THE PRINTABILITY OF THE CONDUCTIVE POLYMER EPOXY ONTO THE PET FLEXIBLE SUBSTRATE

TAN YAN HAU

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

SUPERVISOR'S DECLARATION

I hereby declare that I have read this project and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature	:………………………..
Supervisor's name	:………………………..
Date	:………………………..

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TAN YAN HAU

A report submitted
in fulfilment of the requirements for
the degree of Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2019

DECLARATION

I declare that this project entitled "The Printability of The Conductive Polymer Epoxy onto The PET Substrate" is the result of my own work except as cited in the references.

Signature	:………………………..
name	:………………………..
Date	:………………………..

DEDECATION

To my beloved mother and father

ABSTRACT

The applications of the flexible electronic circuit have increase significantly in the recent years. In order to fabricate a good flexible electronic, there are two important components need to be chosen carefully which are conductive polymer epoxy and substrate. Thus, this project will study about the printability of the conductive polymer epoxy onto the PET substrate. In this study, the conductivity of the conductive polymer epoxy will be measured by using four point probe after printed on the PET substrate with different percentage of filler. The sheet resistance value of the sample in ohms-per-square, the resistivity volume in ohms-cm and the sample thickness can be measured using a four-point probe. After that, morphological analysis will be carried out to observe the surface integrity of the conductive epoxy polymer on the PET substrate. By observing the microstructure of the conductive ink can let to know the quality of the of the conductive ink on the substrate. In addition, the gap of the filler particles can also be observed by microscope, this provided the information about the conductivity of sample with different filler loading. After that, correlate the conductivity vs adhesion property of the conductive polymer epoxy after print on the PET substrate is also one part in the study. This study determines the conductivity of the conductive ink will remain the same or change when the substrate is under bending condition. Due to the flexible electronics have to tolerance bending, vibration and stretching so the study of conductivity under these conditions are very important. Through this study, the printability of the conductive polymer epoxy onto the PET substrate can be determined. The conductivity will decrease with increase of filler percentage and the resistivity will maintain under bending test.

ABSTRAK

Aplikasi litar elektronik fleksibel telah meningkat dengan ketara pada tahun-tahun kebelakangan ini. Untuk menghasilkan elektronik fleksibel yang baik, terdapat dua komponen penting yang perlu dipilih dengan teliti yang epoksi dan substrat polimer konduktif. Oleh itu, projek ini akan mengkaji tentang cetakan epoksi polimer konduktif ke substrat PET. Dalam kajian ini, kekonduksian epoksi polimer konduktif akan diukur dengan menggunakan empat titik probe selepas dicetak pada substrat PET dengan peratusan pengisi yang berlainan. Nilai rintangan lembaran sampel dalam ohm-per-persegi, kelantangan resistiviti dalam ohm-cm dan ketebalan sampel boleh diukur dengan menggunakan kuar empat titik. Selepas itu, analisis morfologi akan dijalankan untuk memerhatikan integriti permukaan polimer epoksi konduktif pada substrat PET. Dengan memerhati struktur mikro dakwat konduktif boleh membezakan kualiti dakwat konduktif pada substrat. Di samping itu, jurang zarah pengisi juga boleh dilihat oleh mikroskop, ini memberikan maklumat tentang kekonduksian sampel dengan pemuatan pengisi yang berlainan. Selepas itu, kaitkan sifat konduktiviti vs sifat lekatan epoksi polimer konduktif selepas cetakan pada substrat PET juga merupakan satu bahagian dalam kajian ini. Kajian ini menentukan kekonduksian dakwat konduktif akan tetap sama atau berubah apabila substrat berada di bawah keadaan lenturan. Oleh kerana elektronik yang fleksibel harus toleransi lenturan, getaran dan regangan supaya kajian kekonduksian di bawah syarat-syarat ini sangat penting. Melalui kajian ini, cetakan epoksi polimer konduktif ke substrat PET boleh ditentukan. Kekonduksian akan berkurangan dengan peningkatan peratusan pengisi dan resistiviti akan mengekalkan ujian lenturan.

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Ag	-	Silver
Cu	-	Copper
C	-	Carbon
Nps	-	Nanoparticles
PET	-	Polyethylene terephthalate
TPU	-	Thermoplastic polyurethane
PC	-	polycarbonate
PI	-	polyimide
Dod	-	Drop-on-demand

LIST OF SYMBOLS

ρ	-	Resistivity
σ	-	Conductivity
A	-	Cross-sectional area of the ink
L	-	Length of sample trace from end to end
R	-	Resistance
l	-	Length of line in mm
W	-	Width in mm
R_{SH}	-	Resistivity of the sheet in *Ohm/sq*, Ω */sq*
V	-	Voltage across the inner pins
I	-	Current between the outer pins
T_m	-	Melting point
E	-	Estimation of error
Ra	-	Average of roughness

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

In recent years, the applications of flexible electronic circuit have increase significantly. This is because the flexible electronic can be applied in many fields such as flexible flat-panel display, smart wearable, medical image sensors, photovoltaics, cell phones, electronic paper and etc (Meena *et al.*, 2010). These electronic devices can hardly meet flexible electronics requirements, and these electronics are considered to be the future trend in the development of electronic devices of next generation. (Wang *et al.*, 2014). In addition, the advantages of the flexible electronic are low cost, ruggedness, light weight, easy to manufacture and so on. All of these advantages of flexible electronic are just some of the important advantages over the rigid electronic circuit.

Figure 1: flexible Electronic

In addition, they need to be highly stable during bending, folding, compressing, and even stretching in certain specific applications, such as wearable devices. In order to understand the electrical behavior and mechanism of flexible electronic devices under mechanical deformation, many studies have been conducted in the last decade. Polymers that are mechanically flexible due to their unique structures were mostly explored for the manufacture of flexible electronic devices among the investigated materials. They are especially promising for flexible fiber-shaped electronic devices with unique and promising advantages compared to the planar structure.(Chen *et al.*, 2012).

The main components of the printed circuits are conductive ink and a flexible PET substrate. The flexible substrates like paper and polyethylene terephthalate (PET) are low price materials so they are enable the wide use of low cost (Zhang *et al.*, 2011). Furthermore, there are many advantages of flexible substrates other than low cost, which are high transparency, exceptionally bright surface, excellent stability, high pressure resistance, light weight and good barrier properties. Thus, the flexible substrates like paper and polyethylene terephthalate (PET) are very suitable to produce flexible electronic.

Conductive ink print on substrate to conduct electricity have been in some talk over the last few years for their applications in Flexible Electronics (FE) and Printed Electronics (PE) as the circuits able to print on paper or some flexible substrate through different kind of printing technology. Some improvements have been made in the electronics sector due to the arise of conductive ink, thus enable the flexible electronics to develop in real world. Some factors such as environmental-wise (due to non-etching manufacturing procedures) good production performances, followed by reductions of material cost as well as lead to the alternative yet efficient way of using conductive inks through PE and FE for end-use applications.

Material selection is one of critical success to fabricate a good quality conductive ink. Basically, there are there main components consist in conductive ink; filler(usually silver), polymer epoxy and hardener (Merilampi *et al* , 2009). When the mixture of all three components is printed to the substrate, it will dry and the random connections of bridge-creating nanoparticles will be established.

1.2 PROBLEM STATEMENT

There are many imperfections after the conductive ink print on the substrate such as the thickness of on the conductive ink is not consistence through the layer, defects inside the conductive layer, the gap among the silver nanoparticle are too large and etc. Hence, observation of the surface integrity should take place to ensure the quality of the conductive layer on substrate. In addition, the accuracy of the adhesion and conductive testing result will increase due to the high quality of the printing.

One of the problems by the conductive ink after printed onto the substrate is the strength of the conductivity though the pattern of conductive ink is different at different location of the printed ink. The reason for conductivity is consistence is the concentration of filler at different location are not same. Therefore, electrical testing used to test the conductivity strength of the conductive ink at different point.

Conductivity of the conductive ink after print on the flexible substrate is a very important study for produce a flexible electronic. This is because the conductivity of the ink on the flexible substrate will be various when the substrate is under bending, twisting and stretching. Hence, conductivity of conductive ink after bending, twisting and stretching should be testing in order to know the suitability of the ink for printing on flexible substrate.

1.3 OBJECTIVE

The objectives of this project are as follows:

1. Observe the surface integrity between epoxy and PET by using microscope
2. Evaluate the strength of resistivity of conductive ink on the PET substrate
3. Evaluate the strength of the resistivity of the conductive ink under bending condition

1.4 SCOPE OF PROJECT

The scopes of this project are:

1. There are many types of polymer epoxy but only one of polymer epoxy will be selected to produce conductive ink and print on the PET substrate.
2. The strength of conductivity of the conductive polymer epoxy after print onto the PET substrate will be measure.
3. The strength of the conductivity of the conductive polymer epoxy will be measure under bending test

CHAPTER 2

LITERATURE REVIEW

2.1 METHOD OF PRINTING

There are many printing methods like inkjet printing, doctor-blading, casting coating, gravure printing, screen printing and etc.

2.1.1 Screen printing

screen printing is a printing process that has been used to manufacture printed circuit boards for many years(Riemer, DE 1989). In recent year, screen printing is simpler and faster compared with other printing tool so it is widely used for manufacturing of solar cells, thin film transistors, and sensors(Suganuma K. 2014). The process of screen printing is shown in figure 2.2. The screen on the printing board is lowered down. Ink is added to the screen's top end, and a squeegee is used to pull the ink along the screen's full length. This presses the ink through the stencil's open areas and imprints the design on the product below.(Hyun, WJ 2015). Due to its simplicity, reproducibility and high compatibility with different inks and substrates, screen printing has advantages; making it a cost-effectiveness approach for flexible device mass printing.

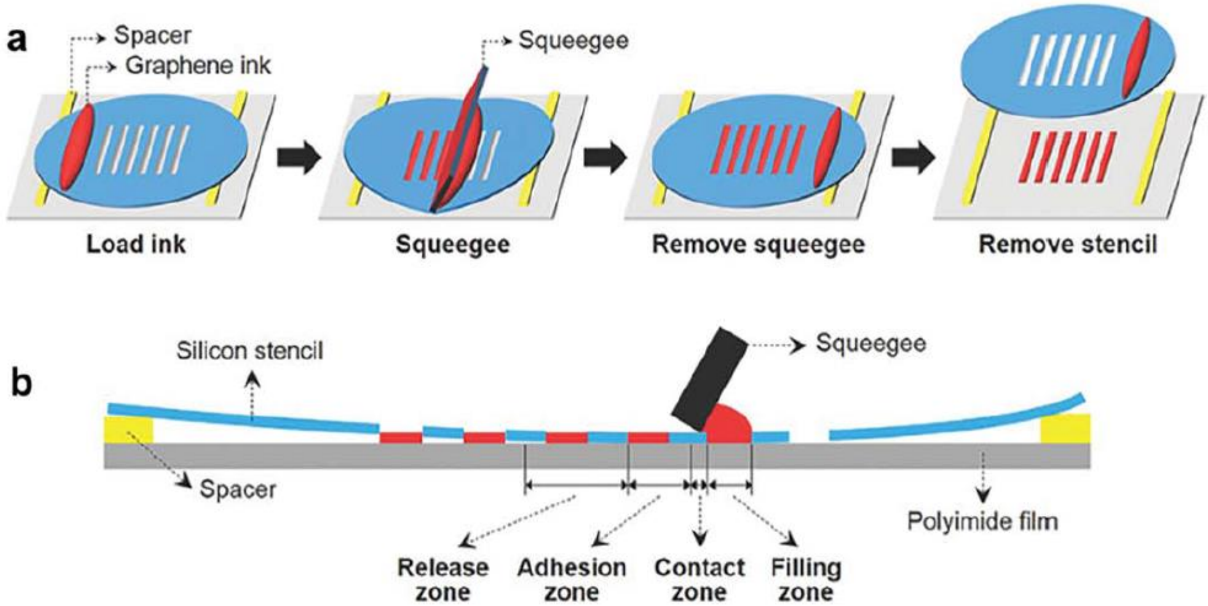


Figure 2.1: (a) Schematic illustration of the screen printing process; (b) Cross-sectional illustration of the screen printing(Hyun *et al.*, 2015b)

2.1.2 Casting coating

In casting method, there is no advanced equipment is required but a well horizontality of surface of substrate is crucial to have. The ink is casted onto the substrate in two forms; liquid portion or isolated drops and then, the ink is being dried at elevated or room temperature. This method is allowed to obtain thick films with nice coating quality, but the accuracy of the film thickness control is what it lacks of (Schwartz, 2010).

2.1.3 Gravure printing

In 2013, Hrehorova, E. et al. represented that gravure printing has four basic elements (Figure 2.2) to each unit of printing; impression roller, doctor blade, ink fountain and an image carrier (engraved cylinder) which the carrier itself is used to deliver the image that will be printed.

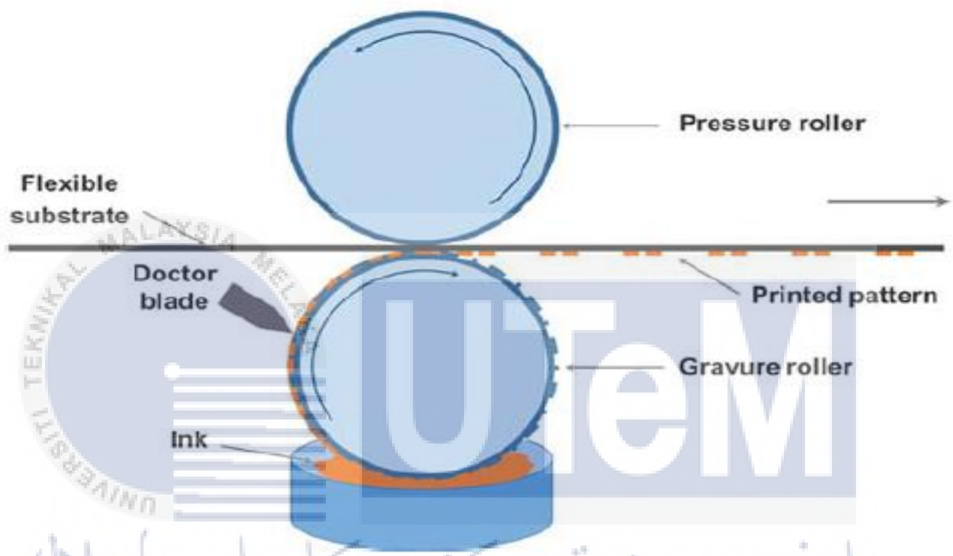


Figure2.2: Schematic illustration of the gravure printing process(Tran, Dutta and Choudhury, 2018)

Some of the benefits that make it an appealing procedure to print the electronic layers consist of its capability to print a wide thickness of ink track and to deposit low viscosity inks; thus, it can be applied for a wide range of ink composition as well as substrates(Hrehorova *et al.*, 2011). In addition, it can produce a printing with great resolution and a long-term stability at increasing printing speeds. It also has the image carrier that comes with the solvent resistance and a special characteristic that other printing methods lack of (Secor *et al.*, 2014).

2.1.4 Doctor-blading

Doctor-blading method will be used in this experiment to deposit ink layers on the substrate. As stated by Krebs, F. C. in 2009, this technique can produce the films‟ formation with a well-predefined thickness; in the range of 10 µm to 500 µm as well as the loss in ink can be reduced to less than 5 *wt*% . In this method, for both flat and groove surfaces, a sharp blade is placed at a specified distance above the surface of the substratum. The ink is placed before the blade and the blade is then moved linearly across the substratum. The value of the thickness of the coated layer is affected by the meniscus between the wet films on the blade trailing edge and the blade due to the blade speed(Hu *et al.*, 2016). According to Nash et al., other aspects that can affect the thickness of films are the surface energy of the substrate and/or its porosity, while the ink viscosity and its surface tension are also included. The final dry of the film thickness as being mentioned by Krebs, F. C. in 2009, will be proportional to the concentration of the solid metal presence in the solution and the width of gap while, the linear speed in this process can be ranged from 1 mm s^{-1} and 100 mm s^{-1}, which related to small shear stress during coating. Another representative for this method is by bar-coating that has the same way but their difference is that the ink is rolled by a bar over the substrate(Ghediya and Chaudhuri, 2015).

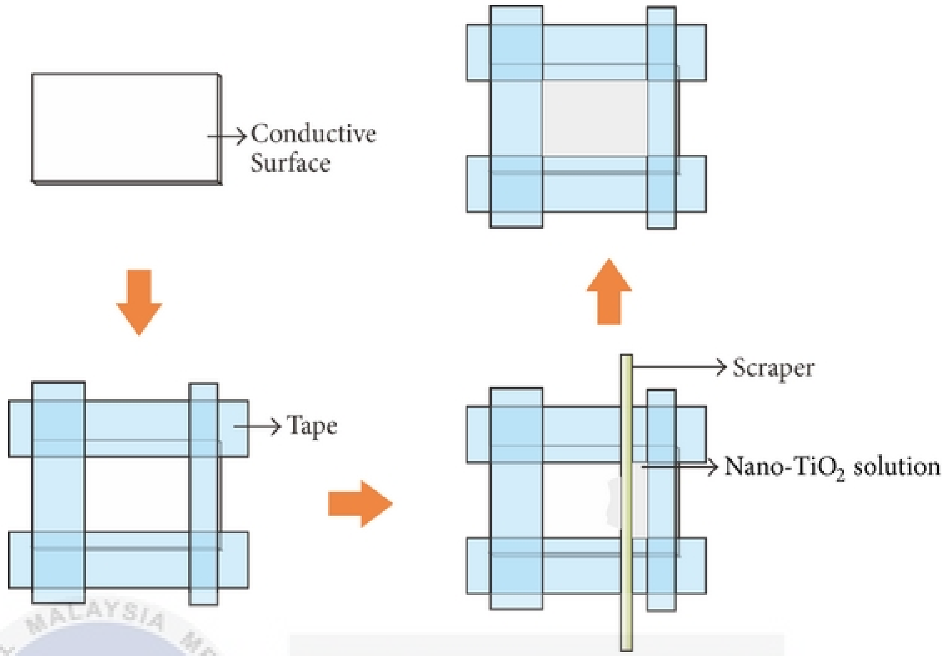


Figure 2.3: Doctor-blading method

2.1.5 Flexographic printing

Bois, C. et al. mentioned in 2012 that in flexographic method, it consists of transferring the required image using relief plates that enforce a low pressure onto the substrate. As illustrated in figure 2.4, the typical flexography system consists of four rollers. The collecting cylinder collects ink and is transferred to the Anilox roller (a cylinder with gravure patterns). The patterns are filled with ink on the Anilox roller and doctored off to remove the excessive ink. Then, through the printer roller, the printed pattern is transferred intermediately to the substratum. The rubber printing roller benefits from this direct contact printing process, which can further reduce the roller impact on the thin film, scratches on the substrates and minimize the creases. The process is very speedy and simple.

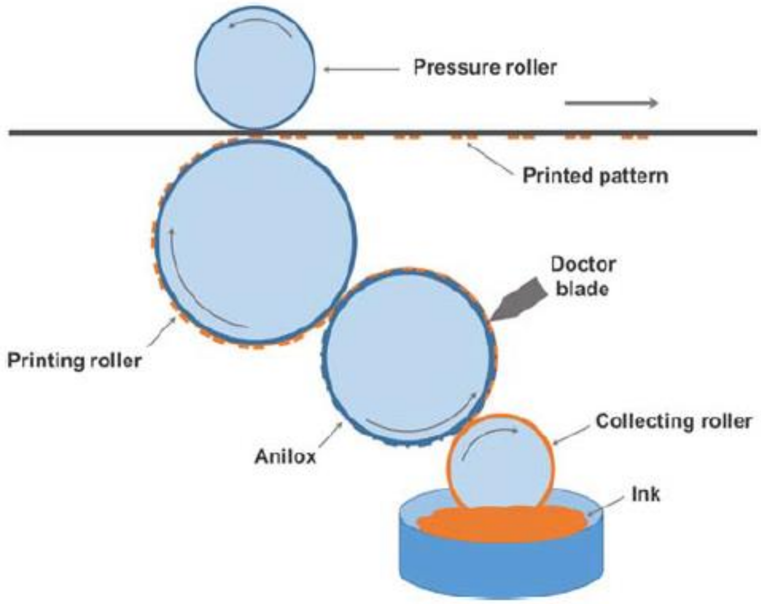


Figure 2.4: schematic illustration of the flexographic printing process(Tran, Dutta and Choudhury, 2018)

2.1.6 Inkjet printing

There are two types of inkjet printing techniques. First technique is continuous inkjet printing and the second one is drop-on-demand inkjet printing (Kim *et al.*, 2006). The difference between them is that continuous inkjet printing can be more than directed at any pixel location by continuous streaming system (Kazani, 2012). And when it's nothing to print, depending on the electric field imposed, the ink is defected into a gutter for recirculation. It is the only one drop per pixel for the drop-on-demand method that was created only when the printing process required (Sumerel, 2016). In continuous mode, ink droplets in an electrical field are generated continuously and charged electrostatically. Then a special collector deflects the charged droplets selectively, allowing only proper droplets to be deposited in a suitable position. In contrast, drop-on-demand mode generates droplets of ink only when required. When the nozzle is in the right place, the electrical pulse is applied to form a single droplet and to locate it on the desired area. Drop-on-demand does not involve droplet charging and is the most commonly used mode.

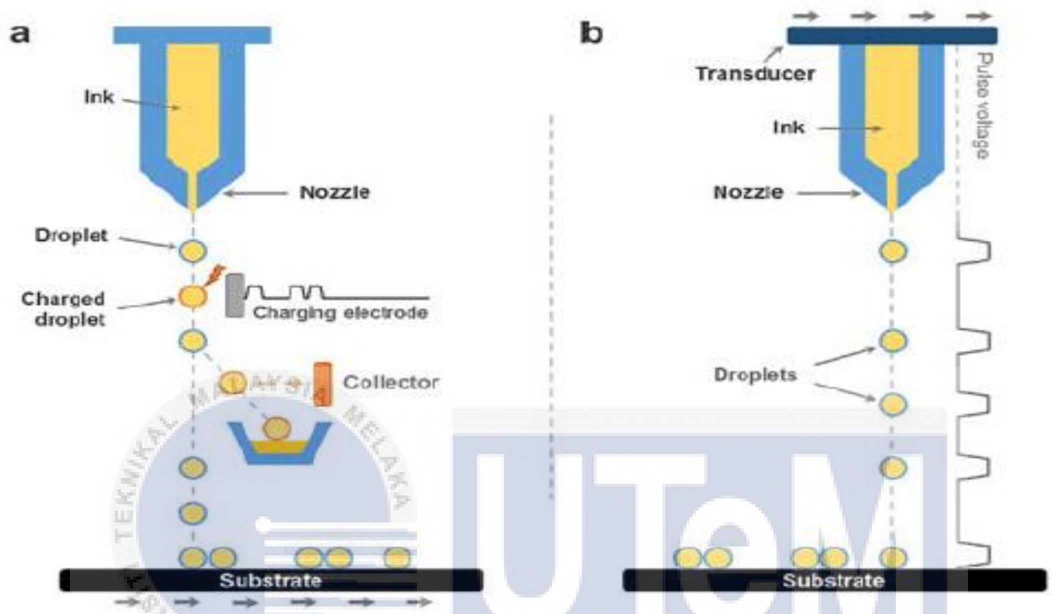


Figure 2.5: (a) continuous mode and (b) drops-on-demand mode of inkjet printing(Tran, Dutta and Choudhury, 2018)

2.2 CONDUCTIVE INK

According to Bhore, inks are specially fabricated in accordance to the demand of a printing process. In 2006, Karwa, A state that are made of the composition of filler with resin, solvents and additives; where the fillers are providing the conductive properties for the ink, while the binder is used to produce the required adhesion to the substrate and cohesion to each other. The factors affected the conductivity of an ink are the particle size of the fillers, the filler loading amount, binder's percentage and the continuity of the printed layer after the processes of printing and dying(Joshi,S.S.,2011).

2.2.1 Metal-base materials

The conductivity of metal-based materials are better compared to carbon, conductive polymers, graphene and etc. Due to the good conductivity of metal-based materials ink, it is widely used in produce flexible electronics (Kamyshny, 2011). According to Nir, M.M., Zamir, et al, the basic specification for metal. Based inks should show good electrical conductivity of the printed pattern as well as the ink should show high resolution and printability with the least maintenance of the printer and show that the ink is compatible with the substrate. After that, the ink should be annealing or curing to achieve higher conductivity and make the adhesion become stronger.

2.2.1.1 Silver

The conductivity of the silver is very high so it is widely used in producing of conductive ink. There are three type of silver conductive inks; silver flake, silver nanoparticle and silver nanowire(Abou El-Nour *et al.*, 2010). As claimed by Joshi in year 2011, Silver flakes are dried by heating process at temperature and heating time; the same applies to nano-silver inks by removing the solvent and leaving the binder and the film of the substrate conductive material. According to Nir, M. M. , Zamir, et al., modification of the synthesis of silver nanoparticles (in terms of particle size, stabilization) is one of the necessary points in the production of tints with high conductivity printed patterns, thus requiring highly concentrated loading dispersions of silver nanoparticles, usually 20-40% wt. The wt% was obtained when the mass of silver nanoparticles were divided by sum of weight of each ink, thus, for the wt%, it consisted of masses of silver content and masses of the capping agent (Shen *et al.*, 2014). The sintering process of silver nanoparticles can result in high conductivity in the description by Kim et al. when a 20 nm diameter of silver nanoparticles is used and healed at a temperature range of 100-300 ° C for 30 min. The ink of nanoparticles is sintered and often requires temperatures ranging from 100°C to 400°C and time to heal from 5 to 60 min, depending on the temperature. (Joshi, S.S., 2011).

2.2.1.2 Copper

Copper is a low price and high conductivity material so it very suitable used to produce conductive ink. In addition, copper conductivity is comparable to silver but price is a lot cheaper. Copper can be a good choice of material as mentioned by Park et al.in 2007 due to its less electro migration and lower cost than novel metals, but it is rapidly oxidized in the air which may worsen the conductivity. The oxides of copper can be eliminated by using certain methods to protect the copper surface when it exposed to air. Thus, oxides are not conductive that cause the limitation in usage of copper in printed electronics applications.

2.2.2 Carbon-based materials

Referring to (Denneulin *et al.*, 2011) carbon-based inks contain particles such as fullerene, amorphous carbon or graphite ; where graphite contains a number of graphite layers defined as a single sheet of carbon atoms. Kirkor et al. also said that these inks are allowed to dry after being applied to the substrates; using heat treatment up to several hundred degrees Celsius in the range of 50 ° C which is essential to obtain high electrical conductivity. Bernds, A. et al., added that graphite is preferred to be in micro-platelets formation for printed electronics in order to attain good processability and electrical performance.

2.2.2 Polymer

In 2015, Chen et al. reported that conductive polymers have been widely applied in various electronic devices such as light emitting displays and batteries. In regards of their organic nature, the adhesion between conductive polymer thin films and flexible plastic substrates and their mechanical stability are the best, mainly under bending conditions. Among conductive polymers, poly(3,4-ethylenedioxythiophene) or PEDOT is considered as one of the most favourable technologically electrically conductive polymers as it has stable electrical conductivity and versatile processability.

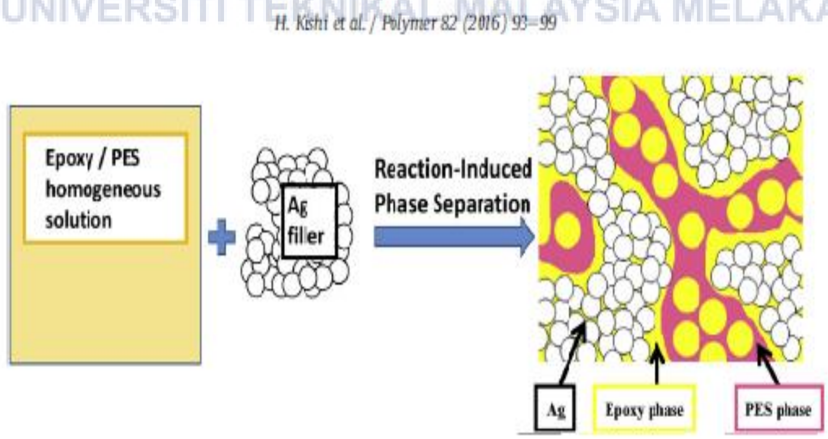


Figure 2.6: concept of electrically conductive Ag adhesive using an epoxy/PES blend as the matrix polymers with reaction- induced phase separation(Kishi *et al.*, 2016).

2.3 HEAT TREATMENT

Conductivity of the conductive ink after printing on the substratum is not good. Additional post-printing treatment should be performed to increase the conductivity of the conductive ink, mainly provided by a heating process. The heating or thermal procedure affects the resistance and the resistivity of the printed structure after the procedure can be just slightly higher than the bulk value of the materials(Jan Felba et al, 2009). Curing and sintering are therefore important in order to achieve high electrical conductivity that can be achieved by heating thermal in the oven, electrical sintering or other techniques to remove the organic between silver nanoparticles(Allen *et al.*, 2008). The time of sintering and curing temperature influence the resistivity of printed patterns according to Ismail, M. and Jabra, R.

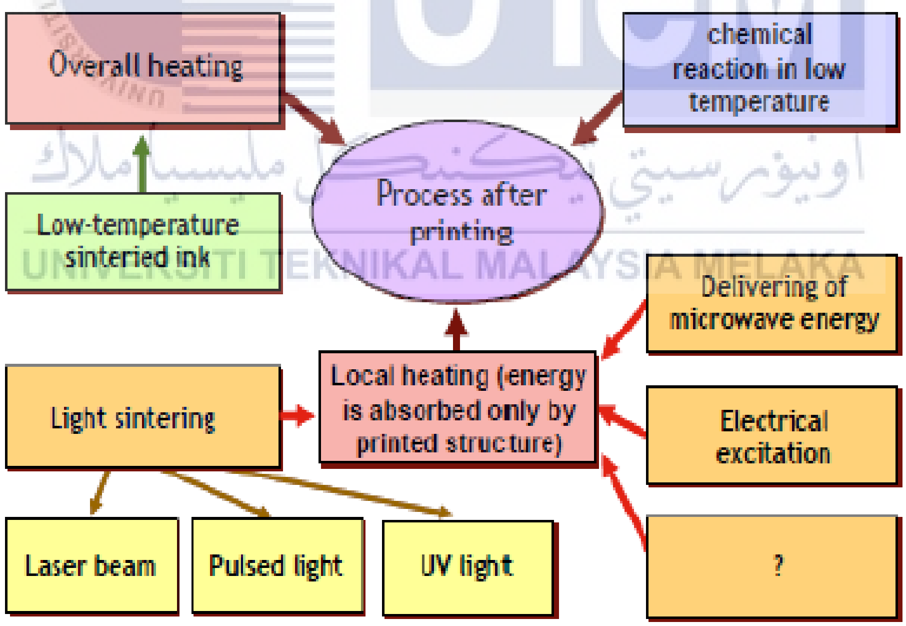


Figure 2.7: Options of processes after printing (Jan Felba et al. 2013)

2.3.1 Sintering

Sintering is a thermal process used to bind particles to a solid object. Sintering is crucial for the success of several engineering products as a thermal treatment, including most ceramics and cemented carbides, several metals and certain polymers.(Randall M. 2014). According to Nir, M.M., Zamir, et al, sintering is a process of binding particles together below their melting point at temperature. The sintering at a higher temperature was more well defined, proven by the lower resistivity (1.1-3 times of that bulk metal) when the printed patterns are sintered at temperature above 200-250°C, up to 400°C(Chen, Sun and Xiao, 2010). As stated by Ahn, B. Y. et al. in 2009, it has been recorded that silver nanoparticles can show high conductivity when being sintered at range of temperature 200 °C – 350 °C.

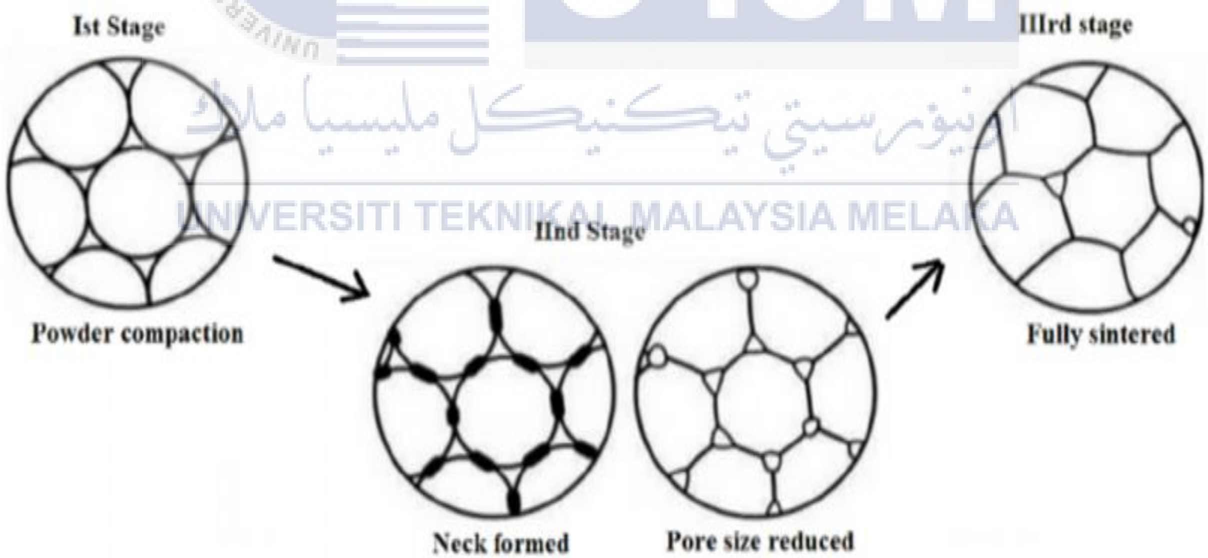


Figure 2.8 stages of sintering

2.3.2 Curing process

The process of curing can be achieved by exposing the printed pattern to heat, intense light (photonic curing), oven healing and electrical healing. To avoid destructive material heating, it is necessary to find the appropriate healing methods. In addition, consideration was given to the economic, low cost and time consumption.

2.3.2.1 Oven Curing process

To sinter the conductive ink particles to get close to each other and evaporate the solvent contained in the conductive ink, the heating process of the oven was used to heat and heal the conductive patterns printed. Heating to a high temperature (100 ºC and above) was required to burn out all organic contaminants (solvent) to achieve high conductivity of the conductive ink track. The heating coil inside the oven at the top and bottom can provide heat supply and maintain or fix a certain level of temperature. However, the defect was found to be deformed by using the process of curing the oven, which is the substrate.

2.3.2.2 Electrical curing process

The electrical curing process method is to apply a voltage over the printed structure that causes current flow through the structure, resulting in local heating by dissipating energy. The benefits of this process are the short healing time (within 10 seconds) and the reduced heating of the substrate (Kamyshny and Steinke, 2011).

2.4 MATERIAL OF SUBSTRATE

The adhesion between the ink and the substrate material is one of the basic factors in the production of conductive structure in printable electronics and the ink should be heated at temperatures below 150 ° C or better than 120 ° C for flexible substrates according to Dang, M. C. et al. There are two options to develop the adhesion between organic substrate and silver ink; which are control the silver ink composition and surface treatments of substrate such as UV-ozone, plasma or corona to increase the surface energy which leads to the improvement of an ink wetting. For high adhesion implementation, it is important to have a low contact angle as close as possible to 0° and great wetting(Chien Dang *et al.*, 2015).

2.4.1 Polymer substrates

For printed electronics, polymer substrates are widely used; unlike paper films, uncoated polymer substrates have no porosity, homogeneous properties and smooth finishing. Although in some topics it brings different advantages, non-absorbing and smooth surface in other topics is unsatisfactory.

In Van Osch's description, T. H. Polytetraflourethylene (PTFE, Teflon) has a high temperature stability and chemical and aging resistance. But, somehow it's expensive and has little surface energy that can create challenges with "line bulging" that can be categorized as unneeded, a local expansion of printed structure.

Öhlund, T. mentioned that polyethylene terephthalate (PET), polycarbonate (PC) and polyethylene (PE) are mostly used for printed purposes as less expensive flexible substrates.

Their softening points are below 150 ° C, so if high sintering temperatures are required, they are not suitable with oven technique.

Chan, M.C. Et al. submitted that substrates of polyimide (PI, Kapton) could maintain their flexibility and could withstand extended exposure to 300 ° C. The high surface energy leads to a large ink wetting, making it impossible to achieve the smallest feasible structures without decreasing surface energy. The cost of substrates of this type is somewhat expensive.

2.4.3 Glass substrates

For printed electronics, substrates that come with greater dimensional stability than polymer or paper substrate has the ability to upgrade device operation, registration and resolution (Hrehorova *et al.*, 2011). Glass slide will be used as a base substrate to find the best formulation of ink in the experiment. Hrehorova, E. et al. classified that glass has been applied in the electronic field for being a very appealing substrate for very sensitive materials commonly used in organic printed electronics in which caused by its features; a) great resistance to moisture and oxygen, b) chemical barrier, c) good quality of surface and d) highly stability of combination between thermo-mechanical.

2.4.4 Paper substrates

In 2014, Öhlund, T. explained that paper-based substrates for printed electronics are captivating for its being cheap, flexible and environmental friendly, but it is also inconsistent and fibrous which is the major problem of applying paper as a substrate. Other than that, surface roughness of uncoated paper and high porosity will cause the problem when the planned functionality is achieved. Paper is easily influenced by factors of environmental such as humidity and temperature that might affect the roughness and dimensional as well as the mechanical properties.

2.5 Conductive Ink Characterization

Merilampi, laine-Ma, & ruuskanen explained that the purpose of characterizing the conductive silver ink is to find out which parameters can be improved to fulfill the desired ink properties in accordance with the condition decided by different applications.

2.5.1 Electrical Properties

Before the concept of conductivity and resistivity is understood, the law of Ohm must first be explained. According to Ohm's law, if physical conditions (such as mechanical stress, temperature) remain unchanged and the potential difference between two ends of a conductor is proportional to the current through a conductor. The formula as shown below:

$$V=IR \tag{2.1}$$

Where,

V= Voltage

I= Electric Current

R = Resistance (Ω)

Voltage is the different or electrical pressure electrical potential to move the charge between two points. Electrical current is a flow of electrical charge performed in the conductor by moving electron. To understand the resistance, it is important to understand the relationship between resistance and resistivity when describing the printable driver(Rd Kharilhijra,2016).

Resistance is a measurement for an object that resists or opposes a flow of electrical current through it. This can explain why the conductivity of metal is better than wood. Compared to the thin wire, the thicker wire has less resistance and the longer wire also has more resistance than a shorter wire. The material's electrically resistive nature is an intensive property called resistivity. The resistance depends on the physical form and pattern, but the resistivity depends on the material's nature. The relationship between resistance and resistivity is shown in below formula:

$$\rho = RA/L \tag{2.2}$$

Where,

ρ = Volume Resistivity, Ω.m

A = Cross-section area , m^2

L = Length, m

R = Resistance, Ω

Electrical conductivity is a measure of a material's ability to transfer or conduct an electrical current. The higher resistivity is the lower conductivity. It is commonly represented by the Greek letter σ (sigma) and the formula for conductivity show as below:

$$\sigma = 1/\rho \tag{2.3}$$

Where,

σ = conductivity

ρ = Volume Resistivity, Ω.m

2.5.2 Conductivity versus Adhesion

There are many conditions have to tolerate by the conductive patterns and the substrate when using flexible materials which are bending, stretching, thermal shock and vibration. Because of this, the adhesive between the conductive ink and the substrate must be strong enough and there are defect free in the microstructure of the printed conductive ink (S. Merilampi 2009). To obtain a good quality of flexible electronic, electrical properties, adhesion, tensile strength and bending should be measured. In addition, the electrical conductivity of the ink on the substrate need to be measure after the substrate was elongate or bend. This testing used to know the change of electrical conductivity after elongate and bend. The data are very important for producing a flexible electronic.

2.6 SUMMARY

Literature review provide the information about the formulation of conductive ink, difference type of substrate, method of printing, methods of production to fabricate ink and the knowledge of electrical properties. After study the literature review, the filler element for produce the conductive ink is nanoparticle silver and doctor-blading method is selected as the printing method. In addition, Polyethylene terephthalate (PET) will be use as the baseline. Furthermore, relationship between the materials and methods of study through parameters also can be found out once the literature review about the related topics has been studied.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter will described the method for the fabrication of the conductive ink, the materials and apparatus used and type of tests that was carried out on the conductive ink in this research experiment. The flow chart of methodology is shown in the figure 3.1. First, different types conductive inks will be prepared. All the conductive inks are made up of epoxy polymer, conductive fillers, solvents and additives. Only change the percentage of filler to make different type of inks. After that, print the conductive inks onto the PET substrate respectively. After printing, the surface integrity of different type of conductive ink on PET substrate were observed by using a microscope. Then, bending test (mechanical test) was used to evaluate the adhesion of conductive inks on PET substrate. After that, conductivity of the conductive inks on the PET substrate will be measured. Lastly, Analysis will be presented on the bending and conductivity test.

3.2 FLOW CHART

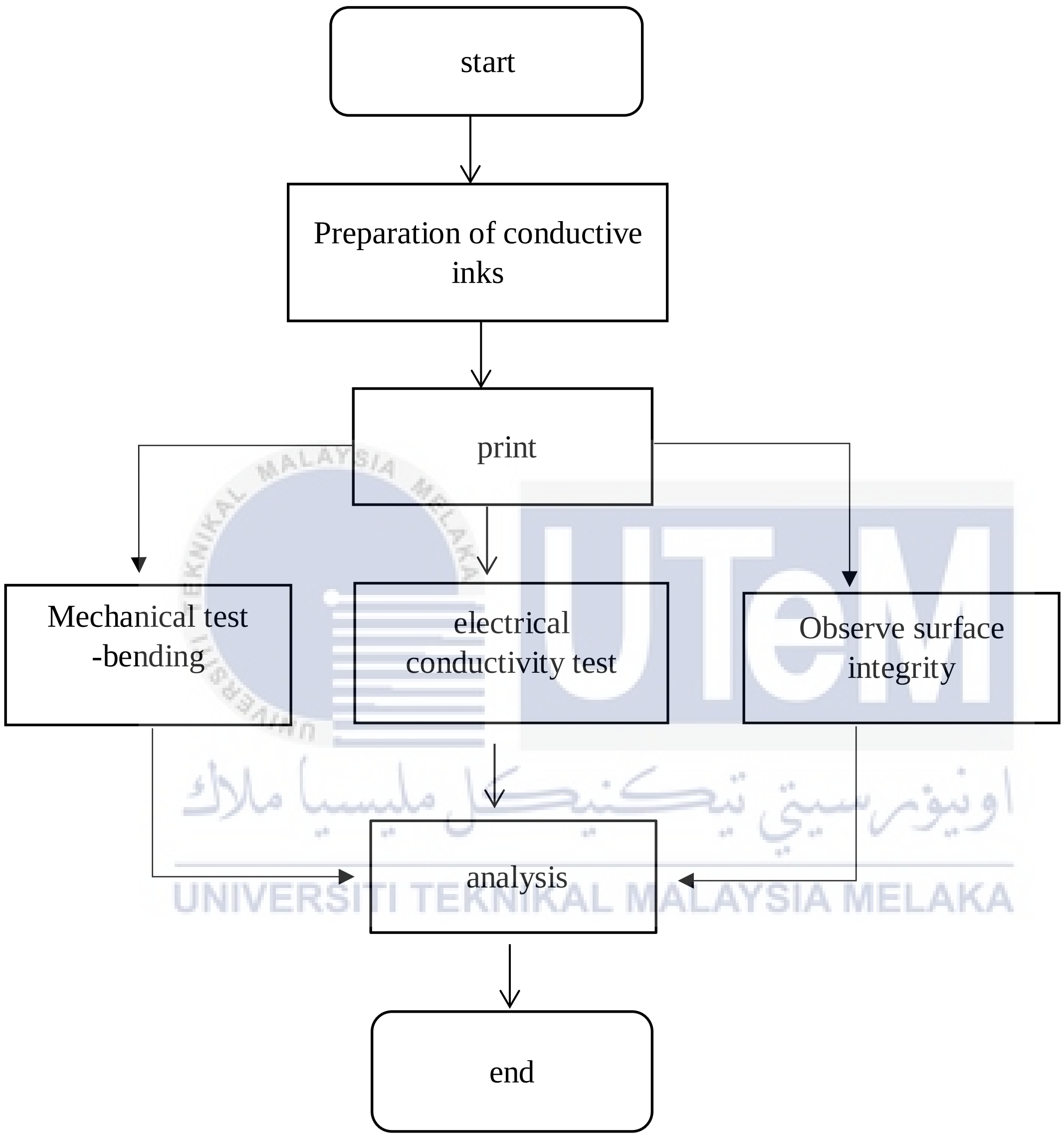


Figure 3.1: Flow chart of methodology.

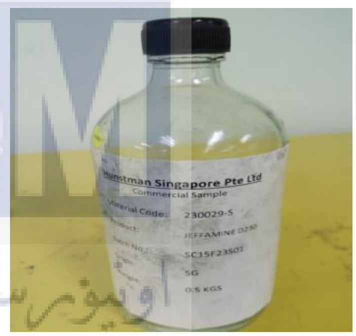
3.3 PREPARATION OF CONDUCTIVE INKS

The following steps show how conductive inks will produced.

3.3.1 Raw materials

The materials involved were prepared as listed in the table below to produce the samples of conductive ink in this experiment.

Table 3.1: materials for produce conductive inks

Materials		
Carbon conductive paint 	Epoxy 	hardener 
Used as filler element	Used as the binder element, to bind the particle together	Used to harden or dry the mixture

3.3.2 Formulation of conductive inks

The materials used to formulate conductive ink in this experiment were shown in the Table 3.1 and epoxy will be mixed with different wt% of carbon black conductive ink to produce new conductive ink. The loading of the hardener is 30% off the loading of the binder. The total value of the conductive inks is the sum of the amount of filler loading and binder loading as the total value was decided before the process of formulation began which is 2g and the wt% of carbon black conductive ink is 30wt%, 60wt%, 90wt% and 100wt%. To begin, the weighing process should be started first and the material and apparatus involved were listed in Table 3.2.

Table 3.2: Material and apparatus involved

Material/Apparatus	Descriptions
Digital Analytical Balance	To weigh the material
Beaker	Used for mixing and stirring processes
Scoop	As a transfer medium of the material

The analytical balance door was opened and the beaker was placed at the center of the weighing pan. (Figure 3.2) but not with bare fingers as the fingerprints will affect the required mass of the materials. Then the door was shut and the unit was considered to be stabilizing for a few seconds. To obtain an accurate reading of the weighted substance, the weight of the beaker was cancelled out by pressing the tare button until 0.0000 g was displayed on the display screen.

Figure 3.2: Beaker positioned on the center of pan

The door was opened after the 0.0000 g was achieved and the materials being weighed were carefully added or reduced until the display displayed the required weight according to the weight value in the table as shown in the figure 3.3.

Figure 3.3: Value of weight being adjusted

But the weight should be slightly higher than the desired value; considering the mixing of the materials into a tolerance of + 0.05 g as the weight loss was taken into account. The process described has been repeated three times since for each sample there are three materials involved.

3.3.3 Mixing and stirring processes

There are some crucial steps in this operation before the final sample can be reached and these steps are processes of mixing and stirring. The materials and apparatus that will be used were listed in the table below.

Table 3.3: Material and apparatus involved

Material/Apparatus	Descriptions
Glass rod	To stir the mixture
Container	To store the extra mixture produced
Desiccator	To store the dry mixture
Multipurpose wipe	Used in cleaning process by wiping the apparatus
Acetone	As a medium to clean the apparatus

They will be mixed into a single beaker after weighing all the related materials. Since all materials have different viscosities, materials with lower viscosity that are carbon black conductive ink and hardener will be poured into a beaker that contains the highest viscosity material that is epoxy. The aim of this mixing method is to prevent materials with high viscosity from losing weight as they tend to stick to the beaker while being poured because of their properties. The next steps to be taken was stirring process where in 2014, Shen, W. et al. The ink mixture was continuously stirred at 65 ° C for one hour. For this experiment, a glass rod is used to continuously take the stirring process at room temperature for five minutes.

To obtain a well-dissolved mixture, the stirring process was proposed to be either clockwise or anti-clockwise in one direction and the speed to stir the mixture must be consistent throughout the stirring process.

Upon completion of the stirring process, the entire mixture was stored in a container (Figure 3.5) and they were placed in an electronic desiccator with different labels. Desiccator is a sealed cabinet which is used by the absorption of moisture to preserve the dryness of the material inside the desiccator.

Figure 3.5: Well-dissolved mixture in the container

After completion of the overall processes, all the devices used were cleaned with acetone by soaking the multipurpose wiper into the acetone. Washing the apparatus used in this experiment is a bit stressful because the residue of the mixtures is difficult to remove as their consistency is a bit thicker. Next, the apparatus was wiped with the acetone-soaked wiper until it was fully clean and then being rinsed with the water and let to dry.

3.3.4 Printing process

Before the process of printing, pattern of the conductive ink was created by using the scotch tape with tape surround a rectangular area. The dimension off the conductive ink are 8cm in length and width is 0.5cm. The mount for printing is show in figure 3.6.

Table 3.4 Material and apparatus involved

Material/Apparatus	Descriptions
Scotch tape	Used to create the ink gap on the substrate
Razor blade	Used to apply the ink on all over the gap
PET	Act as the substrate

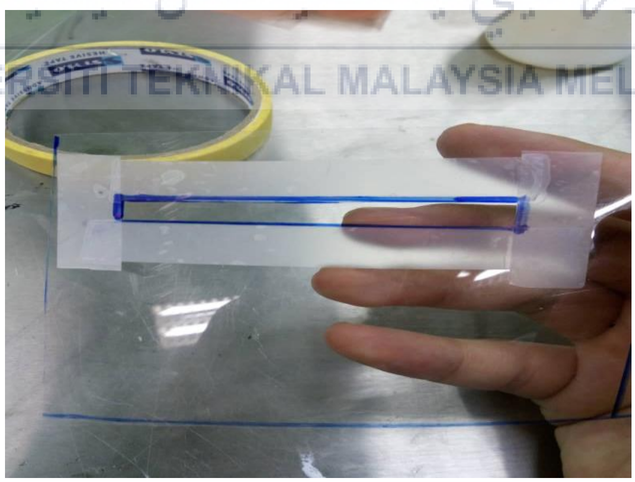


Figure 3.6: pattern of printing

The melting point of Polyethylene terephthalate (PET) is 250-260°C. It does not conduct either electricity or heat. During the experiment, the substrate properties must be kept constant so that the result of this experiment will not be affected.

Thus, the PET is used as the substrate for ink characterization purposes; to investigate the adhesion between the ink and substrate and to identify which ink loading composition will produce the lowest ink resistivity, thus indirectly identifying the best conductivity load.

Only then can the printing process be carried on after the construction of tracks on the PET substrate has been completed (Figure 3.6). The method used in this experiment to print the ink mixture on the substrate was a doctor-blading technique; from this technique it is possible to manually control the thickness and coverage of the ink.

In the method of doctor blading, Nash, C. Et al. described that the ink drops were placed between the scotch tape tracks in order to allow permanent mounting between the substrate and the blade during printing as shown in Figure 3.7.

Figure 3.7 : Ink was put on the PET substrate

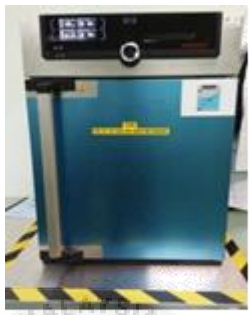
A sharp blade has been used to move across the substratum at a constant speed of approximately 0.01 m / s (Nash, C. et al., 2015) to ensure full space ink coverage. The blade was moved from the top side of the gap to the end of the gap in one move only to ensure that the ink distribution was the same across the gap as shown in Figure 3.8 as well as the ink thickness. The exact same steps can be repeated until satisfied to adjust the ink coverage over the gap.

Figure 3.8: Blade was moved along the gap

3.3.5 Curing process

Curing process is needed to enhance bonding between filler, binder and hardener particles. Curing is also used for the purpose of melting the epoxy and thus hardening the mixture with the help of the hardener to improve the adhesion between the ink and the substrate.

Table 3.5 materials and apparatus

Materials / apparatus	Descriptions
Oven 	Heat source to cure sample
Tray	Sample was put on it in the oven

Matsuhisa, N., 2015. Et al. revealed that the curing temperature of more than 150 °C decreased the resistivity of conductive inks and the resistivity of 2 – 3 μΩ cm carbon conductive inks, only 25 – 88% higher than bulk carbon inks. In addition, in this experiment, the epoxy melting point (Tm) is above 177 ° C; the oven has already been set up at 150 ° C and a time of 30 minutes during the printing process. The purpose of setting up the oven earlier rather than on the spot as it took some time for the oven to raise the temperature to the desired temperature.

Thus, the printed sample placed on a tray can be placed in the oven immediately after the printing process has been completed by setting the oven up earlier, thus not affecting the sample from external factors such as room temperature. It was then allowed to be completely dried at room temperature after the printed sample was cured, so the next procedure can be performed.

3.4 SAMPLE CHARACTERIZATION

After printing, the surface integrity of conductive layers was observed with a microscope from samples printed with different type of conductive ink on PET substrate. Then, conductivity of the conductive inks on the PET substrate will be measured. After that, the conductivity will be measured again after bending test.

3.4.1 Microscopy

Lu, K., in 2013. Microscopy defined as a method for measuring particle shape and size, morphology and nanoparticles arrangements by producing two-dimensional images of three-dimensional objects.

Figure 3.9: microscope with computer

The microscope used in this experiment was connected to the computer for direct display on the screen of the images from the microscope. The sample was placed on stage and held as shown in Figure 3.10(a) by the stage clips.

To secure the position of the required power objective lens, the rotating turret was turned. Three power target lens that are 10x, 20x and 50x were used in this experiment. Next, the focus knob was turned to move the stage up or down without having the objective lens in contact with the sample until the light was directed to the desired position (Figure 3.10(b)), for example at the labeled position 1. The focus knob has been turned into focus mode until the image is in focus mode.

(a) (b)

Figure 3.10: (a) Sample was held by stage clips (b) Light was directed to the position

The image was recorded in the computer after a clear image of the sample was obtained and the image scale was set at 100 µm. In this experiment, three images with different resolutions (10x, 20x and 50x) were required for another power objective lens as at each point.

3.4.2 Electrical testing

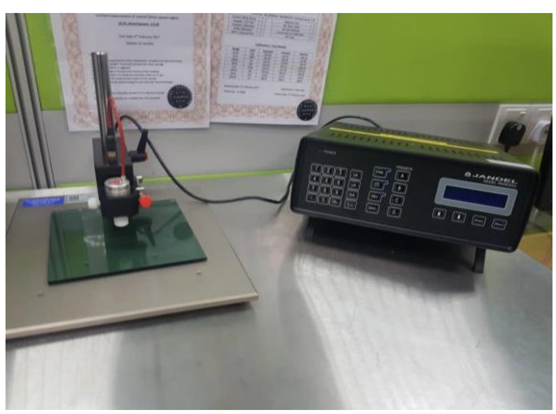


Figure 3.11: four point probe with display meter

The sheet resistance value of the sample in ohms-per-square, the resistivity volume in ohms-cm and the sample thickness can be measured using a four-point probe. The four-point probe works by forcing a constant current along two outer probes and then reading the voltage out of the two inner probes.

Before the sample's resistivity measurement was taken, calibration should be the first to be performed for a reference sample. The reference sample was an indium-tin-oxide (ITO) coated glass to ensure that the four-point probe system works well or not.

The reference glass was placed on the base plate (Figure 3.12) and the probe pin was lowered until the sound of,, tik "reached its limit. The height level was adjusted until the reference glass was in solid contact or touched and waited for the measurement to be stable until the satisfied sheet resistance values were reached.

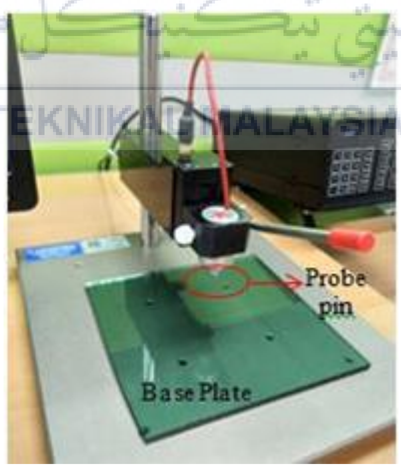


Figure 3.12: based plate and probe pin

While performing the reference calibration, it is necessary to take several measurements from random points across the central reference glass area rather than from a fixed point. Their satisfied values from different random points for the reference glass must be very stable and in agreement within the verified region measurement, which is 12.55 ohms / square 0.25.

The same method as the reference sample was used for the PET in this experiment to obtain the sheet resistance values. The sample was placed on the base plate and adjusted the contact between the track of the ink and the pin of the probe. Before recording the result of the sheet resistance values, it must be in a stable state and the appropriate current has been achieved as it may take some time to reach the best suited current.

Three sheet resistance readings will be taken at each constructed points of the ink track, resulting in their average values. Since each sample has three constructed points, the total readings taken for one sample will be 9 sheet resistance values.

The display meter, however, will display comments such as "contact limit" or "out of range" may be due to poor contact; too close or not at all. A suitable action should therefore be taken to enable the display meter to read the values. Poor contact can be detected by high standard deviations from several measurements taken from similar areas to be discussed in the next chapter.

3.4.4 Adhesion vs conductivity testing

This testing involved the measurement of the conductivity of the conductive ink after the sample was bending.

3.4.4.2 Bending test

The resistance of sample was measure during bending test as a function of change in angle. The bending was performed using a mechanical bending test machine (figure 3.13).

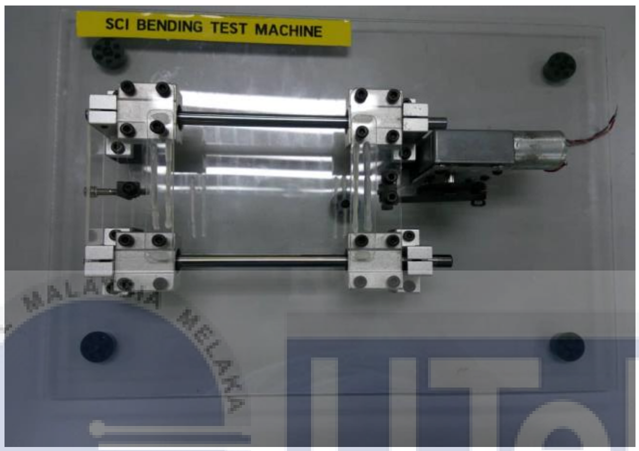


Figure 3.13: bending test machine

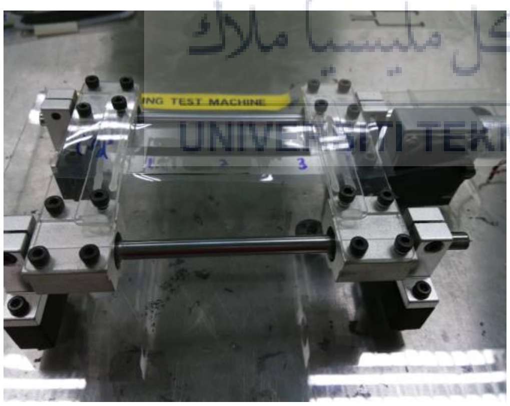


Figure 3.14: sample before bending

figure 3.15: sample under bending

First, fix the sample with two gripping jigs in the mechanical bending test machine which shown in figure 3.14. Then, using multimeter to the measure the resistance from point 1 to point 3 which is origin position of the sample. After that, move the moving part closer to the fixed part until it touch each other which shown in figure 3.15. Measure again the resistance from point 1 to point 3. In a similar manner, the moving back to its origin position and measure the resistance again. Repeat the steps with three time for all samples.

SUMMARY

Literature review provide the information about the formulation of conductive ink, difference type of substrate, method of printing, methods of production to fabricate ink and the knowledge of electrical properties.

After study the literature review, the filler element for produce the conductive ink is nanoparticle silver and doctor-blading method is selected as the printing method. In addition, Polyethylene terephthalate (PET) will be use as the baseline. Mechanical testing was taken place to test the printability of the conductive inks on the PET substrate.

The experimental work will include the testing of the adhesion versus conductivity of the sample. The conductivity of the sample will be measure after bending and stretching. This experiment will show that how the conductivity change when the sample is under bending condition. All the data will be recorded after experiment.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

In this chapter, the resistivity, the stability, the microstructure of and the conductivity after bending test will be discussed to determine the printability of the conductive ink on the PET substrate based on the data gained through four points probes, multimeter and the images from the microscope.

4.2 ANALYSIS OF ELECTRICAL PROPERTIES

The conductivity of the sample with different filler loading will be measured and all the data will be recorded in the chapter.

4.2.1 Result of resistivity

From the Table 4.1, there are four different samples which are 30%wt, 60%wt, 90%wt and 100%wt of carbon conductive paint. Three points were selected to be measure where each point measure three times by using four point probe.

Table 4.1 Resistivity results

% wt	sample	Resistivity(Ω/sq)					
		Point 1	average	Point 2	average	Point 3	average
60	1	9157.26	9240.35	13381.91	13348.22	10337.50	10503.38
		9170.40		13284.46		10487.97	
		9393.40		13378.29		10684.68	
	2	10042.44	10033.38	12520.76	12407.15	14805.54	14655.52
		10029.75		12467.73		14706.73	
		10027.94		12232.95		14454.28	
	3	12239.29	12240.05	9092.45	9048.94	13725.47	13358.22
		12243.37		8881.69		13591.76	
		12237.48		9172.67		13357.44	
90	1	140.83	140.18	162.07	161.97	180.49	180.62
		140.06		161.87		180.43	
		139.66		161.96		180.94	
	2	167.93	167.35	191.86	191.21	144.04	143.65
		167.20		190.09		143.77	
		166.93		191.68		143.13	
	3	159.83	159.88	206.85	205.97	216.86	215.13
		159.90		205.49		215.69	
		159.91		205.56		215.13	
100	1	51.81	51.63	62.32	62.10	66.04	65.86
		51.62		62.09		65.86	
		51.44		61.87		65.67	
	2	48.77	48.72	59.96	59.98	82.49	82.51
		48.72		60.01		82.49	
		48.68		59.96		82.54	
	3	48.72	48.71	59.56	59.51	80.31	80.34
		48.72		59.56		80.36	
		48.68		59.42		80.36	

Its average resistivity is the highest among three of the filler percentage at 60 percent of the filler; therefore, it can produce the lowest conductivity as the resistivity value is proportional to the conductivity value. The 90 percent values were much lower than the 60 percent values; as shown in the table, there was a huge gap between their values.

Table 4.2: average result

%wt \ sample	1	2	3
	Resistivity(Ω/sq)		
60	11030.65	12365.35	11549.07
90	160.92	186.38	193.66
100	65.41	63.73	62.85

In addition, the graph showed the total average resistivity against the filler loading percentage in Figure 4.1. It showed that even the ink formulation used in different samples was the same; it still showed some differences in resistivity value. The graph indicated that the average resistivity will alternatively decrease as the amount of conductive materials in the sample increased when the percentage of filler loading increased.

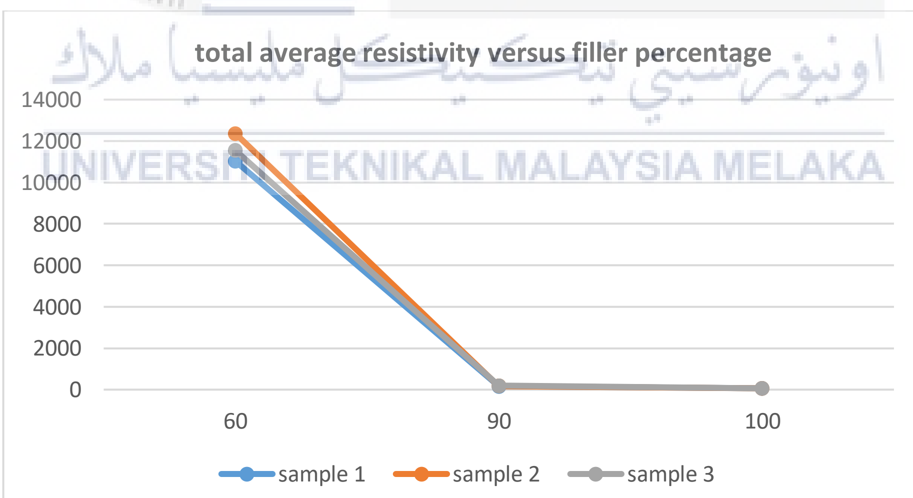


Figure 4.1 Graph of total average resistivity versus filler percentage

For carbon-conductive paint at 90%wt and carbon-conductive paint at 100%wt, their average resistivity value is gradually reduced by increasing the carbon-conductive paint percentage. The highest conductivity sample is 90% conductive paint. The 100% carbon-conductive paint is only used as a reference when compared to other samples.

As for the 30%wt carbon-conductive ink, the four-point probe did not detect any resistivity value of the printed ink as it may lack the percentage of conductive materials in the ink. It showed that as can be seen in Figure 4.1, the lower the percentage of conductor filler, the lower the conductivity with increased resistivity. Therefore, for this analysis, they were not included in the data tabulation.

4.2.2 Analysis of stability

There were two types of comparison in this analysis, which were: comparison of standard deviation between the percentage of carbon-conductive paint and comparison of error estimation between the percentage of carbon conductive paint.

4.2.2.1 Comparison of standard deviation between filler percentage

There is no standard benchmark for determining the standard deviation, but it must be in the lowest value to know the best standard deviation. It can show how tightly the data is collected around the mean or average or how far from the mean or average the data is spread.

Table 4.3 Standard deviation results

Filler %wt	sample	Standard deviation			Overall Standard deviation
		point 1	point 2	point 3	
60	1	132.712	55.251	174.103	120.687
	2	7.901	153.171	181.148	114.073
	3	3.0173	150.291	186.232	113.180
90	1	0.595	0.100	0.279	0.325
	2	0.517	0.974	0.467	0.653
	3	0.046	0.766	0.883	0.565
100	1	0.185	0.225	0.185	0.198
	2	0.045	0.029	0.029	0.0343
	3	0.023	0.081	0.029	0.0443

From table 4.2, the overall standard deviation of 60%wt of carbon conductive paint show a very high value which mean the data is widespread on average, indicated they have the highest resistivity on average. After the carbon conductive paint increase to 90%wt, the overall standard deviation is become much lower compared with the 60%wt which mean the range of the data are very small. While for the carbon conductive paint at 100%, the data has a small standard deviation which indicates that data is collected close to the average, proving that 100% wt of carbon-conductive paint has the lowest and most stable average resistivity.

4.2.2.2 Comparison of estimation of error between filler percentage

It is known that data in the range of 5% and below should obtain the standard E. It showed from Table 4.3 that 100% of the filler had the highest E of all the fillers, be it from Sample 1 to 3, which means it was completely out of range from standard E.

Table 4.4 Average estimation of error between filler percentage

Filler %wt	sample	Average Estimation Error (%)		
		point 1	point 2	point 3
60	1	16.23	21.01	4.78
	2	18.86	0.34	18.52
	3	5.98	21.65	15.66
90	1	12.89	0.65	12.24
	2	10.21	2.59	22.93
	3	17.44	6.36	11.09
100	1	21.07	5.06	0.69
	2	23.55	5.88	29.47
	3	22.50	5.31	27.83

The estimate of error, E is calculated based on the equation:

$$E = \left|\frac{resistivity\ at\ on\ point - Average\ resistivity}{Average\ Resistivity}\right| \times 100\%$$

For example, to calculate the maximum error estimate, E for Sample 1 is 60% filler loading from the Table 4.1 and Table 4.2:

Resistivity at sample 1 = 9240.35, 10033.48, 12240.05; Average resistivity = 11030.65

Thus, $$E = \left|\frac{9240.35 - 11030.65}{11030.65}\right| \times 100\% = 16.23\%$$

$$E = \left|\frac{13348.2 - 11030.65}{11030.65}\right| \times 100\% = 21.01\%$$

$$E = \left|\frac{10503.4 - 11030.65}{11030.65}\right| \times 100\% = 4.87\%$$

The average estimate error of the carbon conductive paint at 60%wt were in the range between 0 ~ 22%, 0 ~ 23% for 90%wt and 0 ~ 30% for 100%wt of conductive. Most of the average estimate error for all sample were relatively high. The unstable range of E that occurred may be due to two reasons; the technique of printing and the measurement of four point probe.

4.2.3 Sources of potential error

There are two sources of potential errors that had been presumed, which were due to printing technique and four point probe measurement.

4.2.3.1 Printing technique

In 2006, in order to produce fewer flaws of conductive tracks with good resolution, Kim, D. et al. proposed that there is a need to alter the silver ink composition and some printing states, consisting of the speed when printing which is linked to the firing frequency, the temperature of substrate and the inter-spacing interval among the dots.

In this case, the inconsistent range of *E* may be due to the printing technique; doctor-blading method. During the printing process, the ink may be not well-distributed all over the gap between the Scotch tape on the PET substrate when the blade was moved across the gap due to the speed or the viscosity of ink.

When the speed of moving the blade is high, it may be the loss in ink where the ink may not cover all over the gap region. While for the viscosity of ink, as the filler content is higher, it will follow to increase as well. Ink with high viscosity was hard to be printed in compliance to the texture of ink; more concentrated texture.

Thus, it may affect the thickness of ink tracks printed on the PET substrate. Some regions may have different thickness, thin or thick which leads to the different spreads of conducting material, carbon conductive paint on the substrate where region with high content of silver will have low resistivity and vice versa.

4.2.3.2 Four point probe measurement

Another factor of the instability in is the time condition while taking the measurement using the four point probe. The error happened may be caused by there was no adequate time spent before taking the data. The time is needed due to RC (resistance-capacitance) delay in the highest resistive samples as the current requires some times to climb up to the value of saturation.

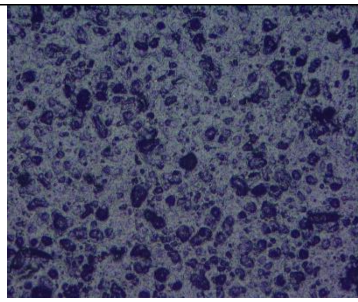
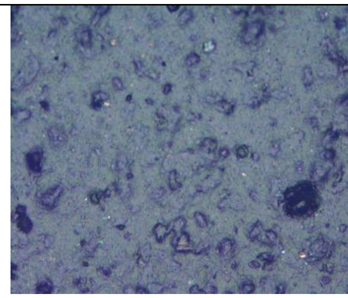
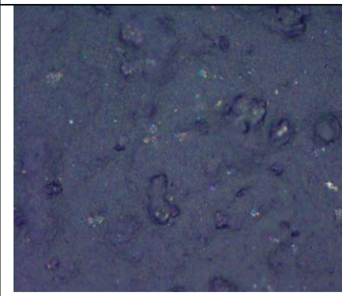
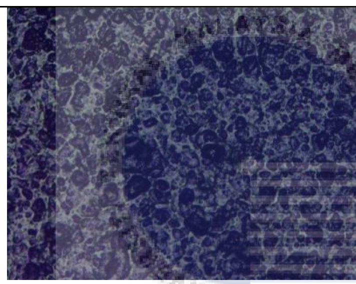
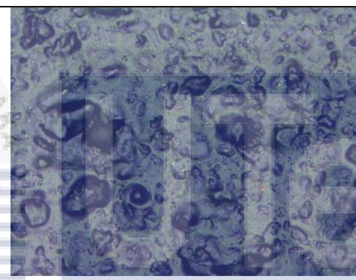
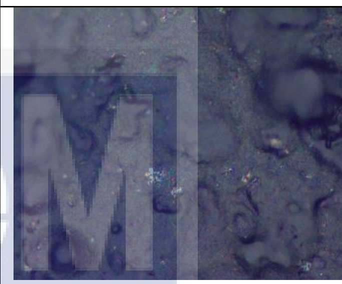
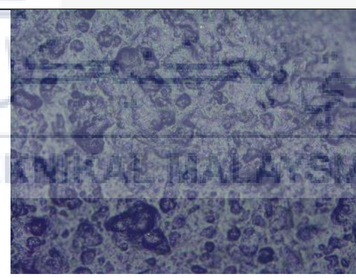
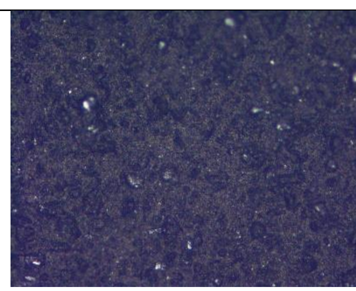
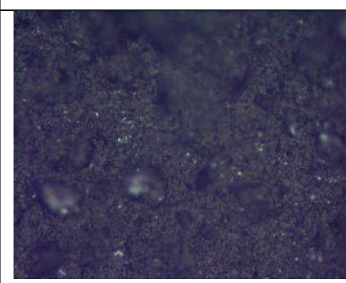
E

Once the data is stable, only then a certain point of measurement can be taken and the average value can be obtained. Hence, when the time spent is inconsistent, so does the resistivity value where it will be unstable too.

4.3 ANALYSIS OF MORPHOLOGICAL

Light microscopy was used to observe the surface integrity of the printed conductive ink on the substrate. In this section, microscopy can show the microstructure image of the sample and the strength of the conductivity can be explained through the microstructure image. There are three scale of magnifications which were 10x, 20x and 50x.

Table 4.5 Microstructure

Filler (%wt)	Magnifications		
	10×	20×	50×
30			
60			
90			
100			

In Table 4.4, the microstructural transformation of the carbon conductive ink based on the filler loading of 30%wt, 60%wt, 90%wt and 100%wt is shown. For 30%wt of conductive paint, it can clearly see that there was a very big gap between the carbon particle. It exhibited the frequency of gaps between carbon particles that will affect the conductive ink layer's electrical properties as the increasing number of gaps will cause the resistance to increase due to the high voltage required to ensure current flow between the carbon particles.

Next, the gap between carbon particle in the 60%wt carbon conductive paint relatively small compared to 30%wt. Thus, 60%wt of sample can conduct electricity but the gap between the carbon particles cause the resistivity of the sample are very high. For 60%wt of carbon conductive paint, the ink layer had the presence of granular-like particle and according to Kim, D., & Moon, J. in 2005, For conductive ink, the granular particle should contain a 3D conductive connection leading to particle necking. Kim, D., and Moon, J. It is also believed that the neck growth provides a continuous connection and that once the interparticle neck is produced, the granular-like particle will be conductive even if it is still porous.

For 90%wt of carbon conductive, the dark color of the microstructure represented the presence of carbon while the brighter region was the binder after it had already melted and major changes since most of the particle barriers had disappeared. This indicated that the ink layers have a close-packed structure where the particles created a strong bonding between each other. The close-packed structure causes the conductivity of the sample consist of high conductivity.

For 100%wt, the dark colour region in the microstructure are very similar with the 90%wt. But, it clearly shown that the carbon particles were closer pack compared with the 90%wt one. Once the carbon particles were in contact with each other, the particles became more continuous rather than discrete and spherical in shape, thus increasing the area of contact between particles. This situation causes the conductivity of 100%wt is higher than 90%wt.

4.4 ANALYSIS OF RESISTIVITY UNDER BENDING

The result of the resistivity of the samples before and after bending are shown in table 4.5.

Table 4.6 the resistivity before and after bending

Filler (%wt)	sample	Resistivity(kΩ)			
		Before bending	Average	After bending	Average
60	1	102.4	102.8	101.7	102.1
		103.2		102.8	
		102.8		101.9	
	2	108.8	107.4	107.3	104.1
		108.2		103.6	
		105.1		101.5	
	3	137.6	138.9	135.4	136.3
		138.7		137.9	
		140.3		135.7	
90	1	2.137	2.070	2.065	2.062
		2.048		2.042	
		2.026		2.078	
	2	2.164	2.215	2.176	2.174
		2.243		2.124	
		2.237		2.221	
	3	2.054	2.154	2.194	2.187
		2.191		2.215	
		2.217		2.153	
100	1	0.642	0.643	0.671	0.656
		0.643		0.656	
		0.643		0.641	
	2	0.714	0.742	0.763	0.751
		0.762		0.729	
		0.750		0.761	
	3	0.797	0.793	0.795	0.795
		0.787		0.793	
		0.796		0.798	

For filler of 60%wt, there are three samples and they resistivity have been measured before and after bending. The resistivity for the sample 1 is 102.8 kΩ at the original state and 102.1 kΩ under bending condition. Based on the result, the resistivity of the sample before and after bending condition are almost same. The result for the sample 2 and 3 of the 60%wt of carbon conductive paint are similar with the result of sample 1. The measurement of the resistivity of sample 2 and 3 before and after bending are very small so consider as no resistivity change in the testing. This situation also happens for all samples of 90%wt and 100%wt of carbon conductive paint. In conclusion, bend the sample will not affect much for the resistivity of conductive ink.

The resistivity change before and after bending are very less, indicating a good stability due to their bridging effect and strong interconnections with carbon particle. This is because the carbon particles still connect to each other after the sample had been bend. Bend a very thin flexible substrate which thickness is less than 0.5mm doesn't not increase much the distance between the particles, this make the carbon particles still connect to each other and this cause the resistivity change of the conductive ink are very less.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 INTRODUCTION

From this chapter, a summarized conclusion was described after all the experiment had been done so that the requirement from three object will be fulfilled where the relationship between sheet resistivity, mechanical testing and morphological for carbon-filled epoxy conductive ink has been identified.

5.2 CONCLUSIONS

The formulation of ink in the study is carbon conductive paint, epoxy and hardener. Then, polyethylene terephthalate (PET) was selected as the substrate. Then, the processes need to carry out to fabricate the ink on the substrates are the process of mixing, printing and curing. After the sample has been fabricated though all this process, the behavior of the ink can be investigated through the electrical testing, mechanical testing and microscopy testing.

There are four different of %wt filler of conductive inks had been fabricated which were 30%wt, 60%wt, 90%wt and 100%wt respectively. All the sample were test by four point probe and found that 30%wt cannot conduct electricity, 60%wt can conduct electricity but the resistivity were very high, 90%wt can conduct electricity with low resistivity and the highest conductivity sample is 100%wt of carbon conductive paint.

In bending test section, the result of resistivity testing for all the samples with different %wt before and after bending are almost same. Thus, bend the sample is not affect the conductivity of the conductive ink.

As for the morphological analysis, the surface integrity was observed by a microscope. The microstructure of the sample provided the information of the conductivity of the conductive ink. The is because the distance among the carbon particles can be observed. The relationship between the %wt of carbon conductive paint in conductive ink and the resistivity was determined through morphological.

5.3 RECOMMENTADION

For future works in this project, it is recommended that the different type of epoxies can be used to fabricate the conductive ink. Try different type of epoxies to fabricate conductive ink can find out the most suitable epoxy that can print on the Polyethylene Terephthalate (PET) with strong adhesion between the conductive ink and substrate. On the other hand, the suggestion of the filler used to produce conductive ink can be change to silver nanoparticle, silver flake, copper and others in order to produce a better conductive ink.

REFERENCE

Abou El-Nour, K. M. M. *et al.* (2010) ‘Synthesis and applications of silver nanoparticles’, *Arabian Journal of Chemistry*. doi: 10.1016/j.arabjc.2010.04.008.

Allen, M. L. *et al.* (2008) ‘Electrical sintering of nanoparticle structures’, *Nanotechnology*. doi: 10.1088/0957-4484/19/17/175201.

Chen, T. *et al.* (2012) ‘An integrated “energy wire” for both photoelectric conversion and energy storage’, *Angewandte Chemie - International Edition*. doi: 10.1002/anie.201207023.

Chen, X., Sun, M. and Xiao, F. (2010) ‘Preparation of nano silver pastes applied in printed electronics’, in *Proceedings - 2010 11th International Conference on Electronic Packaging Technology and High Density Packaging, ICEPT-HDP 2010*. doi: 10.1109/ICEPT.2010.5582419.

Chien Dang, M., Dung Dang, T. M. and Fribourg-Blanc, E. (2015) ‘Silver nanoparticles ink synthesis for conductive patterns fabrication using inkjet printing technology’, in *Advances in Natural Sciences: Nanoscience and Nanotechnology*. doi: 10.1088/2043-6262/6/1/015003.

Denneulin, A. *et al.* (2011) ‘Impact of ink formulation on carbon nanotube network organization within inkjet printed conductive films’, *Carbon*. doi: 10.1016/j.carbon.2011.02.012.

Falat, T. *et al.* (2009) ‘An Approach to measurement and evaluation of the thermal conductivity of the thermal adhesives in electronic packaging', in *ISSE 2009: 32nd International Spring Seminar on Electronics Technology: Hetero System Integration, the path to New Solutions in the Modern Electronics - Conference Proceedings*. doi: 10.1109/ISSE.2009.5207017.

German, R. M. and German, R. M. (2014) *Sintering: from Empirical Observations to Scientific Principles, Sintering: from Empirical Observations to Scientific Principles*. doi: 10.1016/B978-0-12-401682-8.00011-2.

Ghediya, P. R. and Chaudhuri, T. K. (2015) 'Doctor-blade printing of Cu2ZnSnS4 films from microwave-processed ink', *Journal of Materials Science: Materials in Electronics*. doi: 10.1007/s10854-014-2628-1.

Hrehorova, E. *et al.* (2011) 'Gravure printing of conductive inks on glass substrates for applications in printed electronics', *IEEE/OSA Journal of Display Technology*. doi: 10.1109/JDT.2010.2065214.

Hu, Q. *et al.* (2016) 'Large-area perovskite nanowire arrays fabricated by large-scale roll-to-roll micro-gravure printing and doctor blading', *Nanoscale*. doi: 10.1039/c5nr08277c.

Hyun, W. J. *et al.* (2015a) 'High-resolution patterning of graphene by screen printing with a silicon stencil for highly flexible printed electronics', *Advanced Materials*. doi: 10.1002/adma.201404133.

Hyun, W. J. *et al.* (2015b) 'High-resolution patterning of graphene by screen printing with a silicon stencil for highly flexible printed electronics', *Advanced Materials*. doi: 10.1002/adma.201404133.

Kamyshny, A. (2011) ‘Metal-based Inkjet Inks for Printed Electronics’, *The Open Applied Physics Journal*. doi: 10.2174/1874183501104010019.

Kazani, I. *et al.* (2012) ‘Electrical conductive textiles obtained by screen printing’, *Fibres and Textiles in Eastern Europe*.

Khirotdin, R. K., Cheng, T. S. and Mokhtar, K. A. (2016) ‘Printing of conductive INK tracks on textiles using silkscreen printing’, *ARPN Journal of Engineering and Applied Sciences*.

Kim, D. *et al.* (2006) ‘Ink-jet printing of silver conductive tracks on flexible substrates’, *Molecular Crystals and Liquid Crystals*. doi: 10.1080/15421400600930458.

Kishi, H. *et al.* (2016) ‘Self-assembled three-dimensional structure of epoxy/polyethersulphone/silver adhesives with electrical conductivity’, *Polymer*. doi: 10.1016/j.polymer.2015.11.043.

Meena, J. S. *et al.* (2010) ‘Flexible metal-insulator-metal capacitor using plasma enhanced binary hafnium-zirconium-oxide as gate dielectric layer’, *Microelectronics Reliability*. doi: 10.1016/j.microrel.2010.01.046.

Merilampi, S., Laine-Ma, T. and Ruuskanen, P. (2009) ‘The characterization of electrically conductive silver ink patterns on flexible substrates’, *Microelectronics Reliability*. doi: 10.1016/j.microrel.2009.04.004.

Riemer, D. E. (1989) ‘The Theoretical Fundamentals of the Screen Printing Process’, *Microelectronics International: An International Journal*. doi: 10.1108/eb044350.

Schwartz, M. (2010) ‘Casting’, in *Innovations in Materials Manufacturing, Fabrication, and Environmental Safety*. doi: 10.1201/b10386.

Secor, E. B. *et al.* (2014) ‘Gravure printing of graphene for large-area flexible electronics’, *Advanced Materials*. doi: 10.1002/adma.201401052.

Shen, W. *et al.* (2014) ‘Preparation of solid silver nanoparticles for inkjet printed flexible electronics with high conductivity’, *Nanoscale*. doi: 10.1039/c3nr05479a.

Suganuma, K. (2014) ‘Printing Technology’, in *Introduction to Printed ELectronics*. doi: 10.1007/978-1-4614-9625-0.

Sumerel, J. *et al.* (2006) ‘Piezoelectric ink jet processing of materials for medical and biological applications’, *Biotechnology Journal*. doi: 10.1002/biot.200600123.

Tran, T. S., Dutta, N. K. and Choudhury, N. R. (2018) ‘Graphene inks for printed flexible electronics: Graphene dispersions, ink formulations, printing techniques and applications’, *Advances in Colloid and Interface Science*. doi: 10.1016/j.cis.2018.09.003.

Wang, X. *et al.* (2014) ‘Flexible energy-storage devices: Design consideration and recent progress’, *Advanced Materials*. doi: 10.1002/adma.201400910.

Zhang, R. *et al.* (2011) ‘A simple, low-cost approach to prepare flexible highly conductive polymer composites by in situ reduction of silver carboxylate for flexible electronic applications’, *Composites Science and Technology*. doi: 10.1016/j.compscitech.2011.01.001.

APPENDICES

Topic	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Articles and Journals Reading															
Experiment design															
Formulation of conductive ink															
fabrication															
Discuss with supervisor about the process															
Make improvement to the experiment design															
Fabricate the sample and measure the resistivity															
Preliminary data analysis															
Report Writing															
Submission of PSM I Report															

Gantt Chart for PSM 1

Topic	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Articles and Journals Reading															
Preparation of formulation of conductive ink															
Formulation of conductive ink															
Print the conductive ink onto PET substrate															
Measure of resistivity of the sample															
morphological															
Bending test for the sample															
Results and Discussion															
Report Writing															
Submission of PSM I Report															

Gantt Chart for PSM 2