ELECTRICAL, MORPHOLOGICAL AND SURFACE ROUGHNESS ANALYSIS OF SILVER NANOPARTICLES-FILLED EPOXY CONDUCTIVE INK

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ABSTRACT

Conductive ink has become a potential alternative to replace the conventional circuitry in electronic applications. Due to this, various efforts have been conducted to obtain the optimum ink formulation that can fulfill the current demand. The objective of this study is to determine the performance of different formulations of silver nanoparticles-filled epoxy conductive ink with various filler loadings in terms of electrical conductivity. The main investigated parameter was the sheet resistance of the composition. The changes of morphology of the ink surface and surface roughness were also examined, which directly correlated to the sheet resistivity. The obtained results showed that the minimum threshold of silver nanoparticles (AgNP) filler required was 60 %wt for the ink to conduct electricity. However, this filler loading was not acceptable because of the wide dispersion of data. The ink filler loadings that can conduct electricity also showed the presence of granular particles on the ink layer surfaces, which also increased their surface roughness. The sheet resistance value also achieved a saturated value with filler loading of 90 %wt. It means that further addition of filler loading is not going to further improve the sheet resistivity of the composition. Based on this study, it can be summarised that the percentages of AgNP filler loadings of conductive ink that can fulfil the acceptable performance are between 70 to 90 %wt.

Keywords: *Silver nanoparticles conductive ink; resistivity; morphology; surface roughness; filler loadings*

1. INTRODUCTION

Conductive ink has been considered as one of the most magnificent findings in the field of electronic circuitry. It can be used to replace conventional wiring and is beneficial for simple circuitry that requires low-manufacturing costs (Mokhlis *et al*., 2020). Recently, there have been many other various applications of conductive ink, such as for the construction of batteries (Kazemzadeh *et al.*, 2020), supercapacitors (Lehtimäki *et al*., 2014) and sensors (Lynch *et al*., 2020). With the advancement of the technology and more research efforts being conducted in this field, it opens more possibilities of performance enhancement of conductive ink. Various factors can contribute to the performance of conductive ink and the main factor is the filler of the ink. It plays an important role for conducting electricity by allowing the electron flow between the filler particles with low resistivity (Ismail *et al.*, 2020). Furthermore, the composition and printing methods are also important in determining the performance of the conductive ink (Yunos *et al*., 2020).

Silver nanoparticles (AgNP) have been used as the filler of the composition of conductive ink. It is due to the well-known inherent properties of silver as a good electrical conductor. Besides that, it also possesses excellent optical and chemical properties (Bose *et al*., 2018). They also possess high sintering efficiency (Huang *et al*., 2014), undemanding sintering conditions (Chen *et al*., 2020), and chemical stability (Titkov *et al*., 2018). Unfortunately, the requirement of having heat treatment

through sintering limits the utilisation of substrates to be used with the conductive ink. This heat treatment is required to remove hazardous organic solvents, such as toluene, xylene and alkane (Park *et al.*, 2018). However, AgNP has high formulation stability and can be printed using various different printing techniques (Kosmala *et al*., 2011).

For the preparation of AgNP filled epoxy conductive ink, various types of printing methods have been introduced by previous studies. These include inkjet printing, gravure printing and screen printing (Saad *et al*., 2020). For the purpose of this study, it does not require a high resolution of ink pattern. Due to this, the simplest method of printing is utilised to produce the ink tracks on the substrate, which is the doctor blade method. It can be considered as a practical option because of the capability to produce a thick film in a single pass. This reduces tremendously the processing time (Rajan *et al*., 2016).

In studying the performance of AgNP filled epoxy conductive ink, some important parameters need to be examined as the baseline for more advanced investigation works. The first is the minimum content of AgNP in the ink composition that has the capability to work as conductive ink. For this, the measurement of sheet resistivity is performed as it can directly translate to the capability of the ink formulation to conduct electricity. In contrary to metal to metal contact that produces contact resistivity, the more prominent parameter that influences the electrically conducted material for the thin sheet is sheet resistivity (Peng *et al*., 2015). Then, morphological analysis is performed to study the microstructure of the ink layer surface that contributes to the capability of conducting electricity. Different ink formulations produce different ink layer microstructures. In addition, most of the morphological analysis is carried out by referring to the captured images of the microstructure. It requires changes in the physical structure of the ink to explain the results of the analysis. Therefore, surface roughness analysis is performed to obtain the correlation between the morphology of the ink with its physical attributes (McGrath *et al*., 2021).

This study aims to provide initial insight of using AgNP as the filler element for the production of conductive ink. A stable ink formulation that has the capability to conduct electricity was obtained by determining the sheet resistance of the ink formulation at different filler loadings. Then, morphological analysis was performed to study the microstructure of the ink layer that contributes to the electrical conductivity capabilities. The morphological analysis was conformed to the physical features of the ink layer by obtaining the surface roughness of the ink with different filler loadings.

2. METHODOLOGY

2.1 Test Samples Preparation

The material used for this experiment is AgNP as the filler element, epoxy as the binder, and hardener to harden the mixture composition. The composition weight of the ink loading shown in Table 1 was used to produce the total weight of 2 g for each sample.

The mixture composition was blended using the manual stirring process with a glass rod. For filler loadings of 10 to 50 %wt, they were stirred for 5 min at room temperature, while for filler loadings of 60 to 90 %wt, the stirring time was increased to 10 min. This was to ensure that consistency of the mixture due to the increased viscosity of the composition with the increment of filler loading (Htwe *et al*., 2019). Then, the produced ink was printed on the glass substrate using the doctor-blade technique. It was performed by covering the whole glass slide with cellophane tape except for the opening gap for the printed ink. Then, the ink was dropped on the glass slide and a sharp blade was used to spread the ink by moving across the substrate at a constant speed and filling in the gap.

The printed ink track is illustrated in Figure 1. For each sample, two tracks were printed, which were labelled as Tracks A and B. For each position, three points were determined for the measurement. Overall, six points were investigated for each sample. The purpose of having two ink tracks for one glass slide substrate is to examine the homogeneity of the conductive AgNP in the mixture by using a

limited amount of formulated mixture. It is one of the main characteristics of determining the electrical conductivity of mixture composition (Cai *et al*., 2019). Then, the samples were cured in an oven at 160 ºC for 1 h. The purpose of the curing process is to anneal the ink mixture by removing the excess solvent that can become an obstruction to the conductive network paths, which results in better electrical conductivity. Furthermore, it can enhance the adhesion of the ink tracks to the substrate (Htwe *et al*., 2019).

Table 1: Composition of ink loading.

Sample	Filler		Binder		Hardener (g)	Total (g)
	(%)	(g)	(%)	(g)		
1	10	0.2	90	1.8	0.54	2
2	20	0.4	80	1.6	0.48	2
3	30	0.6	70	1.4	0.42	2
4	40	0.8	60	1.2	0.36	2
5	50	1.0	50	1.0	0.30	2
6	60	1.2	40	0.8	0.24	2
7	70	1.4	30	0.6	0.18	2
8	80	1.6	20	0.4	0.12	2
9	90	1.8	10	0.2	0.60	2

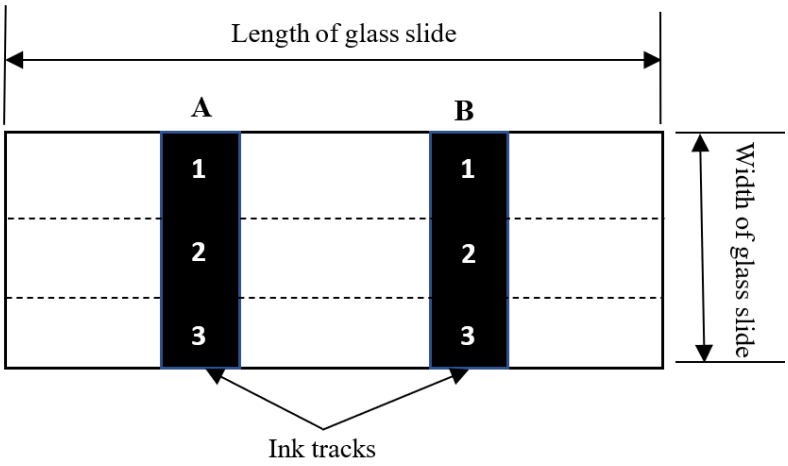


Figure 1: Printed ink tracks on the glass slide substrate.

2.2 Measurement of Sheet Resistivity

For the sheet resistivity measurement, a four-point probe was used. This is because it is suitable for low resistivity thin film and its independence of the square (Ismail *et al*., 2020). For each point, three sheet resistivity readings were taken. Then, the standard deviation of the data was calculated to obtain its dispersion behaviour. Then, the estimation of error (*E*) for the sheet resistivity values were calculated using the following equation:

$$E = \left| \frac{Resistivity\ at\ one\ point - Average\ resistivity}{Average\ Resistivity} \right| \times 100\% \quad (1)$$

2.3 Surface Morphology

For the morphological analysis, the microscopy technique was used to obtain the images of the ink layer surface. Three different resolutions were used to capture the image for every investigated point, which where 5x, 10x and 20x. All the images were recorded in the computer attached to the microscope. Surface morphology is a qualitative analysis by utilising the graphical images of the ink layer surface in terms of the particles' size, shape, profile and structure. With the addition of different filler loadings, it changes the ratio of material composition. Therefore, it affects the atomic and molecular arrangement of the compound (Kulkarni, 2014), which is also portrayed on the ink layer surface. The correlation between the changes of microstructure surface and sheet resistivity can then be obtained.

2.4 Surface Roughness Measurement

For surface roughness, the measurement was taken using a surface roughness sensor. It was performed by moving the sensor probe along the ink layer surface. For this study, two directions of the movement were used, which were the horizontal and vertical directions. Horizontal direction indicates movement along the length of the glass slide, while vertical direction indicates movement along the width of the glass slide. For every investigated point, three surface roughness measurements were taken to increase the accuracy and reliability of the obtained data. Surface roughness measurement can provide qualitative data in reinforcing the surface morphological analysis to determine the effect of filler loadings on sheet resistivity (Buszewski *et al*., 2010). It has a direct correlation with the presence of AgNP granules on the ink layer surface.

3. RESULTS AND DISCUSSIONS

3.1 Sheet Resistivity

The sheet resistivity of different points for different percentages of filler loading is shown in Figure 2. The conductivity of composites is not a straightforward phenomenon. As mentioned by Merilampi *et al*. (2009), there several different controlled parameters involved such as conductive filler, surface resistance of the filler and hopping conductivity. This experiment attempts to discover the direct correlation between the amount of conductive filler and sheet resistivity behaviour. Even though the experiment was carried out for filler loadings starting from 10 until 90 %wt of AgNP, the sheet resistivity results can only be obtained for the filler loadings starting from 60 to 90 %wt. This is because low content of filler loading does not produce any electrical conductivity.

The presence of AgNP in the composition is inadequate to produce a continuous conductive path that allows the electrons to flow in the composition, which produces sheet resistivity. By adding the amount of AgNP in the conductive ink composition, it increases the network path of silver particles and effectively allows the current to flow (Mou *et al*., 2020). Thus, by having more amount of AgNP inside the composition, it reduces the sheet resistivity. This is proven by the collected data, which shows that filler loading of 60 %wt produces the highest sheet resistivity both for Tracks A and B with values of 150.53 and 369.65 Ω/sq respectively. With the addition of filler loading to 70 %wt, it significantly reduces the values of sheet resistivity. Consequently, the values of sheet resistivity for filler loadings of 80 and 90 %wt also show substantial reduction but with minimum difference between Tracks A and B. The reduction trend as illustrated in Figure 2 also shows that the curve achieves a plateau at filler loading 90 %wt. This indicates that sheet resistivity already has achieved a saturated level and any addition of AgNP in the ink mixture composition of more than filler loading of 90 %wt will produce insignificant improvement in sheet resistivity. It only increases the cost of using an unnecessary amount of AgNP in the composition.

The dispersion of sheet resistivity data is shown in Table 2. It shows that filler loading of 60 %wt has the worst data disparity as compared to other the filler loadings. By increasing the filler loading to 70 %wt, it significantly reduces the values of standard deviation. High standard deviation values indicate that the sheet resistivity values are scattered in a wider range across all the examined points. It implies

inconsistency of AgNP in the composition, whereby some portion of the ink paste has a higher content of AgNP as compared to others. This occurrence is because of the limited amount of AgNP in the mixture. Furthermore, it shows the preparation method used, in particular the manual mixing process is unable to create consistent distribution or dispersion of AgNP inside the compound (Mou *et al*., 2020). The value for filler loading of 60 %wt should become the low threshold value for filler loading when preparing AgNP filled conductive ink, which is prepared using a manual mixing process. Even though it can measure the occurrence of electrical conductivity and generates sheet resistivity, the data dispersion is unacceptable to be used for further analysis. On the other hand, filler loading of 90 %wt shows the smallest values of standard deviation. It indicates the minimum distribution of sheet resistivity as compared to the average values. This is caused by the increased amount of conductive material inside the composition, which allows consistent values of electrical conductivity to exist at all measured points of the sample (Merilampi *et al*., 2009). Furthermore, the very low values of sheet resistivity generated by filler loading of 90 %wt also contribute to the low dispersion data.

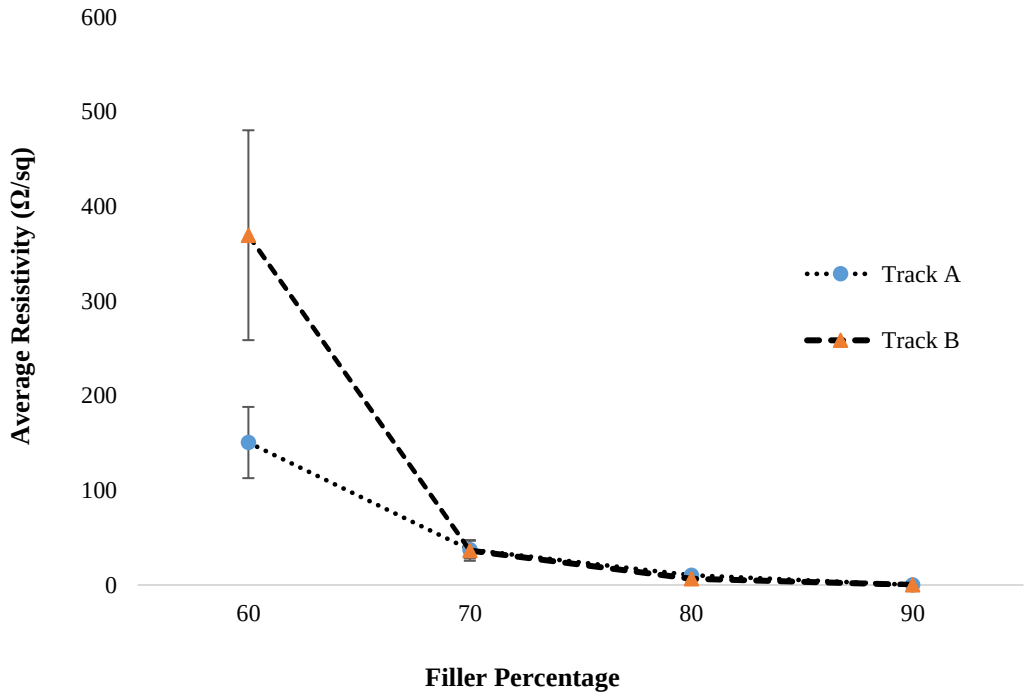


Figure 2: Average sheet resistivity vs filler percentage.

Table 2: Standard deviations of sheet resistivity for different filler percentages.

Filler (%)	Track	Standard Deviation			Overall Standard Deviation
		Point 1	Point 2	Point 3	
60	A	12.16	25.66	101.35	77.82
	B	13.11	59.30	147.68	289.86
70	A	2.70	3.92	5.19	2.69
	B	20.39	4.85	6.31	7.20
80	A	4.67	2.29	4.54	5.29
	B	0.83	0.81	2.56	1.04
90	A	0.04	0.00	0.01	0.08
	B	0.07	0.08	0.03	0.03

The data of estimation of error for sheet resistivity at every measured point is tabulated in Table 3. It is expected that low filler loading produces higher error and vice versa. However, the obtained data shows a random pattern of estimation error. This occurrence is caused by two factors, which are the printing technique and the measurement using the four-point probe. The doctor-blade printing technique requires consistent speed to ensure that the ink is well-distributed in the required gap to produce the ink strips. When the movement speed is too high, it causes ink loss and inaccurately fills the gap (Khirotdin *et al*., 2017). Furthermore, it produces inconsistent thickness of ink, which leads to uneven distribution of ink content as shown in the surface microstructure images in Figures 3 and 4. On the other hand, the measurement using a four-point probe requires delay time for the readings to become stable before they can be recorded. This is due to resistance-capacitance (RC) delay, especially for high resistive samples as the current requires some time to climb up to the saturation value.

Table 3: Estimation of error percentages of sheet resistivity at different filler percentages.

Filler (%)	Average Estimation of Error (%)					
	Track A			Track B		
	1	2	3	1	2	3
60	9.99	21.19	32.24	6.42	17.48	16.24
70	40.48	35.95	35.68	40.63	28.21	40.79
80	50.92	32.12	30.73	27.58	27.44	36.69
90	27.58	0.00	33.33	53.33	31.25	30.56

3.2 Surface Morphology

Morphological analysis was carried out by investigating the surface microstructure images of the ink samples. It is divided into two separated tabulated images, which are the microstructures with no electrical conductivity and microstructures with electrical conductivity. Figure 3 shows the ink microstructure for filler loadings from 10 to 50 %wt that produce no electrical conductivity. In general, the presence of AgNP can be recognised with the formation of dark spots in the microstructure images. For filler loading of 10 %wt, almost no trace of AgNP can be found. The figure portrays light-coloured images, which indicate that the amount of binder and hardener overwhelms the quantity of filler in the composition with the formation of voids. Due to this, it produces no electrical conductivity. Meanwhile, for filler loadings of 20, 30 and 40 %wt, they show scattered existence of AgNP, with these particles portrayed as dark spots in the microstructure images. The frequency of gaps between the silver nanoparticles can affect the electrical properties of the conductive ink layers. The increased gap distance prevents the formation of conductive path and causes the resistance to increase due to the high voltage required to ensure there is a current flow among the AgNP particles (Woo *et al*., 2013). For filler loading of 50 %wt, even though it has the same ratio of filler to the binder, the presence of AgNP is still insignificant. It is represented with very light-coloured microstructure images to indicate the scarce existence of AgNP particles in the composition.

Figure 4 shows the microstructure images for the samples with filler loadings of 60 to 90 %wt that produce electrical conductivity. Based on these images, the samples with filler loadings of 60 to 80 %wt show the presence of granular-like particles on the ink layer. These granular particles contain the 3D connection of conduction, which leads to the existence of particle necking. These particles become conductive although they possess porous characteristic because the growth of the interparticle neck produces the continuous connection between particles (Park *et al.*, 2007). The comparison between these samples of different filler loadings does not display significant difference in terms of microstructure, either for size or shape of the particles.

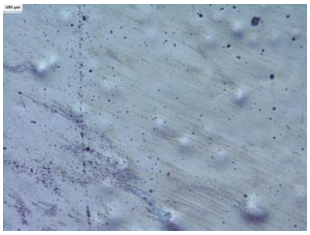
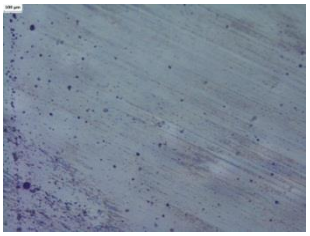
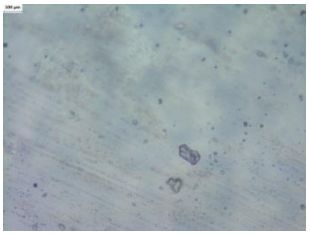
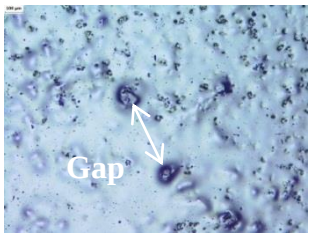
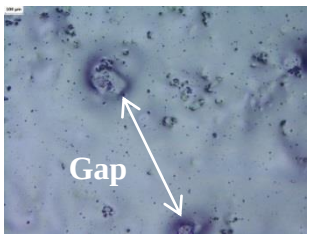
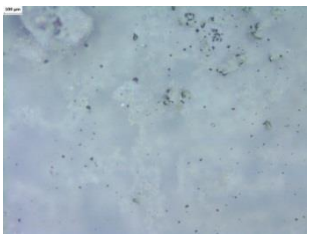
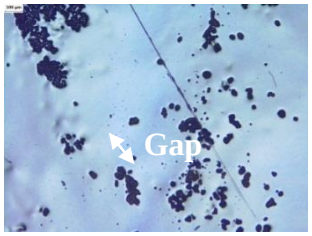
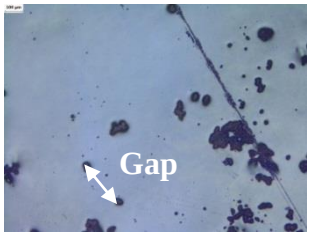
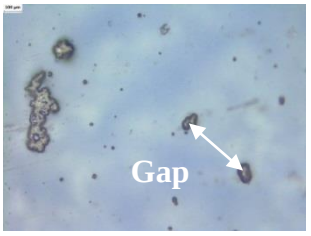
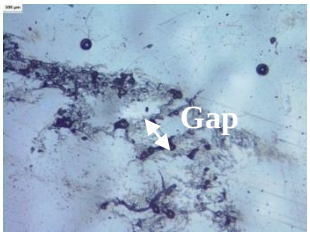
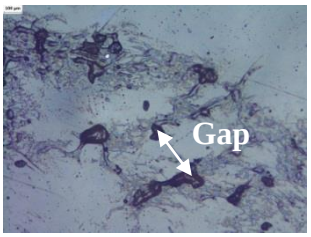
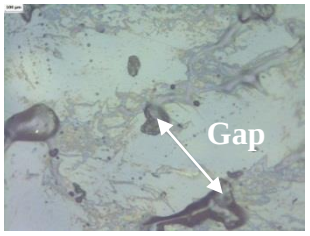
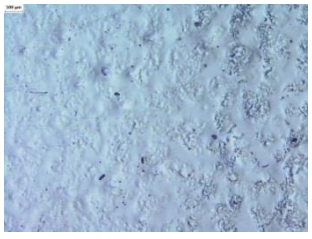
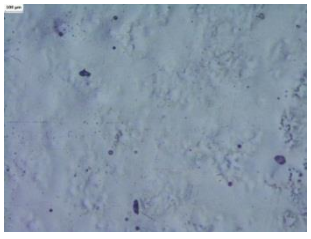
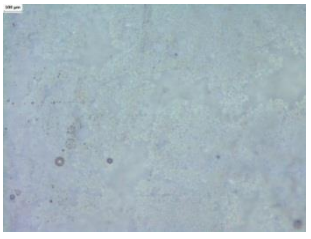
Filler (%)	Magnifications		
	5x	10x	20x
10			
20			
30			
40			
50			

Figure 3: Microstructures with no conductivity.

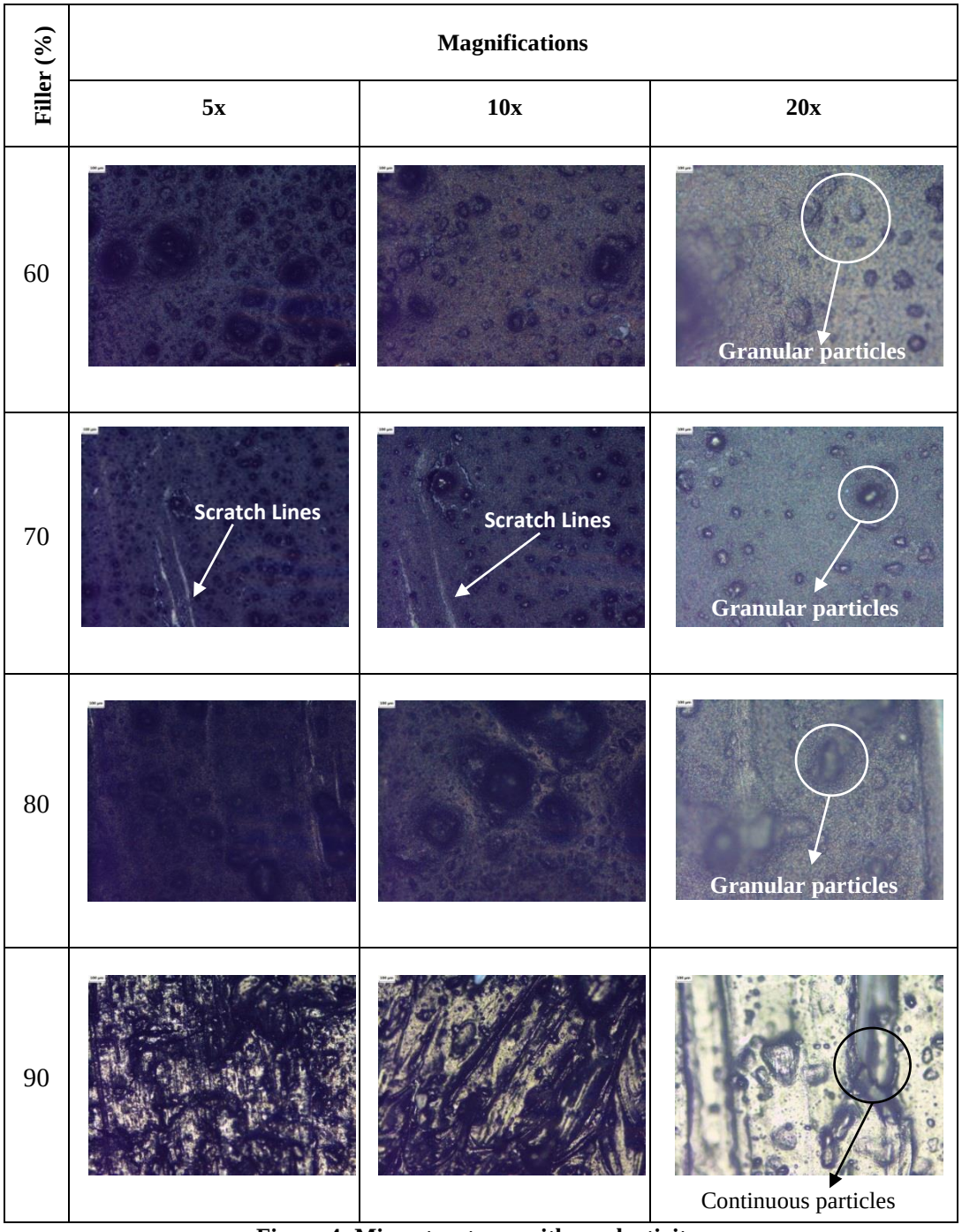


Figure 4: Microstructures with conductivity.

For the filler loading of 70 %wt, it shows evidence of scratch lines on the ink layer surface. This defect is caused by the printing process, where excessive pressure was applied on the razor blade when spreading the ink on the substrate. The filler loading of 90 %wt shows a more obvious presence of AgNP on the ink layer. The increased amount of AgNP creates ink layers with a close-packed structure, where the particles create strong bonding between each other. This is displayed by the continuous particles in the microstructure image to illustrate the contact of particles with each other rather than discrete and spherical shape of particles, which increases the contact area between particles (Zhou *et al*., 2014).

The increase of electrical conductivity because of the increased particle contact area can be described by the percolation threshold. This is because several conducting bridges are formed and an electrical conductivity higher than several orders of virgin polymer is manifested (Khan *et al*., 2019). However, Figure 4 also shows the heterogeneity of AgNP dispersion in the composition. It indicates that the electrical conductivity of the composition can be further improved by forming a more homogenous mixture with a better molecular arrangement (Kulkarni, 2014). One of the methods that this can be done is by improving the annealing process with more suitable operating parameters for the composition. The precise heat addition provides enough molecular energy to form a homogeneous molecular arrangement and prevents agglomeration (Jiang *et al*., 2013).

3.3 Surface Roughness

The measurement of ink layer surface roughness of the samples in this study was conducted for two different movement directions, which were vertical and horizontal directions. The purpose is to eliminate the inconsistency of the obtained data that is sourced from the inaccuracy of the printing technique. Figure 5 illustrates the data and trend of average ink layer surface roughness for the different filler loadings. For the samples with low content of filler loading that do not have the capability to conduct electricity, which are from 10 to 50 %wt, they produce relatively small values of average ink layer surface roughness. The recorded average values are in the range of 0.2 to 0.5 µm. However, with the increment of filler loading starting from 60 %wt, which in the range that the ink composition can conduct electricity, the values of surface roughness increase exponentially. The recorded average values are in the range of 3.5 to 5.5 µm. The increased surface roughness is because of the amalgamation of the AgNP in the composition. The higher amount of AgNP causes more clustering and penetration inside the mixture composition.

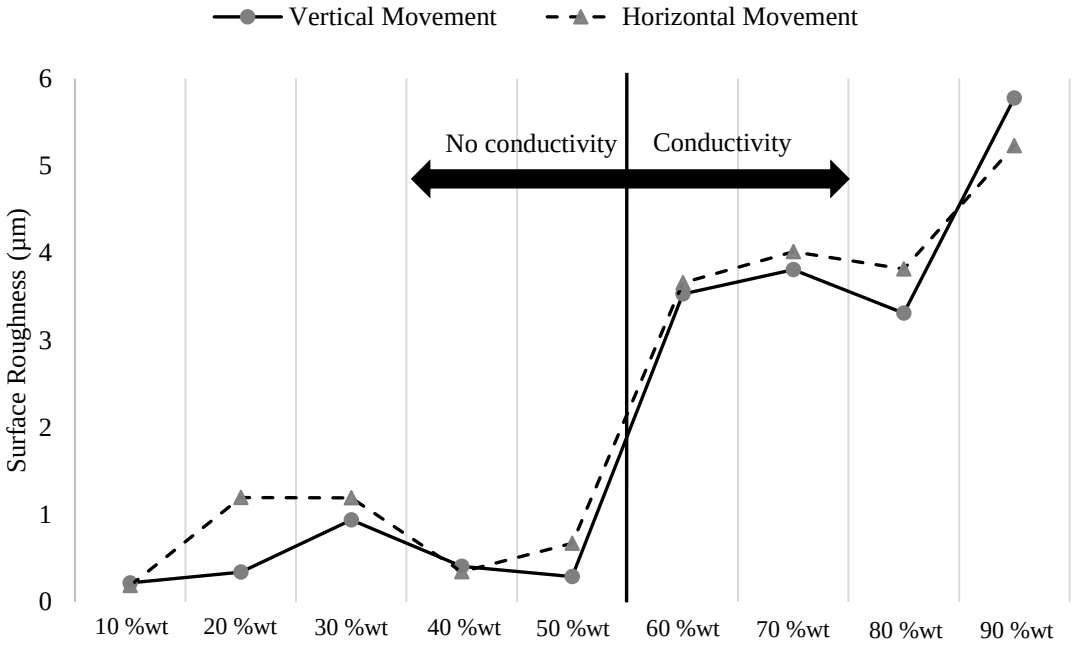


Figure 5: Surface roughness at different filler loadings for horizontal and vertical directions of measurement.

Furthermore, the larger distribution of particle sizes also causes rougher surfaces of the samples (McGrath *et al*., 2021). By referring to Figure 5, the same trend is shown for both the vertical and horizontal directions of movement. This eliminates the influence of inconsistency due to the printing method and the analysis can be solely done on the mixture composition of the ink. Based on the

previous research by Mendez-Rossal *et al.* (2019), there is no direct correlation between surface roughness and electrical conductivity. However, both parameters can potentially be caused by the same source. By focusing on the mixture composition, the results of this study show that higher average surface roughness and higher electrical conductivity are originated from larger distribution of AgNP in the composition.

4. CONCLUSION

The investigation about the resistivity, morphology and surface roughness of silver nanoparticles conductive ink has been conducted. The composition mixture was prepared using a manual mixing process and the doctor-blade technique was chosen as the printing method to form ink tracks on the glass substrate. The correlation between the three investigated parameters was obtained. The minimum required filler loading for the composition to have the ability to conduct electricity is 60 %wt. However, this minimum threshold shows a discrepancy in the tabulated data, which is unacceptable. The resistivity values also reach a plateau with for filler loading of 90 %wt. It indicates that further addition of filler loading is not going to give a significant improvement in electrical conductivity performance.

In terms of morphological analysis, filler loadings of 60 to 80 %wt show the presence of granular particles that contain 3D connection of conduction. This produces the electrical conductivity characteristic. For filler loading of 90 %wt of, it shows the presence of continuous particles that becomes the connecting bridges of higher electrical conductivity capabilities. For the surface roughness analysis, the increment of filler loading increases the surface roughness. Larger distribution of AgNP sizes contributes to higher surface roughness. Based on the results, the usable range of AgNP for conductive ink application is in the range of 70 to 90 %wt. The electrical conductivity performance shows a direct correlation with ink layer surface morphological characteristics but not surface roughness.

ACKNOWLEDGEMENT

The authors would like to thank to the Advanced Academia-Industry Collaboration Laboratory (AiCL) and Fakulti Kejuruteraan Mekanikal (FKM), Universiti Teknikal Malaysia Melaka (UTeM) for providing the laboratory facilities.

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