EFFECT OF TYPE OF SUBSTRATES TO CONDUCTIVE INK UNDER THERMAL PERFORMANCE

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A report submitted

In fulfillment of the requirements for the degree of

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DECLARATION

I declare that this project report entitled “Effect of type of substrates to conductive ink under thermal performance” 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 with Honours.

Signature : ……..

Supervisor's Name :……...

Date : ……..

DEDICATION

This report is dedicated to my beloved late father and my beloved mother,

Azmi bin Saad and Zaitun binti Ibrahim

ABSTRACT

This research project were done to investigate the effect of type of substrates to conductive ink under thermal performance. Two type of substrates were use which is Thermoplastic Polyurethanes (TPU) and Polyethylene Terephthalate (PET) were considered to be use in this research project. Carbon conductive ink from company *Bare Conductive* were used to be printed on the substrates using screen printing method. Instead of conventional method of screen printing method which use mesh as ink template, two tape were tape parallel to each other with the gap of 3 mm to create the ink template. The consistency of the ink printed where considered before continue with the resistivity test of the substrate. The conductive ink were printed for both TPU and PET substrates to be use for the resistivity test & surface roughness test. The electrical resistivity were tested using four-point probe from Jandel's RM3000 test unit, with using ASTM F390-98 as guideline. In terms of the substrate's resistivity, it can be conclude that the TPU resistivity is much lower than PET resistivity where TPU at room temperature have 149.2 Ω/sq while PET have 370.8 Ω/sq at room temperature which is twice the amount of TPU. For the resistance against temperature's result, the resistivity of the conductive ink on both substrates will decrease as the temperature applied to the conductive ink increase where at 100°C, TPU resistivity drop till 52.8 Ω/sq while PET resistivity at 100°C is 102.3 Ω/sq. The surface roughness of both of the substrates and the conductive ink will also decrease when the temperature applied increase where for TPU the mean surface roughness are 2.958µm at room temperature and drop to 1.17µm at 100°C while PET's mean surface roughness for room temperature are 1.921µm and drop to 1.332µm for 100°C. For the adhesion test, a new batch of TPU and PET samples were made to be conduct for this test. The test were conducted using ASTM D3359 as guideline. For the adhere test, the conductive ink appear to be easily detached from the TPU substrates compare to PET and the same results occurred as the temperature increase. However this result seems to be contradicted to the fact that higher surface roughness give better adhesion. Therefor, it can also be conclude that this conductive ink are not suitable as it is very easily detach from the substrates.

ABSTRAK

Projek penyelidikan ini dilakukan bagi menyiasat kesan jenis-jenis substrat kepada dakwat konduktif dibawah prestasi termal. Dua jenis substrat telah digunakan iaitu Poliuretana Termoplastik (TPU) dan Polietilena Tereftalat (PET) telah dipertimbangkan bagi digunakan didlam projek penyelidikan ini. Dakwat konduktif karbon daripada syarikat Bare Conductive telah digunakan untuk dicetak pada substrat mengunakan kaedah percetakan skrin. Daripada mengunakan kaedah konventional percetakan skrin iaitu mengunakan jerat sebagai templat dakwat, dua pita di pita selari sesamanya berjurangkan 3 mm untuk dibuat ruang sebagai templat dakwat. Konsistensi dakwat dicetak telah dipertimbangkan sebelum meneruskan ke ujian rintangan kepada substrat. Dakwat konduktif ini telah dicetak pada kedua-dua substrat TPU dan PET untuk kegunaan ujian rintangan dan ujian kekasaran permukaan. Rintangan elektrik telah diuji menggunakan prob empat titik daripada Jandel RM3000 unit ujikaji, dengan mengunakan ASTM F390-98 sebagai garis panduan. Dari segi rintangan substrat, ia boleh dirumuskan bahawa tahap rintangan TPU lebih rendah berbanding tahap rintangan PET dimana di suhu bilik, TPU mempunyai rintangan sebanyak 149.2 Ω/sq dan PET pula 370.8 Ω/sq iaitu dua kali ganda lebih banyak berbanding TPU. Untuk keputusan rintangan terhadap suhu, rintangan dakwat konduktif pada kedua-dua substrat akan berkurangan jika suhu yang dikenakan kepada substrat bertambah dimana di suhu 100°C, rintangan pada TPU menurun ke 52.8 Ω/sq dan PET pul menurun ke 102.3 Ω/sq. Kekasaran permukaan kedua-dua substrat dan dakwat konduktif juga akan berkurang apabila suhu yang dikenakan bertambah dimana untuk TPU purata kakasaran permukaan adalah 2.958μm untuk suhu bilik dan menurun ke 1.17μm pada suhu 100°C manakala purata kekasaran permukaan bagi PET di suhu bilik adalah 1.921μm dan menurun ke 1.332μm ke 100°C. Untuk ujian kelekatan, kumpulan sampel TPU dan PET yang baru telah dihasilkan untuk digunakan di ujian ini. Ujian ini telah dijalankan mengunakan ASTM D3359 sebagai garis panduan. Untuk ujian kelekatan, dakwat konduktif didapati mudah untuk tercabut daripada substrat TPU berbanding substrat PET dan hasil yang sama juga berlaku apabila suhu ditambah. Namun keputusan ini kelihatan bercanggah dengan fakta dimana kekasaran permukaan yang tinggi mampu memberikan kelikatan yang lebih baik. Oleh itu ianya boleh dikonklusikan bahawa dakwat konduktif ini adalah tidak bersesuaian untuk kegunaan kerana ianya mudah untuk tercabut daripada substrat.

ACKNOWLEDGEMENTS

First and foremost, I would like to express my gratitude and give a sincere acknowledgement to my supervisor Dr Mohd Zaid bin Akop from Faculty of Mechanical Engineering of Universiti Teknikal Malaysia Melaka (UTeM) for his guidance, knowledge, support, lecture, tips, and encouragement throughout the completion of this project.

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Lastly I would also give a big thanks to Abdul Mueezz bin Abdul Raheem, Muhammad Aiman bin Suhaimi, Muhammad Amin bin Othman, Nurul Hasanah binti Sobri and Nurul Syafika binti Rozali, my supportive colleagues who always have my back in answering all the questions I asked that many moments that I in doubt.

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LIST OF ABBREVATIONS

FEC	Flexible electric circuit
PCB	Flexible electric circuit
TPU	Thermoplastic polyurethanes
PET	Polyethylene terephthalate
IoT	Internet of things
CNT	Carbon nanotubes
SPC	Stretchable printed circuit
EMF	Electromotive force
Ag-MWNT	Silver multi-walled carbon nanotubes
AFM	Atomic force microscope
SP	Nano Silica Particle
RMS	Root mean square roughness
RA	Mean roughness
$(Mg_3Ca(CO_3)_4)$	Huntite/hydromagnesite mineral
TGA	Thermogravimetric analysis
DSC	Differential scanning calorimetry
Tg	Glass transition temperature

DBD	Dielectric barrier discharge
ASTM	American Society of Testing and Material

LIST OF SYMBOLS

°C	=	Celsius
V	=	Volt
A	=	Ampere
Ω/sq	=	Ohm per sheet square
σ	=	Conductivity
ρ	=	Volume resistivity
mm	=	Millimeter
μm	=	Micrometer
ℓ	=	Sample range length

CHAPTER I

INTRODUCTION

1.1 Background

Flexible electric circuit (FEC) or flexible circuit board is an revolutionized technology for printed circuit board (PCB) where its serve the same purpose which is connect electrical and mechanical component into a device. For PCB, it have a solid substrate property while the FEC have a flexible substrate property. This difference is one of the main point of interest in FEC as it can lead to creation of device or machine which is flexible to do a certain task.

The study on the FEC were conduct by many researcher which consist of combination of varies material of substrates, various combination of filler, binder and solvent to create a conductive ink, the thickness of the conductive ink as well as the width of the conductive ink as well. Not only the combination of filler, binder and solvent to create conductive ink will give a various kind of results data, the combination of substrates and conductive ink as well can give different data for its properties and reaction between the combination make it that there are million possibilities for the data to obtain.

In this study, the type of substrates used where compared between Thermoplastic Polyurethane (TPU) into Polyethylene terephthalate (PET). Both type of substrates will undergo the same experiment which is experiment test in room temperature and designed temperature of 40°C, 60°C and 100°C on both substrates and the ink, adhesion test, surface roughness test and the resistivity measuring of the conductive ink.

1.2 Problem statement

In the industry, the PCB were commonly use in making product. However there are a lot of limitation of PCB which searching for new alternative have been the aim of researcher to research for the new method for the electric circuit. Firstly the limitation of the size of the PCB where it is very hard to store. Bigger PCB mean that there are a lot of components can be attach to the board. However, bigger PCB mean bigger board and this will make it hard to transport or store. FEC reduce this problem where FEC can be reshape which it can be fold to reduce the area to store.

FEC usage is also new in the industry. Its combination between various substrates as well as various conductive ink are potential to have thousands of variety. Therefore there are lack of information and data of the usage of the FEC. This research also include the temperature effect on the combination of carbon conductive ink with TPU and PET substrate.

1.3 Objectives

The objectives of the study is to:

1) To analyst the resistivity of TPU and PET substrates with carbon conductive ink.
2) To compare the resistivity of both TPU and PET sample when different range of temperature applied on the sample.
3) To compare the surface condition of the TPU and PET sample before and after the temperature applied on the sample.
4) To analyst the adhesion capability of the carbon conductive ink on both TPU and PET sample with and without the temperature applied on the sample.

1.4 Scope of project

The scope of this study are to :

1) Make a comparison on the behavior of the substrates and ink when on temperature test of 40°C, 60°C and 100°C.
2) Make a study on the TPU and PET sample surface condition before and after the temperature test.
3) Make comparison on adhesion test result of the TPU and PET sample with no temperature applied and 100°C temperature applied samples.

1.5 Planning and execution

In this project, several action that need to be carry out to complete it which is:

1) Literature review

   Review any related journal, article, or any materials related with the project subject

2) Making sample

   Print the carbon ink onto the substrate to obtain the TPU and PET sample using a suitable ASTM as guidance.

3) Taking data

   Obtaining the resistivity on the TPU and PET sample by conduct the experiment on the sample with and without apply temperature to the sample using suitable ASTM as guidance.

4) Surface roughness

   Conduct experiment on surface roughness on the TPU and PET sample.

5) Adhesion test

   Make a new sample for TPU and PET sample with different dimension according to the ASTM for the adhesion test.

6) Result analysis

   Analysis the result obtain from all variables applied on the FEC.

7) Report writing

   Write a report based on data obtain from the experiment by the end of the experiment.

By referring Table 1.1 below, the research activities for PSM1 illustrate from start of the PSM title selection, literature review, design of experiment, sample preparation and testing, data analysis, submission of progress report 1, final report draft submission and PSM seminar 1.

Table 1.1 PSM 1 planned schedule

Week/Activities	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Research title selection														
Literature review														
Experiment design														
Sample testing														
Data analysis														
Submission progress report 1														
Final report draft submission														
PSM 1 seminar														

In PSM 2, the activities planned were compiled in a schedule as Table 1.2 below. The research start with continuing the literature review from PSM 1 to fill in studies on the test that will be conduct in PSM 2. The research then continued with apply temperature experiment, surface roughness test, and surface morphology study on the same TPU and PET sample which were made in PSM 1. A new set of TPU and PET sample were made for adhesion test. All data will then be analyst and the result will be discuss before it is compiled in the report. The finished report will then be submit before the presentation in PSM 2 seminar.

Table 1.2 PSM 2 planned schedule

Week/Activities	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Literature review	■	■	■	■	■	■	■	■	■	■	■	■		
Apply temperature experiment	■	■	■	■	■									
Surface roughness test						■	■	■						
Adhesion test					■	■	■	■	■	■	■			
Analysis					■	■	■	■	■	■	■	■		
Results and discussion					■	■	■	■	■	■	■	■		
Submission progress report 1							■							
Final report draft submission												■		
PSM 2 seminar														■

CHAPTER II

LITERATURE REVIEW

2.1 Introduction

In this chapter, a review of flexible printed circuit, conductive ink, ink printing, ink curing, conductivity of the ink, material of substrate, surface roughness of the conductive ink as well as the behavior of the substrates itself were reviewed from previous studies on this area.

2.2 Flexible electric circuit

Flexible electric circuit (FEC) is a new emerging technology for a electronic device. Its a replacement for the use of printed circuit board (PCB) which commonly used. However, due to its nature of new technology, FEC were tested into various of experiment variation which involve the substrates, ink, and more (Byun et al., 2017; Din et al., 2017).

Initially the PCB which is consist of circuit on top of hard board while FEC is circuit on top of board which is soft and flexible. As the experiment on FEC increasing on testing the properties and performance of FEC, more and more new technologies emerge which revolve around technology of FEC. This include the properties of soft behavior of FEC on flexibility, stretchability, and more. Experiment on mixing both properties of softness of FEC and hardness of PCB were also made to increase the varies in this new technologies (Byun et al., 2017).

According to Norhidayah et al. (2017), the basic component for FEC consist of substrates which is usually made up of polymer and conductor (conductive ink). The FEC were tested by experiment and simulation to find out the effect of mix matching different type of substrates and conductive ink. The effect of fillers composition in the conductive ink with solvent and binder are also studied. A conclusion were made where usually the best curvature angle will give better stretch ability interconnection of the substrates (Norhidayah et al., 2017).

One of the main use of FEC, which is wearable electronics usage is increasing by numbers by days lead to research on improve the gadgets. With the increment of the usage of wearable electronics, it bridging the users with their electronics. This can lead to the users use their gadgets in daily tasks which can lead towards the development of Internet of Things (IoT).

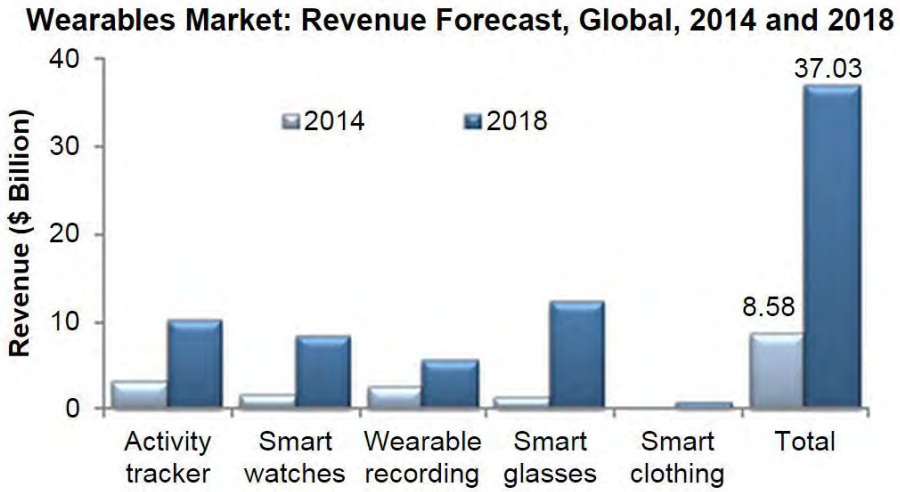

Figure 2.1 Wearable Market Share Revenue for 2014 and Forecast Revenue for 2018 (Suikkola, 2015)

The increment from 8.58 to 37.03 billion in revenue in 2018 is to be expected as it is start to be used by multiple industries. For example, sports industry in product of Pedometer where it is used to count the number of steps of its user and heart beat waist band for checking the heart beat of the users (Qi & Boyce, 2004; Suikkola, 2015).

In a research paper by Din et. al. (2017), it is stated that the most important ingredient in making a good stretchable printed circuit is that it contain conductive filler and a non-conductive stretchable polymer. Fillers such as carbon nanotubes (CNT) and silver nanowires were embedded into conductive polymer making the substrates electrically conductive as the filler materials touching each other. Some polymer such as ionic polymer, which a conductive polymer with low electrical conductivity, need higher amount of filler to make it more conductive. However, this will cause the substrate become more brittle. Each type of fillers have different electrical conductivity value. Thus the amount need to make a substrate with filler and polymer ratio is varies with the filler itself (Din et al., 2017; Yu et al., 2017).

FEC have different characteristic with stretchable printed circuit (SPC) where SPC is test to check on the capability of the printed circuit to stretching while FEC test on the ability to flex. However, the mechanical and thermal properties of both type of printed circuit can be test almost similarly. In this case, a research by Happonen et al. (2015) stated on the capability of FEC to flex after numerous flex cycle.

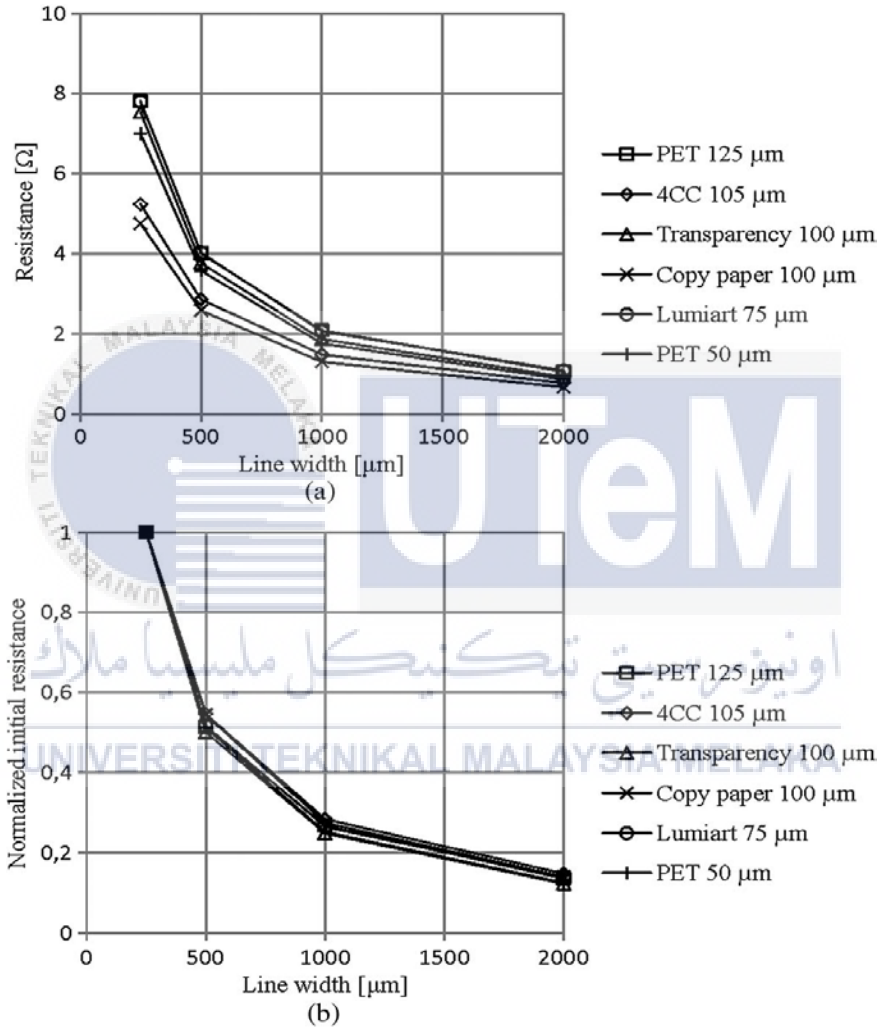

Figure 2.2 (a) absolute and (b) normalized initial resistances of the studied populations as a function of trace width (Happonen et al., 2015)

In this research, the researcher state on through different material of substrates with different thickness, the resistance for the electrical conductivity of the substrates decrease as the width increase. This pattern is consistent across all material which were present and used in the research. An assumption of the research and any future research on this subject can be made where the thickness increase will reduce the resistivity (Happonen et al., 2015: Happonen, 2016).

Happonen et al. (2015) also state in his report which discuss on fatique effect. In the thesis, the fatique means the response of the material which deformed after being applied repeating cycles of loading. Additionally, fatigue can be seen as small crack in the substrate or ink, which then accumulate into bigger crack and physically damaged. However, physical loading is only one of the factors affecting the fatigue life. Others factors which affecting fatigue life can be seen in Figure 2.3 below. Therefore, in order to extend the life of the substrate, these factors need to be considered when use the material for any applications (Happonen et al., 2015).

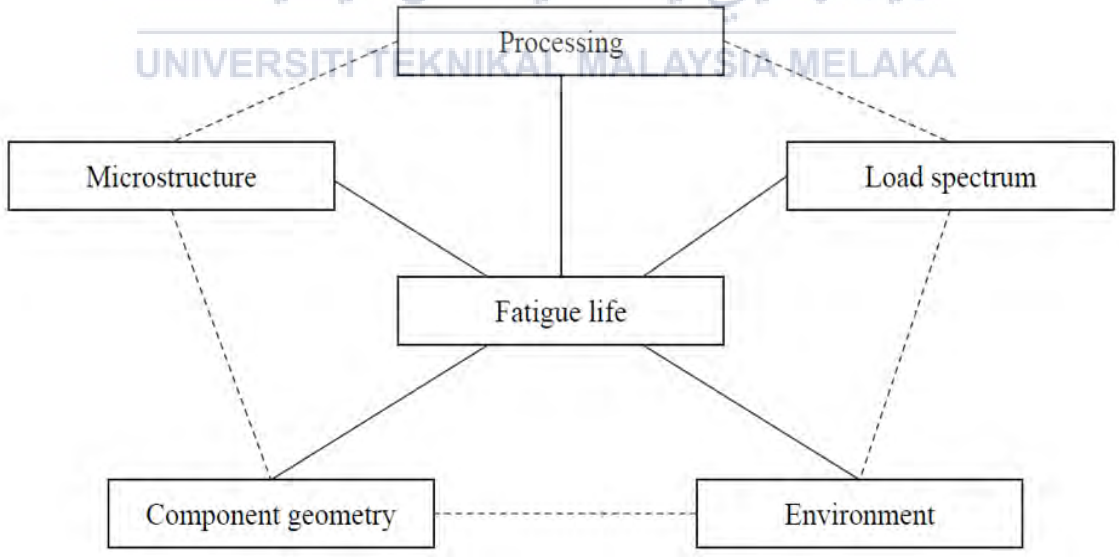

Figure 2.3 Factors affecting fatigue life (Happonen, 2016)

2.3 Conductive ink

In relation of substrate with conductive ink, there are difficulties in print the ink with the substrate. This is because most substrate is hydrophobic and have low surface energy. This indicate that the ink will be having a difficulty to adhere to the substrate the if the substrate have a smooth surface and the ink wettability will make it hard for both of the ink and substrate to stick together. The adhere factors between both substrates and ink can be determine by (Cruz et al., 2016):

a. Substrate properties
b. Ink properties
c. Superficial tension
d. Functional group and their intermolecular forces present in the ink/polymer system
e. Surface topologies and mechanical locking mechanism between ink and polymer

Conductive ink is an ink which were blended by mixing 3 main component which is conducting filler, non-conducting resin/binder and volatile solvent(s). Occasionally some conductive ink were also mixed with additives to further improve the ink. In conductive ink, the conductive filler were blend together with resin (binder) and solvent to make it into either liquid (with additive) or paste type of ink. The different between these two type of conductive ink are the liquid ink contain more solvent but low viscous, and the paste ink is vice versa (Bhore, 2013; phillips, 2017).

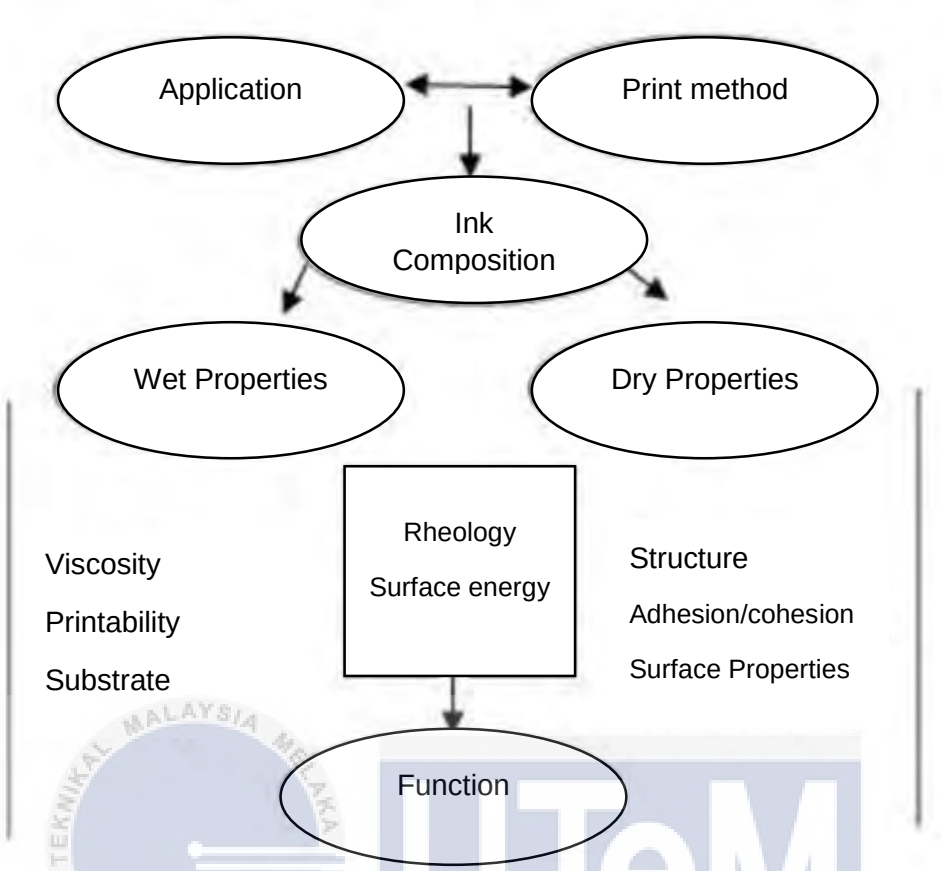

Figure 2.4 Various factors governing the properties of a conductive ink (Bhore, 2013)

When making a conductive ink, the composition ratio between the 3 main component can be varies and as well obtain a different characteristic and properties. It is essential for researchers to make a same ratio when making a batch of the conductive ink as different amount of ratio can differs the electrical conductivity obtain.

In comparing the PCB and FEC or SPC, the conductivity of PCB depend on the copper lining in the circuit and FEC or SPB use conductive ink. PCB circuit lining need to be conventionally solder the interconnection where FEC or SPC use the conductive ink's adhesive properties. The properties of conductive ink have above conventional solder are (Yim & Paik, 2006):

a. Environmentally friendly since conventional soldering need to use lead-base solder as adhesive material of the interconnection.
b. Processing temperature for conductive ink is lower compare soldering.
c. Conductive ink is more flexible compare to soldering.
d. Soldering need higher skill to have a neat and clean finishing while conductive ink does not.
e. Conductive ink have higher option substrates that can be use such as glass and fabric.

2.4 Carbon ink

Carbon ink is commonly used conductive ink as it is consist of conductive filler, polymetric binder and organic solvent which is all three are cheap in cost. Carbon ink's filler consist of carbon and graphite which both material have characteristic which they are easy to process, low current conduction, good surface chemistry and ratioly modify. By adjusting these two ratio as filler, it can give various results (Monteiro et al., 2015; Phillips, 2017; Li & Meng, 2013).

In this research, the carbon ink used is commercialized carbon ink by company Bare Conductive. Due to confidentiality of the product information, no information regarding the ratio of filler, binder and solvent used by the company used is disclosed in this research (n.d, p.1).

2.5 Screen printing

There are few methods that been used in producing FEC were studied to identify the relationship of its flexible interconnect. Few examples are lamination of ink and substrates, inkjet printing, and the commonly used which is screen printing. In this project, screen printing method were used as it is suggested by the commercialize ink product's instruction.

There are 4 main components in screen printing which is ink paste to make the conductive ink, screen for the pattern design, substrates used, and squeegee which is the tools used to spread the paste throughout the screen.

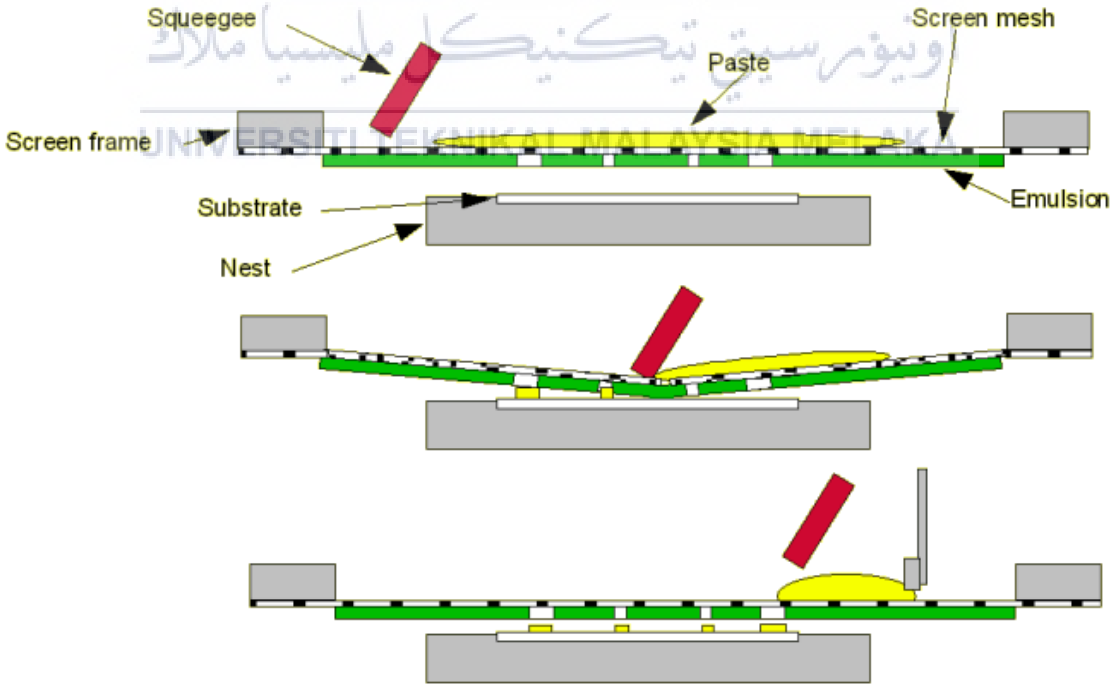

Figure 2.5 Fundamental principle of screen printing (Suikkola, 2015)

By refer the Figure 2.5 above, the concept of screen printing can be seen. The paste were placed above the screen along the substrate. Squeegee were used to spread the paste along the screen. The paste will paste on top of the substrate accordingly to the shape of the screen (Suikkola, 2015; Khirotdin et al., 2016).

2.6 Ink curing

After the conductive ink pasted, the volatile solvent evaporates, the mixture will be left with the filler and binder. The binder is needed for adhere the conductive ink with the substrates while the filler is needed to conduct the electricity. This process is known as ink curing (Phillips, 2017). The composition of the conductive ink during curing process showing in Figure 2.6.

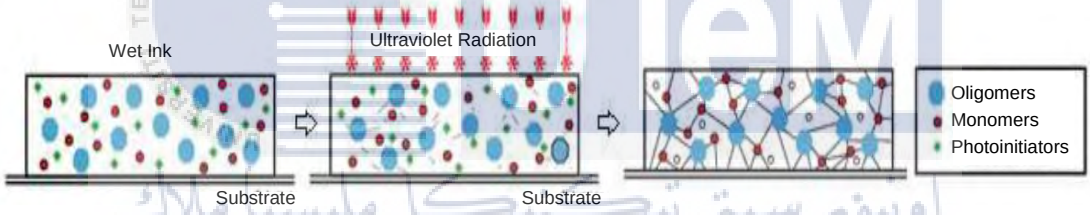

Figure 2.6 Conductive ink reaction to UV radiation in curing process (Bhore, 2013)

Curing is the process which the change of overall electric properties in printed conductive ink were produced by microstructurally change the particle in the ink by heating the ink with a designed work temperature range. By heating the conductive ink to the maximum allowed temperature, the electrical performance of the conductive ink can be improve dramatically (Roberson et al. 2011).

For this research on the effect of type of substrate to conductive ink under thermal performance, the carbon conductive ink used does not have a conventional curing process. Rather than cure the conductive ink using the curing machine, the carbon conductive ink were left at room temperature for 15 min till it dry. The reason it does not need to be cure in the curing machine is due to the warning notes left by the company of this commercialized carbon ink in the conductive ink data sheet (n.d, p.1).

2.7 Conductivity

In an electrical circuit, electromotive force (EMF) is the force which cause the movement on the electrons. EMF does not move the material instead the motion of electron running in the circuit determined by the EMF or voltage of the circuit. The electrical charge determine by the rate of the electron motion in the circuit or also known as current. If there is any opposing flow of electron happen to presence in the circuit in response to the applied voltage, it is known as the resistance.

This three main elements in the electrical circuit can be formulate in to Ohm's Law which is

$$V = IR \quad (1)$$

Where V is the voltage measured in volts (V), I is the current (A) and R is the resistance (Ω).

In this project, assuming the voltage of the circuit is constant, the conductivity which where needed to determine will be varies depend on the resistance in the substrate and ink of the circuit. By referring the Figure 2.7 below, the resistance varies depend on the material used (Banfield, 2000).

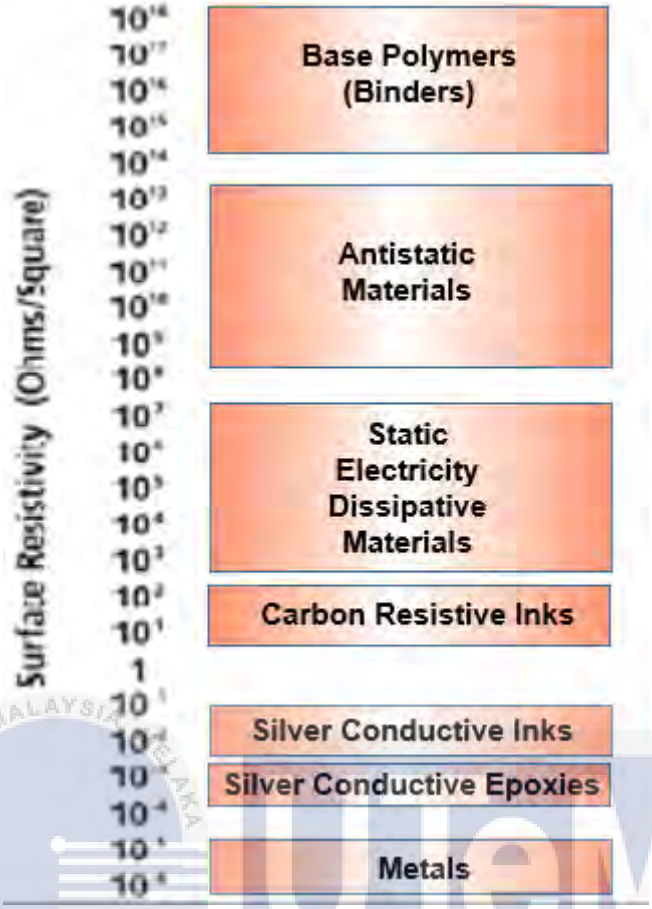

Figure 2.7 Surface-resistivity spectrum (Banfield, 2010)

Electric conductivity is a measurement used to identify the capability of the material to direct electric current in the circuit. Conductivity is directly increase when resistance of the electrical particle to move in the material decrease (Khirotdin, 2016). By determine the resistivity of the material by using (1), we can derive:

$$\sigma = \frac{1}{\rho} \tag{2}$$

Where σ is conductivity and ρ is volume resistivity

2.8 Printed pattern

In searching for the stretchable and the highest endurance on cycle resistance SPC while obtain the highest conductivity ink, the pattern of the printed conductive ink play a major role in the research.

The pattern for printed conductive ink is very important. This is because with all the motion of the substrates whether flex or stretch, the ink need to be stay connected to ensure the circuit complete. If the conductive ink is easily broke, then the circuit will fail as well.

In a journal Kim et al. (2013), the author test on 4 different pattern of printed conductive ink.

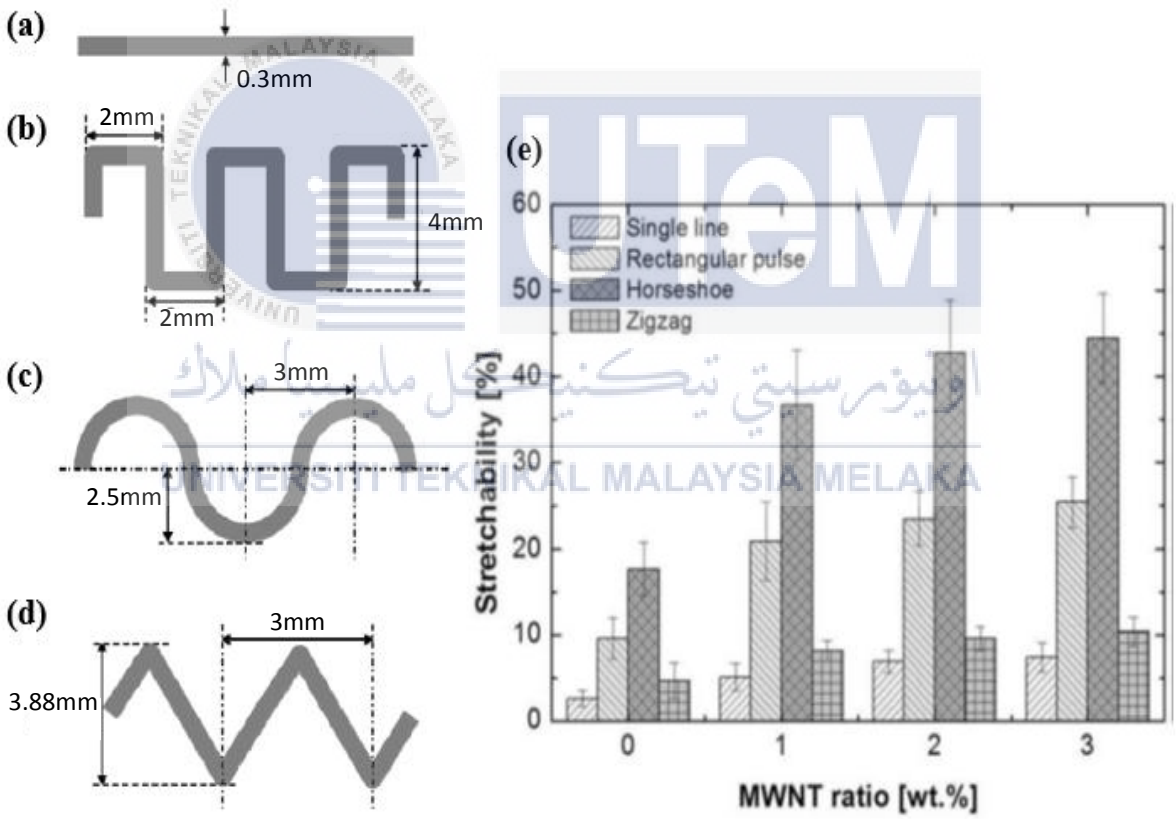

Figure 2.8 Schematics of the various circuit design: (a) single line, (b) rectangular shape, (c) horseshoe/serpentine, (d) zigzag, and (e) the stretch capability of the Ag-MWNT circuits with different mixture ratio (Kim et al., 2013)

By referring Figure 2.8, the ratio of Silver multi-wall carbon nanotubes (Ag-MWNT) in the sample does increase the stretchability of the sample. But in the case of the pattern in relation with the stretchability, the horseshoe have the highest stretchability compare the others, having the highest stretchability percentage which is around 45% from its initial, it is the most suitable in product making. However, the backdraw from this pattern is that it took more ink to print as wanted where single line most probably use less conductive ink to print it shape. It is also conclude from that report that the pattern of the stretchability start with single line being the less stretchable, then zigzag, rectangular pulse and lastly the horseshoe pattern which the horseshoe pattern is the most stretchable (Kim et al., 2013).

Another research by Yu et al. (2016) have few data's on ink pattern stretchability. From the data present in that research show that horseshoes/serpentine pattern of the ink can produce different elastic stretchability percentage by changing the arc angle. The data shows that the higher the arc angle will allow for higher elastic stretchability of the ink. As shown in Figure 2.9, the arc angle of 235° were use and the stretchability keep increasing up until the strain reach 60%. Buckling mode is demonstrated when the interconnect strain reach 80% (Yu et al., 2016).

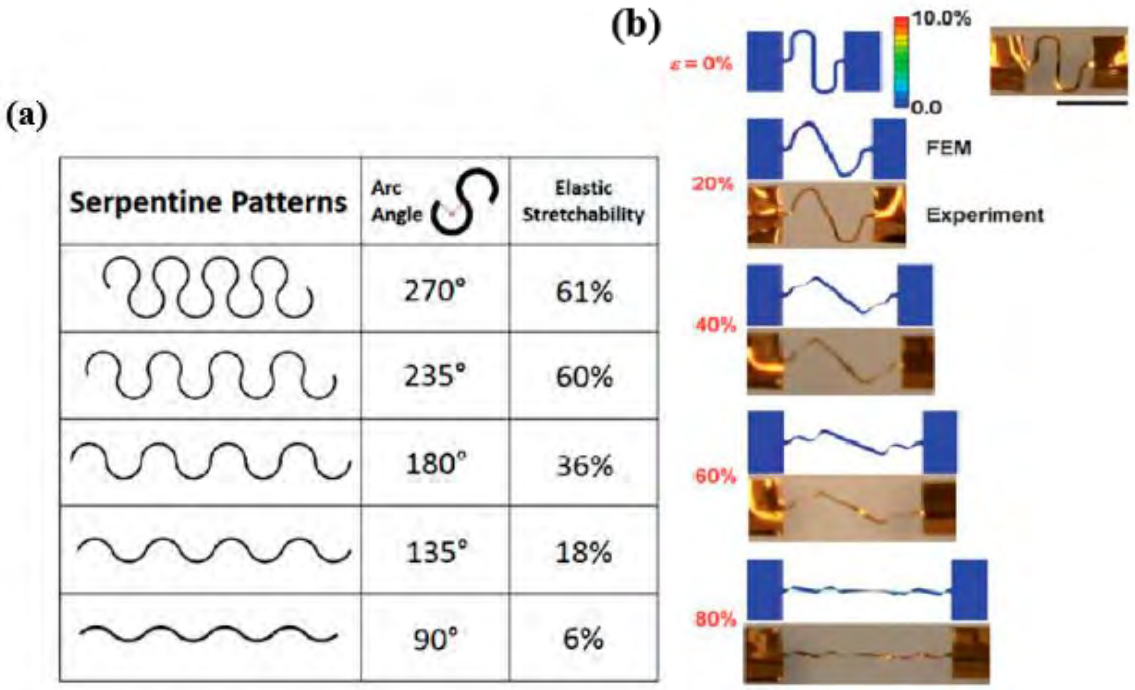

Figure 2.9 a) simulated of elastic stretchability for horseshoe/serpentine patters with different arc angle, and (b) experimental and FEM analysis of sympatric buckling behavior of the horseshoe/serpentine pattern with strain from 0% to 80% (Yu et al., 2017)

2.9 Substrate material

In the use of substrates, there are various material can be choose and use to be made as combining with the conductive ink to make up the printed circuit. Some material is stretchable, some can be flexible, some have high melting point, and more. Depending on the material, it can give different result in conductivity, resistivity, strain properties, thermal results and more.

Polymer which is the basic structure of plastic is a combination of monomer into a macromolecule chains by using chemical process. From here, there are two types of polymer which is Polyaddition and Polycondensation. By refer the Figure 2.10 below, a full list of polymers and plastics currently available commonly use were listed (Klein, 2012).

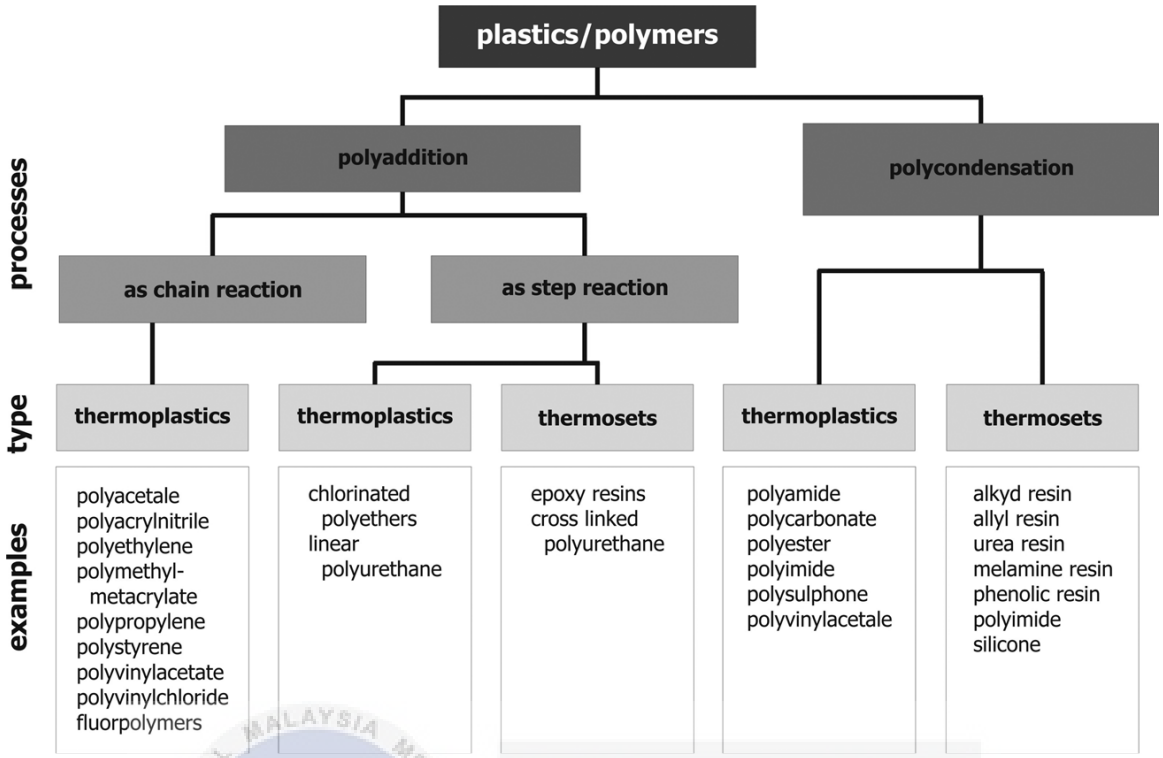

Figure 2.10 Processes of producing plastic and its example (Klein, 2012)

In this research, the main point is to identify the resistivity of the FEC under thermal (heat temperature) load as well the surface roughness after heated. Several important information that need to be take note on the materials when been apply the load are (2011, p.5):

a. Wear and tear resistance of the substrate when applied the load.

b. Maximum temperature that the material can withstand.

c. Flexibility of the substrates when applied with heat.

d. The surface energy effect on the adhesion of the conductive ink.

2.10 Thermoplastic Polyurethanes (TPU)

Thermoplastic Polyurethanes (TPU) is a material which combination of two properties which is stretchable like a rubber but can also be process as plastic. This material particle consist of hard and soft segment which cause this material to be able have a wide range of elasticity as well as strain durability.

A research were made which state that thermoplastic is a material which have high range of combination of hard and soft elasticity which can be used in various application. Soft elasticity consist of elastomer while hard elasticity come from thermoset does not melt easily while it does melt under mechanical, thermal and radiation energy (Klein, 2012).

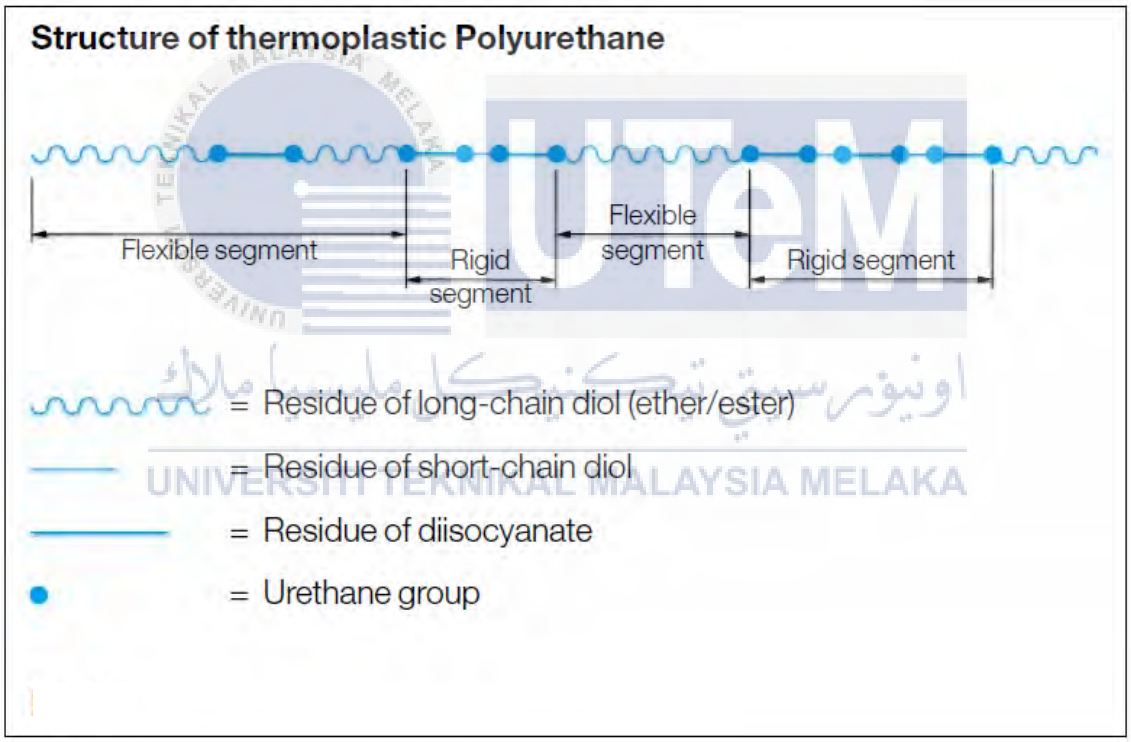

Figure 2.11 TPU structure (2011, p.5)

In many TPU, the hard segment control the rubbery properties of the TPU which govern the tensile strength of the material while the soft segment control the stretchability of the TPU where it govern the elongation of the TPU. Different ratio of hard and soft properties give different ratio of tensile strength and elongation of the TPU (Qi & Boyce, 2004).

According to Cho et al. (2017), a experiment were made on identifying the true strain and true stress of TPU comparison when different ratio of soft and hard properties is used. In their report, they stated that the ratio as Table 2.1 below were used and the TPU then applied with a deformation of maximum strain of the TPU (Cho et al., 2017).

Table 2.1 Max stress of TPU with different Hard and soft TPU segment

	TPU_A	TPU_B	TPU_C
Hard Segment	30%	43%	56%
Soft Segment	70%	57%	44%
Max Stress	10MPa	20MPa	36MPa

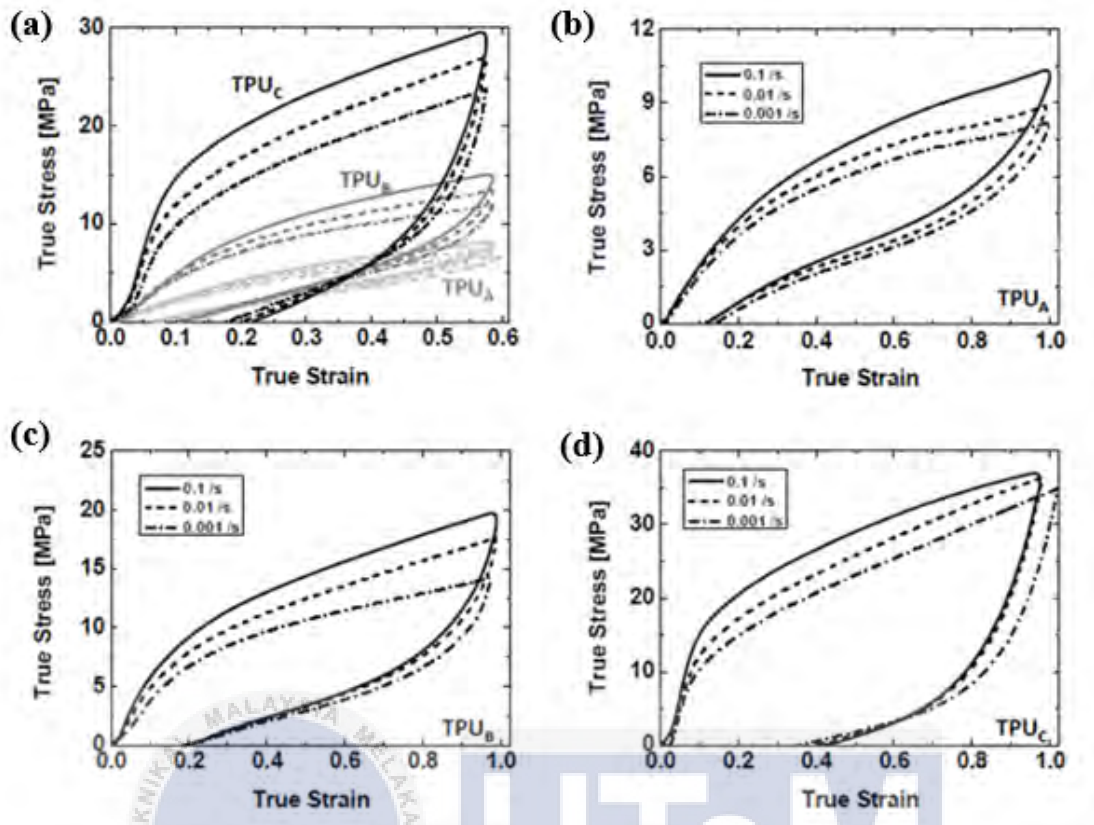

Figure 2.12 Stress-strain behaviors of TPUs when under a large strain compression acting on (a) overall TPU, (b)TPUa , ©TPUb , and (d)TPUc (Cho et al., 2017)

As seen in the Figure 2.12, the maximum stress will increase as the ratio of hard segment increase in the TPU. In the experiment, the strain rate were also test at 0.001, 0.01 and 0.1 /s in each ratio where the smaller the strain rate also effect the behavior of the TPU.

TPU behavior can be seen different using atomic force microscope (AFM) when it is synthesize with another material. Since TPU have a smooth and low surface energy, it does not adhere nicely with many type of conductive inks. Cruz et al. (2016) have conduct experiment by synthesizing TPU with Nano silica particles (SP).

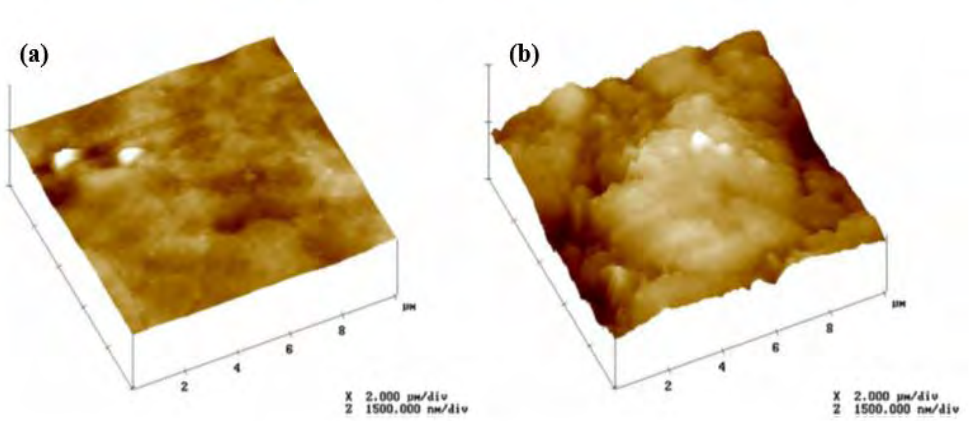

Figure 2.13 AFM topographical images of the (a) TPU and (b) SP–TPU surface (Cruz et al., 2016)

In the research, by combining SP and TPU, the SP deposition and surface cleaning process were made as sample preparation. It is later thermally treated to achieve the particle sink-in which able the conductive ink material to adhere better on top of the surface of TPU. This can be proved where the data in Table 2.2 below. The increment of root-mean-square roughness (RMS) and mean roughness(Ra) mean that the synthesize increase the surface roughness of TPU which mean the adhere between conductive ink and polymer can be increase (Cruz et al., 2106).

Table 2.2 Root mean square (RMS) and mean (RA) roughness of TPU and SP-TPU

	RMS Roughness (nm)	**RA Roughness (nm)**
TPU	27.056	19.574
SP-TPU	194.94	152.40

2.11 Polyethylene Terephthalate (PET)

Polyethylene Terephthalate or also widely known as PET is a thermoplastic polymer resin from polyester group. PET is commonly used by people all over the world as it is a material which is good mechanical and thermal properties, good chemical resistance and low cost. Having this properties is the reason that PET have a wide range of application in daily life as it possess a good balance in its material properties (Kahraman, 2015; Gao et al., 2017; Miranda et al., 2017).

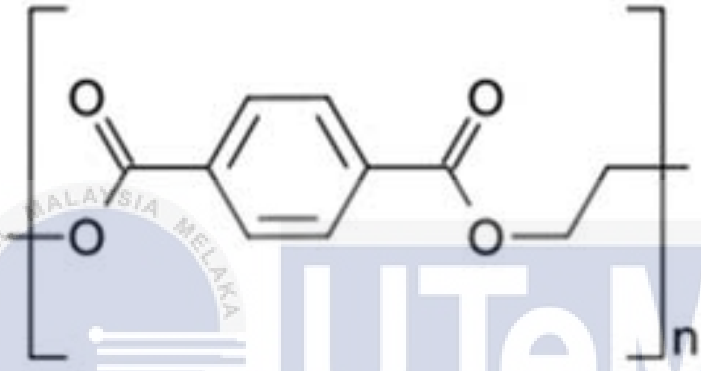

Figure 2.14 Structure of PET (Miranda et al., 2017)

Due to PET usage in various of application, a research by made Li et al. (2016) on the aging effect on the usage of the material. The researchers believe that the usage may be at an environment which exposed to high risk of radiation need to be emphasis for the awareness on the safety of the users of the product from this material.

By thermally control the sample inside a test chamber, the thermal aging effect can be simulated on the PET sample (Li et al., 2016). The data obtain from the research shows that the increase of aging time do effect the surface potential which it is reduce rapidly but will gradually become stable. The increase of aging time and radiation dose, the surface potential decay rate for sample PET will be increasing.

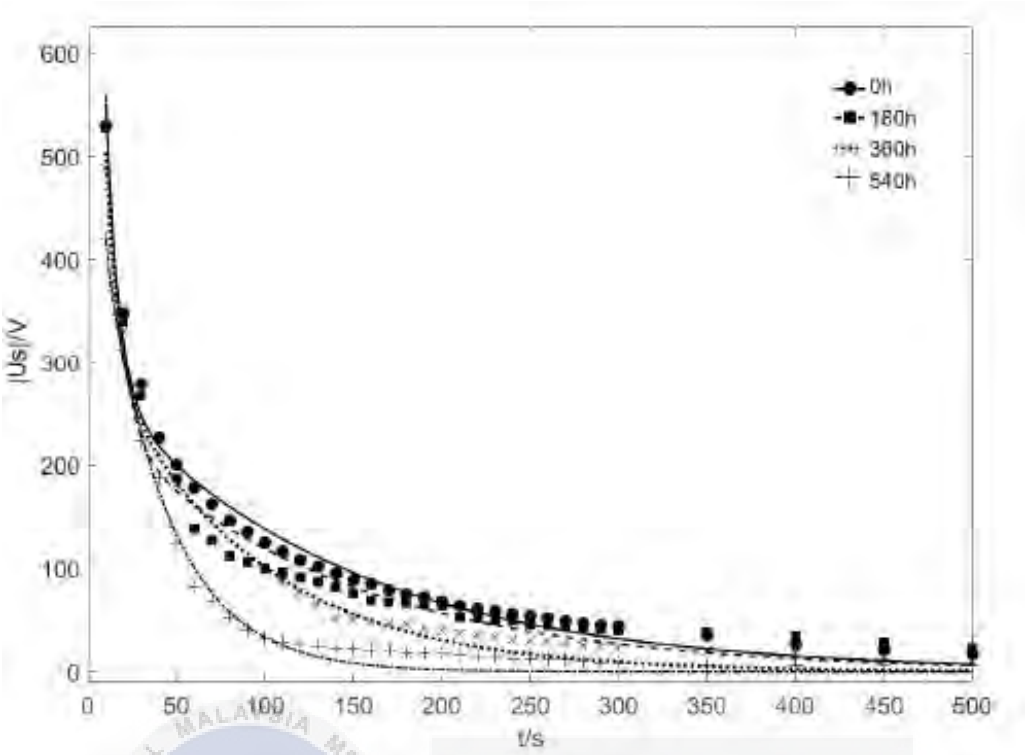

Figure 2.15 Surface potential amplitude and decay time of thermally aged sample (Li et al., 2016)

With the change of the surface potential, it will also change the conductivity where conductivity will decrease if the surface potential decrease. The aging time and radiation dose however will effect the volume conductivity of the sample which the increment of aging time and radiation dose will increase the volume conductivity (Li et al., 2016).

Other research on PET behavior is on the thermal stability of the material were made by Kahraman (2015), where they research on improving the thermal stability of PET by combining PET with Huntite/hydromagnesite mineral ($Mg_3Ca(CO_3)_4$). Huntite is a non-corrosive and environmentally safe to use material as it is flame retardant additives. Therefore many studies were made on Huntite to used as filler (Kahraman, 2015).

Table 2.3 Formulation content for PET and Huntite ration (Kahraman, 2015)

Sample name	*m*-Cresol (mL)	PET (g)	Huntite (g)	Huntite (%)
PetHt0	40	1	–	0
PetHt1	40	1	0.1	10
PetHt2	40	1	0.2	20
PetHt3	40	1	0.3	30
PetHt4	40	1	0.4	40

By using thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) to determine the thermal properties of the composites, results obtained as Table 2.4 below.

Table 2.4 Thermal properties of composite for PET and Huntite (Kahraman, 2015)

Sample name	$T_{\%5}$ (°C)	$T_{\%10}$ (°C)	$T_{\%50}$ (°C)	Char yield (%)	T_g (°C)
PetHt0	173	372	424	0	73
PetHt1	377	398	457	14.4	98
PetHt2	365	396	533	22.0	103
PetHt3	361	386	538	24.0	111
PetHt4	362	391	543	24.8	146

By compare both of the Table 2.3 and Table 2.4, it is can be said that the glass transition temperature, T_g increase as the huntite percentage in the composite increase. It can be conclude that the composite of PET/Huntite is more thermal stable compare to normal PET (Kahraman, 2015).

Gao et al. (2017) investigate on the surface roughness of PET films by using dielectric barrier discharge (DBD). The electrode arrangement of the of DBD generate a non-uniform distribution of plasma to the film create the formation of three region on the film which is central zone, boundary zone and diffuse zone.

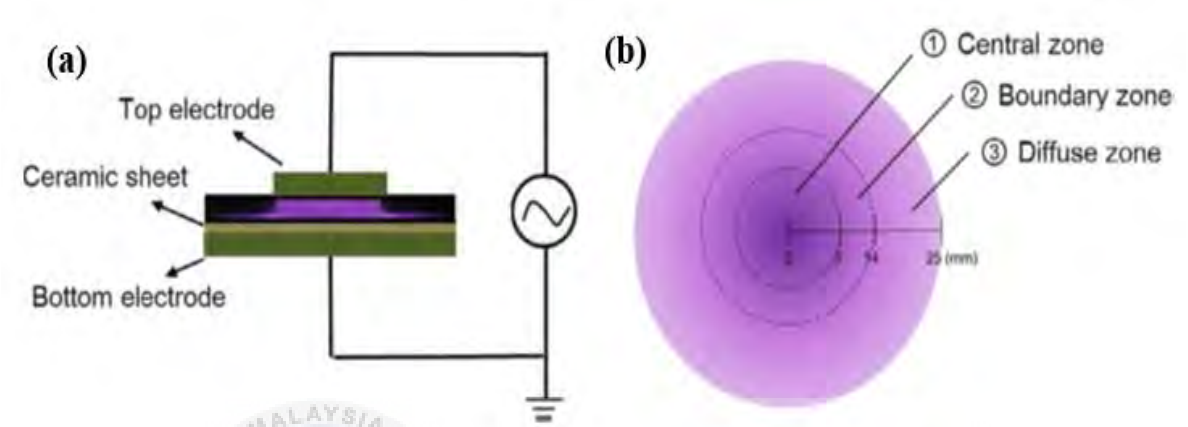

Figure 2.16 A dielectric barrier discharge plasma sketch with (a) typical discharge image of plasma system. (b) the schematic picture of the surface modified different areas (Gao et al., 2017)

It is proven that polymer surface can be roughen by using plasma, but zones of different surface roughness produced from the process.

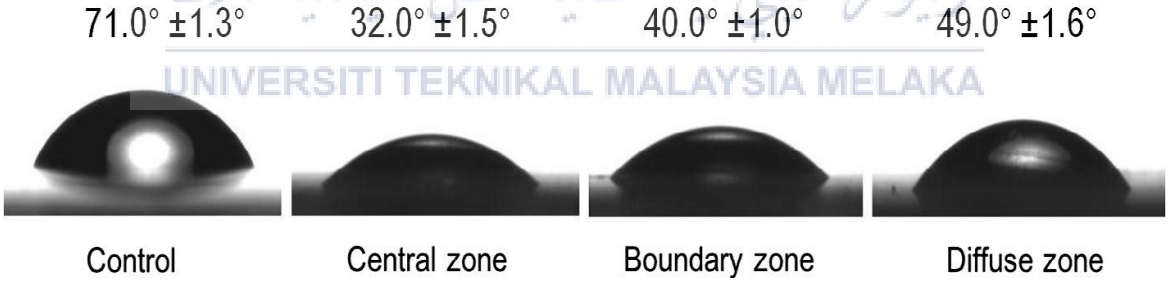

Figure 2.17 Image of water droplet on top of the PET film surfaces (Gao et al., 2017)

In a normal case, the contact angle is around 71.2°, but the contact angle on the zones which were processed have a lower contact angle than the controlled sample as shown in Figure 2.17 (Gao et al., 2017). It can be conclude that the surface roughness of PET can change and it can effect the adhere of the conductive ink on the substrate. This will indirectly effect the conductivity of the circuit.

This can further prove as the thesis made by Eshkeiti (2015), where a part of the research focus is on comparing the resistance vs the roughness of the material of glass, PET and paper. The characteristic of the substrates can be seen in the Table 2.5 below.

Table 2.5 Summary of different characteristic substrates

Substrate	Surface energy (dynes/cm)	Thickness (µm)	Roughness (µm)
Paper	53.9	177 ± 12	0.175
PET	43.8	127 ± 1	0.015
Glass	58.21	629 ± 1	0.005

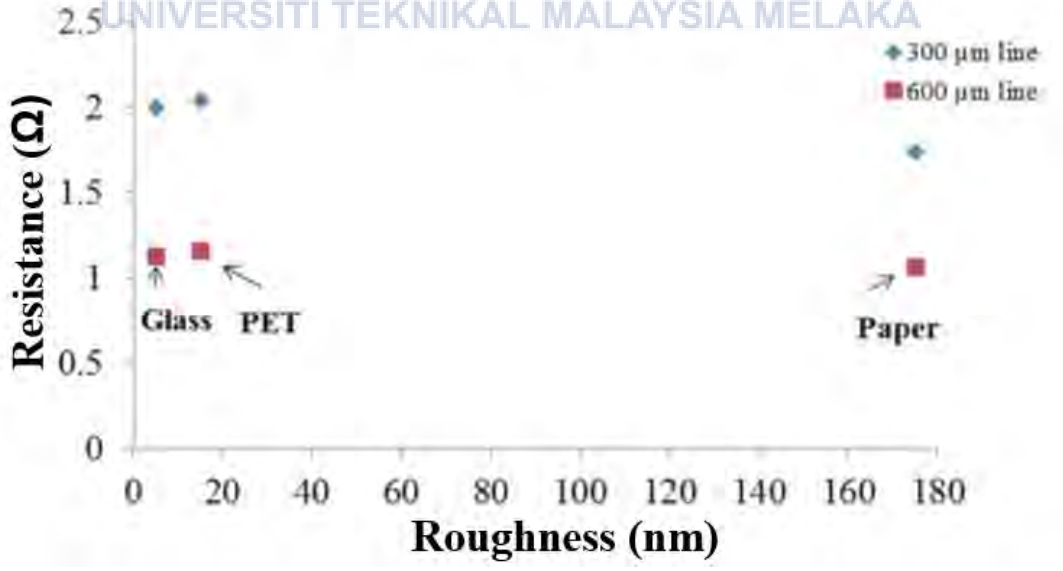

Figure 2.18 Effect of roughness on resistivity of printed line (Eshkeiti, 2015)

2.12 Surface roughness

Surface roughness of the substrates play an important role in the conductivity of the conductive ink. One of the importance are the conductivity itself where the higher surface roughness will give a higher conductivity and lower resistivity. As stated by Wood Et al. (2005), where their research regarding "Paper substrate and inks for printed electronics", they conduct the experiment on a sample of UniTherm Sharp substrate provided by Stora Enso to compare the conductivity of the substrate before and after the surface smoothed by calendar process.

Table 2.6 Effect of surface roughness on conductivity (Wood et al., 2005)

Calendaring Conditions	**Roughness (microns)**	**Thickness (cm)**	**Conductivity** $(S.cm^{-1})$
None	1.58	0.001	1177
10# 1 Pass 1 Side	1.31	0.001	945
40# 1 Pass Each Side	1.25	0.001	786
10# 2 Pass 1 Side	1.24	0.001	812
50# 3 Pass Each Side	1.15	0.001	852

As shown in Table 2.6 above, the substrates were calendared into few conditions to obtain different surfaces roughness. When the surface roughness at normal condition, the surface roughness is the highest. At this condition, the conductivity obtain is the highest. However, the conductivity decrease when the surface roughness is reduced. Therefore it can be conclude that the surface roughness of any substrates can effect the conductivity of the circuit as well instead of the conductive ink only. Also can be note that the thickness of the substrates does not effect the conductivity of the substrates on any circumstance of the surface roughness.

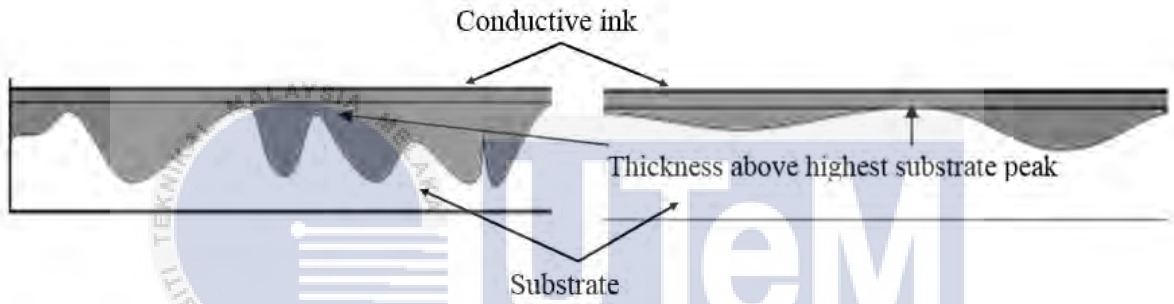

Figure 2.19 Schematic adhesion between conductive ink and substrate (Ryan & Lewis, 2012)

Another important of surface roughness for the substrates is the adhesion capability of the conductive ink with the substrates. According to Ryan & Lewis (2012) the ideal adhesion capability can be obtain which the higher the surface roughness, the higher the effectiveness of the adhesion between the surface of the substrates and the conductive ink. As shown in Figure 2.19 above, the conductive ink deposited under the thickness above the highest substrate peak represent the adhesion capability of the conductive ink. However a good balance between surface roughness of the substrates are needed as smooth surface roughness will make the adhere of the conductive ink became weaker and high surface roughness will cause discrepancies of the ink thickness.

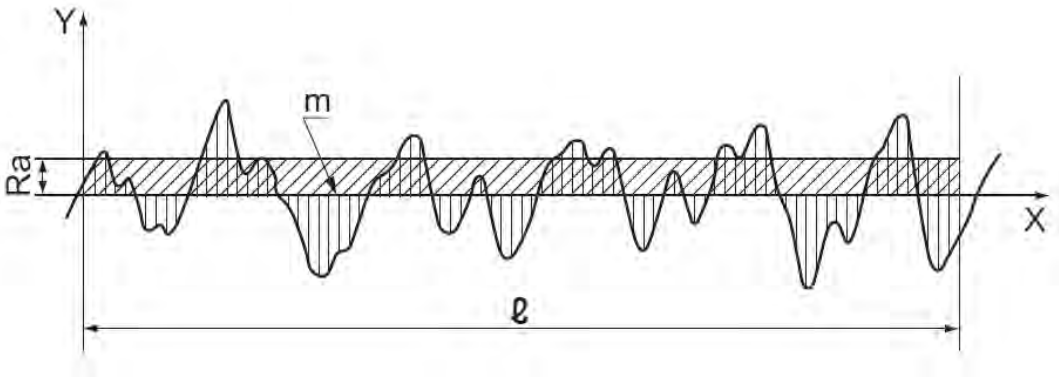

Figure 2.20 Mean average roughness, Ra (2001, p.1)

In theoretical, the Figure 2.20 above show the mean arithmatic sketch of the surface roughness value of the substrate (Ra). Ra is obtained from using the formula below which the roughness curve expressed in terms of y = f(x), where the X-axis to the mean line direction and Y-axis is the mean of the roughness curve value along the range sample of length, ℓ. (2001, p.1)

$$\mathrm{Ra} = \frac{1}{\ell} \int_0^{\ell} \{f(x)\}\, dx \qquad (3)$$

CHAPTER III

METHODOLOGY

3.1 Overview of research

In this chapter, the method of sample preparation, jig producing, sample testing as well as method of taking data result from the sample were explained. Also included in this chapter are description of the materials, tools and apparatus used throughout this research project. By referring to the Figure 3.1, the summary of the procedures for this research project were shown. The research project can also be divided into few main activities which is:

a. The preparation of TPU and PET printed circuit sample.
b. Listing suitable tools, apparatus, and materials accordingly with the procedures in this research project.
c. Conduct experiment test with designed temperature (RT, 40°C, 60°C and 100°) on the TPU sample accordingly with the research plan.
d. Conduct surface roughness test on TPU sample which pre-heated and heated (40°C, 60°C and 100°).
e. Conduct adhesion test on TPU sample
f. Re-do all test on PET sample

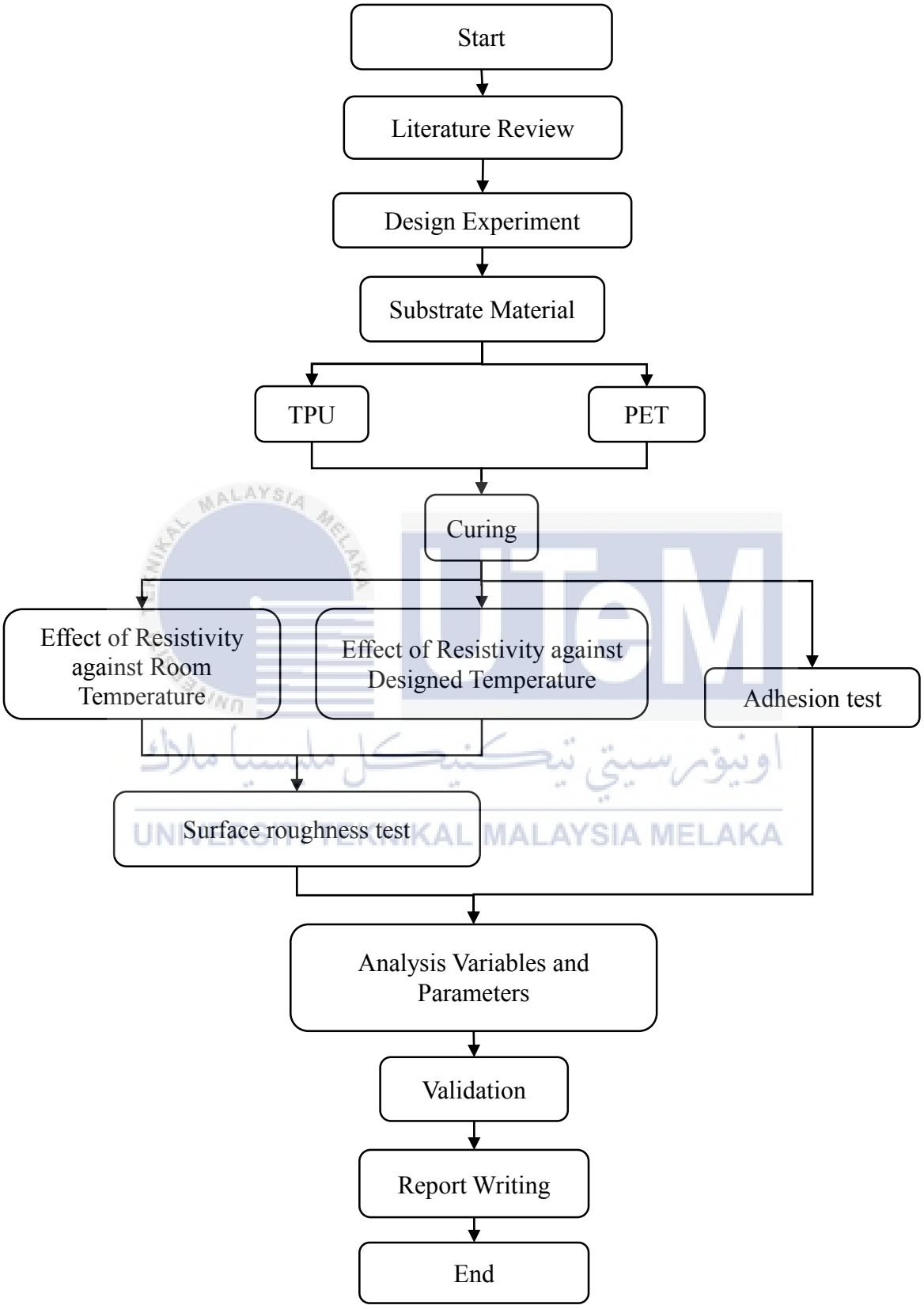

Figure 3.1 General flowchart of methodology

3.2 Sample preparation

Refer to Chapter 2.5 regarding screen printing, there are 4 main components in screen printing which is commercialize carbon ink paste as the conductive ink, tape for the pattern design, substrates used, which is TPU and PET for this research project, and scrapper as the squeegee which for spread the paste through the pattern.

Figure 3.21 Image of (a) scrapper, (b) TPU roll, (c) Bare Conductor conductive ink, and (d) 0.04mm tape

Firstly the TPU substrate is cut in a small size with scissor which is then the TPU substrates were taped on top of the table. Important note before taping it is to make sure that the TPU substrate (clear) side is facing upward and not the substrate cover (pink).

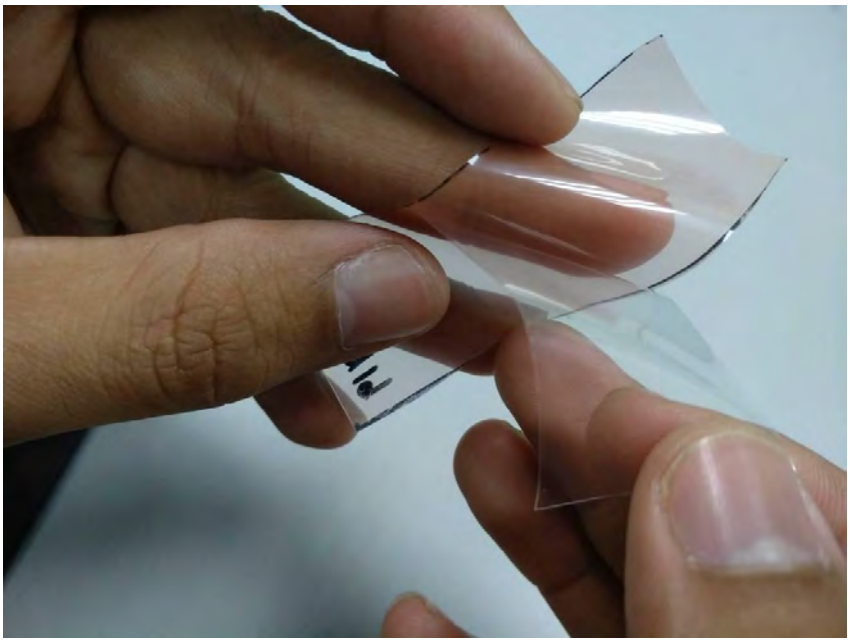

Figure 3.3 TPU (Clear) and TPU cover (pink)

The TPU substrate then taped at the upper side of the substrate which then a 3 mm gap were made using a ruler and marker pen to make a gap for the ink. The marked location is then taped to make a parallel taped line with 3 mm wide for replacement of the stencil's print pattern. Side note of this part is that this part need be measure and tape precisely as it is difficult to paste the tape.

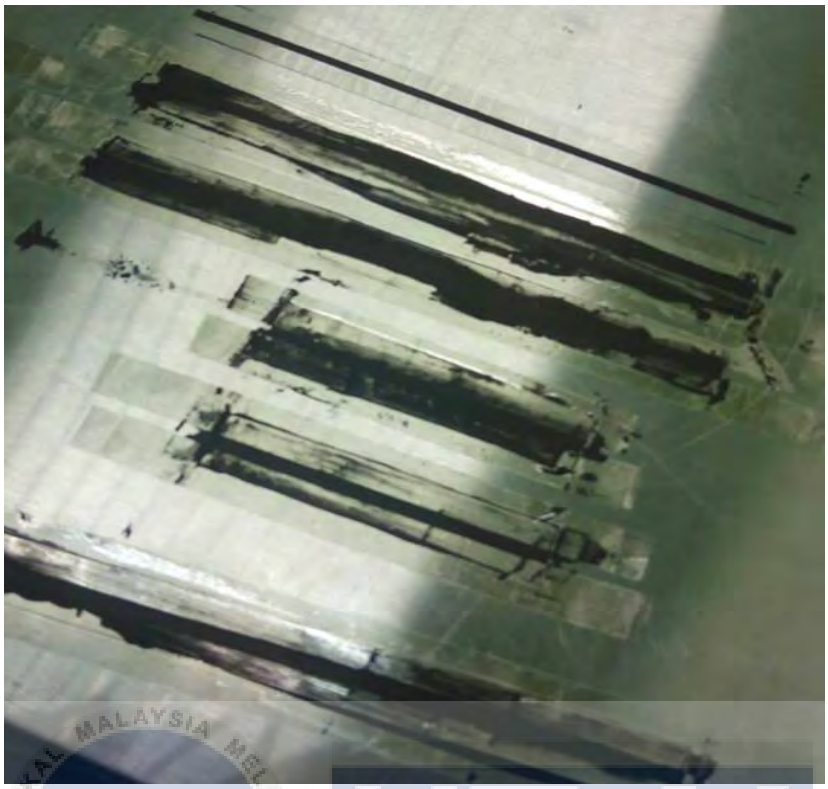

Figure 3.4 Taped TPU substrates for making a sample

By referring the Figure 3.5 and Figure 3.6 below, the test samples were made consistently with the suggested dimension.

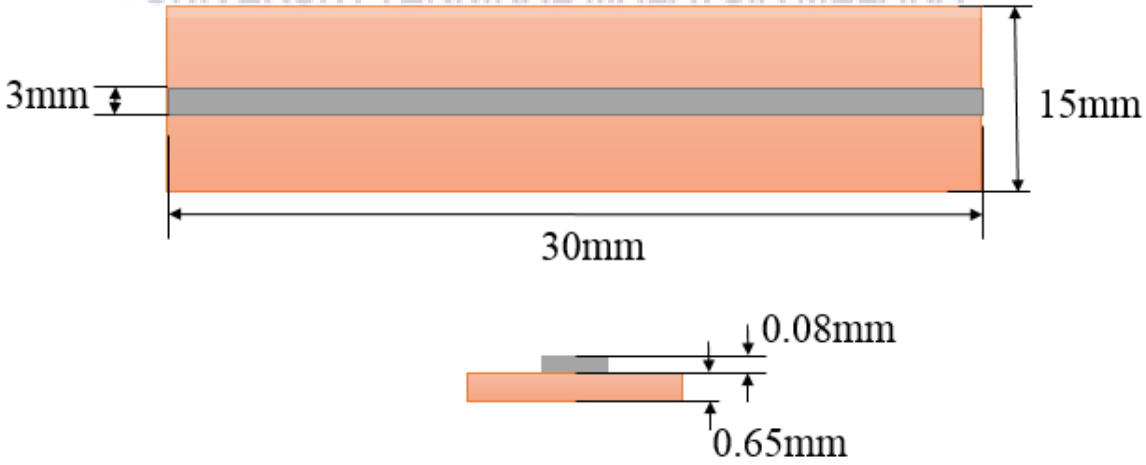

Figure 3.5 Sample dimension

Figure 3.6 Example sample

For this research project, the ink were printed as stated in Figure 3.5 above where the printed area are 3 mm wide and 0.08 mm thick. The wide of 3 mm obtain by making a marked gap using ruler and marker pen. The thickness of 0.08 mm however, obtain by tape the marked point twice. This is because the thickness of the tape used are 0.04 mm.

To print the conductive ink onto the TPU substrate, the conductive ink paste were placed on top of the print pattern using small spoon, which were then spread across the parallel of the pattern line using scrapper. The scrapper must be at consistent angle with low force pulling the conductive ink.

3.3 Curing

For this process, the conductive ink does not need to be cure in the oven. As specified in the technical specification of the commercialize conductive ink, the conductive ink only need to be cure in room temperature for 15 mins. After the conductive ink paste were spread on top of the substrate, the sample were left for 15 mins before removal of the tape.

3.4 Sample labeling

Before any experiment test were conduct to the sample, the sample first need to be label. The sample will be labelled as the dimension shown in Figure 3.7 below. The gap is needed to give a space for the four-point probe needle to be placed. This mean that all four pin will be place within the two line labelled. The 5 sections made are for the 5 location of the pin to be place.

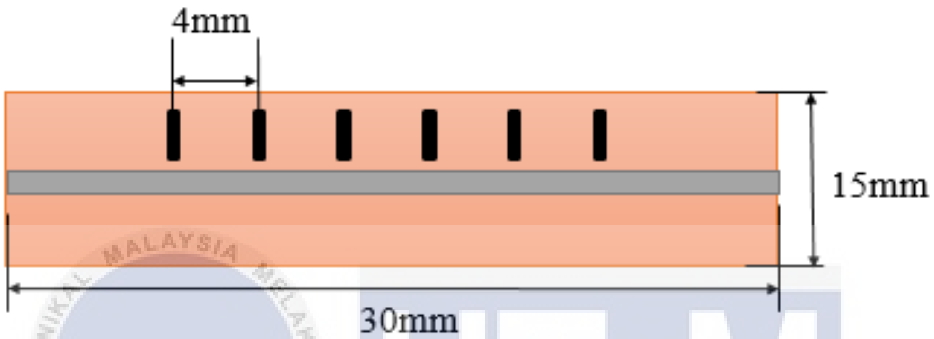

Figure 3.7 Sample labeling

Figure 3.8 Example sample labelled

3.5 Resistivity against temperature experiment setup

In this research project, four-point probe were used as the apparatus to measure resistivity of the carbon conductive ink. Four-point probe are connected to the computer which the data sheet resistance (Ω/sq) value can be obtain through it. In taking the data, the use of four-point probe were referred to a guideline from American Society for Testing and Material (ASTM) which is ASTM F390-98. The four-point probe used is from company model RM3000 test unit.

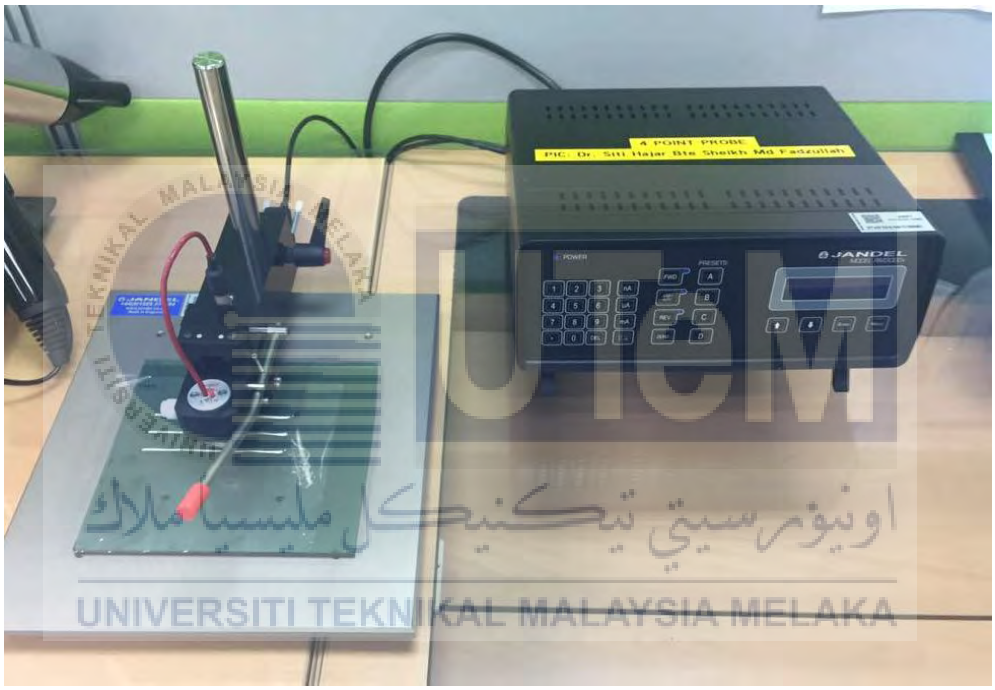

Figure 3.9 Jandel's Four-point probe model RM3000 Test Unit

For the method of taking the data, the four-point probe were slowly positioned above the ink until the value from the machine monitor displayed. The pin from of the four-point probe need to be on top of the ink for the reading to be taken. If there is no reading, the method repeated by lift the pin up and slowly lower it again at the surface of the conductive ink until a reading is obtain. Save the data for every section around 3 times to obtain the average value of each section.

3.5.1 Room temperature against resistivity

As stated in the review obtained in Chapter 2, the resistivity of the conductive ink are believe can be effected by the temperature of the conductive ink itself. Therefore, to prove this the conductive ink need to be tested pre-heated as a datum for the data. In this case, the room temperature (RT) which is 28°C are used as a constant that there are no addition of heat applied onto the substrate and conductive ink.

Figure 3.10 Data taking process for resistivity against room temperature

The data obtain in this experiment will be used as a master data, where the change observation or behavior after any thermal experiment data obtained will be compare to. This experiment were also conducted to observe the physical condition of the substrates before it were heated.

3.5.2 Designed temperature against resistivity

For this experiment, similar with Chapter 3.5.1, the method of taking the data are still be same. However, slight change on this experiment is that the substrate will be heated to different range of temperature which is 40°C, 60°C and 100°C.

Figure 3.11 Heating experiment setup

The tool that were use in heating the substrates into the designed temperature are hair dryer. Since the hair dryer heat generated are uneven which some part are concentrated while some are not, the concentrated heat from the dryer were aimed at the substrates to ensure the are heating happen at the substrates.

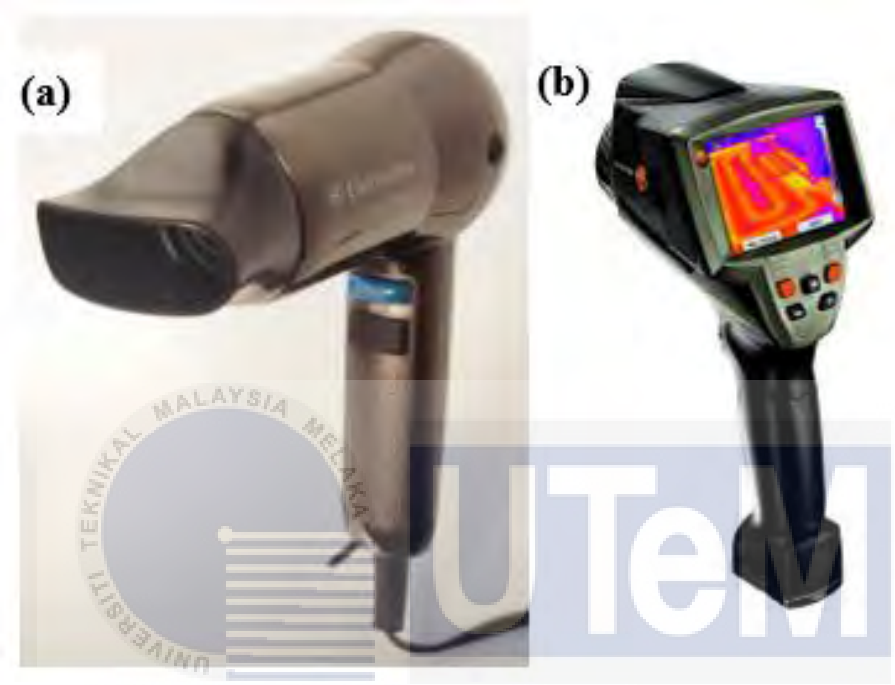

Figure 3.12 Image of (a) hair dryer, and (b) thermal imaging camera

However, thermal imaging camera were used to further ensure that the concentrated heat were aimed at the location that need to be heated which is at the substrates. This is by rendering the infrared radiation of a given space aimed by the thermal imaging camera, it can show the thermographic image of heat of anything aimed at.

3.6 Surface roughness test setup

For surface roughness, the apparatus needed are 3D non-contact profilometer. This apparatus allow the user to check on the roughness of the surface of the substrates in microscopic size. As for guideline, the apparatus were used according to the apparatus manual of operation provided by the company. The brand of the 3D non-profilometer are Shedensha.

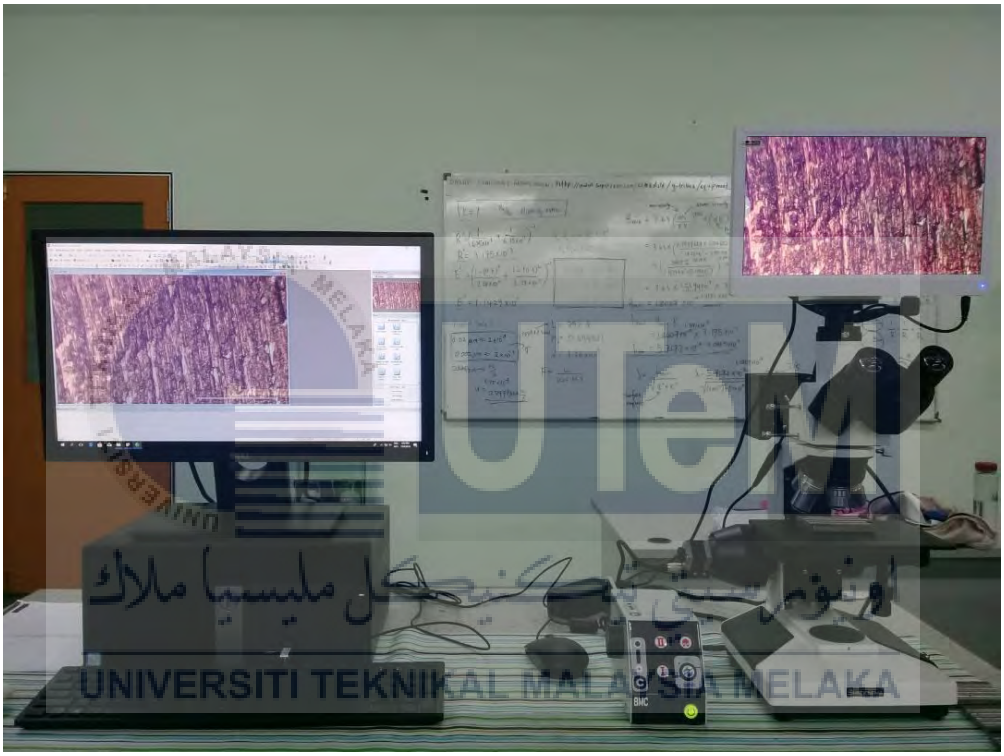

Figure 3.13 Surface roughness test setup

The method of using the apparatus firstly place the substrate on top of glass slide. This is because the small size of the substrate can easily be move using the glass slide. Then the substrates placed underneath the apparatus microscope. The light from the apparatus aimed at the location of the surface of the conductive ink which the location for the research of the conductive ink will be.

From the location aimed at the conductive ink, the image of the surface of the conductive ink will be transfer to the screen of the 3D non-contact profilometer as well as the linked computer. Using the program Winroof, the image from 3D non-contact profilometer can further extract data of 3D cross-section and roughness data of the conductive ink.

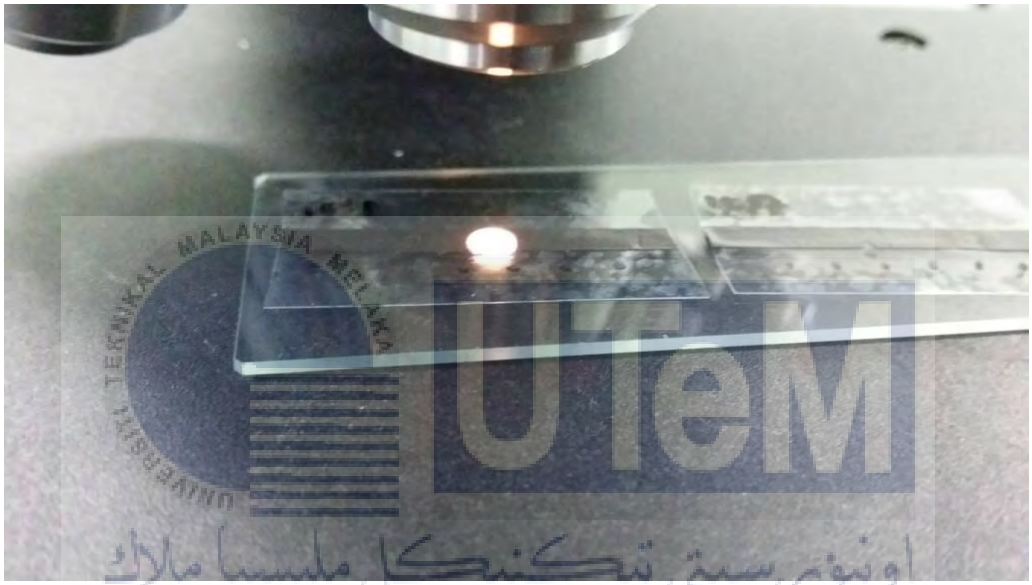

Figure 3.14 Data taking process for surface roughness sample

3.7 Adhesion test setup

For adhesion test, the guideline used are ASTM D3359 where the setup are using test method B which is cross-cut tape test. In the ASTM D3359, there are two test method which test method A is X-cut tape test and test method B are cross-cut test method.

As stated in the guideline, the selection method between these two test methods are that test method A are more suitable to be use at field or job sites while test method B are more suitable to be use in laboratory. Test method B also conditioned that it is more suitable to be use for sample which is thicker than 5 mils (125 µm). Since this test were conduct in laboratory and the sample itself is thicker than 125 µm which is 650 µm.

the apparatus need are few simple tools such as knife, tape, ruler and marker. The marker and ruler used to mark the location that need to be cut. The knife or any cutting tool are used to do the cutting task and tape were used to pull the conductive ink from the substrate. The tape brand are specifically use scotch tape (Pawel & McHargue, 1988).

Figure 3.15 Magic's scotch tape used for adhesion test

New sample were made using the same steps in Chapter 3.2 but with different dimensions. This new sample new dimensions are as Figure 3.16 below.

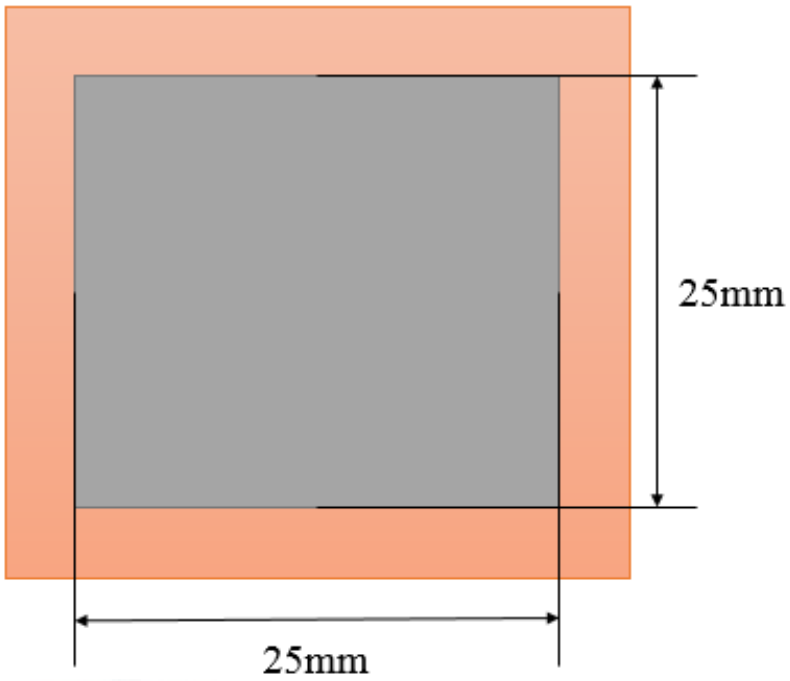

Figure 3.16 Adhesion sample dimension

3.7.1 Room temperature

By following the guideline of ASTM D3359, the conductive ink will be slit into 6 line vertical and 6 line horizontal to obtain 25 small square cut. The total gap between each slit is 20 mm where gap between slit are 4 mm. Marker and ruler were used to mark the position of the slit.

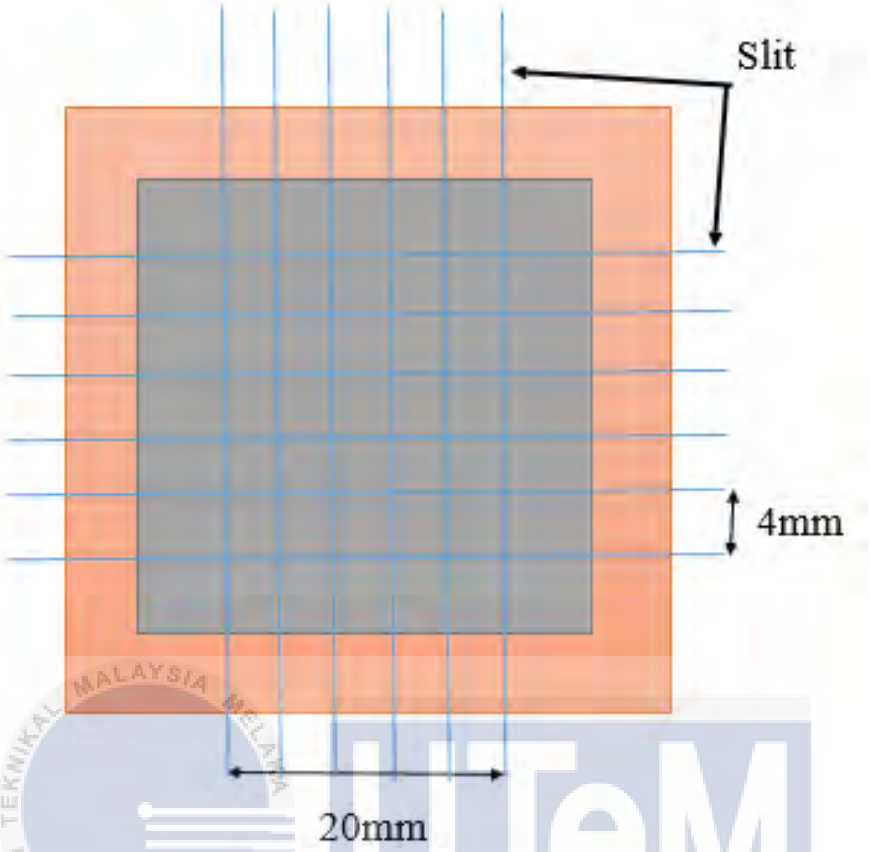

Figure 3.17 12 slit cut for adhesion test samples

Without apply any temperature and working in room temperature, a strip of tape were placed on top of the 25 small square cut. Make sure each square are adhere with the tape by rubbing slowly the tape where the square are adhere to ensure that the tape adhere evenly to all 25 square cut.

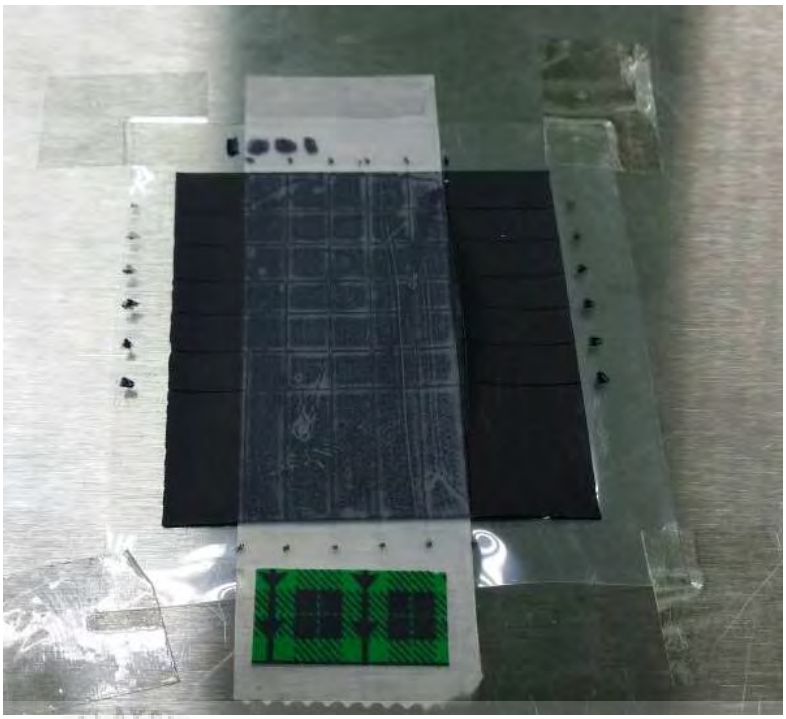

Figure 3.18 Taped sample

Within 90 ± 30 s of tape the 25 square cut area, the tape were removed slowly at angle of as closer to 180° as possible from the tape and pull until the free end of the conductive ink. By observing the amount of square cut of conductive ink still attached to the substrate, the percentage area of conductive ink removed can be obtain. And observation and data obtain from this test will be use to compare with the data and observation obtain when the sample heated

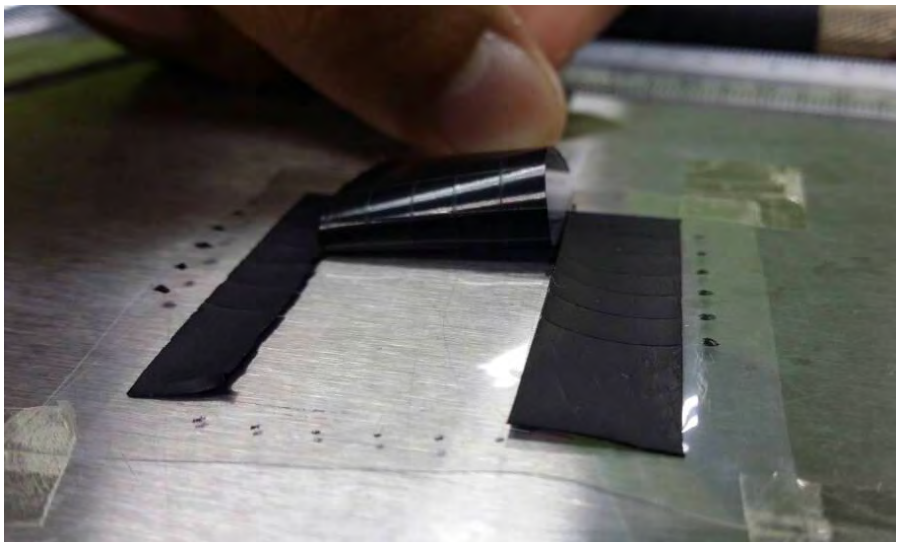

Figure 3.19 Pulling process of adhesion test

3.7.2 Designed temperature

The sample were heated first before conducting the test. The sample heated until 100°C which then proceed with repeating the same procedure in Chapter 3.7.1 which consist of marking, cutting, taping and tape removing. The data then compared with the room temperature data obtained

CHAPTER IV

RESULT AND DISCUSSION

4.1 Overview of research

In this research, the main focus finding that wish to find are the resistivity of the conductive ink under the different range of temperature as well as the reaction of the conductive ink surface roughness and adhesion with the TPU and PET substrate before and after heated with the different range of temperature. Throughout this chapter, the data obtain and any findings which affecting the results for the data obtain will be discuss. This also include assumptions made to justify for the findings.

4.2 Physical observation of conductive ink and substrates

Before start the data taking for resistance, an observation were made for the conductive ink and both of the TPU and PET substrates. This is to see the reaction of all three materials on its changes when the heat applied, and make any hypothesis based on the reaction the materials shows.

4.2.1 Conductive ink

For the conductive ink, the ink at room temperature is more flexible and stretchable together with the substrate. When the conductive ink were heated from room temperature to 40°C, 60°C and 100°C, the conductive ink getting drier as the heat increase causing the ink at 100°C is brittle.

As for the dryness of the conductive ink, the conductive ink is dry after the curing process of the ink when preparing the sample. However, as stated in Chapter 2.6 and Chapter 3.3, curing is a process where the conductive ink is heated to achieve a certain state. The process of heating however is also encouraged by the same process as curing. This cause the conductive ink very dry after heated to 100°C.

Table 4.1 Conductive ink properties after heated

	RT	**40°C**	**60°C**	**100°C**
Conductive ink	Flexy	Lean	Delicate	Brittle
Ink dryness	Dry	Dry	Very dry	Very dry

4.2.2 Thermoplastic polyurethane (TPU) and polyethylene terephthalate (PET) substrates

An observation can be made that the substrates and conductive ink do have physical and resistivity change when it were heated from room temperature to 40°C, 60°C and 100°C. The observation can be simplify as shown in Table 4.2 below.

Table 4.2 TPU substrate properties after heated

	RT	**40°C**	**60°C**	**100°C**
Softness	Rough	Firm	Soft	Silky
Stretchability	Stretchy and retract	Stretchy and retract	Stretchy	Stretchy

Physically the substrates during room temperature are the TPU substrate is stiff and stretchy. When pull, it will stretch and retract back to its original shape quickly. However this properties change as the temperature on the substrate increase. It is slowly swell and slowly lose its retract to the original shape properties. The substrate also getting silkier compare to before heated. By the end of 100°C, the substrate is swell compare to its original shape and the substrate is softer.

Like TPU substrate, PET substrate were also undergo the same experiment of heated to designed temperature which room temperature to 40°C, 60°C and 100°C. There are slight difference between both substrates in term the physical state of the substrates and resistivity of the conductive ink using the substrate.

Table 4.3 PET substrate properties after heated

	RT	**40°C**	**60°C**	**100°C**
Softness	Rigid	Rigid	Rigid	Rigid
Flexibility	Flexible	Flexible	Flexible	Flexible

As shown in Table 4.3 above, in terms of the physical state of the substrate, PET substrate remain almost similar throughout the heating process. The rigidity remain same without any changes. The same goes with the flexibility of the substrate where it can flex normally at room temperature as well after heated in either 40°C, 60°C or 100°C.

To compare the difference between the substrates, firstly is the physical state of the substrates before and after heated. For TPU, it is more stretchable, retractable and flexible before heated. However the TPU substrate changed after heated where it lost its retractable properties and become more silkier. PET however have no obvious changes that can be seen easily. The substrate is flexible and retain it shape throughout the pre and post heating process.

4.3 Resistivity against temperature

The conductive ink contain resistivity to electricity which this research is study on. The rise and drop of resistivity is research in order to identify which situation suit the conductive ink in order to produce the best result of electric conduction.

With the rise of the temperature from room temperature to 40°C, 60°C and 100°C on the conductive ink, the result of the resistivity of the conductive of both TPU and PET substrates can be seen below.

On the resistivity changes of the TPU substrate, the resistivity data result can be seen in Figure 4.1 below. On average, the data resistivity of the conductive ink is around 149.2 Ω/sq at room temperature. This value then decrease with positive slope as the temperature increase by 40°C, 60°C and 100°C.

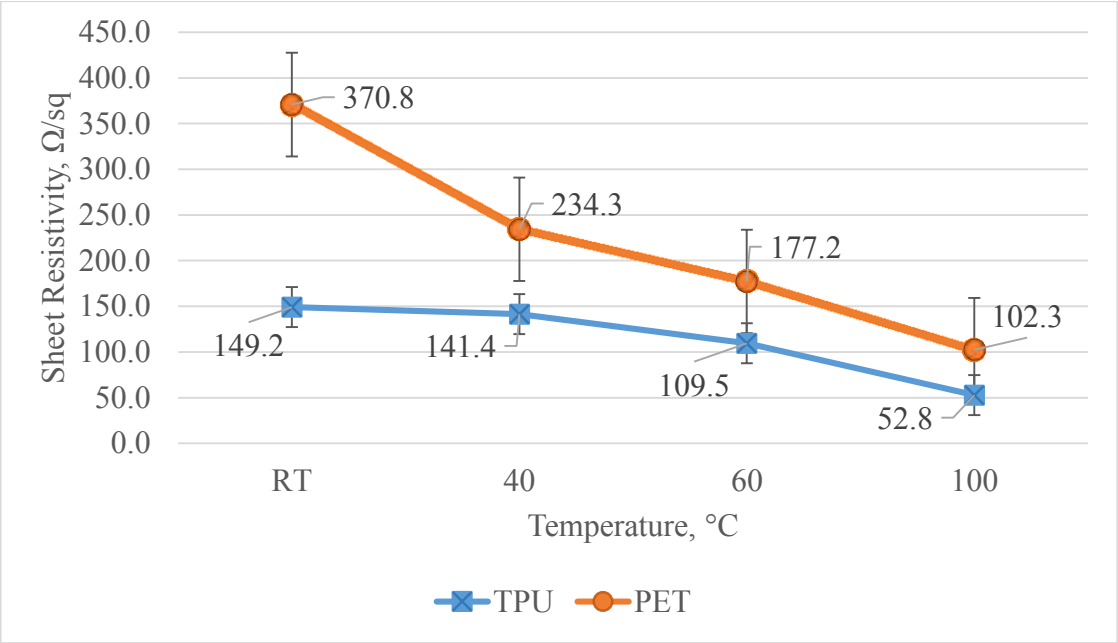

Figure 4.1 TPU and PET sheet resistivity, Ω/sq vs temperature, °C

While the range of average value for room temperature and 40°C is almost similar, it can be conclude that there are small difference between these two temperature which around 10-14°C. A huge difference can be seen in room temperature average value and 100°C where the 100°C is three times smaller than the room temperature average data which around 52.8 Ω/sq.

For the resistivity of PET, the graph are almost linearly negative slope where the resistivity decrease as the temperature increase. The resistivity at room temperature on average is 370.8 Ω/sq. The resistivity however decrease in linear slope where the average resistivity at 100°C are three times lower than average resistivity at room temperature. The average resistivity at 100°C are 102.5 Ω/sq.

To do the comparison between both substrates, the similarities of the conductive ink on both substrates are heated from room temperature to 40°C, 60°C and 100°C respectively. Both substrates also show that the resistivity of the conductive ink decrease as the heat increase by threefold of the room temperature resistivity. We can conclude that the heat can reduce the resistivity of the conductive ink given to any type of substrate.

Also can be compare are that the resistivity of the conductive ink on both substrates where conductive ink on TPU substrates give lesser resistivity compare to conductive ink on PET substrates. On average of room temperature, the resistivity of PET substrates is three times higher compare to TPU substrates.

4.4 Surface roughness test result

Surface roughness refer to the roughness of the surface of a material. In this project, the surface roughness of dried carbon conductive ink on substrates were researched to see the co-relation between the temperature applied on the substrates against the surface roughness of the carbon conductive ink as well as the co-relation of surface roughness with the resistivity of the conductive ink.

By referring Figure 4.2 below, the carbon conductive ink on top of the TPU substrate have a mean roughness for room temperature is 2.958 μm. The surface roughness also decrease as the temperature increase where the highest temperature of 100°C give the lowest amount of the mean roughness at 1.17 μm.

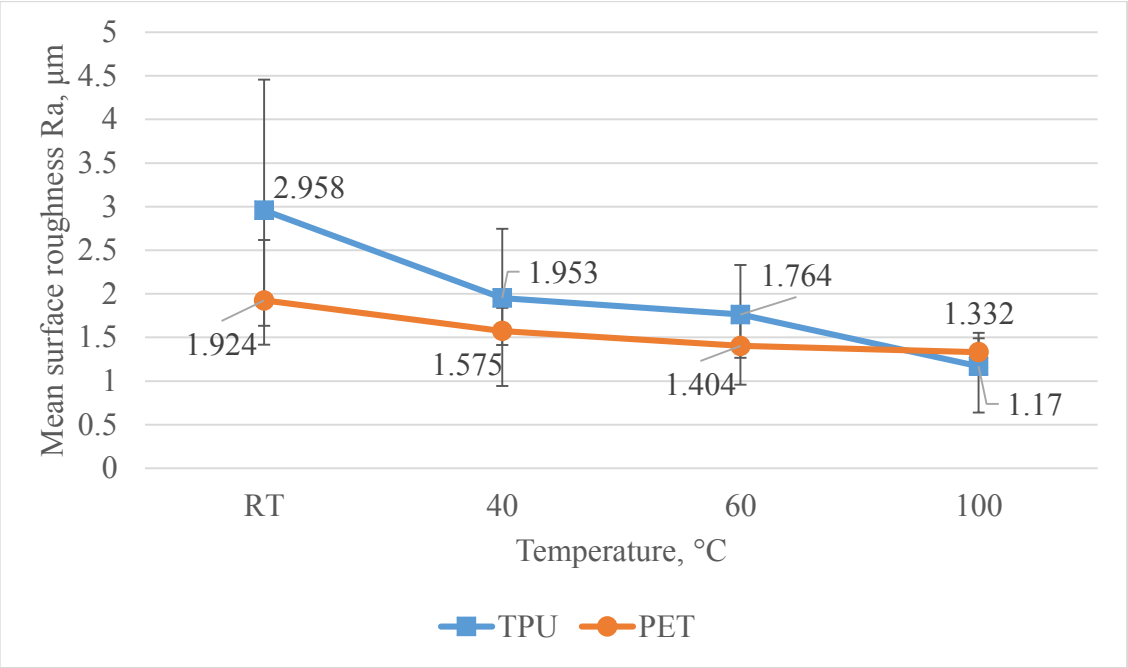

Figure 4.2 TPU and PET mean surface roughness, Ra vs Temperature, °C

For the conductive ink surface roughness on PET substrate, it have lower mean roughness which around 1.92 µm. By referring Figure 4.5 below, like TPU substrate, the mean roughness were also slowly decrease as the temperature increase to 100°C where the mean roughness decrease to 1.332 µm.

The graphical image result for the an average surface roughness of the conductive ink on top of TPU substrates can be seen in Figure 4.3 below where the 3D image of the surface roughness high of the carbon conductive ink can be seen here. If a section of 2D plane image were taken from the front view of the 3D section, it can be seen that the roughness is non-uniform throughout the sample length shows jut how rough is the conductive ink roughness when combine with a certain substrates which in this case the TPU substrates were use. The up and down of the graph were averaged to obtain the mean average roughness of the carbon conductive ink. This mean average roughness is the indicator whether the carbon conductive ink is very adhere or not to other substrates.

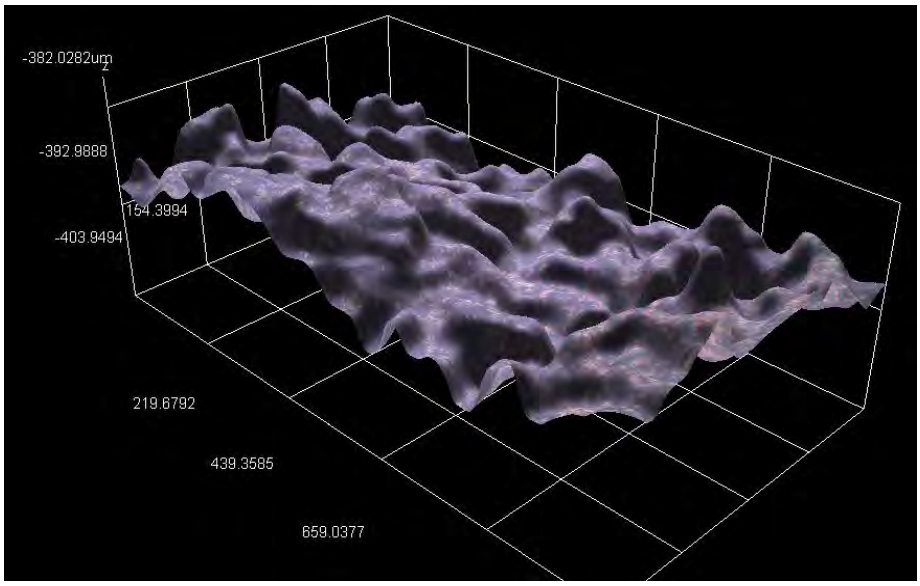

Figure 4.3 Graphical image of TPU sample surface roughness

For the graphical image of the conductive ink on PET substrate samples, on average the carbon conductive ink is more smooth rather that spiky up and down waves. The roughness itself is very smooth and with less cavity or hole or gap between the waves. The smooth roughness of the 3D image of the surface reflect itself in the Figure 3.6 data which the smooth surface will give less the less mean roughness.

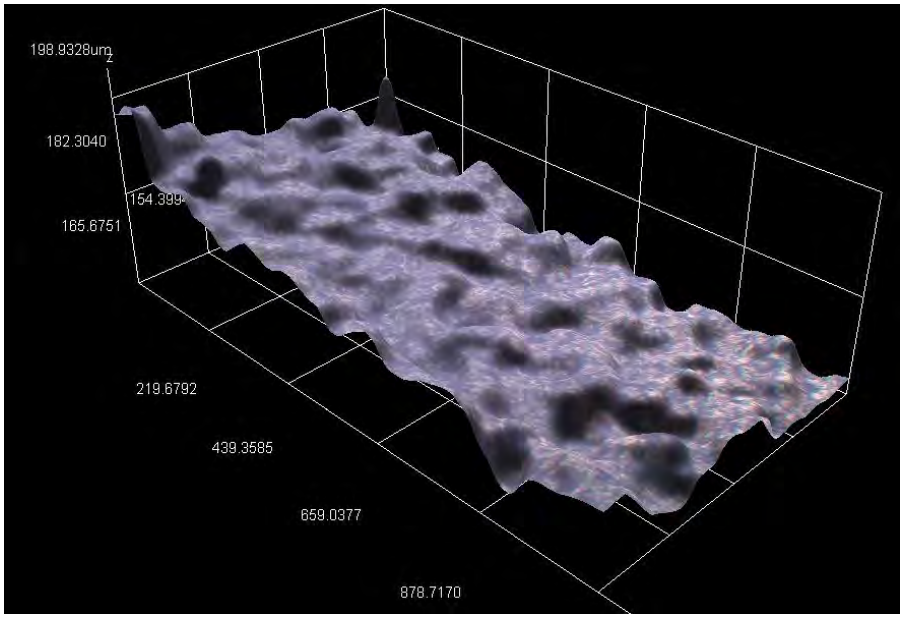

Figure 4.4 Graphical image of PET sample surface roughness

For a comparison of both carbon conductive ink on top of both TPU and PET substrates, firstly is that the TPU have higher mean roughness compare to PET mean roughness which the difference is around 35.05% differences at room temperature.

However for the similarities itself, as shown in Figure 4.5 below where the physically image the carbon conductive itself does not change a lot in terms of its surface roughness when applied to different substrate. Therefore it can be said that the substrates surface roughness is only important for the adhere properties between the conductive ink and the substrates. But this does not change the data obtain for the resistivity change for both substrates.

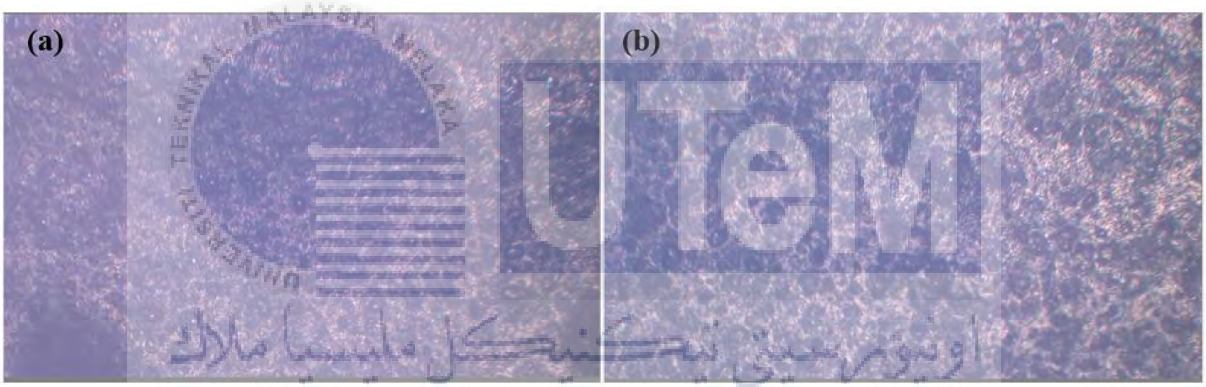

Figure 4.5 Comparison of conductive ink surface roughness on a) TPU substrate and b) PET substrate

In terms of the data obtain, several key points need to be taken into consideration are the usage of the 3D Non-Contact Profilometer itself as it is difficult to take an accurate average data. During the iteration of data taking process for single data, the data taken are sometimes blur even though the process and methods use to take the data are same. As a result, an error occurs once in a while for the graphical image of the carbon conductive ink. Although the problem can be reduce by doing the a repetition of this process, sometimes it

is hard to define whether the data obtain are reliable when the eyes getting blurry from the repetitive process of looking at the screen for the physical image of the conductive ink.

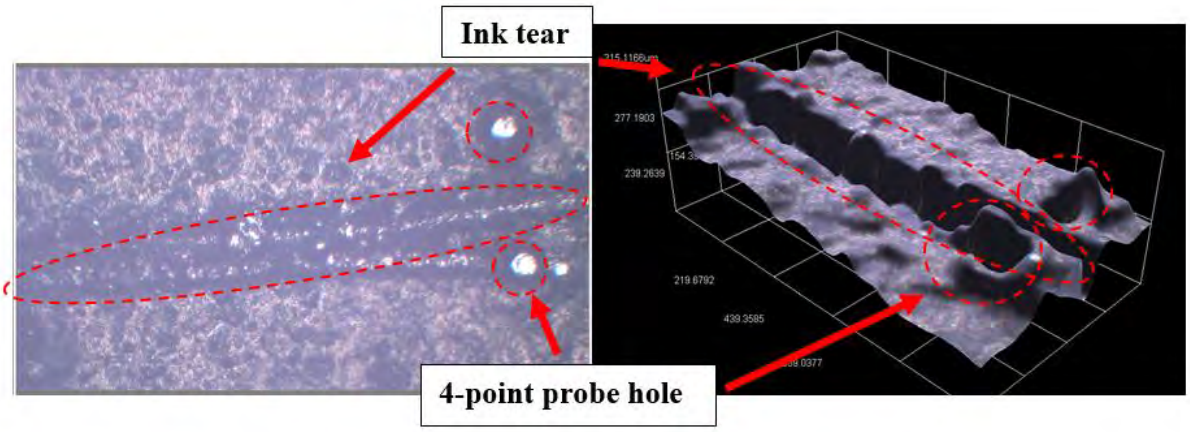

Figure 4.6 Example damage on the conductive ink surface

For another key point that need to be consider are the gap or hole in the conductive ink. This happen due to the tear of the conductive ink when under process such as heating and resistance checking from 4-point probe. As shown in Figure 4.6 above, the mark from using 4-point probe left a circle hole on the surface of the conductive ink. The tear that happen from heating and sometimes from improper printing during the ink printing created as shown as well. This defect on average will not only effect the mean roughness of the conductive ink, it also increase the chance of the conductive ink to peel off from the substrate.

4.5 Adhesion test result

Ink adhesion relate to the ability of the conductive ink to stick to the substrates. For this adhesion test, the TPU and PET samples were tested with test method B which is the cross-cut test method. The number of small square cut remain are multiply with percentage to define its ability to stick to the substrate.

For TPU at room temperature, the result can be seen in Figure 4.7 for room temperature and Figure 4.9 for 100°C. The amount of black column in the cross-cut template are indicate the amount of carbon conductive ink still remain at the substrates after the tape removed from the substrates.

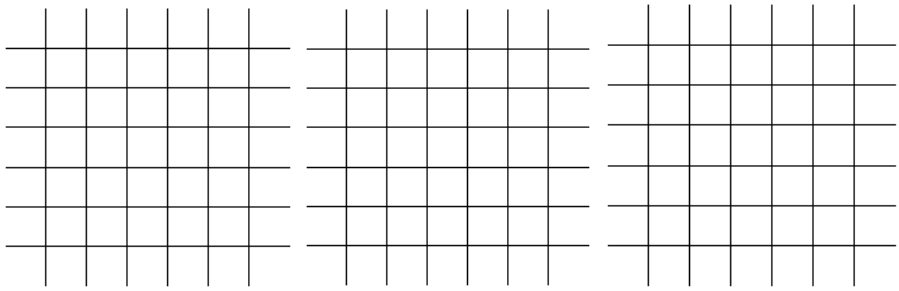

Figure 4.7 Schematic diagram of TPU at room temperature adhesion test

Figure 4.8 Image of TPU sample after tape pulling process for room temperature

For room temperature substrates, the amount of carbon conductive ink remain are zero where the percentage is 0% for all three samples of TPU substrate. The conductive ink instantly removed together with the tape when the tape pulled.

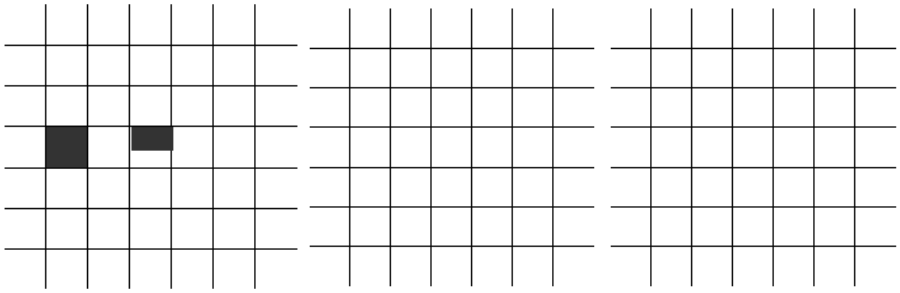

Figure 4.9 Schematic diagram of TPU at 100°C adhesion test

Figure 4.10 Image of TPU sample after tape pulling process for 100°C

However for 100°C, the amount of conductive ink remain are almost similar except for sample no 1. The sample has 6% of conductive ink stick to the substrates. This indicate that after heated to 100°C, there are possibility for the ink to stick better to the TPU substrates due to the properties of TPU substrate itself. However the 6% is still too low to say that the conductive ink is very adhere since the possibility to stick is still to low. Need to take into consideration as well is the brittleness of the conductive ink after heated where the ink start to chipping after heated.

For the conductive ink do stick to the PET substrates nicely. By referring the Figure 4.11 below for room temperature test, the conductive ink on two sample does not peel off with the tape when pulled. On sample 1, around 72% which is can still be consider a good adhesion between carbon conductive ink with PET substrates.

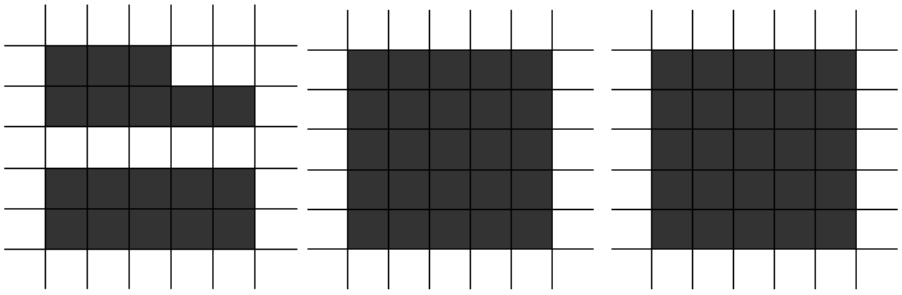

Figure 4.11 Schematic diagram of PET at room temperature adhesion test

However, the result for the 100°C temperature test on the PET substrates are shown in Figure 4.12 below. The three samples result are all different with another. The 1st sample have roughly around 4% of conductive ink remain on the sample. The 2nd sample have 0% where all the conductive ink were peeled off from the substrates. The last sample, the remain conductive ink is around 82% of the total conductive ink.

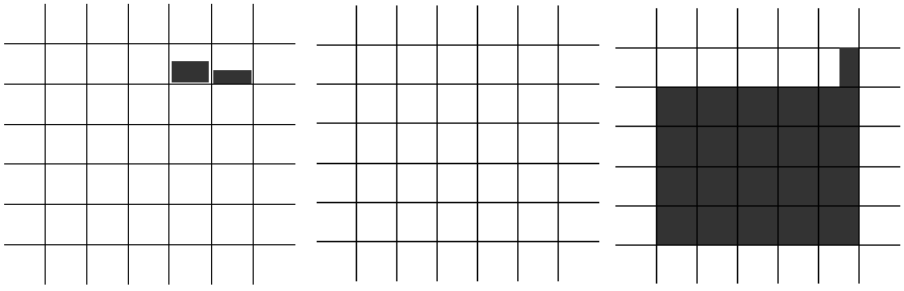

Figure 4.12 Schematic diagram of PET at 100°C adhesion test

From this data in PET substrates, it can be consider that the brittleness and dryness of the conductive ink have effect on the adhesion of the conductive ink with the substrates. Even if the ink still intact like 3rd sample, the ink is too brittle and dry where the conductive ink start to chipping bit by bit.

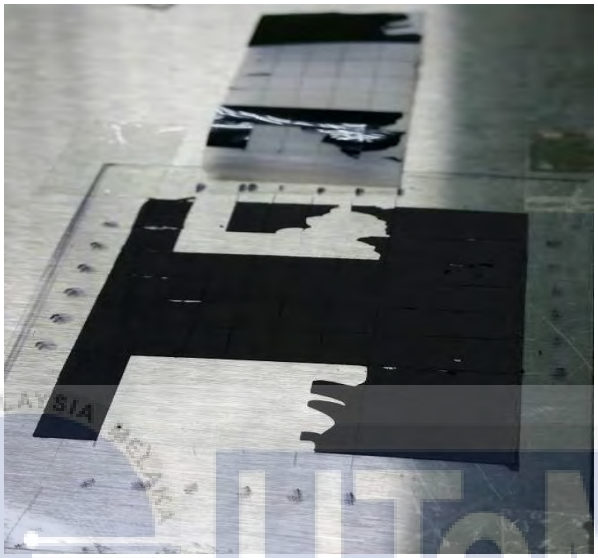

Figure 4.13 Image of PET sample after tape pulling process for 100°C

To discuss on the similarities, the TPU and PET substrates are both undergo the same process and treatment in the same period of time. Unlike the sample for temperature analysis and surface roughness test, the process of making new sample for adhesion test, the process of curing, the process of adhesion test itself as well as the temperature heating to 100°C of both TPU and PET substrates were conduct at the same time. There for the gap of time error during the process such as weather as well as storage period can be reduce.

For comparison, both conductive ink and substrates react as state in Chapter 4.2.1 and Chapter 4.2.2 where the conductive ink become more brittle and dry as the temperature increases while the TPU substrates swell and silkier, and PET substrates remain same as temperature increase.

As for data comparison, the carbon conductive ink can be conclude that it does not have any adhesion capability with TPU substrates. Although there are increase percentage of conductive ink remain when the substrates heated to 100°C, there are possibilities that the remain conductive ink are due to few minor external variables.

For PET substrates, the room temperature samples can be say that the carbon conductive ink can adhere nicely to the substrates. On average, the 2 samples out of 3 still have 100% conductive ink remain adhere while the 1st sample have above 70% of the conductive ink still remain adhere. For the samples which heated to 100°C, the data obtain are inconsistence whether it is can adhere or not. This is due to the data obtain are vary for all three samples. One sample have few conductive ink remain, one have none and one have a lot.

Although this results is inconsistence, it can conclude that the heated conductive ink will lose its adhere. The chipping of the heated conductive ink indicate that the conductive ink might be shrinking from the heat and particle of conductive ink moved from the surface of the substrates. And when there are external force pull or push it such as air-condition breeze, the chipping happen.

4.6 Surface roughness against resistivity and temperature

The three main important variable in this research are the co-relation of the surface roughness, the resistivity of conductive ink and the temperature applied to the samples. To conclude the finding, the co-relation were separate into few sections of comparison.

4.6.1 Surface roughness against resistivity

As shown in Chapter 2.12 and Chapter 4.4, the surface roughness for conductive ink, TPU and PET substrates give of different value of roughness. The surface roughness of the substrates itself will effect the resistivity of the conductive ink along with the conductive ink's resistivity. When the surface roughness is high, more conductive ink can be deposit on top of the substrates which will cause more electric current flow through it.

4.6.2 Surface roughness against temperature

For the surface roughness vs temperature, the temperature will reduce the surface roughness. Theoretically, the heat will slowly trying to melt down the substrate. The rough surface will melt down and became smooth which reduce the substrate surface mean roughness. This can be seen in Chapter 4.4 and Chapter 4.4 which the graph are sloping down when the temperature increase.

4.6.3 Resistivity against temperature

To discuss on the resistivity and temperature, the increment of the temperature heat up the atom in the conductive ink. The particle will then merge thus increase the surface contact between atom. The electric current which flow along the conductive ink will flow much easier when the surface contact is higher.

4.6.4 Overall result

In this research, overall the TPU substrate have higher surface roughness than PET substrate. The resistivity for TPU is also higher than PET which is to be expected. From the overall result as well the temperature increment will cause the resistivity decrease which TPU is still the highest.

4.7 Research finding

In this section, several points need to be discuss as to for any factors that cause any misreading, misleading and failure in the samples.

4.7.1 Stencil

Stencil were used to print the ink paste on top of the substrates by using the screen print method as stated in Chapter 2.6 on information for the method of screen printing. Initially the substrates were printed using stencil instead of tape. However due to a finding on this method lead to change of the screen printing using tape instead.

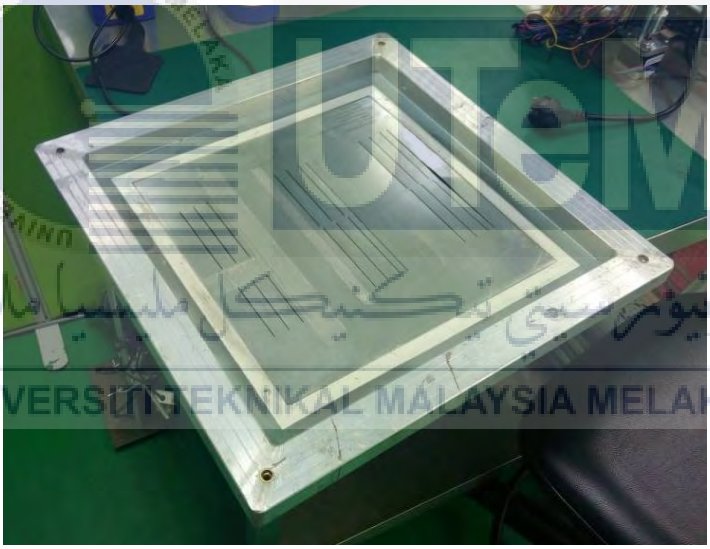

Figure 4.14 Stencil used for print screening process

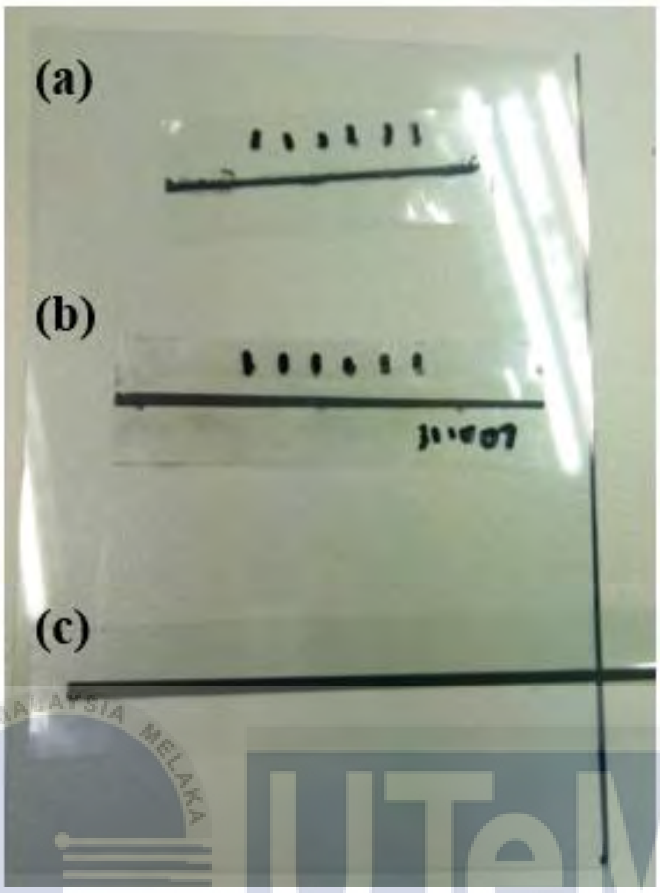

Figure 4.15 Image of test sample of (a) stencils, (b) 1 mm tape and (c) 2 mm tape

As can see in the Figure 4.15, the sample on the left is the sample which is printed using stencil while on the right is the sample which printed using tape. A first observation can be easily see as the sample which were printed using stencil have an inconsistency straight line pattern, while the sample printed using tape have more reliable and eyes pleasing straight line pattern.

This finding can be then seen in the data taken result where the data from sample using stencil does not provide a consistent result. Since the result is inconsistent whether its either no reading obtain from the four-point probe, or the sample will tore first. A counter measure of changing the tools for screen printing method from stencil to tape are due to the observation of many sample from stencil give error compare to tape's sample.

4.7.2 Ink dimension

In this research, initially the width of the ink printed on the substrates were 1 mm as per used by most of the previous research that been made by many researchers. However, the width dimension were change after few attempt on taking the data. A responsive outcome were obtain as the observation of the ink of few samples does not either tore, broke or detach from the substrate.

From the initial of 1 mm, the width of new sample were change to 3 mm which is triple the dimension of the initial sample and the data thickness is increased twice the initial amount which from 0.04mm to 0.08mm. A finding were obtain which show that the thickness and width increment will increase the adhere potential of the conductive ink with the substrates.

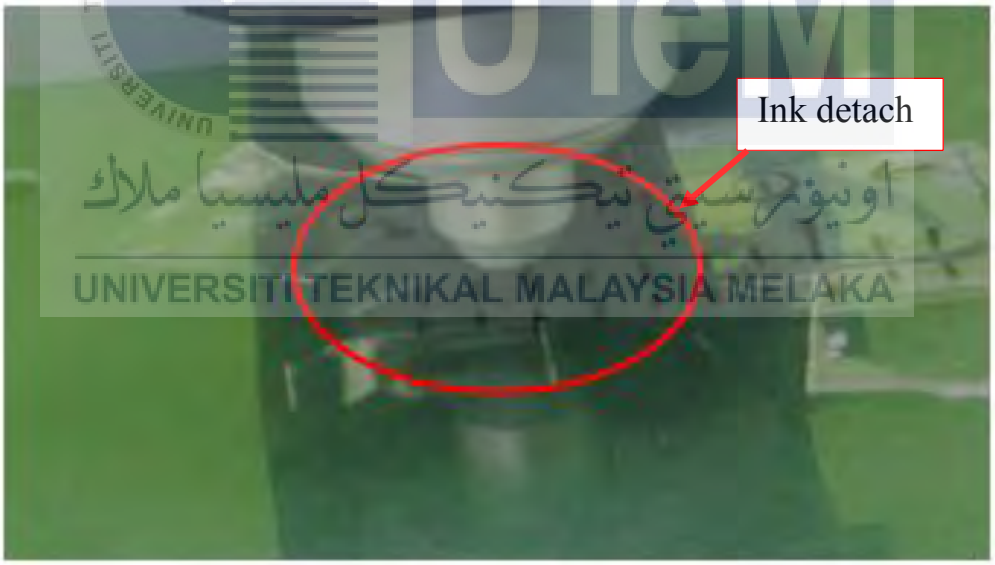

Figure 4.16 Conductive ink detach from the substrate

4.7.3 Ink adhesion

Ink adhesion relate to the ability of the ink to stick to the substrates. In this discussion, the ink adhesion with the width of the ink are to be believe have a relation which affect the failure of the test sample. The ink detach from the substrate relate with the ink adhesion with the substrates itself. Adhesive of the ink relate with the viscosity of the ink and the surface energy of the substrate (Cruz et al., 2016).

Also to be noted is that the adhesion against the width of the ink. It is to be believe that the width of the ink do effect the adhesion. As the ink getting wider, the ink adhere to the surface area of the substrates increase, which is can be summarize to able the ink stick better to the substrate.

Since both ink and substrates were commercialized product, it is hard to state the adhesion potential from both product since some of the information is confidential from the company. However, with enough time and data from test samples, an ideal ratio can be made to give this research project a better results.

CHAPTER V

CONCLUSION & RECOMMENDATION

5.1 Conclusion

In this research for effect of type of substrates to conductive ink under thermal performance, the type of conductive ink play a very important role in this research. The result obtain depend highly on the conductive ink itself whether the flexible electric circuit (FEC) is applicable for use in daily life.

The conductivity rely on the resistance of the electricity flow through the conductive ink. When the resistance is low, the amount of electric flow through will be higher. In this research, the ideal result will be for the resistance to be low without high impact on the substrates and the conductive ink.

For the substrates resistivity between thermoplastic polyurethane (TPU) and polyethylene terephthalate (PET), the overall resistivity of PET is higher than TPU. For the resistivity when temperature applied on the substrates, both substrates shown that the resistivity of the conductive ink reduce as the temperature increase from room temperature to 100°C.

As for the adhesion of the conductive ink on both substrates, it can be conclude that the conductive ink are not really adhere to any of the substrates. Although theoretically there are value of surface roughness of both substrates and conductive ink to say that both of it can adhere with each other, in actual the conductive ink are really unreliable and easily detach from both of the substrates.

Lastly from the physical observation, although TPU substrate can be stretch, when heated it became more stretchy and easy to stretch but cannot retract back to its original shape. This is not desired as it is not useful for repetitive motion or movement. For PET, the substrate does not effect by the increment of temperature. Therefore PET will maintain it shape until higher heat applied which will melt the substrate itself. For the conductive ink, the ink will be more brittle and dry after heated which make it easier to detach or break apart from the substrates which the FEC can be consider fail.

5.2 Recommendation for future works

Flexible electric circuit (FEC) offer many use compare to normal printed circuit board (PCB). It can reach out many new possibilities of uses rather than normal use which no one have ever thought of. However to achieve this new milestones, the FEC need to be tested more in terms of its usability and its applicable in various environment as well as it compatibilities between materials.

One of the main focus that recommended for further study is the material itself where the material for substrates which have various range of surface roughness and conductibility and the material of the conductive ink as well. The combination of 2 new material of substrates and conductive ink can increase the knowledge on the new kind of FEC.

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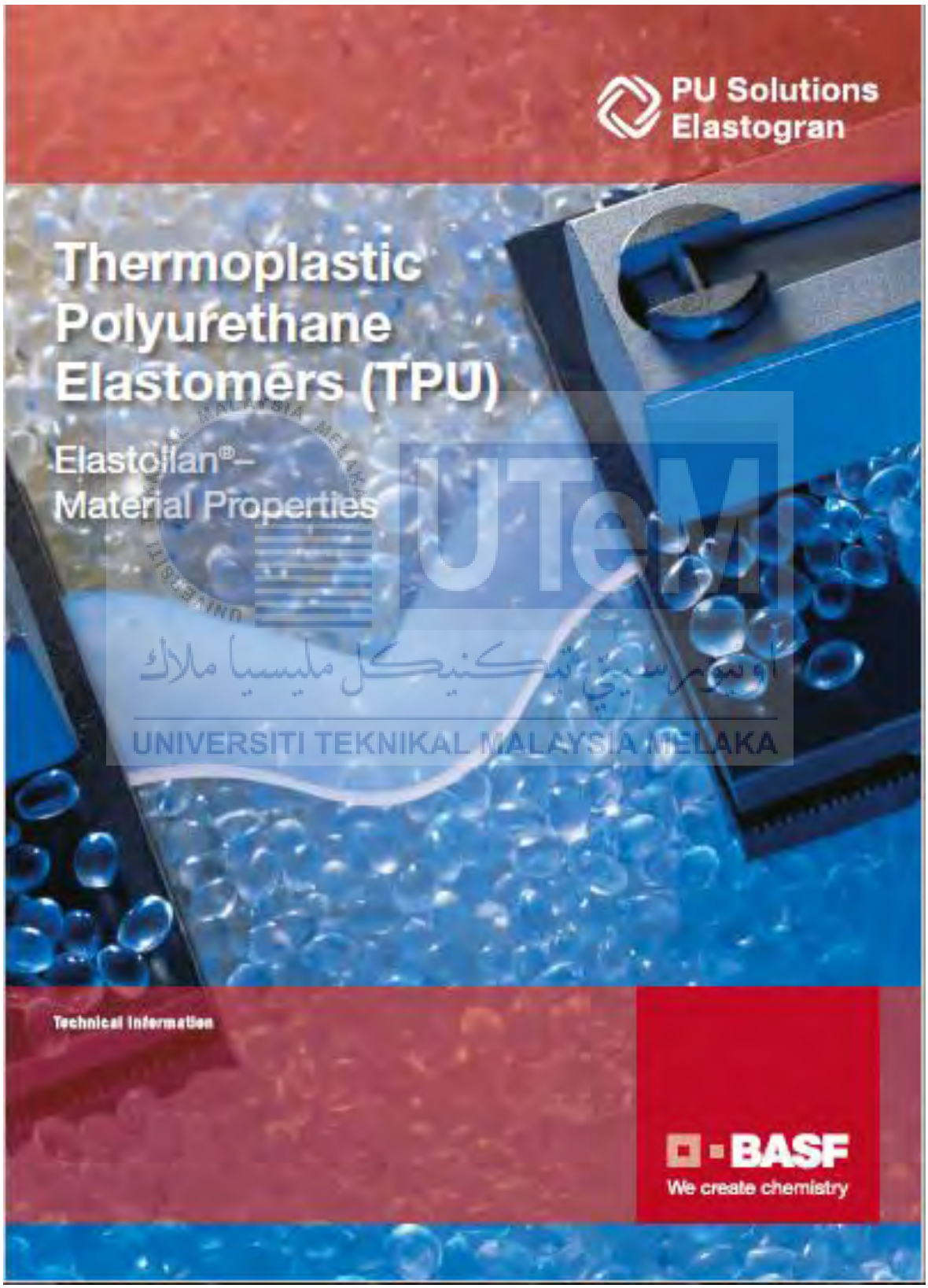
APPENDICES



Chemical structure

Elastollan is essentially formed from the inter-reaction of three components:

1. polyols (long-chain diols)
2. diisocyanates
3. short-chain diols

The polyols and the short-chain diols react with the diisocyanates through polyaddition to form linear polyurethane. Flexible segments are created by the reaction of the polyol with the diisocyanate. The combination of diisocyanate with short-chain diol produces the rigid component (rigid segment). Fig. 1 shows in diagrammatic form the chain structure of thermoplastic polyurethane.

The properties of the product depend on the nature of the raw materials, the reaction conditions, and the ratio of the starting materials. The polyols used have a significant influence on certain properties of the thermoplastic polyurethane.

Either polyester-based polyols (B, C, S, 500 and 600 grades) or polyether-based polyols (1100 grades) are used in the production of Elastollan.

The products are distinguished by the following characteristic features:

Polyester polyol:

- highest mechanical properties
- highest heat resistance
- highest resistance to mineral oils

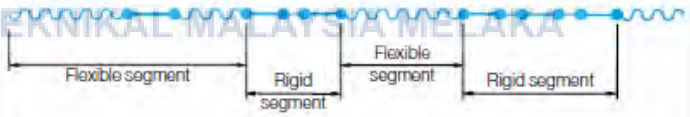
Polyether polyol:

- highest hydrolysis resistance
- best low-temperature flexibility
- resistance to microbiological degradation

In addition to the basic components described above, many Elastollan formulations contain additives to facilitate production and processability. Further additives can also be included to modify specific properties.

Such additives include mould release agents, flame retardants, UV-stabilizers and plasticizers for flexible grades. Glass fibres are used to increase rigidity (RTPU, Elastollan R grades).

Structure of thermoplastic Polyurethane



= Residue of long-chain diol (ether/ester)

= Residue of short-chain diol

= Residue of diisocyanate

= Urethane group

Fig. 1

Designation: D3359 – 09$^{\varepsilon 2}$

Standard Test Methods for Measuring Adhesion by Tape Test[1]

This standard is issued under the fixed designation D3359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Footnote 5 and 5.2 were corrected editorially in June 2010.
ε[2] NOTE—Footnote 5 and 5.2 were corrected editorially and moved into Note 4 in 5.3 in July 2010.

1. Scope*

1.1 These test methods cover procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film.

NOTE 1—This test method has been reported being used to measure adhesion of organic coatings on soft substrates (for example, wood and plastic). Issues with plastic substrates are noted in Appendix X1. A similar test method, ISO 2409, permits tests on soft substrates (for example, wood and plaster). Precision and bias data on the later is lacking. Test Methods D3359 was developed with metal as the substrate and, in the absence of supporting precision and bias data, is so limited.

1.2 Test Method A is primarily intended for use at job sites while Test Method B is more suitable for use in the laboratory. Also, Test Method B is not considered suitable for films thicker than 5 mils (125μm).

NOTE 2—Subject to agreement between the purchaser and the seller, Test Method B can be used for thicker films if wider spaced cuts are employed.

1.3 These test methods are used to establish whether the adhesion of a coating to a substrate is at a generally adequate level. They do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required.

NOTE 3—It should be recognized that differences in adherability of the coating surface can affect the results obtained with coatings having the same inherent adhesion.

1.4 This test method is similar in content (but not technically equivalent) to ISO 2409.

1.5 In multicoat systems adhesion failure may occur between coats so that the adhesion of the coating system to the substrate is not determined.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products

D823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels

D1000 Test Methods for Pressure-Sensitive Adhesive-Coated Tapes Used for Electrical and Electronic Applications

D1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting

D2092 Guide for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting (Withdrawn 2008)[3]

D2370 Test Method for Tensile Properties of Organic Coatings

D3330/D3330M Test Method for Peel Adhesion of Pressure-Sensitive Tape

D3924 Specification for Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials

D4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved June 1, 2009. Published June 2009. Originally approved in 1974. Last previous edition approved in 2008 as D3359 – 08. DOI: 10.1520/D3359-09E02.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

2.2 *Other Standard:*
ISO 2409 Paint and Varnishes — Cross-cut test[4]

3. Summary of Test Methods

3.1 *Test Method A*—An X-cut is made through the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale.

3.2 *Test Method B*—A lattice pattern with either six or eleven cuts in each direction is made in the film to the substrate, pressure-sensitive tape is applied over the lattice and then removed, and adhesion is evaluated by comparison with descriptions and illustrations.

4. Significance and Use

4.1 If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because the substrate and its surface preparation (or lack of it) have a drastic effect on the adhesion of coatings, a method to evaluate adhesion of a coating to different substrates or surface treatments, or of different coatings to the same substrate and treatment, is of considerable usefulness in the industry.

4.2 The limitations of all adhesion methods and the specific limitation of this test method to lower levels of adhesion (see 1.3) should be recognized before using it. The intra- and inter-laboratory precision of this test method is similar to other widely-accepted tests for coated substrates (for example, Test Method D2370 and Test Method D4060), but this is partly the result of it being insensitive to all but large differences in adhesion. The limited scale of 0 to 5 was selected deliberately to avoid a false impression of being sensitive.

TEST METHOD A—X-CUT TAPE TEST

5. Apparatus and Materials

5.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting devices. It is of particular importance that the cutting edges be in good condition.

5.2 *Cutting Guide*—Steel or other hard metal straightedge to ensure straight cuts.

5.3 *Tape*—25-mm (1.0-in.) wide semitransparent pressure-sensitive tape with an adhesion strength agreed upon by the supplier and the user is needed. Because of the variability in adhesion strength from batch-to-batch and with time, it is essential that tape from the same batch be used when tests are to be run in different laboratories. If this is not possible the test method should be used only for ranking a series of test coatings.

NOTE 4—Permacel P99 tape, previously identified as suitable for this purpose, was withdrawn from manufacture in July 2009. Current supplies of Permacel 99 on the market at this time have a shelf life that runs out in July 2010. Subcommittee D01.23 is assessing alternative tapes and a new interlaboratory study is planned to take place in 2010. Alternative tapes with specifications similar to that of Permacel P99 tape are available. Users of alternative tapes should check whether the alternative tapes give comparable results to the Permacel P99 tape. If more information is required about the tapes being evaluated in the D01.23 interlaboratory study, please contact the Committee D01 staff manager.

5.4 *Rubber Eraser,* on the end of a pencil.

5.5 *Illumination*—A light source is helpful in determining whether the cuts have been made through the film to the substrate.

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the adhesion is to be evaluated.

6.2 For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the adhesion.

NOTE 5—Applicable test panel description and surface preparation methods are given in Practice D609 and Practices D1730 and D2092.

NOTE 6—Coatings should be applied in accordance with Practice D823, or as agreed upon between the purchaser and the seller.

NOTE 7—If desired or specified, the coated test panels may be subjected to a preliminary exposure such as water immersion, salt spray, or high humidity before conducting the tape test. The conditions and time of exposure will be governed by ultimate coating use or shall be agreed upon between the purchaser and seller.

7. Procedure

7.1 Select an area free of blemishes and minor surface imperfections. For tests in the field, ensure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

7.1.1 For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

7.2 Make two cuts in the film each about 40 mm (1.5 in.) long that intersect near their middle with a smaller angle of between 30 and 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion.

7.3 Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another X in a different location. Do not attempt to deepen a previous cut as this may affect adhesion along the incision.

7.4 At each day of testing, before initiation of testing, remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

7.5 Place the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of a pencil. The color under the transparent tape is a useful indication of when good contact has been made.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

7.6 Within 90 ± 30 s of application, remove the tape by seizing the free end and pulling it off rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

7.7 Inspect the X-cut area for removal of coating from the substrate or previous coating and rate the adhesion in accordance with the following scale:

5A No peeling or removal,
4A Trace peeling or removal along incisions or at their intersection,
3A Jagged removal along incisions up to 1.6 mm (1/16 in.) on either side,
2A Jagged removal along most of incisions up to 3.2 mm (1/8 in.) on either side,
1A Removal from most of the area of the X under the tape, and
0A Removal beyond the area of the X.

7.8 Repeat the test in two other locations on each test panel. For large structures make sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

7.9 After making several cuts examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone before using again. Discard cutting tools that develop nicks or other defects that tear the film.

8. Report

8.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred that is, between first coat and substrate, between first and second coat, etc.

8.2 *For field tests report the structure or article tested, the* location and the environmental conditions at the time of testing.

8.3 For test panels report the substrate employed, the type of coating, the method of cure, and the environmental conditions at the time of testing.

8.4 If the adhesion strength of the tape has been determined in accordance with Test Methods D1000 or D3330/D3330M, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

8.5 If the test is performed after immersion, report immersion conditions and method of sample preparation.

9. Precision and Bias[5]

9.1 In an interlaboratory study of this test method in which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion, the within-laboratories standard deviation was found to be 0.33 and the between-laboratories 0.44. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than 1 rating unit for two measurements.

9.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 1.5 rating units.

9.2 Bias cannot be established for these test methods.

TEST METHOD B—CROSS-CUT TAPE TEST

10. Apparatus and Materials

10.1 *Cutting Tool*[6]—Sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between 15 and 30° that will make either a single cut or several cuts at once. It is of particular importance that the cutting edge or edges be in good condition.

10.2 *Cutting Guide*—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straightedge or template to ensure straight cuts.

10.3 *Rule*—Tempered steel rule graduated in 0.5 mm for measuring individual cuts.

10.4 *Tape*, as described in 5.3.

10.5 *Rubber Eraser*, on the end of a pencil.

10.6 *Illumination*, as described in 5.5.

10.7 *Magnifying Glass*—An illuminated magnifier to be used while making individual cuts and examining the test area.

11. Test Specimens

11.1 Test specimens shall be as described in Section 6. It should be noted, however, that multitip cutters[7] provide good results only on test areas sufficiently plane that all cutting edges contact the substrate to the same degree. Check for flatness with a straight edge such as that of the tempered steel rule (10.3).

12. Procedure

12.1 Where required or when agreed upon, subject the specimens to a preliminary test before conducting the tape test (see Note 5). After drying or testing the coating, conduct the tape test at room temperature as defined in Specification D3924, unless D3924 standard temperature is required or agreed.

12.1.1 For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

12.2 Select an area free of blemishes and minor surface imperfections, place on a firm base, and under the illuminated magnifier, make parallel cuts as follows:

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1008. Contact ASTM Customer Service at service@astm.org.

[6] Multiblade cutters are available from a few sources that specialize in testing equipment for the paint industry.

[7] The sole source of supply of the multitip cutter for coated pipe surfaces known to the committee at this time is Paul N. Gardner Co., 316 NE First St., Pompano Beach, FL 33060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

12.2.1 For coatings having a dry film thickness up to and including 2.0 mils (50 µm) space the cuts 1 mm apart and make eleven cuts unless otherwise agreed upon.

12.2.2 For coatings having a dry film thickness between 2.0 mils (50 µm) and 5 mils (125 µm), space the cuts 2 mm apart and make six cuts. For films thicker than 5 mils (125 µm), use Test Method A.[8]

12.2.3 Make all cuts about 20 mm (¾ in.) long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area.

12.3 After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings.

12.4 Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts.

12.5 Brush the area as before and inspect the incisions for reflection of light from the substrate. If the metal has not been reached make another grid in a different location.

12.6 At each day of testing, before initiation of testing, remove two complete laps of tape and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

12.7 Place the center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser on the end of a pencil. The color under the tape is a useful indication of when good contact has been made.

12.8 Within 90 ± 30 s of application, remove the tape by seizing the free end and rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

12.9 Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. Rate the adhesion in accordance with the following scale illustrated in Fig. 1:

5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
4B Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
0B Flaking and detachment worse than Grade 1.

12.10 Repeat the test in two other locations on each test panel.

[8] Test Method B has been used successfully by some people on coatings greater than 5 mils (0.13 mm) by spacing the cuts 5 mm apart. However, the precision values given in 14.1 do not apply as they are based on coatings less than 5 mils (0.13 mm) in thickness.

CLASSIFICATION OF ADHESION TEST RESULTS

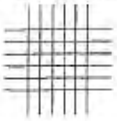
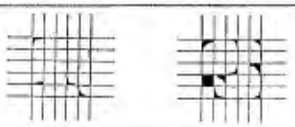
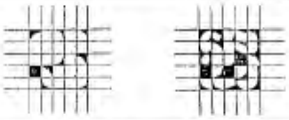
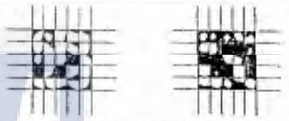
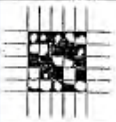
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

13. Report

13.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred, that is, between first coat and substrate, between first and second coat, etc.

13.2 Report the substrate employed, the type of coating and the method of cure.

13.3 If the adhesion strength has been determined in accordance with Test Methods D1000 or D3330/D3330M, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

13.4 If the test is performed after immersion, report immersion conditions and method of sample preparation.

14. Precision and Bias[5]

14.1 On the basis of two interlaboratory tests of this test method in one of which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion and in the other operators in six laboratories made three measurements on two panels each of four different coatings applied over two other coatings, the

pooled standard deviations for within- and between-laboratories were found to be 0.37 and 0.7. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than one rating unit for two measurements.

14.1.2 *Reproducibility*—Two results, each the mean of duplicates or triplicates, obtained by different operators should be considered suspect if they differ by more than two rating units.

14.2 Bias cannot be established for these test methods.

15. Keywords

15.1 adhesion; crosscut adhesion test method; tape; tape adhesion test method; X-cut adhesion test method

APPENDIX

(Nonmandatory Information)

X1. COMMENTARY

X1.1 Introduction

X1.1.1 Given the complexities of the adhesion process, can adhesion be measured? As Mittal (1)[9] has pointed out, the answer is both yes and no. It is reasonable to state that at the present time no test exists that can precisely assess the actual physical strength of an adhesive bond. But it can also be said that it is possible to obtain an indication of relative adhesion performance.

X1.1.2 Practical adhesion test methods are generally of two types: "*implied*" and "*direct*." "Implied" tests include indentation or scribe techniques, rub testing, and wear testing. Criticism of these tests arises when they are used to quantify the strength of adhesive bonding. But this, in fact, is not their purpose. An "implied" test should be used to assess coating performance under actual service conditions. "Direct" measurements, on the other hand, are intended expressly to measure adhesion. Meaningful tests of this type are highly sought after, primarily because the results are expressed by a single discrete quantity, the force required to rupture the coating/substrate bond under prescribed conditions. Direct tests include the Hesiometer and the Adherometer (2). Common methods which approach the direct tests are peel, lap-shear, and tensile tests.

X1.2 Test Methods

X1.2.1 In practice, numerous types of tests have been used to attempt to evaluate adhesion by inducing bond rupture by different modes. Criteria deemed essential for a test to warrant large-scale acceptance are: use of a straightforward and unambiguous procedure; relevance to its intended application; repeatability and reproducibility; and quantifiability, including a meaningful rating scale for assessing performance.

X1.2.2 Test methods used for coatings on metals are: peel adhesion or "tape testing;" Gardner impact flexibility testing; and adhesive joint testing including shear (lap joint) and direct tensile (butt joint) testing. These tests do not strictly meet all the criteria listed, but an appealing aspect of these tests is that in most cases the equipment/instrumentation is readily available or can be obtained at reasonable cost.

X1.2.3 A wide diversity of tests methods have been developed over the years that measure aspects of adhesion (1-5). There generally is difficulty, however, in relating these tests to basic adhesion phenomena.

X1.3 The Tape Test

X1.3.1 By far the most prevalent test for evaluating coating "adhesion" is the tape-and-peel test, which has been used since the 1930's. In its simplest version a piece of adhesive tape is pressed against the paint film and the resistance to and degree of film removal observed when the tape is pulled off. Since an intact film with appreciable adhesion is frequently not removed at all, the severity of the test is usually enhanced by cutting into the film a figure *X* or a cross hatched pattern, before applying and removing the tape. Adhesion is then rated by comparing film removed against an established rating scale. If an intact film is peeled cleanly by the tape, or if it debonds just by cutting into it without applying tape, then the adhesion is rated simply as poor or very poor, a more precise evaluation of such films not being within the capability of this test.

X1.3.2 The current widely-used version was first published in 1974; two test methods are covered in this standard. Both test methods are used to establish whether the adhesion of a coating to a substrate is at an adequate level; however they do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required. Major limitations of the tape test are its low sensitivity, applicability only to coatings of relatively low bond strengths, and non-determination of adhesion to the substrate where failure occurs within a single coat, as when testing primers alone, or within or between coats in multicoat systems. For multicoat systems where adhesion failure may occur between or within coats, the adhesion of the coating system to the substrate is not determined.

X1.3.3 Repeatability within one rating unit is generally observed for coatings on metals for both methods, with reproducibility of one to two units. The tape test enjoys widespread popularity and is viewed as "simple" as well as low

[9] The boldface numbers in parentheses refer to the list of references at the end of this test method.

ELECTRIC PAINT®

Technical Data Sheet

PRODUCT DESCRIPTION

Electric Paint is a nontoxic, water based, water soluble, electrically conductive paint. It can be used in circuits as a painted resistor element, a capacitive electrode or can function as a conductor in designs that can tolerate high resistivity. It is intended for applications with circuits using low DC voltages at low currents. **Electric Paint** adheres to a wide variety of substrates and can be applied using screen printing equipment. Its major benefits include low cost, solubility in water and good screen life. It is black in colour and can be over-painted with any material compatible with a water-based paint.

ADVANTAGES / PRODUCT BENEFITS

- High resistivity
- Nontoxic
- Water-soluble
- Can be used to create capacitive touch and proximity sensors
- Can be used as a potentiometer or resistive circuit element
- Compatible with many standard printing processes
- Low cost

TYPICAL PROPERTIES

Colour /	Black
Viscosity /	Highly viscous and shear sensitive (thixotropic)
Density /	1.16 g/ml
Surface Resistivity /	55 Ω/Sq/50 microns
Vehicle /	Water-based
Drying Temperature /	**Electric Paint** should be allowed to dry at room temperature for 5 – 15 minutes. Drying time can be reduced by placing Electric Paint under a warm lamp or other low intensity heat source.

See below summary table of typical properties.

PROCESSING AND HANDLING

Screen Printing Equipment /	Manual
Screen Types /	Polyester, stainless steel (43T – 90T gauge mesh)
Typical Cure Conditions /	Room temperature (24°C) for 15 minutes
Typical Circuit Line Width /	0.5 – 10mm (43T-mesh stainless steel screen)
Clean-up Solvent /	Warm water and soap
Surface Resistivity /	32Ω/Sq when using a brush or manual screen printing
Shelf Life /	6 months after opening
Storage /	**Electric Paint** should be stored, tightly sealed in a clean, stable environment at room temperature. Composition should be thoroughly mixed prior to use.

See below graph to predict resistance using manual screen printing.

Bare Conductive Ltd
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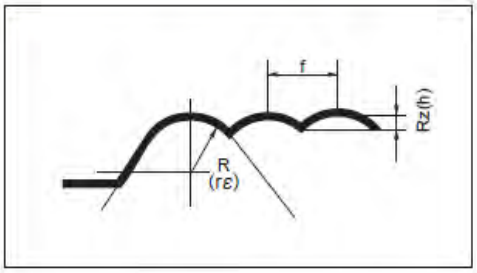
Surface Roughness (JIS B 0601-2001)

Theoretical (Geometrical) Surface Roughness

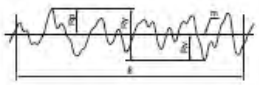
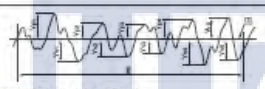
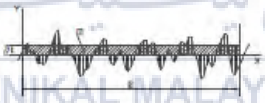
Theoretical Surface Roughness at Turning indicates the minimum roughness value from the cutting conditions and it is shown by the formula as follows:

$$Rz(h) = \frac{f^2}{8R(r\varepsilon)} \times 10^3$$

Rz(h) : Theoretical Surface Roughness (μm)
f : Feed Rate (mm/rev)
R(rε) : Corner Radius of Insert (mm)



How to Obtain Surface Roughness Values

Type	Symbol	How to Obtain	Explanation
Max. Height Roughness	Rz	Ry is obtained from the distance in micron meter between the highest peak and the lowest valley in the range of sampled reference length (ℓ) to the direction of mean line of the roughness curve. Rz=Rp+Rv	
Ten Points Mean Roughness	RzJIS	Rz is obtained from the total in micron meter of the mean value of the each distance between the mean line and 5 peaks (Yp) from the highest one, and the mean value of the each distance between the mean line and the 5 valleys (Yv) from the lowest one, of the roughness curve in the range of sampled reference length "ℓ". $RzJIS = \frac{(Yp1+Yp2+Yp3+Yp4+Yp5)+(Yv1+Yv2+Yv3+Yv4+Yv5)}{5}$	 *Yp1, Yp2, Yp3, Yp4, Yp5:* Distance from the mean line to highest 5 peaks in the range of sampled reference length "ℓ" *Yv1, Yv2, Yv3, Yv4, Yv5:* Distance from the mean line to the lowest 5 valleys in the range of sampled reference length "ℓ"
Arithmetical Mean Roughness	Ra	Ra is obtained from the following formula in micron meter when the roughness curve is expressed by y=f(x), taking X-axis to the mean line direction and Y-axis to the vertical magnification of the roughness curve in the range of sampled reference length "ℓ". $Ra = \frac{1}{\ell}\int_0^{\ell} \lvert f(x) \rvert dx$	

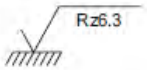
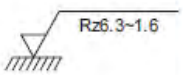
Relationship with Triangle Symbol

Arithmetical Mean Roughness Ra(μm)	Max. Height Roughness Rz(μm)	Ten Points Mean Roughness RzJIS(μm)	Note: (Relationship with Triangle)
0.025 0.05 0.1 0.2	0.1 0.2 0.4 0.8	0.1 0.2 0.4 0.8	▽▽▽▽
0.4 0.8 1.6	1.6 3.2 6.3	1.6 3.2 6.3	▽▽▽
3.2 6.3	12.5 25	12.5 25	▽▽
12.5 25	50 100	50 100	▽

Note: Finishing symbol (Triangle ▽and wave~) was abolished from JIS standard from 1994 Revision.

· How to Indicate
Example
①When Ra is 1.6μm→1.6μmRa
②When Rz is 6.3μm→6.3μmRz
③When RzJIS is 6.3μm→6.3μmRzJIS

Indication in JIS Standard

Example of Ra Indication		Example of Ry, (Rz) Indication	
①When indicating the upper limit only (when upper limit is 6.3μmRa)		①When indicating upper limit onlyIndicate surface roughness following the parameter symbol.	
②When indicating both lower and upper limit (when upper limit is 6.3μmRa, lower limit is 1.6μmRa)		②When indicating both lower and upper limit Indicate surface roughness as (upper limit ~ lower limit) following the parameter symbol.	

Note: The indications of Ra and Rz are different.

Surface Roughness Symbol Caution

The above information is based on JIS B 0601-2001.
However, some symbols were revised as shown in the right table in accordance with ISO Standard from JIS B 0601-2001 version.
Ten Points Mean Roughness (Rz) was eliminated from 2001 version but it still remains as RzJIS reference, since it was popular in Japan.

Type	Symbol of JIS B 0601-1994		Symbol of JIS B 0601-2001
Max. Height Roughness	Ry	→	Rz
Ten Points Mean Roughness	Rz	→	(RzJIS)
Arithmetical Mean Roughness	Ra	→	Ra

Designation: F 390 – 98 (Reapproved 2003)

Standard Test Method for Sheet Resistance of Thin Metallic Films With a Collinear Four-Probe Array[1]

This standard is issued under the fixed designation F 390; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the sheet resistance of metallic thin films with a collinear four-probe array. It is intended for use with rectangular metallic films between 0.01 and 100 μm thick, formed by deposition of a material or by a thinning process and supported by an insulating substrate, in the sheet resistance range from 10^{-2} to $10^{4}\Omega/\square$ (see 3.1.3).

1.2 This test method is suitable for referee measurement purposes as well as for routine acceptance measurements.

1.3 The values stated in Si units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 1 Specification for ASTM Thermometers

F 388 Method for Measurement of Oxide Thickness on Silicon Wafers and Metallization Thickness by Multiple-Beam Interference (Tolansky Method)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *thin film*—a film having a thickness much smaller than any lateral dimension, formed by deposition of a material or by a thinning process.

3.1.2 *thin metallic film*—a thin film composed of a material or materials with resistivity in the range from 10^{-8} to $10^{-3}\Omega\cdot$cm.

3.1.3 *sheet resistance, R_s—in a thin film, the ratio of the potential gradient parallel to the current to the product of the current density and the film thickness; in a rectangular thin film, the quotient of the resistance, measured along the length of the film, divided by the length, l, to width, w, ratio. The ratio l/w is the number of squares.*

4. Summary of Test Method

4.1 A collinear four-probe array is used to determine the sheet resistance by passing a measured direct current through the specimen between the outer probes and measuring the resulting potential difference between the inner probes. The sheet resistance is calculated from the measured current and potential values using correction factors associated with the geometry of the specimen and the probe spacing.

4.2 This test method includes procedures for checking both the probe assembly and the electrical measuring apparatus.

4.2.1 The spacings between the four probe tips are determined from measurements of indentations made by the tips in a suitable surface. This test also is used to determine the condition of the tips.

4.2.2 The accuracy of the electrical measuring equipment is tested by means of an analog circuit containing a known standard resistor together with other resistors which simulate the resistance at the contacts between the probe tips and the film surface.

5. Apparatus

5.1 *Probe Assembly*:

5.1.1 *Probes*—The probe shaft and tip shall be constructed of tungsten carbide, Monel, hardened tool steel, or hard copper and have a conical tip with included angle of 45 to 90°. Alternatively, the tip may be formed from a platinum-palladium alloy and resistance welded to the shaft. The tip shall have a nominal initial radius of 25 to 50 μm. In all cases all of the four paths from the electrical measurement equipment inputs to the film surface must be identical.

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputtered Thin Films.

Current edition approved May 10, 1998. Published October 1998. Originally published as F 390 – 73 T. Last previous edition F 390 – 97.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Discontinued; see *1992 Annual Book of ASTM Standards*, Vol 10.05.

5.1.2 *Probe Force*—The probes shall be uniformly loaded to exert a force sufficient to deform the metal film but insufficient to puncture the film. A rough guide for loading is a load of 20 g/Mohs (unit of hardness) of the film material on each probe.

5.1.3 *Probe Characteristics*—The probes shall be mounted in an insulating fixture such as a sapphire bearing in a methyl methacrylate or hardened polystyrene block in an equally spaced linear array. The electrical insulation between adjacent probe points shall be at least 10^5 times greater than the *V/I* ratio of the film. The spacing shall be 0.64 to 1.00 mm inclusive (0.025 to 0.040 in. inclusive) as agreed upon between the parties concerned with the test. The precision and reproducibility of the probe spacing shall be established according to the procedure of 7.1.

5.1.4 *Probe Support*—The probe support shall allow the probes to be lowered perpendicularly onto the surface of the specimen so that the center of the array is centered on the specimen within ±10 % of the specimen length *l* and width *w*.

5.2 *Electrical Measuring Apparatus*:

5.2.1 The electrical apparatus shall consist of a suitable voltmeter, current source, ammeter, and electrical connections (see 7.2).

5.2.2 *Voltmeter* with input impedance 10^4 times the *V/I* ratio of the film. A vacuum-tube voltmeter, a digital voltmeter, or similar high-impedance input apparatus is suitable.

5.2.3 *Current Source* with current regulation and stability of ±0.1 % or better. The recommended current range is from 0.01 to 100 mA.

5.2.4 *Ammeter* capable of reading direct current in the range from 0.01 to 100 mA to an accuracy of ±0.1 % or better.

5.2.5 The current source and ammeter are connected to the outer probes; the voltmeter is connected to the inner probes.

5.3 *Specimen Support*—A copper block at least 100 mm (approximately 4 in.) in lateral dimensions and at least 40 mm (approximately 1.5 in.) thick, shall be used to support the specimen and provide a heat sink. It shall contain a hole that will accommodate a thermometer (see 5.4) in such a manner that the center of the bulb of the thermometer shall be not more than 10 mm below the central area of the top of the block where the specimen is to be placed.

5.4 *Thermometer* having a range from − 8 to 32°C and conforming to the requirements for Thermometer 63C as prescribed in Specification E 1.

5.5 *Vernier Calipers*.

5.6 *Toolmaker's Microscope* capable of measuring increments of 2.5 µm.

6. Test Specimen

6.1 The specimen shall consist of a continuous rectangular thin metallic film with a thickness greater than 0.01 µm and less than 100µ m. Thickness variation shall be less than ±10 % of the nominal thickness for thickness from 0.01 µm to 0.1 µm, inclusive; for greater thicknesses, the variation shall be less than ±5 % of the nominal thickness. The specimen shall be used as prepared by deposition of a material or by a thinning process, with no further cleaning or preparation. The test specimen shall be supported by a substrate consisting of a suitable insulating material.

6.2 *Geometry*—Measure the length, *l*, and width, *w*, of the specimen with vernier calipers. Record the values.

6.3 Measure the thickness, *t*, of the film in accordance with Method F 388.

7. Suitability of Test Equipment

7.1 *Probe Assembly*—The probe spacing and tip condition shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

7.1.1 *Procedure*:

7.1.1.1 Make a series of indentations on the surface of the specimen to be tested or other surface of similar hardness with the four-probe array. Make these indentations by applying the probes to the surface using normal point pressures. Lift the probes and move either the specimen surface or the probes 0.05 to 0.10 mm in a direction perpendicular to a line through the probe tips. Again apply the probes to the specimen surface. Repeat the procedure until a series of ten indentation sets is obtained.

NOTE 1—It is recommended that the surface or the probes be moved twice the usual distance after every second or every third indentation set in order to assist the operator in identifying the indentations belonging to each set.

7.1.1.2 Place the specimen so indented on the stage of the toolmaker's microscope so that the *Y*-axis readings (Y_A and Y_B in Fig. 1) do not differ by more than 0.15 mm (0.006 in.). For each of the ten indentation sets record the readings *A* through *H* (defined in Fig. 1) on the *X*-axis of the toolmaker's microscope and the readings Y_A and Y_B on the *Y*-axis.

7.1.2 *Calculations*:

7.1.2.1 For each of the ten sets of measurements calculate the probe separations, S_{1j}, S_{2j}, and S_{3j}, from the equations:

$$S_{1j} = [(C_j + D_j)/2] - [(A_j + B_j)/2],$$

$$S_{2j} = [(E_j + F_j)/2] - [(C_j + D_j)/2], \text{ and}$$

$$S_{3j} = [(G_j + H_j)/2] - [(E_j + F_j)/2]$$

where the index *j* is the set number and has a value from 1 to 10.

7.1.2.2 Calculate the average value for each of the three separations using the S_{ij} calculated above and the equation:

$$\bar{S}_i = \left(\frac{1}{10}\right)\sum_{j=1}^{10} = S_{ij}$$

where the index *i* successively takes the values 1, 2, and 3 (see 7.1.2.1).

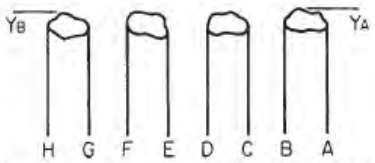


FIG. 1 Measurement Locations for Typical Probe Indentation Pattern

7.1.2.3 Calculate the sample standard deviation s_i for each of the three separations using the $\bar{S}_i$ calculated in 7.1.2.2, the S_{ij} calculated in 7.1.2.1, and the equation:

$$s_i = \left(\frac{1}{3}\right)[\sum_{j=1}^{10} (S_{ij} - \bar{S}_i)^2]^{1/2}$$

7.1.2.4 Calculate the average probe spacing $\bar{S}$ as follows:

$$\bar{S} = \left(\frac{1}{3}\right)(\bar{S}_1 + \bar{S}_2 + \bar{S}_3)$$

7.1.2.5 Calculate the probe spacing correction factor F_{sp} as follows:

$$F_{sp} = 1 + 1.082[1 - (\bar{S}_2/\bar{S})]$$

7.1.3 *Requirements*—For the probe assembly to be acceptable it must meet the following requirements:

7.1.3.1 Each of the three sets of ten measurements for S_i shall have a sample standard deviation s_i of less than 1 % of $\bar{S}_i$.

7.1.3.2 The average values of the separations ($\bar{S}_1$, $\bar{S}_2$, and $\bar{S}_3$) shall not differ by more than 5 % of $\bar{S}$.

7.1.3.3 The probe indentations shall not puncture the film.

7.2 *Electrical Equipment*—The suitability and accuracy of the electrical equipment shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

7.2.1 Measure the current through and voltage across a standard resistor whose resistance value is within a factor of ten of the *V/I* ratio of the film to be measured. Perform ten times.

7.2.2 Calculate the resistance r_i for the ratio of voltage to current for each measurement.

7.2.2.1 Calculate the average resistance $\bar{r}$ as follows:

$$\bar{r} = \left(\frac{1}{10}\right)\Sigma_{j=1}^{10} r_i$$

where:

r_i = one of the ten values of resistance determined in 7.2.1.

7.2.2.2 Calculate the sample standard deviation as follows:

$$s_r = \left(\frac{1}{3}\right)[\Sigma_{j=1}^{10} (r_i - \bar{r})^2]^{1/2}$$

7.2.3 *Requirements*—For the electrical measuring equipment to be suitable, it must meet the following requirements:

7.2.3.1 The value of $\bar{r}$ must be within 1.0 % of the known value of r.

7.2.3.2 The sample standard deviation s_r must be less than 1.0 % of $\bar{r}$.

7.2.3.3 The resolution of the equipment must be such that differences in resistance of 0.05 % can be detected.

8. Procedure

8.1 Connect the voltage measuring apparatus to the two center probes.

8.2 Connect the current source to the outer two probes.

8.3 Equilibrate the specimen at room temperature (23± 2°C) on the heat-sink block. Record the temperature.

8.4 Place the test specimen on the mounting block under the probe with the length parallel to the line of the probe array to within± 2°. Lower the probe onto the test specimen ensuring that the center of the probe array is centered on the specimen within ±10 % of the specimen length l and width w. Establish a current (see 8.5.1) between the outer probes. Record the voltage and current. Perform ten times.

8.5 *Caution*—Spurious and inaccurate results can arise from a number of sources.

8.5.1 It is recommended that, consistent with the desired accuracy, the applied current be as low as possible to reduce specimen heating. In high resistance or very thin films, it may be desirable to reduce the specimen current to prevent resistance heating. A drifting of the voltage reading may indicate a change in the resistance due to heating.

8.5.2 Wear and deformation of the tips in use may make frequent inspection and replacement necessary.

8.5.3 Spurious currents can be introduced into the test specimen by high-frequency generators. If equipment is used near such sources, adequate shielding should be provided.

9. Calculations

9.1 Calculate the specimen resistance R_i from the ratio of measured voltage and current.

9.2 Calculate the average specimen resistance $\bar{R}$ as follows:

$$\bar{R} = \left(\frac{1}{10}\right)\Sigma_{j=1}^{10} \bar{R}_i$$

9.3 Calculate the sample standard deviation as follows:

$$s = \left(\frac{1}{3}\right)[\Sigma_{j=1}^{10}(R_i - \bar{R})^2]^{1/2}$$

9.3.1 *Requirement*—For acceptance of the resistance, the sample standard deviation s shall be less than 1 % of $\bar{R}$.

9.4 Calculate the ratio of the specimen width w (see 6.2) to the average probe separation $\bar{S}$ (see 7.1.2.4). Calculate the ratio of specimen length l to specimen width w. Determine the lateral correction factor c from Table 1 by means of linear interpolation.

TABLE 1 Lateral Correction Factor, c, for Rectangular Thin Films

$w/\bar{S}$	$\ell/w = 1$	$\ell/w = 2$	$\ell/w = 3$	$\ell/w = 4$
1.00	...	...	0.9988	0.9994
1.25	...	...	1.2467	1.2248
1.50	...	1.4788	1.4893	1.4893
1.75	...	1.7196	1.7238	1.7238
2.00	...	1.9454	1.9475	1.9475
2.50	...	2.3532	2.3541	2.3541
3.00	2.4575	2.7000	2.7005	2.7005
4.00	3.1137	3.2246	3.2248	3.2248
5.00	3.5098	3.5749	3.5750	3.5750
7.50	4.0095	4.0361	4.0362	4.0362
10.00	4.2209	4.2357	4.2357	4.2357
15.00	4.3882	4.3947	4.3947	4.3947
20.00	4.4516	4.4553	4.4553	4.4553
40.00	4.5190	4.5129	4.5129	4.5129
∞	4.5324	4.5324	4.5324	4.5324

9.5 Calculation the ratio of the film thickness t (see 6.3) to the average probe separation $\bar{S}$ (see 7.1.2.4). Find the correlation factor $F(t/\bar{S})$ from Table 2 by means of linear interpolation.

9.6 Calculate the geometrical correction factor F as follows:

$$F = c \times F(t/\bar{S}) \times F_{sp}$$

where

F_{sp} = probe spacing correction factor (see 7.1.2.5).

9.7 Calculate the sheet resistance R_s as follows:

$$R_s = \bar{R} \times F$$

10. Report

10.1 For a referee test the report shall include the following:

10.1.1 A description of the specimen, including:

10.1.1.1 Type of film,

10.1.1.2 Specimen identification,

10.1.1.3 Color,

10.1.1.4 Appearance,

10.1.1.5 Source, and

10.1.1.6 Previous treatment and tests.

10.1.2 Dimensions and data, including:

10.1.2.1 Length and width,

10.1.2.2 Average values and standard deviations of probe spacing,

10.1.2.3 Standard resistor value,

10.1.2.4 Measured average value and standard deviation of standard resistor, and

10.1.2.5 Temperature.

10.1.3 Measured values of current and voltage.

10.1.4 Calculated average value and standard deviation of resistance.

10.1.5 Values of correction factors used.

10.1.6 Calculated value of room temperature sheet resistance.

10.2 Fur a routine test only such items as are deemed significant by the parties to the test need be reported.

TABLE 2 Thickness Correction Factor for Thin Films

t/S	F(t/S)
0.4000	0.9995
0.5000	0.9974
0.5555	0.9948
0.6250	0.9898
0.7143	0.9798
0.8333	0.9600
1.0000	0.9214
1.1111	0.8907
1.2500	0.8490
1.4286	0.7938
1.6666	0.7225
2.0000	0.6336

11. Precision and Bias

11.1 *Precision*—A two-laboratory comparative test of the measurement of sheet resistance on two groups of thin metallic films using separate pieces of equipment has yielded agreement to within ±0.44 % of the average value for sheet resistance values in the range from 25 to 40 $\Omega/^2$ and ±1.7 % for sheet resistance values in the range from 0.010 to 0.060 $\Omega/^2$.

11.1.1 *Precision*—Subcommittee F01.17 will conduct an interlaboratory test to confirm the precision of this test method.

11.2 *Bias*——Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

12. Keywords

12.1 collinear four-point probe; electrical resistance; electrical sheet resistance; four-point probe; resistance; thin films; thin conductive films; thin metallic films

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JANDEL ENGINEERING LTD.

Jandel Four Point Probing System

Hand Applied Probe with RM3000 Test Unit

The Hand Applied Probe combined with the RM3000 Test Unit is a high quality four point probe measurement system which incorporates the Jandel Cylindrical probe head. The system can be used to measure a wide range of materials with varying shapes and sizes.

The Hand Applied Probe is ideally suited for use in measuring large substrates and flat panels, however, it can be used to measure wafers and even small samples as well. When measuring small samples, you will sometimes need another piece of the same thickness of material underneath the back side of the probe body so that the entire unit rests on the material in the same plane, i.e., to keep it level. A discussion of using the Hand Applied Probe on small samples can be seen on page four of the following PDF file:
http://www.fourpointprobes.com/jandel-hap.pdf

HAND APPLIED PROBE GENERAL DESCRIPTION

The unit comprises a Teflon body containing a cylindrical brass mass sufficient to cause the probe needles of the 4-point head (loaded up to 200g each) to be completely retracted. The Teflon body incorporates a lead about 1m long to connect to the associated electronic measuring equipment. There is a toggle switch marked 'S' (shorted) and 'R' (read) which permits the probe head to be raised off the sample, or placed on it, with no sparking. The current source is shorted at position 'S' on the hand applied probe independent of the FWD, SBY, REV switch on the power supply. Of course, when the probe head is in position the FWD/REV positions can be used in the usual way to observe forward and reverse readings.

HAND APPLIED PROBE OPERATION

The probe head should be installed so that its acrylic insulating pad (adjacent to the projecting probe needles) lies in the same plane as the lower Teflon surface. Rotate the probe head so that its needles lie at right angles to the longitudinal axis of the Teflon holder, and clamp firmly with the two red screws. To present the probe head to the specimen it is best to make contact with the rear end of the block (where the switch is) and rock the block downwards so that it effectively pivots about the rear. In this way the probe points will retract without scrubbing on the specimen surface. The actual position of the probe points can be seen via the cutaway.

HIGH TEMPERATURE OPTION for the Hand Applied Probe

The Jandel Hand Applied Probe is available in a version that can withstand temperature of up to 200°C in an oven. The "read/standby" switch is removed and the Cylindrical Probe Head is modified to withstand 200°C. A complete four point probing system consists of the **Hand Applied Probe** shown above, combined with any of the four point probe electronics that Jandel Engineering offers.

SPECIFICATIONS

Dimensions:	Length:	approximately 125 mm (4.9") front to rear
	Width:	75 mm (3")
	Height:	approximately 80 mm (3.15"). The wire from probe head projects an additional 30mm (1.18") upwards
	Weight:	approximately 1.6kg (3.5 lbs)
Downward force:	approximately 1.1 Kg (2.4 lbs) - sufficient to easily retract 4 needles with 200g load	
Material:	Virgin Teflon body with nickel plated brass weight to accept Jandel cylindrical probe Ø 25.4mm	
Electrical	4-point probe with Teflon screened lead and Lemo 5-way plug and socket. Toggle shorting switch. Teflon screened lead to 180 degree x 5-way DIN plug.	

Download the Product Brochures for the Hand Applied Probe

Download the Instruction Manual for the Hand Applied Probe

RM3000 Test Unit

The RM3000 Test Unit is a specialty electronics instruments designed specifically for the four point probe measurement. It features high accuracy, an excellent range, and many features which simplify the four point probing measurement. The following are features of the RM3000 Test Unit:

- The measurement range of the RM3000 Test Unit is from 1 milliohm-per-square (10^-3) up to 5 x 10^8 ohms-per-square with 0.3% accuracy. The volume resistivity range is from 1 milliohm-cm (10^-3) up to 10^6 ohms-cm (more conductive materials can be measured if in the form of a thin film).
- The RM3000 includes PC control software which can be used for data logging (storing data in the CSV format) and measurement conversion to ohms-per-square or ohms-cm.
- The RM3000 provides simultaneous readout of input current and either mV, ohms-per-square, or ohms-cm. The ohms-cm readout requires input of the sample thickness for thin films, or tip spacing for bulk samples.
- The RM3000 has onboard non-volatile memory so that up to 50 measurements can be stored internally and then downloaded and saved all at one time using the software. Alternately, each measurement can be saved to a PC as it is made.
- The RM3000 has an auto-range button that can be used to automatically determine the optimum input current for a given material without using the trial and error method.
- The RM3000 has forward (FWD) and reverse (REV) buttons to reverse the direction of current flow. A common way to determine if a measurement is valid is to reverse the direction of current flow and then check to see if the forward and reverse voltage readings correlate well, i.e., the values should be similar, but with the reverse current voltage being a negative value.
- The RM3000 allows input of correction factor when making sheet resistance or volume resistivity measurements
- The RM3000 interfaces with optional AFPP motorized Z-motion arm

SPECIFICATIONS

Superior Current Source

- 10nA to 100mA (99.999mA) current source selectable in steps to 3 decimal place resolution
- Current set numeric keypad
- 4 default preset current programs (user programmable)

Superior Inbuilt DVM

- Input Impedance 1,000,000,000,000 ohms
- Input Bias current 4pA
- DVM 1300mV range and 130mV range
- 130mV accuracy
- 0.2% +/- 5uV resolution (10uV or 1uV) range
- 1300mV accuracy 0.2%+/-100uV resolution
- 100uV Ohms/Square
- Rapid Zeroing null function for DVM

FEATURES

- 28 Key high quality Keypad
- 16x2 line LCD Display for simultaneous indication of Set Current and either
- Ohms/Sq, Ohms-cm, or mV
- Auto-Ranging capability to determine the optimum input current based upon the material being measured.
- Intuitive operation
- Microprocessor controlled
- Reduced Footprint
- Robust Attractive ABS Case
- Accurately measures down to 10's of milliohms/square without external meter
- 4mm socket facility to connect an external meter
- RS232/USB connectivity for control and for collecting data in CSV format

Click here to see the instruction manual (688K PDF file) for the RM3000 Test Unit

Cylindrical Probe Head

The Cylindrical probe head, one of which is included with the Hand Applied Probe , can withstand temperatures from 77K up to 120C in it's standard configuration. A modification to the Cylindrical probe will allow it to withstand temperatures from 77K up to 200C (in an oven). The Cylindrical probe head is built to high standards of quality and accuracy. A brochure regarding the Cylindrical probe can be found here:
http://www.fourpointprobes.com/jandelcylindrical.pdf

An application note with information regarding the constructions and specifications of the Jandel Cylindrical probe can be seen here: http://www.fourpointprobes.com/cylindrical_app_notes.pdf