ADHESION INTEGRITY OF EPOXY PASTE ON THERMOPLASTIC POLYURETHANE(TPU)

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ADHESION INTEGRITY OF EPOXY PASTE ON THERMOPLASTIC POLYURETHANE(TPU)

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This report is submitted
in fulfillment of the requirement for the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering

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DECLARATION

I declare that this project report entitled "Adhesion integrity of epoxy on thermoplastic polyurethane (TPU)" is the result of my own work except as cited in the references

Signature : ....

Name : ....

Date : ....

APPROVAL

I hereby declare that I have read this project report 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 (Structure & Materials).

Signature : ....

Name of Supervisor :..................................

Date : ....

DEDICATION

To my beloved father Tan Yong Lee and mother Toh Mei Mei.

ABSTRACT

This study aspires to have an analysis of carbon filled epoxy conductive ink printed on thermoplastic polyurethane (TPU). The objectives of this study are to evaluate the resistivity of different filler loading of conductive epoxy on TPU substrate, compare the resistivity of epoxy paste under tensile test, and observe the morphological microstructure between epoxy paste and TPU. Specifically, the parameters that were evaluated adhesion integrity which include of sheet resistivity, tensile test and morphological analysis. This project starts with printing of conductive ink on TPU and cured using an oven. In experiment, multimeter is used to measure the sheet resistance value of the sample in ohms-per-square. It can detect the resistivity of different percentage of ink loading. The highest average resistivity is detected in low percentage of ink loading while the lowest one is found in high percentage of ink loading. In tensile test, a self made machine is used to stretch the TPU to test conductivity of conductive ink.The resistivity increase as the conductive TPU stretch, the resisitance decreased. In morphological analysis, light microscope is used to visualize the microscopic image of carbon ink categorized by the electrical properties of the ink. Ink loading with high percentage of carbon particles has conductivity while low percentage has no conductivity. At low percentage, the microstructure image shows no appearance of carbon particles element while for high percentage; the image shows the content of filler loading. The future researchers can use different type of substrate or different materials of conductive ink in order to do the same analysis.

ABSTRAK

Kajian ini bercita-cita untuk menganalisis dakwat konduktif karbon yang mengandungi epoksi. Objektif kajian termasuk menilai berbeza konduktif karbon, berbanding resistiviti semasa ujian tagangan dan memperhati morfologi karbon mikrostrukrur yang cetak atas TPU. Secara khususnya, parameter yang dinilai terdiri daripada resistiviti, ujian tegangan dan analisis morfologi. Untuk mencapai analisis, prob empat titik digunakan untuk mengukur nilai rintangan lembaran sampel dalam ohms-per-kuadrat. Beberapa peratusan kandungan dakwat telah mengesan kehadiran resistiviti dan beberapa peratusan lagi tidak mengesan kehadiran resistivitas. Purata konduktiviti tertinggi dikesan dalam kandungan dakwat yang berperatusan tinggi manakala resistiviti yang paling tinggi didapati dalam kandungan dakwat yang berperatusanrendah. Dari segi tegangan, mesin mudah digunakan di mana sampel untek regang. Sementara itu, bagi sampel yang mengandungi peratusan pengisi tinggi mudah pecah. Dalam analisis morfologi, mikroskop cahaya digunakan untuk memvisualisasikan imej mikroskopik dakwat konduktif karbon yang dikategorikan oleh sifat-sifat elektrik dakwat. Kandungan dakwat dengan peratusan karbon yang lebih tinggi mempunyai kekonduksian manakala dakwat yang berperatusan rendah tidak mempunyai konduktiviti. Pada peratusan rendah, imej mikrostruktur tidak memperlihatkan unsur nanopartikel perak manakala peratusan yang tinggi; imej menunjukkan kandungan pengisi yang berkilau. Penyelidik masa depan boleh menggunakan jenis substrat yang berlainan atau bahan berlainan dalam dakwat konduktif untuk melakukan analisis yang sama.

ACKNOWLEDGEMENTS

Thanks to my supervisor Dr. Ghazali bin Omar for giving me this opportunity to do final year project with him. He never hesitated to give me advice and guidance whenever I confronted problems. I am thankful for his patience and advice while leading me in this project.

I revere the patronage and moral support extended with love, by my parents whose passionate encouragement made it possible for me to complete this project and not to forget my late father who is always in my prayers.

I am deeply indebted to all concerned persons who cooperated with me in this regard for their valuable help in preparing this project. My joy knows no bounds in expressing my cordial gratitude to my friends as their keen interest and encouragement were a great help throughout the course of this project.

I humbly extend my thanks to all people that involved either directly or indirectly in order to complete this project.

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LIST OF ABBREVIATIONS

ρ	-	Resistivity
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

Recent few years, stretchable and flexible conductive products have attracted considerable attention for their potential applications in wearable electronics, smart textiles, soft robotics, and structural health monitoring. Numerous efforts put on development of high sensitive and stretchable sensor which can be compliant and sensitive to detect the process of body motions and work to monitor deformations (Chang,2015). Therefore, conductive epoxy paste with electrical stabilty and material under large strains should be considered.

Thermoplastic polyurethane (TPU) is a linear block copolymers with domains of varying length resulting in a polymer with both hard and soft domains with varying degrees of crystallization. TPU is widely used for properties like high abrasion resistance, high performance at low temperature, high shear strength, elasticity, transparency and oil and grease resistance.

Conductive epoxy used to form electrically conductive bond between dissimilar materials eliminating need for soldering. Polymer composite materials are perfect candidates as lossy media, since their conductivity can be improved by adding fillers(Valentini,2014). Good adhesion epoxy paste can minimize the stress risers at soft interfaces like TPU that develop during stretching and can avoid to substrate failure.

Instead of bulky and rigid wires, a stretchable conductive technology can be used to carry the electrical signals. The conductive ink is often screen printed onto TPU. Reliability is a concern in many applications due to stretching, compression and bending. Interface adhesion between printed materials like TPU and contacts (Au, Ag, Cu, Al, etc.) is important for print quality and reliability (Steven,2016). Conductive epoxy paste are regularly printed on plastic sheets like TPU. Poor electrical performance, mechanical strength, and adhesion integrity limits interest in printing technology for the semiconductor industry.

1.2 PROBLEM STATEMENT

Nowadays, the use of conductive polymer is become a trend of topic since it involved in a variety of applications in electronic devices, electronic packaging, military and health care industry.The bonding and joining between epoxy paste and TPU is an important issue. In this case, different filler loading is important because it will affect the electricity of object. Besides, stretchable conductive technology are widely used. The stretching of printed conductive adhesive will affect conductivity.

In this study will focus on promoting the adhesion integrity of epoxy paste on thermoplastic Polyurethane (TPU) by identify the effective adhesive material for TPU polymer because defect of printed conductive ink lead to failure of conductivity.

1.3 OBJECTIVE

The objectives of this project are as follows:

1. To evaluate the resistivity of different filler loading of conductive epoxy on TPU substrate
2. To compare the resistivity of epoxy paste under tensile test
3. To observe the morphological microstructure between epoxy paste and TPU

1.4 SCOPE OF PROJECT

The scopes of this project are include the preparation of conductive epoxy paste, printing of epoxy paste on TPU and undergo test on adhesion integrity of epoxy paste and TPU. To test resistivity and mechanical property of conductive epoxy paste, conductivity test, tensile test and observation are carried out.

1.5 THESIS CONTENT

The actions that need to be carried out to achieve the objectives in this project are listed below.

1. Literature review

Journals, articles, or any materials regarding the project will be reviewed.

2. Preparation of printing

Conductive epoxy paste prepared for printing on TPU which consist of polymer epoxy, conductive fillers, solvents and additives.

3. Printing

Print the conductive epoxy on TPU substrate respectively.

4. Testing of Epoxy Paste Printing

Do mechanical testing like tensile test . Besides, conductivity test to test whether there’s any conductive electricity after tensile. Observe the adhesity of TPU and epoxy using light microscope.

5. Analysis

Analysis will be presented on the tensile test, bending test and conductivity test. Adhesity integrity between epoxy paste and TPU is analyse by tests.

6. Report writing

A report on this study will be written at the end of the project.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The sources from previous studies on TPU, conductive epoxy, methods of printing, heat treatment, material of substrates, method of mechanical testing and the conductive ink characterization are being presented with the sources from previous studies in this field.

2.2 CONDUCTIVE INK

According to Ilda Kazani, conductive inks have been used in many applications include electronics, computers and communications. PCBs (Printed circuit boards), RFID tags (Radio-frequency identification) or wiring boards are example of applications of conductive inks. Diverse technologies such as notably screen printing have been applied at the most. Primarily, conductive inks are composed of a filler, binder,solvent(s) and additives (Sughosh, 2013). The conductive fillers are what provide the conductive properties, while the binder provides the needed adhesion to the substrate and cohesion to each other. Thus, the conductivity of an ink depends upon the amount of filler loading, particle size of the fillers, percentage of binder used and continuity of the printed layer after printing and drying.

2.2.1 EPOXY

Epoxies are mixture of resin and hardener. Normally, epoxy resins are widely used in industrial applications due to their properties which include toughness, strong adhesion, chemical resistance and other specialized properties. The preferred epoxy resin for use with the present invention is a solid or liquid epoxy resin (YueXiao, 2000).

2.2.2 Metal-based materials

In 2007, Hye-Jin Cho state that the metal nanoparticles used in a conductive ink composition include one or more nanoparticles from a group consisting of silver (Ag), gold (Au), copper (Cu), nickel (Ni), palladium (Pd), platinum (Pt), and alloys thereof. f. The particle size of the metal nanooparticles may be 20 to 50 nm. According to Ilda Kazani, metals like copper, silver and gold are used in conductive inks. The conductive ink are made of a dispersion of metal particles and suitable resins in an organic or inorganic solvent. The less electrimigration property of copper make it a good candidate material but the unstable thermodynamical property in atmospheric conditions will lead to oxidation and deterioriate the conductivity (Park,2007). Compared to gold ink, silverbased ink is better option due to lower price.

2.2.2.1 Silver

According to Shangjie Chang state that silver nanowires have high optical and electrical properties. Thus, there are widely used in flexible devices. Mohammed Mohammed Ali stated in 2017 that silver nanowire which print on TPU substrate can maintain electrical conductivity for tensile strain ranging from 16%-30%. Normally, the viscous nature and good adhesion capabilities makes silver compatible with screen printing process. Besides, Wenfeng Shen stated that silver nanoparticles were easily stored without degeneration and can synthesized with epoxy in an aqueous phase in 2014. The inks printed on paper in different patterning techniques. The resulting high conductivity of the printed silver patterns makes it suitable in the fabrication of flexible electronic devices.

2.2.2.2 Copper

Michael Grouchko states in Journal of Materials Chemistry that copper nanoparticles widely used in inkjet printing of conductive patterns because it is a low-cost replacement for silver and gold nanoparticles which are currently used in . According to Park, B. K., copper is an excellent candidate material due to its lower cost and less electromigration effect than novel metals. The oxidation of copper can be happened spontaniously which lead to the failure of conductivity.

2.2.2.3 Carbon nanotube (CNT)

Li Li Zhang state that carbon-based materials include activated carbons (ACs) to carbon nanotubes (CNTs) are the most widely used due to their excellent physical and chemical properties.For example, carbon-based material have properties like low cost, different form of carbon, ease of processability, relatively inert electrochemistry, and controllable porosity.

2.2.3 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.

2.3 METHODS OF PRINTING

According to Cian Nash, to achieve continuous metallic pattern in a different kind of substrates, the direct printing of metals is important technique. Conductive inks used in different application as conductive connectors printed on paper or substrates which have strict requirements in terms of performance and stability. The inks can be applied on a variety of substrates using different printing method to obtain the desired patterns. For example, coating techniques include spin, spray, curtain, casting, painting, knife-over-edge coating, doctor blading are the printing methods used in different situation.

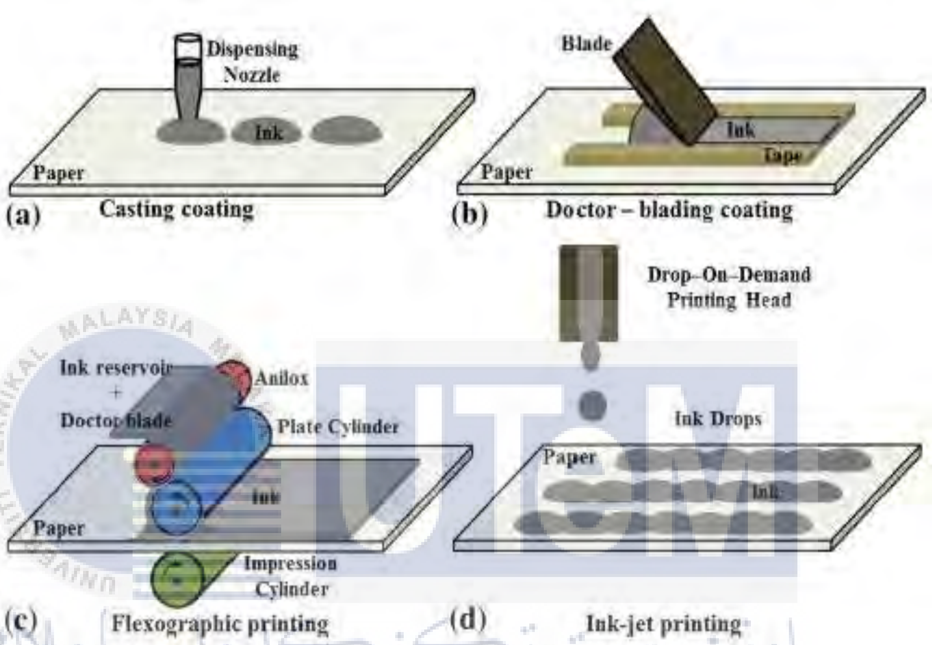


Figure 2.1 Methods of depositing the ink tracks

2.3.1 Doctor-blading

Paul D. Fleming III state that doctor blade is a printing method that removes the excess ink using blade. In the paper coating processes, doctor blading method are also used broadly. The doctor blade used to wipe the paste on the substrate with controlled adjustable angle.

According to Cian Nash, film formation that have 10 lm to 500 lm thickness range are allowed to use this printing method. Doctor blading method can minimized the loss of ink. In the end of printing, the thickness of the film will proportional to the width of gap and the filler loading of the solid material.

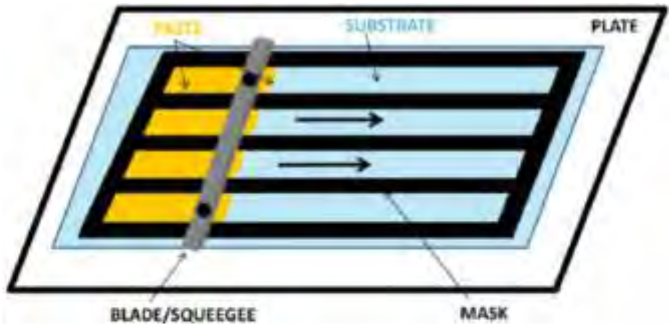


Figure 2.2 Basic principle of doctor-blade printing

2.3.2 Screen printing

According to Ilda Kazani, there's a traditional printing method was found at the end of the 9th century which is screen printing. It is low-cost and easy process. Thus, it is widely used. In screen printing, a design is prepared on screen and the blank areas covered with other substance. After that, the ink is printed through the mesh.

2.3.3 Ink-jet printing

Recently, inkjet printing technology is widely used in various applications due to the properties of low cost, material savings and scalability to large-area manufacturing especially on flexible electronics. Wenfeng Shen state that inkjet technology which can used different materials provides a digital, non-contact and maskless additive patterning process.

2.3.4 Aerosol-jet printing

Aerosol jet printing is developed by Optomec which is another material deposition technology for printed electronics. According to Cruz stated that the liquid formed ink is placed into an atomizer. The aerosolized ink is transported into the using nitrogen gas flow. Then, jet stream print the aerosol onto the substrate. This printing method is suitable for large production due to its non-planar capability and different complex designs can be printed .

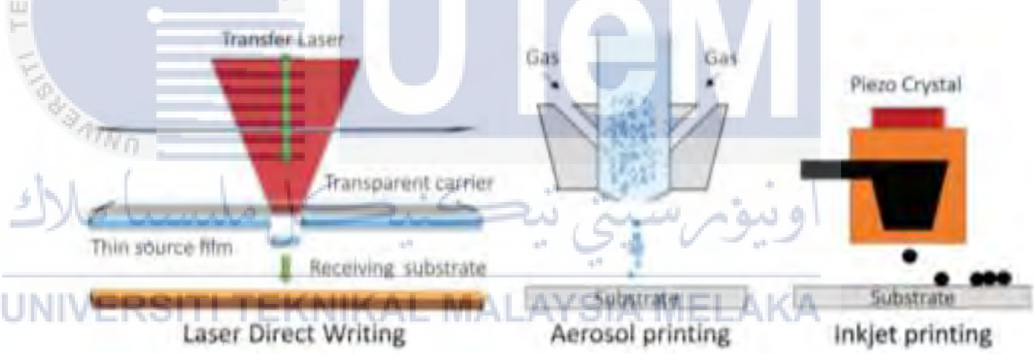


Figure 2.3 Method of Aerosol-jet printing

2.4 HEAT TREATMENT

Heat treatment processing removed the organic component of the coating to leave a thin silvery film. According to Kamyshny, the inks need an additional post-printing treatment after the printing to improve high conductivity of ink.

2.4.1 Curing

According to Tan, curing process is the process that expose printed pattern to heat. The advantage of curing is economic, low cost and time consume. The printed silver patterns were sintered in the argon oven to become close to each other and evaporate the solvent contain in the conductive ink, oven curing process was used to heat and cure the printed conductive patterns. To achieve high conductivity of the conductive ink track, heating to a high temperature (100 ℃ and above) were required to burn-out all organic contaminants (solvent). Inside of the oven had heating coil to provide heat or maintain the temperature in certain level.

2.4.2 Sintering

According to Alexander Kamyshny, sintering is a solid compacting process at temperatures below their melting point. The step of sintering is to expose the printed pattern to heat. This method can avoid destructive heating of common flexible substrates polymeric substrates. For example, PET and polycarbonate are suitable in sintering.

2.5 MATERIAL OF SUBSTRATES

In printable electronic structure, Dang stated that the basic factors to produce it is adhesion between the ink and the material of substrate. He also showed that the ink with flexible substrate should be going through heat treatment at temperatures below 150 °C.

There are two options to develop the adhesion between organic substrate and silver ink; which are surface treatments of substrates such as UV-ozone, plasma or corona to increase the surface energy which leads to the improvement of an ink wetting, and the second option is to control the silver ink composition. For high adhesion implementation, it is important to have a low contact angle as close as possible to 0° and a great wetting (Dang, et al., 2014).

2.5.1 TPU

Recently, flexible and stretchable electronic devices wildely used in the biomedical, military and tactile robotic industries.The flexible and stretchable capabilities can help in implementation of wearable electronic devices. Wearable devices require flexible and stretchable electrodes that provides high conductivity and mechanical stability for varying strains. According to Mohammed Ali, the flexible and stretchable TPU (ST604) from Bemis Associates, Inc, was used as a substrate for the fabrication of the strain sensor in the experiment.

Mohammadali Razeghi state that thermoplastic polyurethane (TPU) is a linear block copolymer from polyaddition reaction which contain soft segments and hard segments. Soft segments are created by reaction polyols reaction with diisocyanates and hard segment contain high polar groups which is combination of dissocyanates with short chain diols.

2.6 ADHESIVE TEST

According to Sughosh Satish Bhore, adhesive test were performed with Scotch tape to determine the adhesion of the ink film to the PET substrate. The effect of binder and carbon content on adhesion was studied. Tests were performed manually by placing the tape on the printed samples and pulling the tape by hand.

2.7 CONDUCTIVE INK CHARACTERIZATION

According to Tsai, C.-Y, copper, silver and gold are metals usually used in conductive inks. The conductive ink that is applied in screen printing consists of organic or inorganic solvent and conductive metal particles. The epoxy that mix with copper is thermodynamically unstable in atmospheric conditions due to oxidation. Since goldbased ink is very high cost, silverbased ink is the best option. The organic solvent used in a conductive ink composition according to an embodiment of the invention is a hydrophilic solvent. The organic solvent may be one of the hydrophilic solvents used alone or may be a two or more solvents used as a mixture.

2.7.1 Electrical properties

According to Ohm's law, the potential difference across two ends of a conductor is proportional to current flowing through a conductor if physical conditions remains unchanged. The formula as shown below:

$V=IR$,where, V= Voltage, I= Electric Current, R = Resistance (Ω)

Sheet resistance can compare the difference of printed conductors where the current travels parallel to the plane of the printed line. The equation below usedto calculate meter resistance (Rs).

$R = R_S (l/w)$ [Ω]

$R_s = R (w/l)$ [Ω/Υ], w = width of the line,

l = length of the line

According to Nash, a Jandel 4 point probe is used to measure the patterns resistance. Before using 4 point probe, the current between the outer pins was set to I = 100 mA, and the voltage V across the inner pins was measured. Then, the probes were placed on each of the lines to measure the values. The resistance (R) per unit length (l) was calculated using $R/l = V/I$. The resistivity ρ was calculated by formulae RA / L where R is resistance and A is cross-sectional area A . The obtained resistivity values were then used to evaluate the conductivity σ of the layer since $\sigma = 1/\rho$.

2.8 IMAGE ANALYSIS

In 2018, Evanko stated that image analysis is a method to get quantitative data using analysis software. Besides, Mendoza also stated that image analysis can used to recognizing, differentiating, and quantifying images. Nowadays, image analysis is widely used due to more conveniently, rapidly and cost effectively.

2.8.1 Scanning Electron Microscopy (SEM)

In 2014, Cian Nash state that the microstructure of the conductive ink was inspected by scanning electron microscopy (SEM) operating at 5 kV. Low magnification top-view SEM images gave information on the quality of ink-jet printed layers surface and high magnification gave information of the shape and the 2D projected area of the silver flakes. Thus, the surface coverage can be evaluated. The SEM investigation of the layer cross-section allowed to observe the flakes distribution and their connectivity.

Nash, C state that SEM investigation of the layer cross-section can estimate the silver amount and observe the flakes vertical distribution and their connectivity. The layers thickness was also estimated from the SEM cross-sections. Low magnification top-view SEM images show the quality of ink-jet printed layers surface and at larger magnification show the shape and the 2D projected area of the silver flakes.

2.8.2 Light Microscope

Kurt Thorn state that the techniques of light microscopy separated into brightfield and fluorescence categories. In thesis, Kurt also showed that how to choose the right experiments for different experiment. The performance of light microscope depend on the details of its components that have to configured and find the properties of light sheet microscope to see whether is suitable for experiment.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter describes the methodology used in this project to obtain 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 the project is shown in Figure 3.1. This project starts by literature review study until the writing of final report in completing the project.The research flows related based on the overall flowchart that can be simplified into data collection, sample fabrication and simple characterization. The experiment procedure start with designing the experiment procedures where the parameters were listed in formulating the sample (conductive ink). After that, the printing and curing of conductive ink on substrate is processed. It is followed by observe the adhesion integrity between epoxy paste and TPU using light microscope.

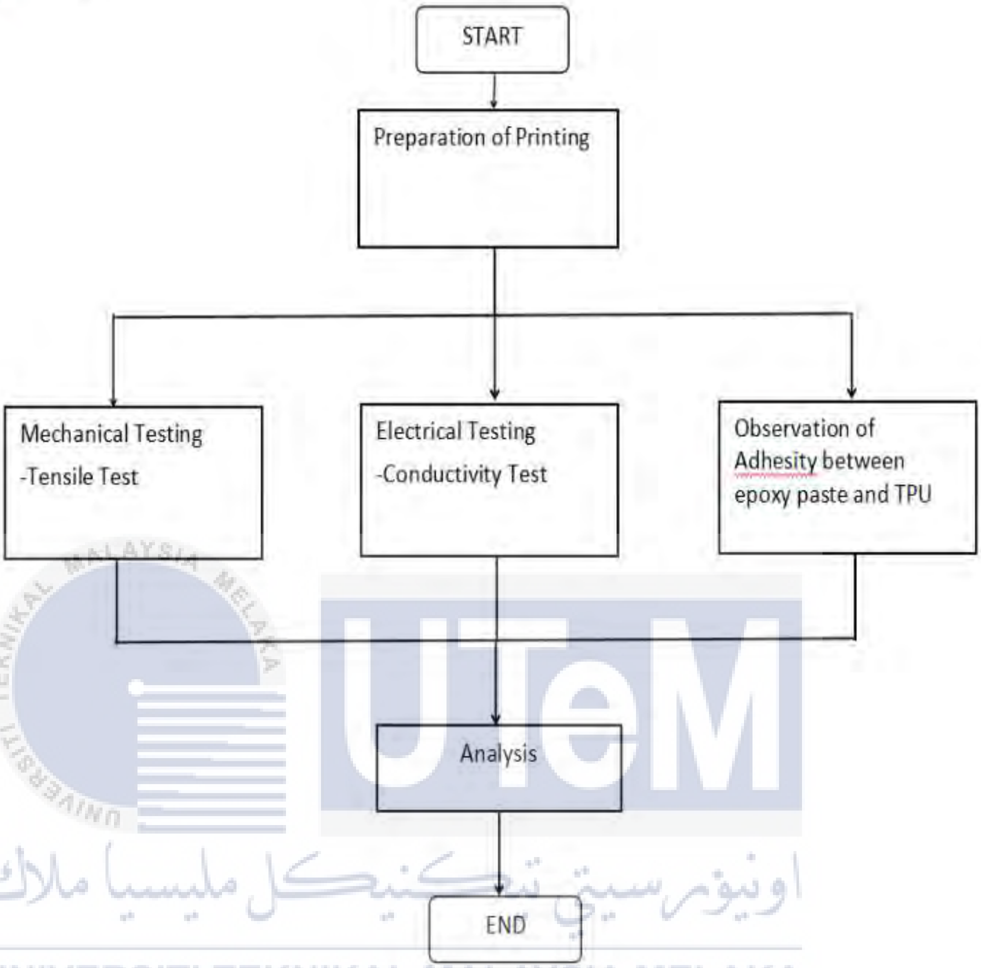


Figure 3.1 Methodology flowchart

3.2 DATA COLLECTION

The process of collecting data in this research is merely based on the study of literature review. The literature review is necessary as it gives the latest information about the method applied, materials used, parameters being investigated and research done by others.

By referring to the literature review, the comparison about the subject in this experiment can be identified and the shortcoming can be make better. This can make the experiment smoothly and more systematically.

3.3 RAW MATERIALS

To produce the conductive ink sample in this experiment, the table 3.1 list out the materials needed.

Materials		
		
Carbon conductive paint: Used as the filler elements.	Epoxy: Used to bind the particles together.	Hardener: Used to harden or dry mixture.

Table 3.1 Materials used

3.4 SAMPLE FABRICATION

Fabrication is the method of processing something from semi-finished or raw materials. In this section, formulation of conductive ink epoxy, printing process on TPU and test will be done.

3.4.1 Formulation of ink

Based on Wenfeng Shen in 2014 state that the powder could not be dispersed well in the 10 g epoxy solution if the amount of silver NP powder used was more than approximately 4 g. Hence, only 5–25 wt% concentration of silver NP inks were prepared and used for printing conductive patterns which wt% includes the masses of the capping agent and silver content. The wt% was calculated by dividing the mass of the silver NP powder by the total weight of each ink.

The epoxy that used in this experiment is Kingkote KK223 interface emulsion. The loading of hardener is 30% off from the amount of binder loading. The total value from the table is the sum of the amount of filler loading and binder loading as the total value was decided before the formulation process started which is 2 g. To begin, the weighing process should be started first and the material and apparatus involved were listed in Table 3.2.

Figure 3.2 KingKote KK223

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

Table 3.2 Material and apparatus involved

The door of the analytical balance was opened and the beaker was put on the centre of the weighing pan using glove(Figure 3.2). After that, the door was shut and a few seconds were considered for the unit to stabilize. The weight of the beaker was cancelled out with the tare button was pressed until the display screen exhibited 0.0000 g to obtain a precise reading of the substance.

Figure 3.3 Beaker positioned on the centre of pan

After the reading stabilize and reach 0.0000g, the door was opened and the materials being weighed were carefully added or reduced until the display exhibited the required weight in accordance to the value of weight in the table as shown in the figure below.

Figure 3.4 Value of weight being adjusted

But the weight should be slightly higher than the desired value; within tolerance of +0.05 g as the loss of weight of the materials was taken into consideration mixing them together. The described process was repeated three times as there are three materials involved for each sample.

3.4.2 Mixing and stirring processes

In this operation, mixing and stirring processes will be done with some crucial steps. The materials and apparatus that will be used were listed in the table below.

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

Table 3.3 Material and apparatus involved

Figure 3.5 Mixing machine

The epoxy and conductive carbon paint will mixed together into one beaker after weighed as shown in Figure 3.3. Since all of the materials have different viscosity, carbon conductive paint and hardener that are low viscosity will be poured into a beaker which consist highest viscosity epoxy. The objective of this mixing method is to prevent the loss in weight of high viscosity materials because they tend to stick to the beaker while being poured due to their properties.

In order to obtain a well-dissolved mixture, the mixture is put inside a container and stirring process was proposed in mixing machine shown in figure 3.4. Once the stirring procedure was completed, the printing process is prepared to be done. All of the apparatus used were fully cleaned after the overall processes were completed.

Figure 3.6 Mixed conductive epoxy

3.4.3 Printing process

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
Glass slide	Act as the substrate

Table 3.4 Material and apparatus involved

Before the printing process started, the gap of ink tracks in two positions, A and B was constructed on the glass slide by using the Scotch tape to create the space of 0.5

cm width on the TPU as in Figure 3.7.

Figure 3.7 Applying the Scotch tape

Thus, the TPU is used as the substrate is for the purpose of ink characterization; to investigate the adhesion behaviour between the epoxy and TPU and test the mechanical and electrical properties of two different types of epoxy paste, thus indirectly the loading with best conductivity will be identified.

After the construction of tracks on the TPU was completed (Figure 3.8), only then the printing process can be carried on. The method used to print the mixture of ink on the substrate in this experiment was doctor-blading technique. In this technique, the thickness and coverage of ink can be controlled manually.

Figure 3.8 Constructed gap

3.4.4 Curing process

Curing is a term refers to the toughening of a polymer material Curing process. It is been processed to increase the bonding between the particles of filler, binder and hardener. The process can improve the adhesion between ink and the substrate.

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

Table 3.5 Material and apparatus involved

Many epoxy compositions can be cured to resins having a high glass transition temperature. The samples were heat-treated at curing temperature within the range of 50–180 C in a drying oven for 30 min. Lastly, the printed sample was cured and let to be fully dried at room temperature, thus their structural morphology and electric properties were investigated.

Figure 3.9 Inside the oven

3.4.5 Electrical testing

In experiment, four point probe to measure the resistivity of conductive epoxy which either bulk or thin film specimen such as the sheet resistance value of the sample in ohms-per-square, the resistivity volume in ohms-cm and the thickness of sample. It works by forcing a constant current along two outer probes and next and the voltage is read out from the two inner probes.

Figure 3.10 Four point probe with display meter

Before the resistivity measurement of the sample was taken, calibration for a reference sample should be the first to carry out. Thus, an indium-tin-oxide (ITO) coated glass was used as the reference sample to ensure whether the four point probe system is working well or not.

The reference glass was put on the base plate (Figure 3.16) and the pin of probe was lowered until 'tik' sound was heard which means it reached its limit. The height level was adjusted until it was in solid contact or touched the reference glass and waited for the measurement to be stable until the satisfied values of sheet resistance was achieved.

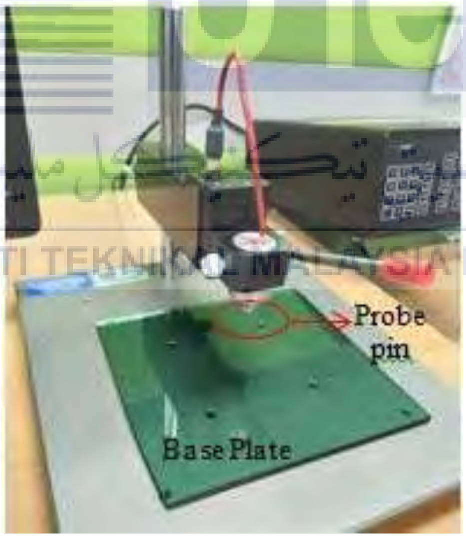


Figure 3.11 Base plate and probe pin

While doing the reference calibration, several measurements must be taken from random points across the central area of reference glass instead of from a fixed point. For the reference TPU, the value should be stable readings.

For the TPU in this experiment, the same method as for the reference sample was used to obtain the values of sheet resistance. The sample was put on the base plate and the contact between the ink track and the probe pin was adjusted. It must be in stable state and the suitable current has been achieved before the result of sheet resistance values was recorded, as it may take some times to reach the suited current.

At each constructed points of the ink track, three readings of sheet resistance will be taken and the average values of them would be the result. Since each sample has two ink layers deposited and three points constructed at each layer, the total readings taken will be 18 of sheet resistance values for one sample.

However, the display meter will show comments such as “contact limit” or “out of range” may be due to poor contact; too close or no contact at all. Thus, an appropriate action should be taken in order for the display meter can read out the values. Poor contact can be detected through high standard deviations of several measurements taken from the similar areas which will be discussed for the next chapter.

3.5 MECHANICAL AND ELECTROMECHANICAL PERFORMANCE

Mechanical performance of printed samples was evaluated after good printability was achieved on both substrate materials with the chosen ink. First, adhesion between ink and substrate material was tested with a tensile test.

3.5.1 Tensile test

Stretchable circuits on TPU is naturally their electrical performance and adhesion of the conductive ink-substrate interface. Since the TPU substrates chosen for the experiments in this thesis are relatively soft and stretchable. Therefore adhesion is chosen to be analyzed roughly by a simple elongation test to test the electromechanical performance. It also can analyze elasticity and plasticity of printed matter.

In order to test peak strain and life time of printed circuits on a stretchable substrate, a custom test system was designed and built. The system comprises a ruler, static clamp and a moving clamp. The moving clamp is moved by a screw. Thus, elongation length can be determined using screw and ruler. The main parts of this test bench are illustrated in Figure 3.13.

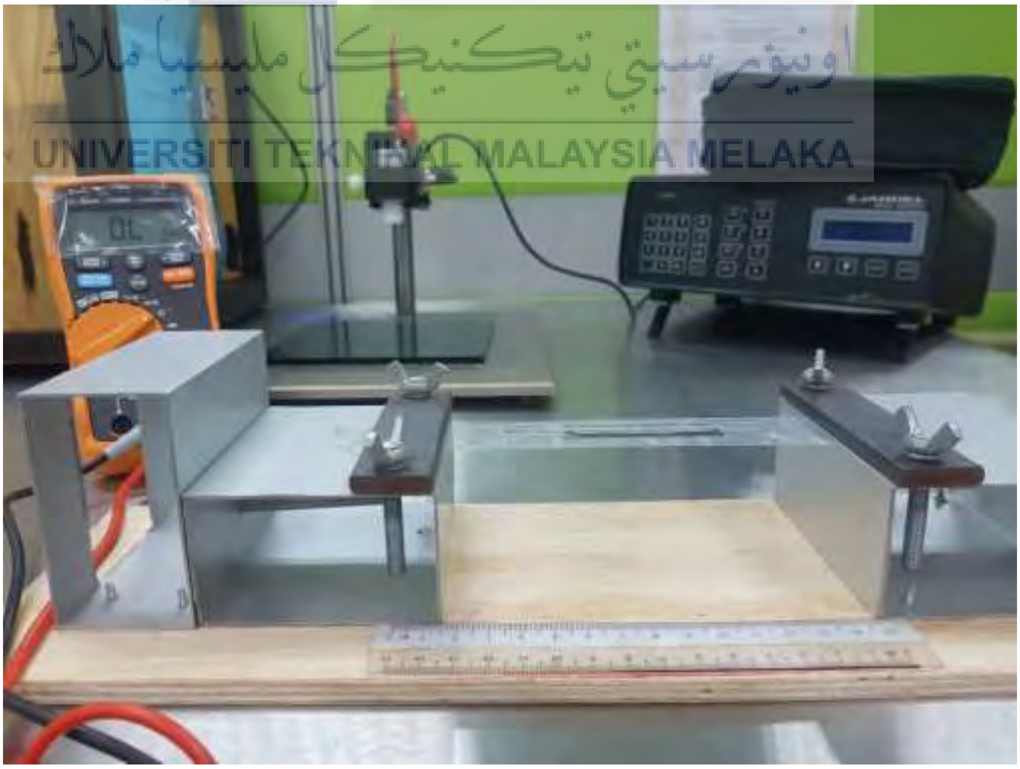


Figure 3.12 Tensile test machine

For the measurement principle, when taking the measurement of resistiviy, the multimeter sensor is put on the surface. Trace length is elongated 1 cm each time and the resistivity is measured using multimeter. First, the sample is clamped on the tensile test machine. The result of resistivity is tested using multimeter shown in Figure 3.14. Next, the sample is elongated 1 cm and the resistivity value is recorded. The experiment is repeated for 1 cm,2 cm, 3 cm and 4 cm.

Figure 3.13   Multimeter

3.6 MICROSCOPY ANALYSIS

The scotch tape on the TPU will be removed to observe the track of ink after the printed sample was fully dried.

3.6.1 Light Microscopy

Light microscopy is used to measure the particle shape and size with capture picture of the object before and after tensile test. The microscope which connected with computer can showed the morphological state on the screen.

Figure 3.14 Light Microscope with computer

The required power objective lens used in this experiment were 10x, 20x and 50x. The focus knob is used to adjust to have a clear and focused image shown. After that, the image shown was captured in the computer. Thus, it repeated for another power objective lens as at each point.

3.7 SUMMARY

The literature study to compare the adhesion integrity of conductive epoxy and TPU substrate. The experiment will be conducted based on 4Ms which are men, method, material and machine that has been stated in this chapter to fabricate the ink onto the substrate. From the study of literature review, the knowledge about the type of materials, formulation of ink, methods of production to fabricate ink and the parameters investigated have been identified. Carbon conductive paint will be used as the filler element to produce ink throughout this project and doctor- blading method will be applied to deposit the layer of ink on the substrate, also the TPU will be used as the substrate.

After the overall steps of ink fabrication have been made for all percentages of filler loading, the next step to be taken is the testing of the ink in terms of electrical properties and microscopy. Ink characteristic has been analysed by using light microscope, four point probe and tensile test machine. Light microscope was used to check the microstructure of the conductive epoxy; the four point probe was used to check the resistivity of epoxy while the tensile test machine is to test whether the conductive epoxy still conduct electricity when it elongate and test the electromechanical of conductive epoxy.

CHAPTER 4

RESULTS AND ANALYSIS

4.1 INTRODUCTION

In this chapter, the resistivity, the microstructure of ink and the tensile of the sample will be discussed to find out the best ink formulation based on the data gained through four point probe, tensile machine, multimeter and the images from the microscope.

4.2 ANALYSIS OF ELECTRICAL PROPERTIES

4.2.1 Result of resistivity

Resistance was measured using a four point probe and set at a range of 10nA and 100nA. Sufficient pressure was applied to avoid contact resistance between the probes and the conductive prints. The initial resistance measurements from each of the tests were used to determine which filler percentage proved best moving forward in the experiment.

Table 4.1 Reststivity value of 30 wt% of carbon conductive ink

SAMPLE 1

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	-	-	-	-	-
Resistivity (R/sq)	-	-	-	-	-

SAMPLE 2

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	-	-	-	-	-
Resistivity (R/sq)	-	-	-	-	-

SAMPLE 3

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	-	-	-	-	-
Resistivity (R/sq)	-	-	-	-	-

Table 4.2 Reststivity value of 60 wt% of carbon conductive ink

SAMPLE 1

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	450.187	207.291	290.880	52.272	250.1575
Resistivity (R/sq)	204.043	93.952	131.839	23.692	113.3815

SAMPLE 2

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	317.859	444.334	149.612	98.569	25205935
Resistivity (R/sq)	144066413.16	201389942.16	67810142.88	44675413.56	114485477.9

SAMPLE 3

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	379.075	182.841	96.925	62.225	180.2665
Resistivity (R/sq)	171.812	82.871	43.930	28.203	81.704

Table 4.3 Reststivity value of 90 wt% of carbon conductive ink

SAMPLE 1

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	33.998	17.328	36.006	19.967	26.82475
Resistivity (R/sq)	15409.25	7853.74	16319.36	9049.84	12158.0475

SAMPLE 2

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	22.901	24.983	58.366	58.281	41.1328
Resistivity (R/sq)	10379.65	11323.29	26453.81	26415.28	18643.0075

SAMPLE 3

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	10	10	10	10	10
Voltage(mV)	22.458	43.247	41.170	51.227	39.5255
Resistivity (R/sq)	10178.86	19601.27	18659.89	23218.13	17914.515

Table 4.1 Reststivity value of 100 wt% of carbon conductive ink

SAMPLE 1

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	100	100	100	100	100
Voltage(mV)	4.545	3.884	4.254	5.427	4.5275
Resistivity (R/sq)	206.00	176.04	192.81	245.97	205.21

SAMPLE 2

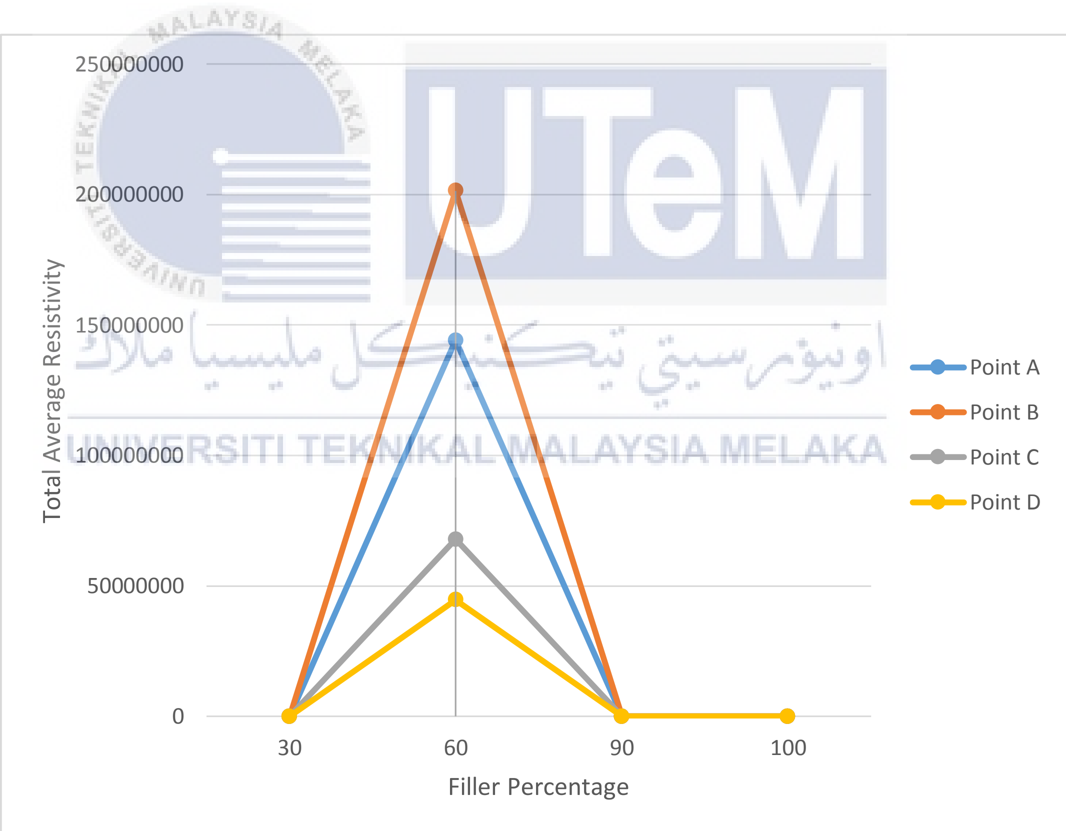
	Point A	Point B	Point C	Point D	Average
Ampere (nA)	100	100	100	100	100
Voltage(mV)	10.816	8.929	3.518	5.872	7.2838
Resistivity (R/sq)	490.234	404.711	159.452	266.126	330.13

SAMPLE 3

	Point A	Point B	Point C	Point D	Average
Ampere (nA)	100	100	100	100	100
Voltage(mV)	7.179	3.106	2.928	5.586	4.6998
Resistivity (R/sq)	325.38	140.794	132.728	253.163	213.02

Wt %	sample	Resistivity (R/sq)	Total average resistivity (R/sq)
30	1	undefined	undefined
	2	undefined	
	3	undefined	
60	1	113.38	38161891.00
	2	114485477.90	
	3	81.70	
90	1	12158.01	16238.52
	2	18643.00	
	3	17914.52	
100	1	205.21	207.95
	2	330.13	
	3	213.02	

Table 4.5: Total average data



Graph 4.2: Total Average Resistivity versus Filler Percentage

At 30% of filler, the resistivity is undefined. There's no resistivity value of conductivity ink detected using four point probe. It proved that it is lack of conductive materials in the ink.

The lower the percentage of conductor filler, the lower the conductivity with increasing the resistivity as can be seen in the graph. For 60% of filler, the average resistivity is the highest among the four pencentage of the filler which is 3816189 Ω/sq. It has the lowest conductivity as the resistivity value is proportional to the conductivity value.

For filler at 90%, their average value of resistivity is decreased compared to 60% of filler which is 16238.52 Ω/sq. It show that an increase of filler content leads to a decrease of resistivity due to the high amount of conducting materials composition in the ink.

In 100% filler percentage, the data showed the lowest resistivity. It being the zero percentage of epoxy have the highest conductivity among the overall filler percentages.

Last, Graph 4.2 showed that the formulation of ink used at positions A, B, C and D have slightly different in the resistivity value. 30 wt% couldn't define the resistivity, 60 wt% had the highest resistivity, followed by 90 wt% and 100 wt% had the lowest resistivity. The graph indicated that the total resistivity against the percentage of filler loading. It showed that the higher percentage of filler loading, the lower the resistivity due to the increased conducting materials.

4.2.2 Potential error sources

In this experiment, two potential error sources which will affect result included printing technique and four point probe measurement.

4.2.2.1 Printing technique

Flexible circuits have been one of the widest range of growing market segments for interconnection products in the last several year. The doctor-blading method was specifically selected because it is an inexpensive, flexible and fast way. But, in this case, the printing error may be occur due to inconsistent range of printing on TPU when blade moved across the Scotch tape.

When printing speed is high, the conductive ink may not cover all over the region. Thus, some of the region will cover more carbon conductive paint. This will affect the different thickness paint on TPU and lead to different spreads of conducting materials. The thicker the conducting carbon conductive epoxy paint on TPU, the higher content of carbon leads to higher conductivity.

4.2.2.2 Four point probe measurement

The square resistance of the printed samples was measured with the four-point probe machine. When using the four point probe measurement, error may be caused when there's no fixed time period when taking data. The current need some time to enough saturation for RC (resistance-capacitance) delay in the highest resistive samples. If the time spent is inconsistent, the resistivity value will be unstable.

4.3 ANALYSIS ON TENSILE TEST

The results of the tensile test measurement is measured resistivity value during stretching with the simple elongation machine. Resistivity recorded when TPU sample elongate 1 cm. Three point of value is analyzed in each sample.

4.3.1 Result of resistivity of tensile test

Table 4.6 resistivity value under tensile test: 30 wt%of carbon conductive ink

Sample 1.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	-	-	-	-
1 cm	-	-	-	-
2 cm	-	-	-	-
3 cm	-	-	-	-
4 cm	-	-	-	-

Sample 2.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	-	-	-	-
1 cm	-	-	-	-
2 cm	-	-	-	-
3 cm	-	-	-	-
4 cm	-	-	-	-

Sample 3.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	-	-	-	-
1 cm	-	-	-	-
2 cm	-	-	-	-
3 cm	-	-	-	-
4 cm	-	-	-	-

Table 4.7 resistivity value under tensile test: 60 wt% of carbon conductive ink

Sample 1.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	59.390	61.450	55.340	58.727
1 cm	55.080	48.190	50.670	51.310
2 cm	42.510	52.070	48.610	47.730
3 cm	33.990	41.250	36.000	37.080
4 cm	33.590	42.480	34.620	36.897

Sample 2.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	65.670	61.250	63.980	63.633
1 cm	60.510	56.850	50.360	55.907
2 cm	57.630	50.750	53.310	53.897
3 cm	50.020	50.630	50.500	50.383
4 cm	48.510	41.060	47.810	45.793

Sample 3.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	61.250	65.480	63.010	63.240
1 cm	57.280	52.650	54.630	54.853
2 cm	49.020	44.210	45.960	46.397
3 cm	44.670	33.580	38.010	38.753
4 cm	37.630	39.250	37.620	38.167

Table 4.8 resistivity value under tensile test: 90 wt% of carbon conductive ink

Sample 1.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	3.048	2.487	2.496	2.677
1 cm	0.707	0.678	0.537	0.641
2 cm	0.121	0.131	0.145	0.132
3 cm	0.091	0.083	0.100	0.091
4 cm	0.069	0.073	0.066	0.070

Sample 2.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	2.389	3.425	2.651	2.820
1 cm	0.651	0.697	0.689	0.679
2 cm	0.087	0.092	0.096	0.092
3 cm	0.073	0.076	0.081	0.080
4 cm	0.067	0.070	0.061	0.066

Sample 3.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	3.528	3.176	4.261	3.655
1 cm	0.658	0.629	0.612	0.633
2 cm	0.149	0.121	0.153	0.141
3 cm	0.082	0.089	0.105	0.092
4 cm	0.070	0.056	0.078	0.068

Table 4.9 resistivity value under tensile test: 100wt% of carbon conductive ink

Sample 1.0

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	1.891	1.348	1.309	1.516
1 cm	0.678	0.781	0.567	0.675
2 cm	0.502	0.488	0.509	0.500
3 cm	0	0	0	0
4 cm	0	0	0	0

Sample 2.0

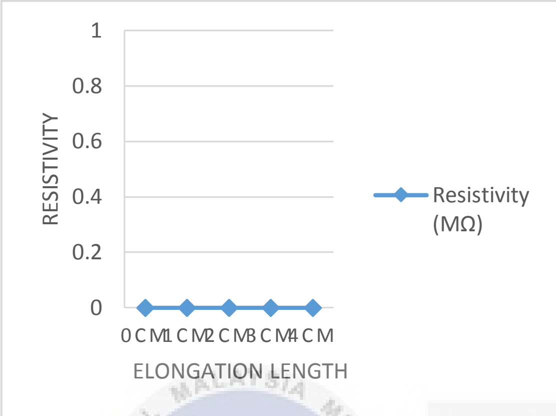
	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	2.040	2.036	1.878	1.985
1 cm	1.813	1.887	1.726	1.809
2 cm	1.726	1.708	1.659	1.698
3 cm	0	0	0	0
4 cm	0	0	0	0

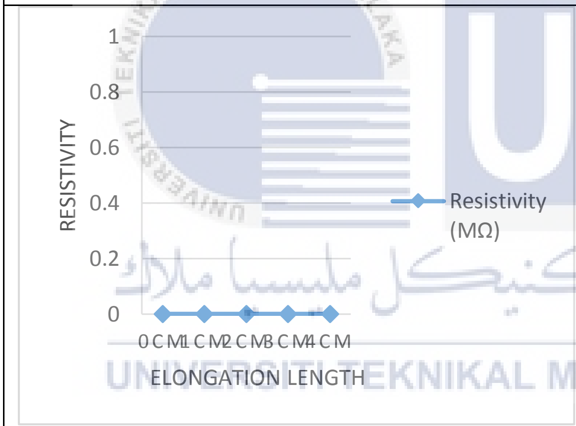
Sample 3.0

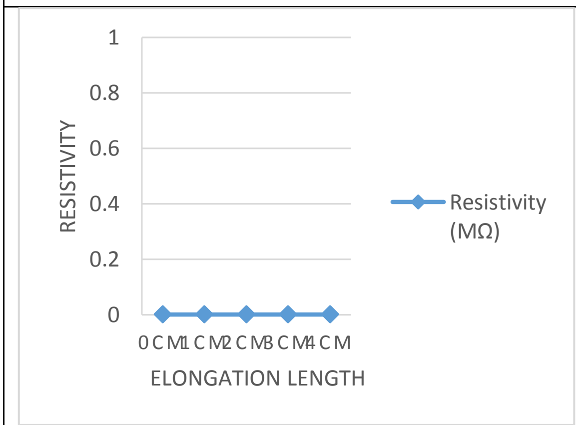
	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
0 cm	2.308	2.078	1.967	2.118
1 cm	1.368	1.282	1.317	1.322
2 cm	0.897	0.865	0.976	0.913
3 cm	0	0	0	0
4 cm	0	0	0	0

Each of the data is translated into the graph that represents the relationship between elongation length during tensile test and the resistivity value.

Table 4.10 Graph and analysis: 30 wt% of carbon conductive ink

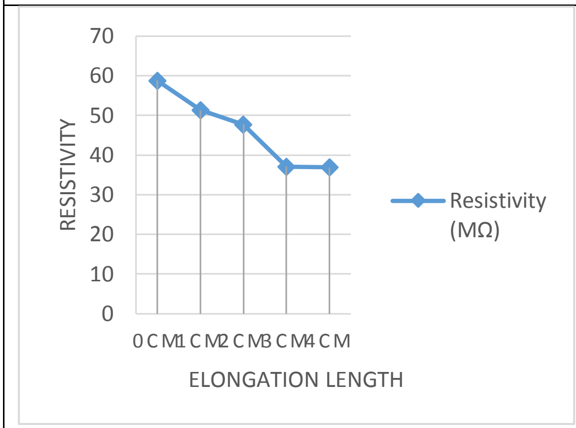
<table>
<tr><th>Result</th><th>Analysis</th></tr>
<tr><td>
</td><td>Sample 1

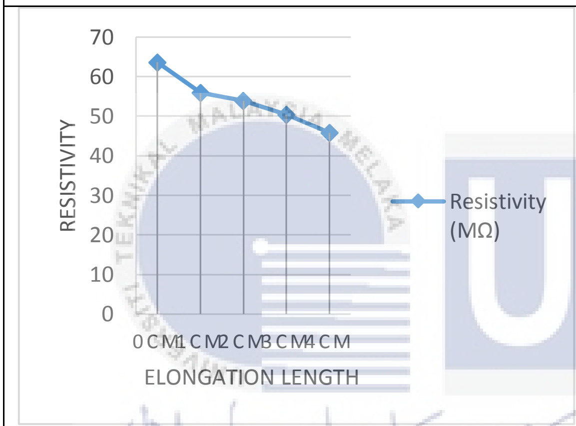
There was no resistivity value was detected.</td></tr>
<tr><td>
</td><td>Sample 2

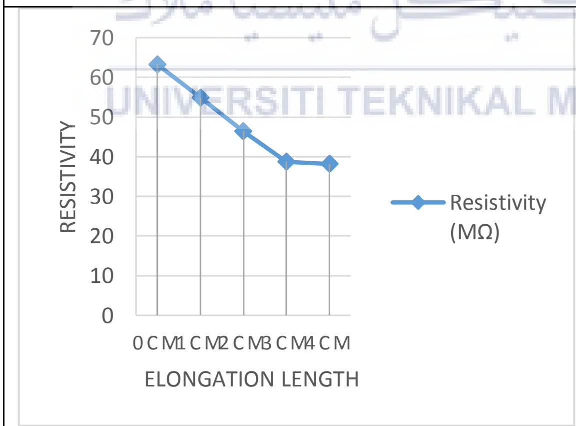
There was no resistivity value was detected.</td></tr>
<tr><td>
</td><td>Sample 3

There was no resistivity value was detected.</td></tr>
</table>

Table 4.11 Graph and analysis: 60 wt% of carbon conductive ink

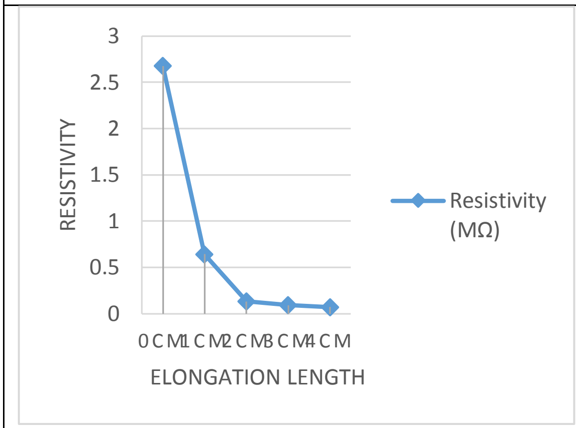
<table>
<tr><th>Result</th><th>Analysis</th></tr>
<tr><td>
</td><td>Sample 1

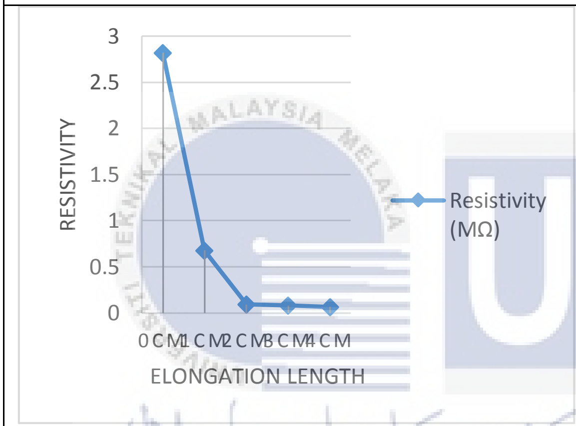
The average value gradually decreases; the resistivity decreases but at tension 3cm and 4cm are approximately the same.</td></tr>
<tr><td>
</td><td>Sample 2

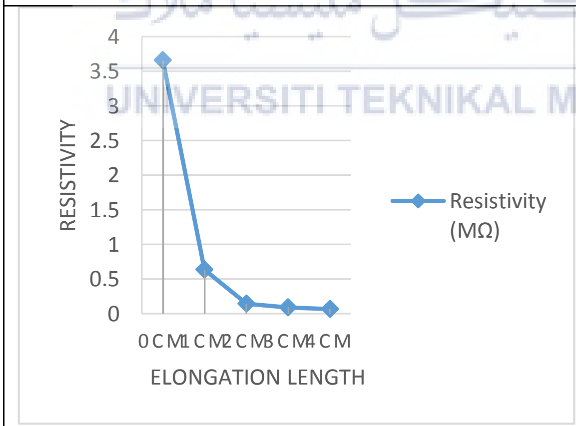
The total average values are gradually decreasing across all of five points.</td></tr>
<tr><td>
</td><td>Sample 3

The average resistivity value gradually decreases but at tension length 3cm and 4cm are approximately the same.</td></tr>
</table>

Table 4.12 Graph and analysis: 90 wt% of carbon conductive ink

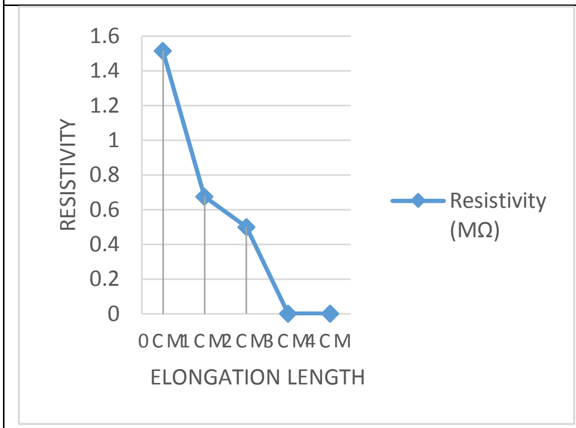
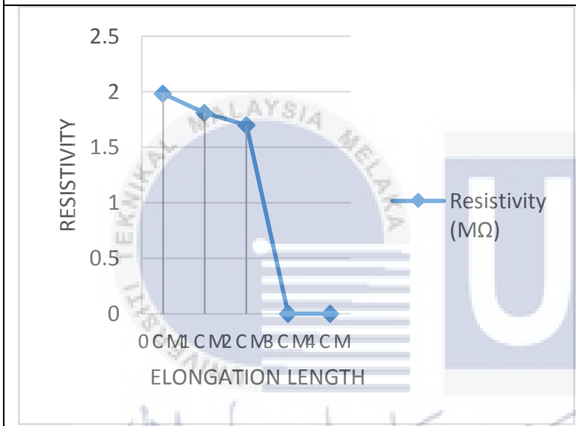
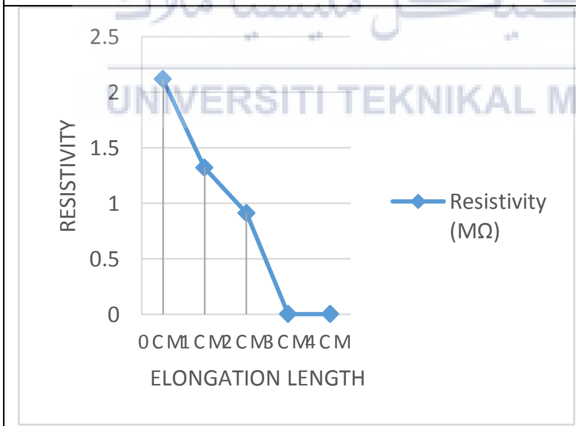
<table>
<tr><th>Result</th><th>Analysis</th></tr>
<tr><td>
</td><td>Sample 1

The total average values are gradually decreasing across all of five points. The longer the TPU elongate, the lower the resistivity.</td></tr>
<tr><td>
</td><td>Sample 2

The graph illustrates decreasing of resistivity compared to the previous point.</td></tr>
<tr><td>
</td><td>Sample 3

The values rapidly decrease from the elongation length of 1 cm. From the graph, the values at 3 cm and 4 cm are approximately consistent while at 2cm, the value is a bit higher.</td></tr>
</table>

Table 4.13 Graph and analysis: 100wt% of carbon conductive ink

Result	Analysis
	Sample 1 The average value at points 3cm and 4cm are zero, the value of resistivity decrease when the elongation length increase in tension test..
	Sample 2 The average value of resistivity is the highest at beginning but the average value starts decreasing when it apply stress to elongate. The values cannot detected after 3cm.
	Sample 3 The total average values are gradually decreasing across all of three points which show the decrease of resistivity. The values cannot detected after 3cm.

4.3.2 Result of resistivity after tensile test

After tensile test, the conductive TPU substrate is return to original state. The resistivity value is measured using multimeter after tensile test

Table 4.14 resistivity value after tensile test: 30 wt% of carbon conductive ink

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
Sample 1.0	-	-	-	-
Sample 2.0	-	-	-	-
Sample 3.0	-	-	-	-
Average				-

Table 4.15 resistivity value after tensile test: 60 wt% of carbon conductive ink

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
Sample 1.0	1.370	1.027	1.370	1.26
Sample 2.0	4.067	9.580	8.675	7.44
Sample 3.0	9.031	6.950	7.670	7.88
Average				5.53

Table 4.16 resistivity value after tensile test: 90 wt% of carbon conductive ink

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
Sample 1.0	2.072	2.407	2.697	2.392
Sample 2.0	2.561	2.270	2.284	2.370
Sample 3.0	6.130	6.120	4.751	5.667
Average				10.429

Table 4.17 resistivity value after tensile test: 100 wt% of carbon conductive ink

	Resistivity (MΩ)	Resistivity (MΩ)	Resistivity (MΩ)	Average
Sample 1.0	-	-	-	-
Sample 2.0	-	-	-	-
Sample 3.0	-	-	-	-
Average				-

4.3.3 Comparison of average resistivity value before and after tensile test

Comparison of the average resistivity result before and after tensile test can evaluate the eletricity condition of carbon conductive TPU substrate after tensile test. The value is measured using multimeter.

Table 4.18 Average resistivity value before tensile test

Filler Loading/ wt%	Average Resistivity (MΩ) SAMPLE 1	Average Resistivity (MΩ) SAMPLE 2	Average Resistivity (MΩ) SAMPLE 3	Average
30	-	-	-	-
60	58.733	63.633	62.240	61.53
90	2.677	2.820	3.655	3.05
100	1.516	1.985	2.118	1.87

Table 4.19 Average resistivity value before tensile test

Filler Loading/ wt%	Resistivity before tensile test(MΩ)	Resistivity after tensile test (MΩ)	Difference
30	-	-	-
60	61.53	5.53	-56.00
90	3.05	10.43	7.38
100	1.87	-	-

4.3.3 Critical discussion in tensile test and resistivity

The soft and stretchable TPU is chosen in this thesis due to the electrical performance and adhesion integrity of the conductive ink-substrate interface. It is tested for adhesion by a simple elongation test to test the electromechanical performance. In this experiment, It also can test elasticity and plasticity of printed TPU.

From the table of results in both directions, the values of resistivity for the sample of 30 wt% of filler were undefined due to lack of conductive carbon particles. 60% of filler were the highest resistivity among all the filler percentage. It showed that the average value of resistivity were gradually decreases when the strain values increases. It also the lowest conductivity among all the filler percentage. When the 60 wt% elongated by machine more than 2cm, the change of resistivity were small. This is because the deformation of carbon adhesive started to get stabilized.

Different filler percentage makes significantly different strain-resistance behavior in increasing of strain. In 90 wt% of filler shown the resistivity gradually decreasing when the TPU elongate. The value of resistivity are approximately to zero when the TPU elongate more than 2cm. In case 100% filler, the conductive carbon paint crack when the force applied to elongate TPU. The electrical pathways were destroyed seriously than the lower content fillers composites. Thus it showed no result on the multimeter when it elongate to 3cm.

In comparison between before and after tensile test, the average resistance of 30 wt% conductive carbon loading were not detected. For 60 wt%, the resistivity value decrease 56MΩ which lead to increase of conductivity. For 90 wt%, the resistance increased 7.38MΩ. The resistance of 100MΩ after tensile test cannot detected because the crack of conductive ink on TPU.

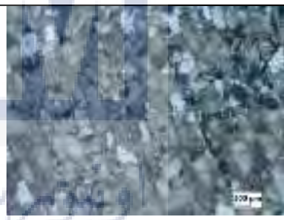
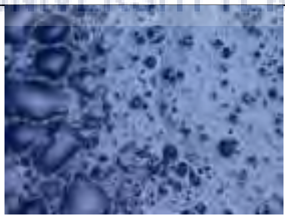
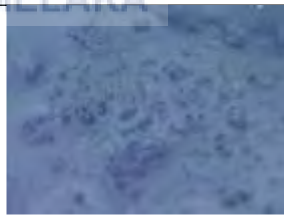
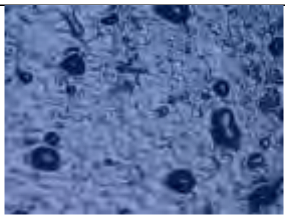
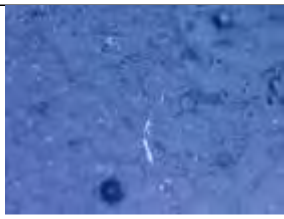
From all of the graphs that had been constructed, 60 wt% is better choice because it have more stable resistivity compared to 90 wt% filler. The samples with lower filler percentage had lower resistivity. It is not easily to break into pieces due to epoxy. When the strain is applied to TPU, the density of dislocations and grain boundary decreased. In this case, the resistivity decreased. Meanwhile, the samples without epoxy have the highest conductivity but is easily break down when stretching.

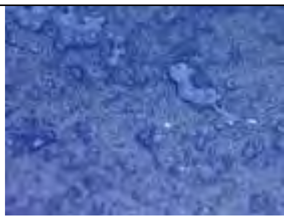
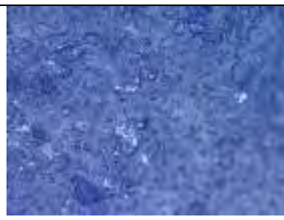
4.4 ANALYSIS OF MORPHOLOGICAL

Light microscope is to observe the microstructure changes such as particle reorientation and failures in the ink film under loading. To observe the sample conductive ink tracks, it is divided into two categories: microstructure that have conductivity and microstructure with conductivity after tensile test. Three scales of microstructure magnification, 10x, 20x and 50x are captured and organized in accordance to their filler loading with three scales of magnification.

Table 4.20 Microstructure with conductivity before tensile test

Figure 4.1 Microstructure before tensile test

Filler (%)	Magnifications		
	10x	20x	50x
30			
60			
90			

100			

Based on Table 4.4, the microstructure transformation of carbon conductive paint epoxy were in the range 30 wt%, 60 wt%, 90 wt% and 100 wt%. For 30% filler loading, the microstructure showed that there was more epoxy paste compared to others. There's lack of carbon conductive filler loading and more amount of epoxy. Thus, there's no conductivity and very high resistivity.

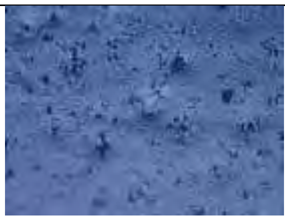
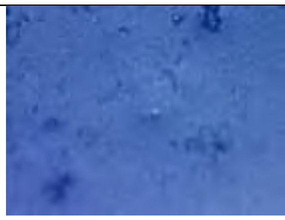
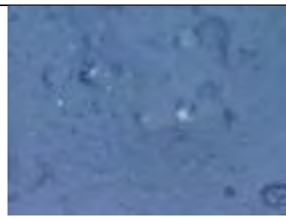
The microstructure transformation conductive paint epoxy of 60 wt%, 90 wt% and 100 wt% showed, the existence of sparkling particles represented the carbon and the dark colour in the microstructure region was the melted epoxy. As the filler percentage increase, the frequency of gaps between carbon particles is smaller. This will affect the electrical conductivity of conductive ink epoxy. The increasing frequency of gaps between carbon particles proved that the increasing gap number of particles and lead to lower resistivity of epoxy. The presence of carbon conductive particles ensure there has current flow. In filler loading of 60% showed the presence of carbon particles but in a very small quantity. Next, in 90 wt% showed more sparkling carbon particles than 60 wt% due to the same ratio of filler to binder.The addition of conductive fillers percentage usually makes polymer composites brittle, and low repeatability. In 100 wt% showed the most carbon particles and there's no epoxy in it.

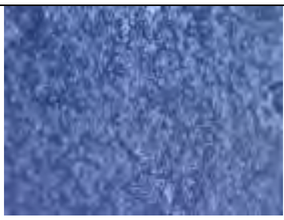
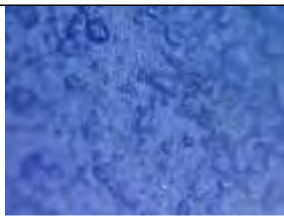
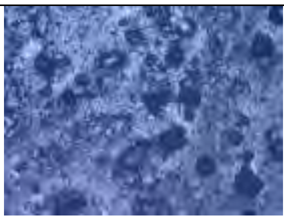
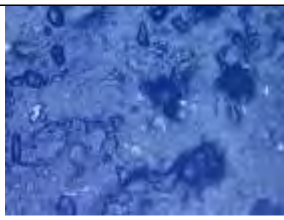
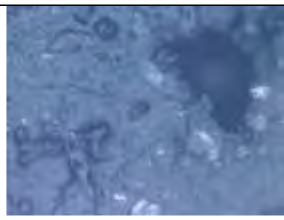
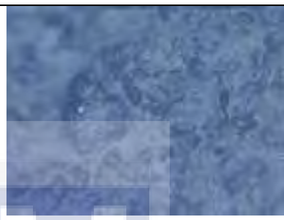
At 60 wt% and 90 wt%, there were presence of granular-like particle which contain electrical conductivity which leads to the existence of particle necking. Kim, D., & Moon, J. also believed that the necking growth provides a continuous connection and once the interparticle neck has been produced, the granular-like particle will be conductive although it is still porous.

Figure 4.5 crack of 100 wt% conductive ink on TPU after tensile test

Table 4.21 Microstructure with conductivity after tensile test

Figure 4.2 Microstructure after tensile test

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

60			
90			
100			

From Table 4.6, the data were obtained shows that 90 wt% contains more carbon compare to 60 wt%, which causes it has better conductivity compared to the 60 wt%. For 60% and 90%, the microstructure showed no outstanding difference between each other either in shape of particle or size compared to the filler loading at 30 wt% and 100 wt%. In 60 wt% and 90 wt% indicated the conductive epoxy have a close-packed structure and formed a strong bonding between each other. When the carbon conductive epoxy were print on TPU, the adhesion of epoxy is strong to return to its original shape when the force is removed same as TPU. 90 wt% samples showed more holes and microcrack compared to 60 wt%.

At the 100 wt% microstructure transformation after tensile test, there had traces of crack and wrinkles developed in the ink layer. The conductive ink break into pieces when it elongate to 3cm. It undergoes plastic deformation due to lack of epoxy added in carbon conductive ink in 100 wt%. The conductive ink cannot stand for tensile test. Matrix cracking mentioned by Merilampi, S in articles 2010. It can be explained by microstructural changes in the ink film. A poor adhesion of the matrix to TPU cause the complete breakage.

After the tensile test, the deformation of TPU and carbon conductive epoxy get stabilized. Only some of the granular particles formed and microcracks occured when increasing of strain. When specimens with different elongation length were observed after the tensile test, it is clearly seen that the carbon particles is slightly further compared to conductive epoxy before tensile test. It has been observed that conductive epoxy return to almost same as before except 100 wt% due to high intrinsic porosity. Carbon conductive paint without any epoxy and hardener cannot prevent from breaking during tensile test. The tensile strength was related to adhesion integrity on TPU.

4.5 RELATIONSHIP BETWEEN ELECTRICAL PROPERTIES, MORPHOLOGICAL ANALYSIS AND TENSILE STRENGTH

In this section discussed three parameters which include total average values of resistivity, total average values of resistivity during tensile test and microstructure. Thus, a study of the relationship between sample composition and those three parameters is constructed.

For 30 wt% of filler, there was no resistivity value was detected by four point probe due to low filler composition. Conductive carbon in epoxy which widely known as conductor. If the amount of carbon is too low, there was no resistivity tracked by 4 point probe and multimeter. Besides,the resistivity value is decreased when the conductive carbon paint percentage increased in epoxy. From 60% and 90% of filler, the resistivity was detected with 60% having the highest total average value of resistivity: 3816189 Ω/sq which is the lowest value of conductivity. The resistivity varies inversely with the conductivity value. Then, 90 wt% have the lower resistivity was 16238.52 Ω/sq.

Furthermore, the adhesion integrity is tested by a simple elongation test for electromechanical performance. In tensile test, the resistivity value of 60% conductive carbon percentage were gradually decreases and the change of resistivity were small start from 2cm due to the deformation of carbon adhesive started to get stabilized. In 90 wt% of filler shown the resistivity gradually decreasing when the TPU elongate. Compared to 90 wt% filler, 60 wt% have more stable resistivity. 90wt% carbon filler have result approximately to zero. 100% conductive carbon paint is the solution without adding any binding epoxy and hardener. Thus, the sample of 100 wt% filler crack into pieces when the tensile stress applied to it. There were not enough adhesive integrity between conductive ink and TPU. In the comparison of resistivity value before and after tensile test, the resistance of 60wt% carbon loading decreased, the resistance of 90 wt% carbon loading increased. However, 90 wt% and 30 wt% cannot detected any resistance after tensile test.

High composition of filler have higher resistivity. Based on morphological analysis, the conductive carbon shown sparkling in it. When the composition increased, there's more sparkling granular-like particles shown in the figure. The existence of sparkling granular-like particles represented the silver and the dark colour in the microstructure region was the melted epoxy. The morphological result of 30 wt%, 60 wt% and 90 wt% after tensile test shown that the carbon particles is slightly further when it return to original condition compared to figure before tensile test. Granular particles formed and microcracks occured when increasing of strain. 90 wt% samples showed more holes and microcrack compared to 60 wt%. However, the 100 wt% showed that the cracking when the strain increased to 3cm due to lack of epoxy.

4.6 SUMMARY

From this chapter, the conductive carbon epoxy can analyzed based on the conductive carbon percentage and the resistivity, the microstructure behavior of conductive ink before and after tensile test, analysis on the conductive carbon performance of resistivity during tensile test. Based on the thesis result, conductive ink with the best conductivity has been found out with the best tensile strength. For the microstructure, it was needed to identify any traces of filler or binder or hardener within the ink.

From the above results, carbon adhesives on TPU proved that it had excellent mechanical properties under stretching, but the electrical conductivity yet still need to be further improved. Compared to all the data, 60 wt% conductive carbon filler percentage is the best choices among the samples. It can be presumed from the ratio of filler was higher that the ratio of binder and hardener which have had lower and more stable resistivity value (in table elongation length against resistivity) and better tensile strength.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 INTRODUCTION

In this chapter, the thesis is summarized that the objectives were fulfilled which are to compare the adhesion integrity of conductive epoxy and TPU substrate, to test the tensile and conductivity of two different types of epoxy paste and to observe the morphological microstructure between epoxy paste and TPU. The relationship between sheet resistivity before and during tensile test and morphological analysis for conductive carbon epoxy conductive ink has been identified.

5.2 CONCLUSIONS

In this project, the objectives set are reached. First, resistivity test is done to evaluate the resistivity of different filler loading of conductive epoxy on TPU substrate. In resistivity testing, 30% filler loading did not have any presence of resistivity and conductivity, where the 60%, 90% and 100% of filler loading showed the presence of conductivity. The highest conductivity was 100 wt% followed by 90 wt% and 60 wt%. Thus, the decision of the best filler loading can choose from the filler of loading 60%, 90% and 100% only. Moreover, at filler in the range from 60% until 90% showed a stable adhesion to the substrate.

Next, the objective to test the resistivity of conductive carbon epoxy and TPU, the tensile test is the highlight of the subject. In this case, it can be summarized from the results of average resistivity value when the substrate is elongated. Through the result, 30 wt% filler did not have any resistivity through the tensile test. 60 wt% and 90 wt% conductive carbon filler percentage showed a stable adhesion to the substrate. When the tensile length increased, the density of dislocations and grain boundary decreased,so the conductive properties were accordingly improved. However, samples of 100 wt% filler loading started to crack and break into pieces when undergoes elongation stress. The samples with high percentage of filler had inconsistent adhesion integrity to the surface.

The microstructure of the sample was needed in order to support the objectives of observe the morphological microstructure between epoxy paste and TPU. The resistivity and adhesion integrity can be analyzed to see whether the samples are acceptable and identify the best print resolution for optimizing the performance of printed electronics.

In summary, carbon as the fillers could construct the conductive networks for improving the electrical conductivity. The tensile properties of the specimen had significant improvement when the epoxy increased. Compared to all the data, 60 wt% conductive carbon filler percentage is the best choices among the samples. It has more stable resistivity value and better adhesion integrity. The highly conductive, flexible, and low cost of conductive carbon epoxy on TPU widely used in flexible electronic devices in the future.

5.3 RECOMMENDATION

Based on the thesis above, some improvement can be done to get a better conductivity and adhesion integrity. TPU that have thicker conductors can inhibit cracking, which leads to more locally focused strain. For the future works in this project, the fabrication process is recommended to produce improved conductive epoxy. The mixing, printing and curing process could be adjusted to get a better conductivity and stronger adhesion integrity ink loading.

Besides, Polyethylene Terephthalate (PET) recommended to test the difference of adhesion integrity. The different conductive filler loading also recommended to produce better resistivity. For example, silver powder, silver flake, gold, copper, aluminium and others can be tested.

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