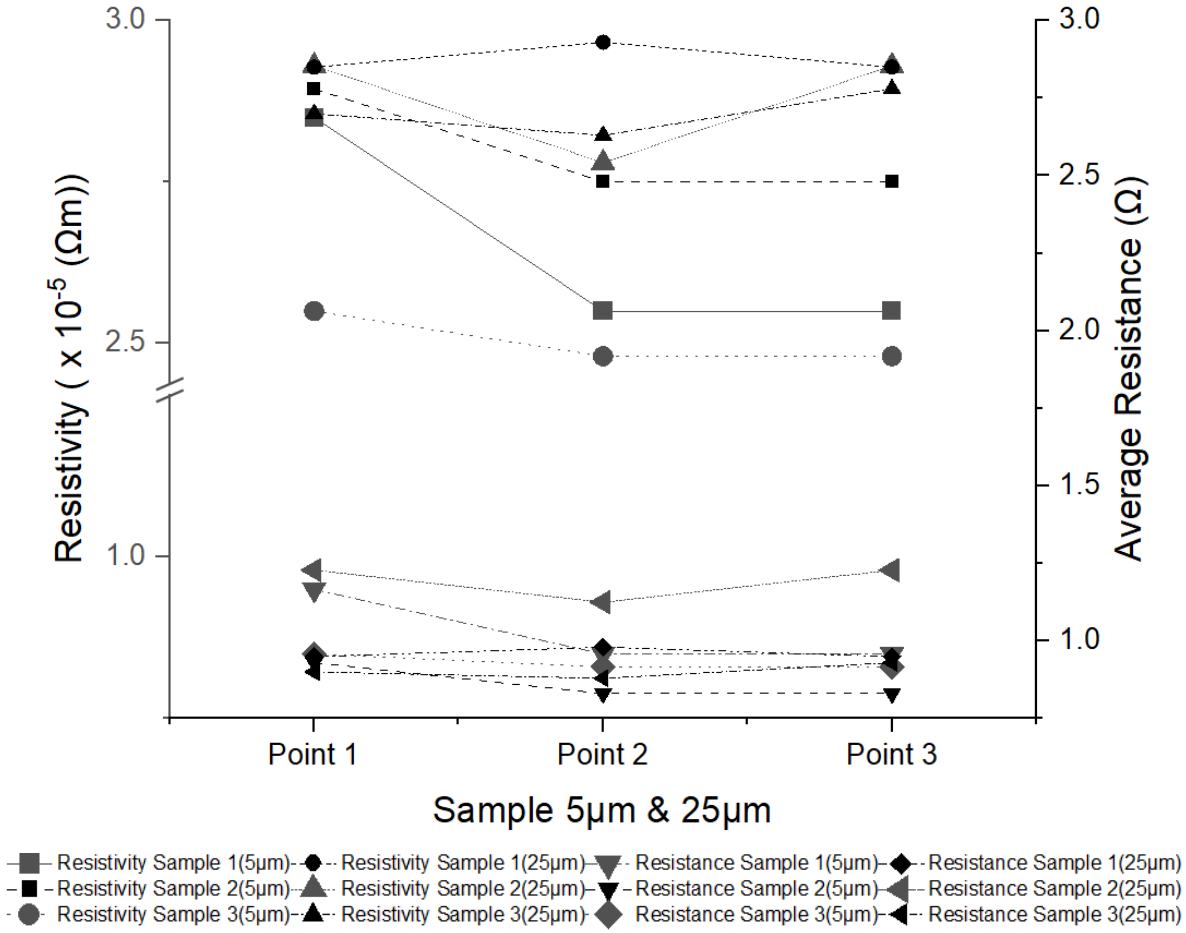


Fig. 7. The resistivity and resistance for GNP samples of 5 µm and 25 µm

Furthermore, the 25 µm samples produce contradictory results. The overall resistivity of 25 µm demonstrates the relatively high resistivity value which is due to the inability to form an ink mixture, it is because the combination of the sample of material produces powder during the mixing, sonication, and stirring processes [26,27]Physical dimensions of a material, such as thickness or cross-sectional area, can influence the resistance and resistivity. Resistance and resistivity differ at different thicknesses due to the differences in the amount of material present and the number of conductive paths available to conduct electric current [28]. The resistance and resistivity of 5 µm samples differ from those of 25 µm samples. This is due to the fact that a thicker material has more material and possesses the availability of more conductive paths for the flow of electrical current, resulting in lower resistance and resistivity.

During the screen-printing process, it is possible that the ink is not evenly distributed throughout the stencil space. Because of the speed during the screen-printing process, there are some uneven spaces when the squeegee moves across the gap [29]. This also affects the readings from the 3 of 5 µm and 25 µm samples. As the squeegee's speed increases, the ink disappears and is unable to cover all of the gaps. However, the average particle size of 5 µm and 25 µm indicates that their resistivity values are at the lowest level compared to both samples. The melting temperature and resistivity are also affected by the particle size. [30]

Microstructure analysis is an effective tool for improving ink screen printing. It can be used to identify potential defects such as poor adhesion, poor ink transfer, erosion, and printing flaws. These happen usually during the curing process. However, this defect can be corrected during the pre-

printing step [31]. Furthermore, microstructure analysis can be used to evaluate the quality of the screen mesh, ensuring that it is defect-free and capable of producing consistent, high-quality prints. To investigate the microscopic condition of the ink, an image analyser is used to determine the image analysis. All microstructure images in this section are divided into two sample categories namely average particle size of 5 µm as shown in Figure 23 and the average particle size of 25 µm as shown in Figure 24.

This microstructure image analysis is organised according to 50 times magnification, which magnifies the objects 50 times larger than what the naked eye can see. The diagram depicts various microstructure images, particularly particle shapes and average particle sizes. Figure 8 shows the fine surface texture conditions for 5 µm. Figure 9 shows that the surface of the 25 µm sample is rough and uneven, with open gaps everywhere. The size selection process is important to obtain maximum conductivity and get good performance, rather than making an inaccurate size selection for the graphene [32]. This demonstrates that a larger average particle size of graphene produces a very significant difference in the final result when doing screen printing and subsequently causes a difference in the measurement data.

In addition, the microstructure shows the homogenous composition of the GNP/Ag/SA conductive ink. It is well-distributed arrangement for all particles. The homogenous composition produces less gap between the conductive particles, which is allow the movement of electrons to flow the activation energy such as current flow between the particles. Thus, the microstructure provide qualitative proof of the performance of the conductive ink.

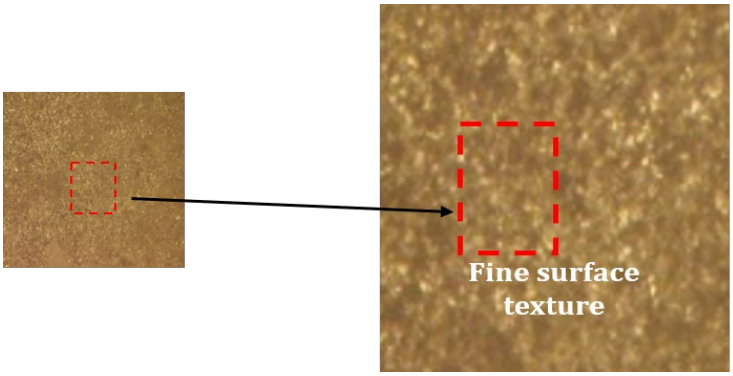


Fig. 8. Microstructure image of graphene average particle size of 5 µm

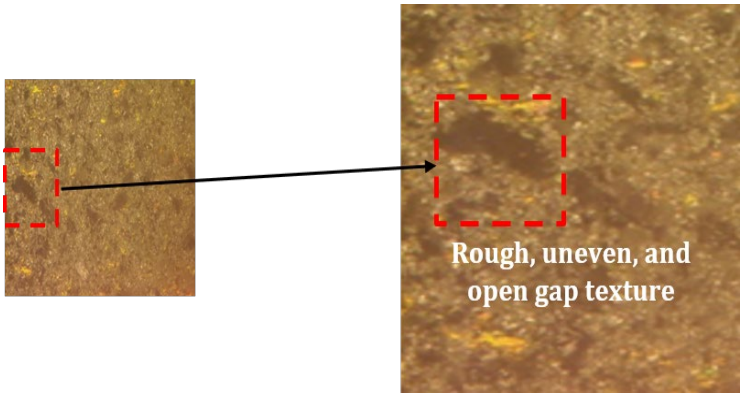


Fig. 9. Microstructure image of graphene average particle size of 25 µm

4. Conclusions

Graphene is one of the carbon-based materials that has been used in conductive inks because of its excellent electrical conductivity. Graphene also has a very high resistivity, which makes it an ideal material for use in a variety of applications. In this study, the screen-printing method was used to make test samples with several different parameters such as particle size of 5 µm and 25 µm. All test samples were characterized in a static state to determine the resistivity as well as to analyse the microstructure behavior. Bulk resistance was measured with a multimeter. The samples have different data and results even though the formulation process in producing the ink paste was made with the same method. The most significant difference was in terms of average particle size which has a different effect on the resistivity of each sample. Particle size also affects the contact area and electrical flow between conductive ink particles. It also shows the difference in the microstructure analysis behavior of the samples. There are several factors that cause the resistance in the conductive ink to increase such as constriction resistance and tunnel resistance. It results from the production, especially the squeegee speed during printing. There are gaps and uneven surfaces that cause the resistance, which is reflected in the value of each sample. However, the difference in terms of particle size of each sample gives a significant difference in the resistance value and the resistivity which involves the particle sizes of 5 µm and 25 µm.

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