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Effect of Low Temperature on the Carbon- based Conductive Ink Resistivity for Flexible Printed Circuit

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Abstract

Nowadays, it is known that printing methodologies can be used to print electrically functional devices on variety substrates includes organic or inorganic materials. Currently, printing technologies have been an attractive alternative printing method to fabricate flexible electronic devices, keeping to its advantages including easy handling, wide use and low cost. However, using flexible substrate open up new possibilities for printed electronic (PE), where certain applications expose the flexible substrate to a mechanical bending, which might decreased the performance or becomes a cause to a functionality failure. Moreover, developing of an appropriate ink at an extreme surrounding temperature with high conductivity and good dispersion of the ink-jet printing is one of the critical issues that need to be solved. Thus, the objectives of this study are determine the effects of cyclic loading on the elasticity and conductivity of carbon-based conductive ink after exposing to low and room temperature. The sample of conductive ink has been tested by resistivity test using 4-point probe after doing cyclic test. This study is focused on the conductivity behavior of printed conductive ink after being exposed to different temperature; low temperature at -6ºC and at room temperature of 26ºC. After being exposed to each of the temperature set, the cyclic loading test of 1000 and 5000 cycles each was carried out while non-cyclic sample was prepared as the bench-marking sample. From the cyclic test results, it is understood that the resistivity of printed ink at low temperature is lower than that of at a room temperature.

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1. Introduction

Printed electronics refers to the printing technologies application to meet industrial requirements for low cost and high volume demand of electronic circuits and devices at low processing temperature. Besides the recognized technological advantages, printed electronic (PE) is estimated to grow up into a multibillion business in the near future [1]. Currently, the ink-jet printing technologies have been an attractive alternative printing method to fabricate flexible electronic devices, keeping to its advantages including easy handling, wide use and low cost [2]. Another advantage in PE is the availability of using various types of flexible substrates on the electrical structure such as polymers, papers and also fabrics. Moreover, the PE belong to an effectively functional conductive ink that can be used high precision conducting circuits on flexible circuit with using simple and low cost techniques [3].

However, using flexible substrate open up new possibilities for PE, where the application needs to be used in a curved form or requires repeated cyclic loading exposes it to a mechanical bending which might cause it

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to lower performance or becomes a failure [1]. The other issue is developing of an appropriate ink at an extreme surrounding temperature with high conductivity and good dispersion of the ink-jet printing. Thus, the objectives of this research are determine the effect of cyclic loading on the elasticity and conductivity of carbon-based conductive ink after exposing at low and room temperature.

In this study, the flexible polyethylene terephthalate (PET) and polyurethane (TPU) thin film was employed as a substrate for carbon-based conductive ink sample. Both of the substrate should be tested to determine which substrate is matching with the carbon-based conductive ink when the process printed exposes to the low and room temperature. Normally, the specific adhesion of the inks onto the substrate is needed in a proper range of substrate energy surface. PET is an excellent commercial thermoplastic polymer resin, low cost, good thermal stability; good spin ability and excellent moisture resistance. However, TPU is unique category of plastic where it has high elongation and tensile strength, more elasticity and ability resist oil, grease, solvents and chemicals. These characteristics make TPU extremely popular among market industries as a substrate [4].

Conductive ink for printed electronics applications is commonly made of metal particles filler in a polymer matrix [5]. There are various types of conductive materials use as the filler for conductive ink such as copper, silver, conductive polymer, graphene, carbon and metal nanoparticles. Normally, the quality of printed ink performance is based on the formulation of the conductive ink including the amount of conductive fillers, binder, dispersing medium, resin, dispersant and other additives [6]. A few years ago until now, carbon-based conductive ink had gained increasing popularity in technologies printed and electronic packaging application because of their low cost, environmental compatibility and lower assembly temperature [7]. There are two form of carbon which are graphite (a grey platelet form) and carbon black (a jet-black amorphous form). Thus, this study is focused on carbon black to investigate its conductivity performance compared to other materials filler. Fig. 1 shows about the microstructure of carbon black conductive ink.

This study utilize carbon black based conductive in as conductive filler that printed onto flexible printed circuit. Currently, carbon black is gaining popularity wearable textiles comparing low cost material than carbon nanotubes (CNT). Previous research mentioned that carbon activated cotton tread was used to harvest electrostatic energy from environment [8,9]. In another previous research stated that nontoxic inexpensive active carbon was printed on a textile as a flexible energy storage device [10].

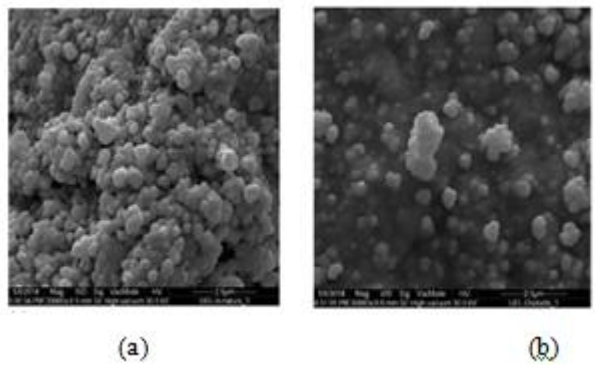

Figure 1: (a) Carbon black (b) Oxidized carbon black

2. Experimental

This section explains details about the methodology that involves in this research to achieve the objectives of this research. It includes the printing of conductive ink and testing process.

Material Selection

Normally, various types of conductive ink are used on printed circuit technology based on their application and performance. For this research, carbon black (BARE conductive electric paint) was selected to investigate its performance when expose to the extreme low temperature. The formulation of conductive ink was formulated from XX Company and the details information are listed in Table 1 below:

Table 1: Information of BARE conductive electric paint

Chemical Name	Water-based dispersion of carbon pigment in natural resin
Ingredients	Water, natural resin, conductive carbon and humectant
Appearance	Liquid
Colour	Black

This research also tested on flexible printed circuit which are polyethylene terephthalate (PET) and polyurethane (TPU) substrate to look either these substrates fully attach or not at low and room temperature.

Sample Preparation

In this study, carbon-based conductive ink was printed manually hand-printing technique so called doctor blading technique ink printing process substrate where the pattern will construct by paper masking tape on the substrate. Sample sizing was constructed based on the technical specification from Supplier Company and decided the sizing of the sample was 40mm x 5mm that shown in Fig. 2. Then, one sample of each substrate was printed with carbon black conductive ink and dried at low and room temperature until 24 hours. Both of samples will be tested either it is fully attach or not applicable.

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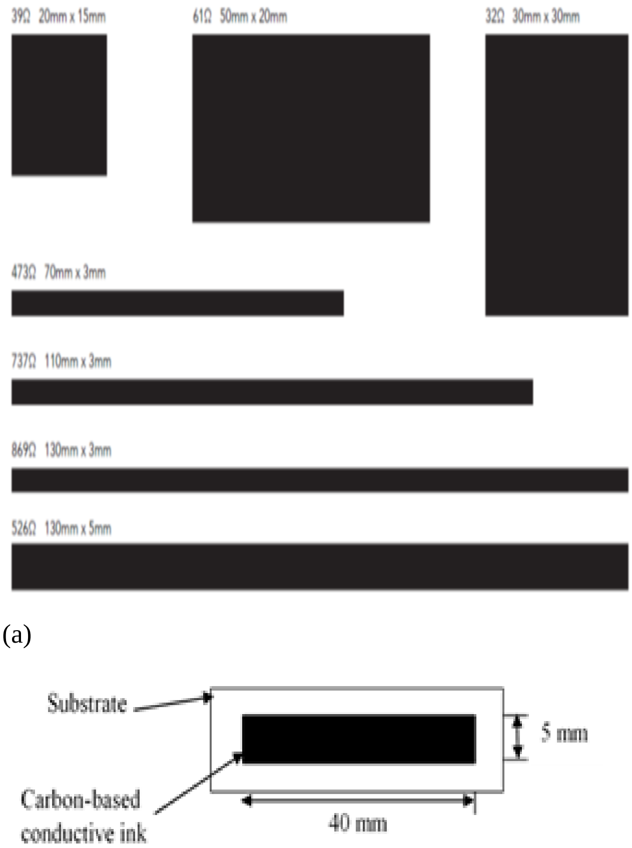

(a)

(b)

Figure 2: (a) Resistance sample visual (b) Sample sizing

After that, the conductive ink was printed on the selected substrate either PET or TPU as much as 48 samples with similar line thickness and width where 24 samples kept into the cold chamber at low temperature around -6 ºC and another samples were exposed at room temperature around 26 ºC almost 24 hours for each sample. Finally, all samples were divided to 6 cases which are non-cyclic (low temperature), 1000 cyclic (low temperature), 5000 cyclic (low temperatures), non-cyclic (room) temperature, 1000 cyclic (room temperature) and 5000 cyclic (room temperature).

3. Analysis

Firstly, the samples need cyclic loading to present the effect of conductive ink because flexible substrate always exposes to the mechanical bending application. Cyclic test machine was generated by 11v DC motor was produced 100 cyclic per minute for 1 sample that shown I Fig. 3. The voltage controlled by power supply around 11v that required from DC motor. The both end side sample were clamped to ensure the sample tend to maximum bend and stretch.

Before doing the cyclic loading test, one sample of each cases were analyzing its microstructure by using Axiaskop 2 MAT Low Power Microscope. Surface morphology on the fracture printed conductive ink is crucial to fine out the defect of surface and cross section of grain. Then, the sheet resistance data will be measured by using JANDEL In-Line Four Point Probe with following ASTM F390 as standard guideline that shown in Fig. 4. The current flow through the probe set up at specified ampere prior of resistivity test. All the data such as current (µA), voltage (mV) and resistivity (R/sq) were appeared in the computer. A total of 5 measurements point were taken for each sample.

Figure 3: Cyclic bending test equipment

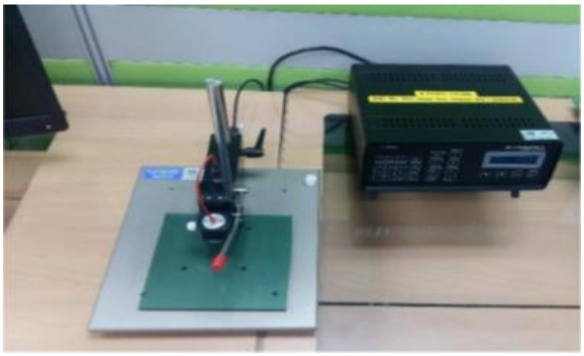

Figure 4: JANDEL In-Line 4 Point Probe test equipment

4. Results And Discussion

Carbon black conductive ink was printed onto two type of flexible substrate which are PET and TPU. As a result, PET substrate looked fully attached with carbon black conductive ink. The specific adhesion of the carbon black inks and a proper range of PET substrate energy surface is a good combination of each other. Besides, PET substrate also have a flexible ad sufficient of smoothness and porosity surface that will be printed of carbon black conductive ink thin film. While, TPU have a unique characteristic which it is too resist of oil grease and solvents [4]. The selected conductive ink (BARE conductive electric paint) is water-based dispersion of carbon pigment in natural resin. Therefore, TPU is not suitable for this conductive ink because of their solution is not enough range energy for its surface.

All the samples were printed on the PET substrates and continued with the cyclic loading test (1000 cyclic and 5000 cyclic). The effect of cyclic loading test on the samples will evaluate through the resistivity test to determine its conductivity performance and comparing with non-cyclic samples. Based on the Fig. 2, most the

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data were acceptable where the resistivity design of 130mm x 5mm specification is 526Ω. Design of this specification is three times bigger than the design research sample (40mm x 5mm). So, the range of resistivity of the sample almost 175Ω. However, a few data were out of range because of printed process temperature and thickness samples.

Resistivity Analysis

Fig. 5 represents about the performance of resistivity for non-cyclic sample between low and room temperature. The bar graph shown that the resistivity sample at low temperature is lower than at room temperature. The average resistivity performance at low temperature is 117.07Ω/sq while at room temperature is 152.95Ω/sq. Thus, the resistivity performance of low temperature is 30.65% lower than room temperature for non-cyclic samples. In fact, conductive ink has own its characteristic and properties. The previous research found that the resistivity of silver is lower when sinter at high temperature (105ºC) while graphite resistivity is lesser when sinter at low temperature (50ºC) [10]. Similarly, carbon black conductive ink need low temperature printed process to decrease its resistivity where this ink very sensitive of the high temperature.

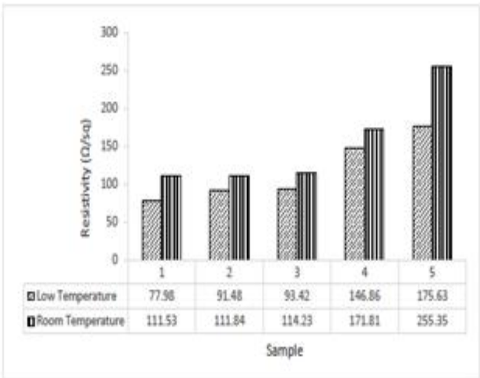

Figure 5: Resistivity performance of non-cyclic samples

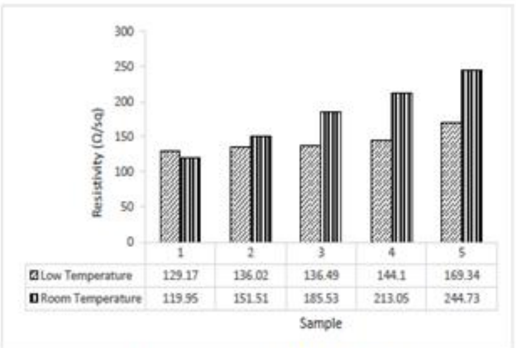

Figure 6: Resistivity performance of 1000 cyclic samples

Fig. 6 demonstrates about the resistivity performance of 1000 cyclic samples between low and room temperature. The figure stated that samples at low temperature were lower than room temperature and more accurate data between each sample. While, all the data at room temperature was increased gradually and inconsistently between each sample. Most of the resistivity data at room temperature is higher than low temperature samples. The average resistivity of room temperature is 182.95Ω/sq and low temperature is 143.02Ω/sq. Hence, the resistivity performance of room temperature is 21.83% higher than lower temperature at 1000 cyclic loading test. The resistivity of room temperature is higher than specific range resistivity around 175Ω. For instance, cyclic loading give tendency high resistivity to the printed electronics.

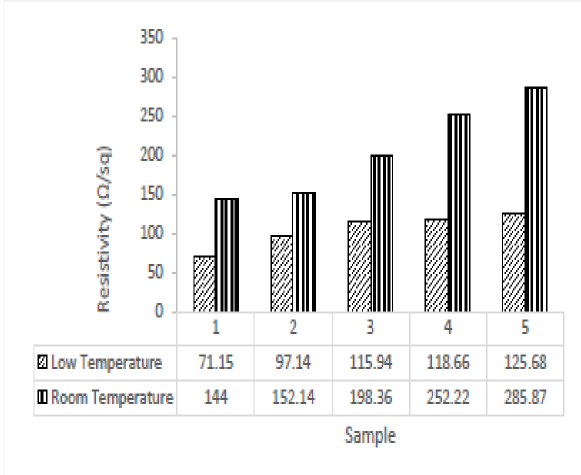

Figure 7: Resistivity performance of 5000 cyclic samples

Cyclic test was continued with 5000 cyclic to investigate its effect when bending and tensile loading is increasing. The resistivity performance for 5000 cyclic loading at low and room temperature was demonstrated in Fig. 7. As seen from the figure, the reading of data between both temperatures samples look like too different. Samples at room temperature appeared that the resistivity performance on a printed circuit is increasing drastically when cyclic loading is increasing. However, the data value was increased gradually by sample and inconsistently data between each sample. The highest resistivity at room temperature is 285.87 Ω/sq and the lower is 144 Ω/sq. Resistivity performance of low temperature is lesser than room temperature. The figure shown that the resistivity was slightly increased by sample with the highest value resistivity is 125.68 Ω/sq. Apart from the analysis; the resistivity performance of room temperature is 66.64% higher than low temperature at 5000 cyclic loading.

Fig. 8 displays the overview data of all samples and cases for resistivity behaviour between low and room temperature. The graph below mentioned clearly that the resistivity performance at room temperature sample was higher than low temperature sample for each cyclic process. From the result also displayed about 5000 cyclic

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loading was appeared more an effect to the conductive ink. At the room temperature the resistivity continued increased, after 1000 cyclic but will be decreased when expose to the low temperature.

The graph also stated about the standard deviation for each sample and the result shows that the data of low temperature more accurately than room temperature. All the data will be acceptable because most of the standard deviation is almost 5% from the data.

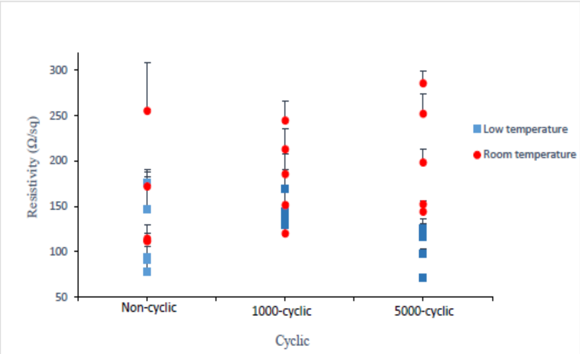

Figure 8: Resistivity behavior between low and room temperature for all samples

Microstructure Analysis

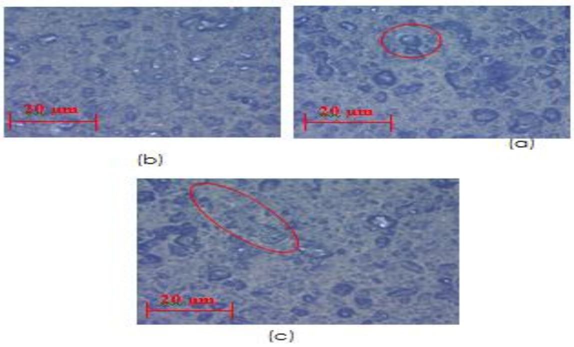

Figure 9: Microstructure of carbon black conductive ink at low temperature (a) Non-cyclic (b) 1000 cyclic (c) 5000 cyclic

Based on the specification, this conductive ink is water-based dispersion carbon pigment. As a result, Fig. 9 shows about the microstructure of carbon black at low temperature around -6ºC and exposed high moisture content. Previous researcher stated that resin is extremely easy to absorb water from the moisture air which leads volume expansion. Carbon basically is non-absorbent, so the resin's swelling generates stress and can cause interface debonding [11–13]. However, this carbon black ink contrast with that statement where its physical properties is partially soluble of water and make it more elasticity. Carbon black ink still had effected after done the cyclic test for instance crack surrounding glumeric and appeared wavy crack along the resin that shown in Fig. 3 (b) and (c).

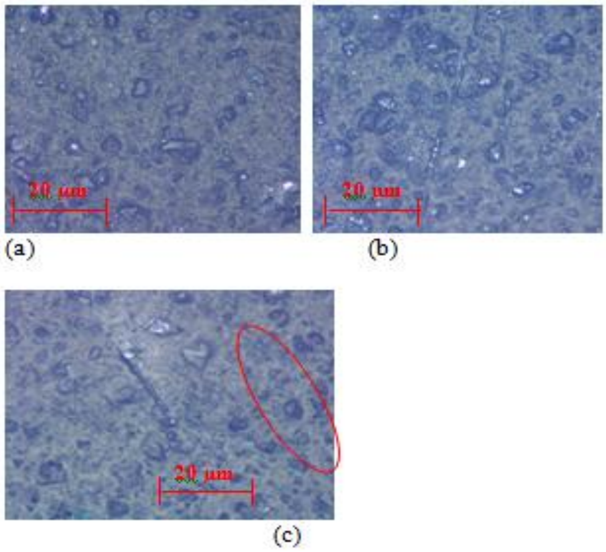

Figure 10: Microstructure of carbon black conductive ink at room temperature (a) Non-cyclic (b) 1000 cyclic (c) 5000 cyclic

The crack and scratch was cleared showed at the sample room temperature rather than low temperature that displayed in Fig. 10. Apart from that, it is proving that the resistivity at room temperature will be higher than low temperature. In fact, when material is expose to low temperature, the grain of structure is finer than high temperature. It will be become difficult to transfer electron from one grain to another grain [14]. However, most of the samples in this study were did cyclic loading testing and the effects of this factor were shown in Fig. 10. So, the effect of resistivity not analyze based on size of the grain but the effect microstructure.

5. Conclusion

Based on the analysis, the following conclusions are arrived:

- Result represented that the resistivity of low temperature samples was lower than room temperature samples after doing the cyclic loading test.
- At low temperature analysis, the resistivity of 1000 cyclic loading was higher than non-cyclic and 5000-cyclic around 117.07Ω/sq. While, the pattern resistivity of room temperature was increased drastically from non-cyclic to 5000 cyclic.
- The resistivity of room temperature samples was increased almost 20% for each level.
- At last, microstructure analysis shown that the effect of room temperature sample is more scratches and cracks especially at glumeric carbon black. The resin surface at 5000 cyclic of both temperature sample appeared wavier crack that make it lower conductivity performance.

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