# EFFECT OF LINE THICKNESS CROSS-SECTIONAL GEOMETRY TO STRETCHABLE PRINTED CIRCUIT UNDER THERMAL PERFORMANCE

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## DECLARATION

I declare that this project report entitled "Effect of line thickness cross-sectional geometry of Stretchable Printed Circuit under thermal performance" is the result of my own work except as cited in the references.



# APPROVAL

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



# DEDICATION

This report is dedicated to my beloved late father and my beloved mother, Suhaimi Sulaiman and Fatimah Ismail.



### ABSTRACT

The stretchable printed circuit are widely used days by days due to the physical characteristic that able to be bend and stretch. The stretchable printed circuit have been used in various field such as health, sport, industry and fashion. The research were done to find out the effect of ink thickness against the resistivity. Next, to find out the effect of temperature against the resistivity and the last one to find out the effect of strain applied against the resistivity. The samples were prepared with four different thicknesses (2, 4, 6 and 8 layers) where the layers was made using cellophane tape to create different thickness. The printing method used was screen printing method and the samples was measured using four point probe in the unit of  $\Omega$ /sq. The samples were tested under three different condition where the first condition the sample will be measure under normal condition (room temperature -32 °C). The second condition, the sample were tested under various temperature (40, 60 and 100 °C). The last condition the sample were tested in the room temperature under various strain applied (20, 40, 60 and 80 %), the Vernier caliper was used to apply the strain on the samples. Carbon was used as the conductive ink and thermalpolyurethane (TPU) was used as the substrate in this studies. The result of the study shows that when the ink thickness increase, the resistivity will decreased. As for the mechanical or strain test when the higher strain applied to the sample, the higher resistivity obtain by the sample.

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### ABSTRAK

Litar bercetak boleh regang digunakan dengan cara meluas hari demi hari kerana karakter fizikalnya yang mampu lentur dan regang. Litar bercerak boleh regang digunakan dalam pelbagai bidang seperti kesihatan, sukan, industri dan fesyen. Kajian ini dilakukan untuk mengetahui kesan ketebalan dakwat terhadap rintangan. Seterusnya, untuk mengetahui kesan suhu terhadap rintangan dan akhir sekali untuk mengetahui kesan ketegangan yang diberikan terhadap rintangan. Sampel disediakan dengan empat jenis ketebalan berbeza (2, 4, 6 dan 8 lapisan) dimana setiap lapisan dihasilkan dengan menggunakan pita selofan untuk menghasilkan ketebalan berbeza. Kaedah cetakan digunakan adalah kaedah cetakan skrin dan sampel diukur menggunakan prob empat mata didalam unit  $\Omega$ /sq. Sampel akan diuji dibawah tiga kondisi berbeza dimana kondisi pertama sampel akan diukur didalam suhu bilik (32 °C). Kondisi kedua, sampel akan diuji didalam pelbagai suhu (40, 60, 100 °C). Kondisi terakhir, sampel akan diuji didalam suhu bilik dibawah pelbagai tegangan (20, 40, 60, 80 %) yang dikenakan padanya, dimana angkup vernier digunakan untuk meregangkan sampel. Karbon digunakan sebagai dakwat konduktif dan poliuretana termal digunakan sebagai substrat didalam kajian ini. Hasil kajian menunjukkan apabila semakin tebal dakwat, rintangan akan berkurangan Bagi ujian mekanikal atau ketegangan pula apabila semakin tinggi tegangan dikenakan pada sampel, semakin tinggi rintangan dihasilkan oleh sampel.

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# LIST OF ABBREVIATIONS

PCB	Printed Circuit Board
SPC	Stretchable Printed Circuit
FR4	Fiber Glass
TPU	Thermoplastic polyurethanes / Thermal polyurethane
PWB	Printed Wiring Board
PDMS	Poly Dimethyl-Siloxane
PET	Polyethylene terephthalate
HTV	UNIVERSITI TEKNIKAL MALAYSIA MELAKA High Temperature Vulcanizing
LTV	Low Temperature vulcanizing
LSR	Liquid Silicone Rubber
CIJ	Continuous Inkjet
DoD	Drop on Demand
ASTM	American Society for Testing and Materials
MSDS	Material Safety Data Sheet

# LIST OF SYMBOLS



# LIST OF APPENDICES



#### **CHAPTER 1**

#### INTRODUCTION

#### **1.1 Background of Research**

The printed circuit board or PCB is a board that connecting electrical component together and actually make the device working and functioning correctly. The part of the PCB is consist of substrate, copper layer, soldermask and silk screen. The existence of the PCB really improve our technologies to another level.

But due to the restriction of the PCB which cannot be bend or stretch it make the PCB less favorable in small devices so there come the new technology which is stretchable printed circuit (SPC) or also known as stretchable printed ink. SPC is a circuit board that are made from combination of substrate but not same as the substrate used on PCB which is FR4 or also known as fiberglass or any solid component that are unbendable or stretchable and conductive ink

The substrate used is something flexible such as fabric, plastic and any flexible material that are suitable to be used, in this research the substrate used is thermoplastic polyurethanes or TPU which is bendable and stretchable. The conductive ink used is carbon and the ink will be printed above the substrate using screen printing method and the purpose of the ink is to conduct electricity through the stretchable printed circuit, this ink work as replacement of the copper layer in PCB.

After completing printing the conductive ink to the substrate, the substrate will be place inside oven for curing process. The last step after curing process is the testing process which is 4 point probe is used to measure the resistivity of the stretchable printed circuit board in unit of ohm/sq. The function of the SPC is same as normal printed circuit board but it is bendable and stretchable which make the SPC more versatile and can be use inside small devices or even in complicated shaped devices.



#### **1.2 Problem Statement**

The technology nowadays keep improving and more complex. In order to make something powerful yet small in size is becoming more and more difficult due to the restriction on printed circuit board (PCB) that not bendable and stretchable. But the PCB has evolved day by day and now the latest technology is stretchable printed circuit (SPC) which are bendable and stretchable. The substrate was made up using stretchable material such as fabric and plastic which allow the printed circuit board to become flexible and stretchable. With this two new features the use of the SPC made something impossible before to something possible, as can be seen the design of the television, phone and other gadget becoming more smaller and unique but the function of the device much better than before. This is due to the flexible and the stretchable properties of the SPC.

There are several problems that causing the limitation of SPC in the industries, such as the thermal properties, physical properties and electrical resistivity of the SPC. These problems will be study based on the parameter related, for example the effect of the temperature and strain to the conductive ink and substrate.

The effect of the line thickness cross-sectional to stretchable printed circuit under thermal performance will be covered in this research. There are different thickness of conductive ink was used to study the effect on resistivity and how to improve the quality of the SPC. The conductive ink and the substrate used in this study are carbon ink and thermal polyurethane (TPU) respectively.

## 1.3 Objective

The objective for this research are :

- 1. To study the effect of thickness of the ink to the resistance.
- 2. To study the effect of the temperature to the resistance.
- 3. To study the effect of the strain applied to the stretchable printed circuit to the resistance.

## 1.4 Scope of Study

The scope of study is listed as below :

- 1. Screen printing process to print the ink on the substrate.
- 2. Curing process to cured the samples.
- 3. 4 Point probe resistance test to measure the resistivity of the samples.
- 4. Mechanical and thermal testing of the specimens based on various temperature and strain.
- 5. Variable ink thickness (2 layers, 4 layers, 6 layers and 8 layers). UNIVERSITI TEKNIKAL MALAYSIA MELAKA

### **1.5** Planning and Execution

The research activities and progress for PSM 1 is been illustrates as Figure 1.1. The figure includes the flow and process of the research such as title selection, literature review, designing the experiment, formulation of samples, material characterization testing that consist of mechanical and thermal testing, data analysis, report writing then followed by report writing and report submission and the last one is PSM 1 seminar. In PSM 2 the research activities was continued. The new sample was prepared and used for the mechanical and thermal characterization test, followed by the data analysis process before compiling the final result in the report. The research activities present in PSM 2 was illustrated in the Figure 1.2.



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Report submission														
PSM 1 Seminar														

Table 1.1 : Gantt Chart for PSM 1

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PSM 2 Seminar														

Table 1.2 : Gantt Chart for PSM 2

#### **CHAPTER 2**

#### LITERATURE REVIEW

## 2.1 Introduction

In this chapter will be focusing on reviewing about the printed circuit board (PCB), stretchable printed circuit (SPC), conductive ink, substrate, printing method, 4 point probe, mechanical behavior, thermal behavior, purpose of research study and overview of research study.

## 2.2 Printed Circuit Board (PCB)

A printed circuit board is a board that connecting all the electrical component together and actually make the board light up and functioning using conductive tracks, pads and other featured etched from copper sheet. It is also act as a medium for microelectronic components such as semiconductor chips and capacitors are mounted. The PCB layers can be single sided, double sided or multi-layer (LaDou, 2006).

#### 2.2.1 History and Evolution of PCB

Basically the PCB is made up from substrate, copper layer, solder mask and silk screen. The substrate usually use is fiberglass which giving the inflexible characteristic to the PCB and the purpose of the substrate is to hold the circuit component or the wire been printed. Next is copper layer, the copper layer will be sandwiching the substrate and giving the conductivity to the board. The solder mask which usually giving the green colour to the PCB will be coated to the copper layer in order to protect the copper layer from corrosion and anything can disturb or harm the conductivity of the copper layer. Last layer is the silk screen which a layer that being use to placing text or part outline on the board (LaDou, 2006).



Before the existence of PCB people having hard time to do wiring and frequent failures occur at the wire junction and when the wire insulation already old or crack, short circuit occurring to the circuit. The old method used before the PCB is called point-to-point wiring or also called as printed wiring board (PWB) because their placing the wire on a rigid surface (LaDou, 2006).



Figure 2.2 : Point-to-Point Wiring (Sparkfun, 2014)

The PWB material can be anything that having rigid surface such as Bakelite, Masonite or even plain wood. The material will be drilled to make holes and after that wires such flat brass would be riveted onto it (LaDou, 2006).

Then the PCB technologies keep improving and getting more modern. The material used, the function and the quality of the PCB also increasing years by years from wood to fiberglass (LaDou, 2006). But people keep trying to improve the PCB and then people begin to study and start inventing a flexible PCB that bendable in order to increase the possibilities of the modern devices (Banfield, 2000; Happonen et al 2016; Suikkola, 2015). But the flexible PCB is only bendable and not stretchable which still limiting the use of the PCB inside small devices. But with the new technology that came up after the flexible PCB which is stretchable PCB technology allow the PCB to be stretch and bend (Norhidayah et al., 2017; Suikkola, 2015).

This increase the possibilities and the use of the PCB inside device and gadget and make something impossible before to something possible. As can be seen how big the role played by the PCB in the technologies world, and the PCB keep evolving and changing to improve and modernize the devices and gadget toward the greater future (Suikkola, 2015).

## 2.3 Stretchable Printed Circuit (SPC)

Printed circuit board is a circuit board that connecting the electronic component, but the substrate is a board which is rigid object or flexible or stretchable, but it is still using a board (Banfield, 2000; LaDou, 2006). Meanwhile the stretchable printed ink also flexible and stretchable but the substrate not from a board. The substrate can be from fabric, paper, glass and polymer or any surface that the conductive ink can be printed on it (Hrehorova et al., 2011; Khirotdin et al., 2016; Merilampi et al., 2010; Pudas, 2004; Sevkat et al., 2008; Suikkola, 2015; Van Osch et al., 2008).



Figure 2.3 : DuPont stretchable printed ink (Rodie, 2015)

The stretchable printed circuit can be used in various kind of field. For example in health. Due to the capability of the ink and the substrate that are capable to be printed on fabric and polymer made the possibilities in medical also increasing rapidly. It made the choice of material and the flexibility of the material better by making better and more attractive equipment to be used. This also can reduce the space used inside the hospital because the machine in smaller size or even possible to be diagnose remotely, so the space inside the hospital can be better and more efficient (Suikkola, 2015).

The stretchable printed circuit can be divided into two main component which is the substrate which act as the base of the stretchable printed ink and the conductive ink that allow the electricity to flow on it (Banfield, 2000).

#### 2.3.1 Conductive Ink

The conductive ink works the same way as normal ink that being used daily in a pen marker or anything that using ink such as printer (Cummins & Desmulliez, 2012; Liimatta et al., 2014; Van Osch et al., 2008), but the difference is the conductive ink can conduct electricity through the ink. The concept of the conductive ink is same and can be said as replacement for the conductive traces that exist on the PCB. The purpose of the conductive ink is to make a route or track for the electricity to flow and move to one place to other place which is same as conductive traces that allow the electricity to move around through the conductive traces.



Figure 2.5 : The Conductive ink traces on TPU (Suikkola, 2015)

The conductive ink is simpler and more environmental friendly compare to the conductive traces on the PCB because of the process to making the traces on the PCB using standard industrial process such as etching copper from copper plated substrates, this process usually producing waste stream that later will be need to be treated. Besides that the process making the printed circuit board also causing a lot of pollution that lead to environmental problem in the future (LaDou, 2006).

The process to manufacturing the PCB also affecting the worker that work in the production department. There was a case that producing the PCB damaging the health of the worker in long term process. It is mention in the journal that the high intensity of the chemical and often sloppy manufacturing processes exposed to the workers causing dangerous health problem that are known to be toxic to human body especially the organ system (LaDou, 2006).

There are various kind of conductive ink, from cheap to expensive, from high conductivity to low conductivity. For example the conductive ink being used so far are silver, gold, cooper and carbon. But gold not really being use because of the expensive value despite having good conductivity (Bhore, 2013). Not just gold, the silver also having good conductivity and frequently being used, but the price of silver ink keep increasing so people need to find another alternative to replace the silver ink (Banfield, 2013; Bhore, 2013; Salam et al., 2011).

In order to solve the cost problem, people tend to use copper ink since it is cheaper and having good conductivity even the conductivity not good as the silver ink and gold ink, the conductivity of the copper ink is 10 magnitude lesser compared to the silver ink but the cost of the copper ink is far cheaper than the silver ink so basically its cost effective (Bhore, 2013; Salam et al., 2011; Tsai et al., 2015). But the main problem using the copper is the characteristic

which is the tendency to oxidize in air, and the oxidized copper does not conducting electricity (Banfield, 2013; Tsai et al., 2015).

The copper ink problem can be solve by doing additional steps, such as hydrazine reductants or using sodium borohydride or sodium hydrophosphate as the reductants. The hydrazine are one of the famous method used in the early times, but the side effect of the hydrazine reductant is the high toxicity which will causing serious pollution and risk the people when producing the sample. As for the sodium borohydride or sodium hydrophosphate will cost more if using them as the reductant since the process using both of the reductant need to be performed in a vacuum environment (Tsai et al., 2015).

So the practical way and the best way is to use carbon ink, since the carbon ink is cheap and suit to be used in this research due to its characteristic. The carbon ink is pretty flexible, stretchable and have quite high durability properties. Besides that, the carbon ink is also having good adhesion to be used on elastomers substrate such as thermal polyurethane (TPU) (Mosallaei et al., 2017).

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Carbon ink is far cheaper compared to silver ink there is some advantages using carbon ink compared to the silver ink. One of the advantages using carbon as the conductive ink over the silver is the inert nature of the carbon which is the carbon resistivity toward any gas or liquid. This making the carbon ink much easier to be used compared to the silver or other metal in case of the compability issue. Besides that the famous characteristic of the carbon is the ability to have many kind of resistivity depend on the formulae used to produce the carbon ink (Bhore, 2013). Electromigration of the silver is one of the weakness using silver, meanwhile carbon does not have any tendency for electromigration which making it more stable and have higher resistance toward chemical. Electromigration can be defined as the movement of any metallic material by influence of the electrical field, this process usually through or across a nonmetallic medium (Bhore, 2013).

The conductive ink are mainly made from three or four component which is filler, binder, solvent and additives. But sometimes additives doesn't need to be used as its name the additive will be used just to enhance the ink. The types of the ink can be divided into two categories depend on the quantity of the solvent. The conductive ink will be called as liquid ink if the quantity of the solvent is high and the viscosity of the ink is low, and the conductive ink will be called as the paste ink if the quantity of the solvent is low which the viscosity of the ink is very high (Bhore, 2013).

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## 2.3.1.1 Filler

The type of filler is making the different between the graphic ink and conductive ink. If using the graphic ink will have the capabilities to provide various kind of colour which are used for printing image on paper or fabric, as for conductive ink the filler are made from material that are able to conduct the electricity such as silver, copper or carbon (Bhore, 2013).

#### 2.3.1.2 Binder

The purpose of the binder is to make the ink possible to stick to the substrate. Binder giving the adhesion and cohesion characteristic to make the ink stay still on the substrate without fall over it. The binder will be mixed with the filler by adding solvent which will be explain in detail later (Bhore, 2013).

The classification of the binder is based on the compatibility of the solvent and the binder. The first class is the binder that soluble in water, so it is called as water-based binder. Next is the binder that are soluble in specific solvent such as alcohol and acetates, hence it is called as solvent based resins (Bhore, 2013).

#### 2.3.1.3 Solvent

In the process making the conductive ink, the material of the binder used usually a thermoplastic, besides that the thermoplastic material can be dissolved using solvent or through adding heat until the thermoplastic melt and turn it from solid to liquid state (Banfield, 2000).

So basically the purpose or the function of the solvent is to mixing the filler and binder together by turn the thermoplastic to liquid form which is act as the binder with the filler. After both of the material mixed together it can be used as conductive ink and print it on the substrate. After the printing process wait for the solvent to evaporated or let the heated thermoplastic (substrate) to cool down so it will turn to solid state back this process also called curing process. Due to this problem sometimes some binder are not suitable to be cured or heated in high temperature because it will melt the thermoplastic back to liquid state (Banfield, 2000; Banfield, 2013; Bhore, 2013).
#### 2.3.1.4 Additive

Additive also one of the conductive ink material but some conductive ink does not use additive because the purpose of the additive is to enhance the performance of the ink. The additive must be lesser than 0.2 to 1.0 part of the ink formulation. The used of the additive can be greatly benefit and improve the functionality of the ink (Bhore, 2013).

The additive can be divided into few classes. For example plasticizers which used to soften the ink and improve the flexibility, adhesion and make it glossier. Next is waxes that used to improve the rub resistance of the ink. Then the wetting agent for reduce the surface tension and increase the wettability of the pigment. There a many more additive such as dispersing agent, shorting compound, defoamer and drier (Bhore, 2013).

#### 2.3.2 Substrate

A substrate act as medium or base to place the conductive ink (Patton et al., 2017). In stretchable printed ink the substrate used are something flexible and stretchable. All of this **UNIVERSITI TEKNIKAL MALAYSIA MELAKA** possible thanks to advancement of the technologies especially the advancement in material. The advancement of the material made the substrate from a rigid board to flexible and stretchable.

The physical properties of the substrate which are flexible and stretchable increase a lot of possibilities in various field such as fashion, health and technologies. The stretchable conductor are possible thanks to the substrate material and it is interesting because the capabilities to handle large deformation during application (Merilampi et al., 2010). For example the camera, it is possible to make such great device due to the possibilities of the substrate to bend and stretch inside the camera which reduce the size, weight but in the same time increase the possibilities of electronic connection (Norhidayah et al., 2017).

There many kind of substrate can be used such as thermal polyurethane (TPU), polydimethyl-siloxane (PDMS) and Polyethylene terephthalate (PET) (Amjadi et al., 2014; Faraj et al., 2011; Huntsman, 2010; Sigma-aldrich Co Llc, 2014; Suikkola, 2015). But only two substrates will be focusing on later which is thermal polyurethane (TPU) and poly-dimethylsiloxane (PDMS) because of the substrate are capable to bend and stretch (Amjadi et al., 2014; Huntsman, 2010; Suikkola, 2015).

## 2.3.2.1 Thermal Polyurethane (TPU)

Thermal polyurethane or also known as TPU is one of the most common substrate use in stretchable electronic device. The first TPU was developed around 1937, TPU was able to be reshape more than once by heating at certain temperature the TPU will be soft and become hard back when cooled. In high temperature the TPU in soft state so it can be shaped and when it is cooled the shaped will be formed as the new form made in cooled state (Suikkola, 2015).

The characteristic of the TPU are stretchable and bendable. Besides that the TPU can be reshape to any form when heated or high temperature and after it being cooled the new shape will be maintained (Suikkola, 2015). Due to this features TPU are quite famous in industries, the purpose of the TPU not only to be as a substrate but also being used as other product (Huntsman, 2010).

Thermal polyurethane really popular in the markets and application, because of the flexible features, the TPU is able to be extruded or injection molded to create plastic product

such as footwear, hose or tube, film and many other industrial product (Huntsman, 2010). So in other word the TPU is widely used in the industry.



Figure 2.7 : Example of Product using TPUs (Huntsman, 2010)

Basically there three main TPU classes based in chemical classes which are polyester UNVERSITITEKNIKAL MALAYSIA MELAKA TPUs, polyether TPUs and polycaprolactone TPUs. The first class is polyester TPUs, this type of TPU are suitable to be use with polyvinyl chloride or commonly abbreviated as PVC and other polar plastic. It providing great abrasion resistance, enhancing properties, not affected by oil and chemical, have a good balance of physical properties and suitable to be used in polyblends (Huntsman, 2010).

The second class is polyether TPUs, comparing to another two classes which is polyester TPUs and polycaprolactone TPUs the specific gravity of polyether TPUs are quite lower. Polyether TPUs offering low temperature flexibility and great abrasion and tear resilience. It is also strong against microbial attack and providing great hydrolysis resistance which making the polyether TPUs suitable for water application (Huntsman, 2010).

The last class which is the third class of the chemical classes of TPUs is the polycaprolactone TPUs. This class famous with it tough and high resistance properties and having high resistance to hydrolysis. Polycaprolactone TPUs are an ideal choice as the raw material to be use for hydraulic and pneumatic seals (Huntsman, 2010).

Even there are several classes of TPUs as mention above but all of the TPU have similar characteristics which making them difference from other plastic materials such as high abrasion resistance, good elasticity, great low temperature and impact strength, resilience to greases, oil and various kind of solvent, have great flexibility under various temperature range, easy for colouring purpose, recyclable, suitable for bonding and welding (Suikkola, 2015).

The flexible and stretchable properties is thanks to the soft and hard segment on the TPU microstructure. The soft segment are made up from polyol and an isocyanate, meanwhile the hard segment was made up from a chain extender and isocyanate. The soft segment is the reason that allowing the TPU to be flexible and having elastomeric characteristic. Meanwhile the hard segment giving the physical performance properties and the toughness characteristic. This made the TPU as one of the best polymer to be used which having high flexibility yet excellent durability (Huntsman, 2010; Qi & Boyce, 2004).



At the room temperature the soft domain will having a characteristic like a rubber, meanwhile the hard domain are though to having permanent deformation, high modulus, hysteresis and tensile strength (Qi & Boyce, 2004).

### 2.3.2.2 Poly-Dimethyl-Siloxane (PDMS)

Poly-dimethyl-siloxane is also known as PDMS are widely used in many industries, not just manufacturing but it is also being used in medical, sports, food and cosmetic. PDMS basically belong to the same group as polymeric organosilicon or commonly known as silicones (Bindu, 2003; McDonald's, 2017; Reuss et al., 2012; Suikkola, 2015).



Figure 2.9 : PDMS chemical structure (Smokefoot, 2012)

PDMS as a substrate can be divided roughly into three categories and it is depend on the curing conditions and the manufacturing processes. The three categories of the PDMS are the high temperature vulcanizing –types (HTV), liquid silicone rubber -types (LSR), and low temperature vulcanizing -types (LTV). Each of the categories having slightly different mechanical properties, such as the fracture point, the ultimate tensile strength and the hardness of the material (Suikkola, 2015).

The common property of the PDMS even between these three categories are the ability to exhibit chemical inertness, highly constant mechanical properties over wide operation temperature range, having high compability, and highly elastic behavior. But there is a problem if using PDMS in printing applications. This is due to the adhesion problem because of its low surface energy. This problem can be solve only when the PDMS undergo surface treatment processes (Suikkola, 2015).

#### 2.4 Printing Method

The process to print the conductive ink to the substrate have several method, such as screen printing, inkjet printing, gravure printing and many more method. The method of printing can be divided into two categories which is impact printing and non-impact printing (Bhore, 2013; Cummins & Desmulliez, 2012).

The impact printing is a printing process that have direct contact with the substrate in order to print or transfer the image to the substrate. In other word, the printing process completed by applying the pressure directly to the substrate without having any extra component or part between the ink and the substrate. The example for the impact printing are gravure printing, screen printing and flexography printing (Bhore, 2013).

Meanwhile the non-impact printing is a printing process that transfer or print the image on the substrate without any impact or pressure. The process occur are indirectly by using intermediate component to print the ink to the substrate. The example for non-impact printing are inkjet printing and laser printing process. Besides that, the printing process have variation and different kind of concept and working principle depend on the method and the machine used (Bhore, 2013).

## 2.4.1 Screen Printing

The screen printing method was known as one of the oldest printing technique or processes ever known by humanity. The process of printing is manual, the main component needed for screen printing method are the printing paste, the screen or desired pattern used to print the ink, the surface used for printing or in other word the substrate, and the last one is squeegee (Liimatta et al., 2014; Suikkola, 2015).

All of the component or element are crucial and important to be understand in order to use the screen printing method. The first element is the printing paste, the printing paste can be any liquid based or paste based ink. In this case the ink use is conductive ink. The second element is screen, you have to place the screen above the substrate so that the pattern will be printed as the screen design. The third element is the surface used for printing which is substrate, the substrate can be glass, TPU, PDMS or any other substrate, but make sure to print on flat surface in order to have neat printing result. The last one is the squeegee, the squeegee is used to spread the ink on the screen and print it to the substrate. Figure 2.5 shows the screen printing process (Liimatta et al., 2014; Suikkola, 2015).



Figure 2.10 : The principle of the screen printing process (Suikkola, 2015)

As can be seen in Figure 2.6 the ink paste was placed above the screen and then using the squeegee to spread the paste throughout the screen. The squeegee will spread the paste by applying pressure and moving forward and backward in order to make sure the paste are spread evenly on the screen. After the squeegee complete spreading the ink paste on the screen, the screen will be lifted and left the substrate to be cured. The curing process are depend on the ink used either can be rest at room temperature or need to use other machine such as oven or Digital Light Process (Khirotdin et al., 2016; Suikkola, 2015).



Figure 2.11 : Screen printing working principle (Liimatta et al., 2014)

## 2.4.2 Inkjet Printing

Inkjet printing are widely used in publishing and graphic industries. In early stage the inkjet printing are used for printing text and graphic that are mainly in office, tailor advertising, customize packaging and desktop publishing. But the usage of inkjet printing have been explore

and improve by adding more and more purpose can be use not just simply printing text or graphic on paper for office purpose (Cummins & Desmulliez, 2012; Van Osch et al., 2008).

The inkjet printing was begin by Felix Savart in 1833, which showing the breakup of the liquid jets into a series of repeated drop that related with the concept of fluid dynamics. There was a tiny nozzles that spraying the ink onto the substrate, and the quality and the dimension of the inject printed are depend on the nozzle size which is by decreasing the size of the nozzle can increase the printing resolution (Cummins & Desmulliez, 2012).

The inkjet printer was first commercialized by Hewlett-Packard in year 1984, since that years the inkjet printer become really popular especially among office and home appliance (Liimatta et al., 2014). So, what actually made the inkjet printer so popular? There are few advantages can be listed using the inkjet printing :

- 1. The printing process was non-impact, so there is no contact between the substrate and the ink which making it is possible to print on non- flat substrate material.
- 2. Reduce the cost because there is no need any printing plates or mold, this can reduce the UNIVERSITI TEKNIKAL MALAYSIA MELAKA initial cost and reduce the cost for per printing process.
- 3. Saving ink better, there wastage of the ink greatly reduce compared to other printing method.
- 4. The process can be digitally controlled easily.
- 5. The cost of the printer cheaper compared to other conventional printing presses.
- 6. The printing process is automated, less monitoring required.

The inkjet technology can be classify into two classes, the first one is continuous mode inkjet (CIJ) and the second one is drop on demand (DoD). The classes can divided depend on

the working principle of the inkjet technology. The working principle will be summarize in the Table 2.1.

DoD method doesn't have any magnetic
charges and there is no need to recycle the ink
because the ink will be ejected only when
they are needed which making the DoD have
much simpler system compared to CIJ. The
transducer will control the formation of the
droplet to prevent from excessive ink droplet
than needed (Liimatta et al., 2014).
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Table 2.1 · Different between CII and DeD working principle	
Table 2.1. Different between Cij and Dod working binnerbi	e



#### 2.4.3 Gravure Printing

Gravure printing has been used for long time, it is mentioned that the gravure printing already being used almost one hundred years. There are several name of gravure printing based on country such as America called gravure printing as rotogravure printing. Based in the history, the gravure printing method was being used initially to producing stamps in early 1990s. But now the term gravure not only being used in stamping but also being used in printing industry. (Morgan, 2016).



Figure 2.13 : Example of Egyptian stamp by Harrison in 1923 (Morgan, 2016)

Gravure printing are well known because of the capability to produce the best quality of the printed product. This made the gravure printing method widely used in graphic art application, not just that it is also being used in various printed electronic due to the low cost manufacturing process. The gravure printing is much simpler compared to other printing processes. It is using four main component which is an engraved cylinder with the pattern, ink fountain, doctor blade and impression roller (Hrehorova et al., 2011).



Figure 2.14 : The component for gravure printing and its basic working principle (Hrehorova

et al., 2011)

The gravure printing method can be divided into two method which are direct gravure printing and off-set gravure printing. In Figure 2.14 the working principle shown is an example for direct gravure printing. It is called direct because the ink is directly to the engraved cylinder and the engraved cylinder print the pattern to the substrate, or in simpler term the image was printed directly from engraved cylinder to the substrate (Serenius, 2011).

The off-set gravure printing working principle is similar to direct gravure printing. But the difference between two of them is the off-set gravure printing not directly print the pattern to the substrate using the engraved cylinder. Instead of printing the image directly to the substrate, the off-set gravure printing method print the image to the intermediate part or also called as transfer roller, and then the transfer roller will print the image to the substrate (Serenius, 2011).



Figure 2.15 : Working principle of (A) Direct gravure printing and (B) Off-set gravure



# 2.5 4 Point Probe

4 point probe is a measurement device that used for measuring the resistivity of the semiconductor samples. As its name 4 point probe so there was four pins which two outer pins represent the current source and measurement and two inner pins for measuring the voltage. The 4 point probe also can be used to measure the thickness of the film, but the 4 point probe are usually used for measuring the sheet resistance and the bulk resistivity (Bautista, 2004).

The theory of the 4 point probe basically because of the fixed current is injected into the wafer through the current pins which is the two outer pins on the probe, and the measurement of the voltage was measured using two inner probes pins. The spacing between the pin is labelled as 's' (ASTM, 2011; Bautista, 2004).



The sheet resistance unit in the 4 point probe is ohm/sq and the basic equation to get the sheet resistance are using this equation, but need to have the value of the current and voltage first before applying the equation (Smits, 1958).

$$R_s = \frac{\pi}{\ln\left(2\right)} \frac{V}{I}$$

(2.1)

Where,

$$\frac{\pi}{\ln(2)} = 4.5324$$
 (2.2)

Where,

V= voltage

I = current

 $R_s = Resistance Sheet$ 

Usually the 4 point probe is connected to computer and the calculation will be done by the software that connected with the 4 point probe. The software can calculated the resistance, voltage, current and even the thickness of the samples (Bautista, 2004).

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### **CHAPTER 3**

#### METHODOLOGY

#### **3.1** Overview of Research

In this chapter, the method and procedure needed to prepare the sample, the apparatus and material used, and the types of test that was conducted on the sample (TPU and glass slide) for the research project will be focused. Figure 3.1 shows the flow and the summary of the methodology of this study. Begin with the literature review study suitable with the project title and ended with the final report writing which mean the completion of the project. The summary for the research of this project are as below:-

- i. Listing out the procedure, parameters and standards used for the research.
- ii. Preparing the apparatus and material needed for the research.
- iii. Preparing the 2 kinds of samples. The conductive ink will be printed on TPUs and glass slides.
- iv. Conduct mechanical and thermal test on the test specimen after the printing process complete and determine the resistance of the samples.



Figure 3.1 : Flow Chart of research project.

### **3.2 Sample Preparation**

The preparation of the material and apparatus need to be prepared before proceeding with the steps and procedure need to be done. The rough steps can be refer in Figure 3.1 in order to get better understanding. So basically the step to preparing the samples consist of three main process which are :

- 1. Preparing the material and apparatus needed.
- 2. The printing of the ink to the substrate process.
- 3. The final process is the curing process.

### **3.2.1 Screen Printing Process**

The screen printing process requires four main component which are razor blade for spreading the ink paste, flat surface or in this research the zinc sheet was used and act base for printing. Next, the cellophane tape to change the thickness of the ink, the substrate which is TPU and glass slide and the last one is the conductive ink which is Bare brand carbon conductive ink.

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Figure 3.3 : Roll of TPU substrate.

So basically the method used to print the samples are following the standard of razor straight line printing method referring to standard ASTM D2739 (ASTM, 2017). The printing process to print the ink on the substrate which are the TPU and the glass are quite same but first the process to print the ink on the glass will be explained as below :

## A) Printing on Glass slide

1. Wipe the glass slide with isopropyl alcohol (IPA) to prevent any dust or dirt on the glass slide surface that can disturb the result later.



Figure 3.4 : Wiping the surface of the glass slide with IPA (Step 1).

2. Measure the thickness of the cellophane tape using Vernier caliper. The thickness of the cellophane tape is 0.04 mm.



Tape layer by layer on the glass slide, there should have 5 samples which are 2, 4, 6, 8 layer. The gap between the tapes is 0.1cm which represent the thickness of the ink.



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Figure 3.6 : Tape layer by layer on the glass slide (Step 3).

4. Before the gap was made, distance of 0.1 cm was marked using marker pen, and then tape on the other side.



5. Print the carbon ink on the samples by placing the carbon ink on the samples and spread it using razor blade.



(A)



Figure 3.8 : (A) Placing the ink on the glass slide and (B) Spread the ink using razor

blade (Step 5).

Left the samples for 30 minutes for curing process, the carbon ink was cured in room temperature and the recommended time for curing is 15 minutes (BareConductive, 2015a).

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Figure 3.9 : Cure the ink for 30 minutes in room temperature (Step 6).

 After 30 minutes remove the cellophane tape carefully. Don't move the tape to fast it will mess up the cured ink.



Figure 3.10 : Remove the tape (Step 7).

8. After complete print all the layer for 5 samples, label and mark every 5cm to guide for measuring using 4 point probe later, it should have 13 points used for glass slide.



Figure 3.11 : Marking point for each 5 mm on the substrate (Glass slide) (Step 8).





## **B)** Printing on TPU

1. Cut the TPU substrate into 5.0 x 2.5 cm dimension using scissor. Few samples were prepared with the similar dimension in order to ease the process for printing later.



Figure 3.14 : Preparing few samples (Step 1).

2. Then tape the TPU on the zinc sheet using cellophane tape. Before taping the TPU make sure to labelled each TPU and also check either the surface of the TPU facing in the right direction to prevent from printing the ink on the sheet cover.



Figure 3.15 : Bottom is TPU (transparent) and top is sheet cover (pinkish-colour) (Step 2).



Figure 3.16 : Label and tape the samples on zinc plate (Step 2).

3. Using the same type and brand of cellophane tape as in glass slide is compulsory to prevent getting wrong data later.

4. After that begin to make the different number of layer to change the thickness of the ink by stacking the cellophane tape. In this research the number of layer will be tested are 2, 4, 6, 8 layers. The gap between the tapes is 1 mm which represent the thickness of the ink.



Figure 3.17 : Preparing the different layers of tape on the substrate (Step 4).

5. After preparing 5 samples which is TPU with 2, 4, 6 and 8 layers the carbon ink will be place on the surface of the TPU and spread by the razor blade evenly.



Figure 3.18 : Spreading the ink using Razor blade (Step 5).

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 Since the carbon ink can be cured at the room temperature, the samples were cured for 30 minute in the room temperature which is twice from the recommended time from the data sheet (BareConductive, 2015a). The oven does not require for the curing process.



Figure 3.19: Curing process (Step 6).

7. After 30 minutes, remove the cellophane tape and the samples are ready to be labelled.



8. The samples will be mark with 5 points where each point gap are 0.4 cm for data average

measurement purpose.



Figure 3.21 : Sample points measurement gap

The dimension of the samples are as below. All of the samples have same dimension but only the thickness are different.



Figure 3.22 : Dimension of the sample

## 3.3 Experimental Setup

The resistivity of the conductive ink will be tested using 4 point probe. There are three different situation or test will be tested on the samples during reading the temperature which are as following : UNIVERSITI TEKNIKAL MALAYSIA MELAKA

- 1. In the room temperature without any strain applied on the samples (Room Temperature against Resistance Test).
- In variable heat (40, 60, 80, 100 °C) without any strain applied on the samples (Designed Temperature against Resistance).
- In the room temperature with variable strain (20, 40, 60, 80 %) applied on the samples (Strain against Resistance).

Before proceeding to the test, the steps and basic procedure to handle and use the 4 point probe will be explained :



Figure 3.23 : 4 point probe setup

- 1. Turn on the 4 point probe and computer.
- 2. After turn both of the computer and 4 point probe, Jandel RM3000+ software was opened and link the software with the 4 point probe. The purpose doing this step because the data can be store inside the computer. MALAYSIA MELAKA
- 3. Then on the 4 point probe forward (FWD) button was pressed, then hold the standby button for auto-ranging process. In this process the suitable current used is 100uA.



Figure 3.24 : The buttons on the 4 point probe.

- 4. Then FWD button was pressed again to get reading and set the resistance reading as ohm/sq.
- 5. Before placing the sample, the 4 point probe was calibrated using resistance test glass. The reading should be 12.55+- 0.25 ohm/sq (JandelEngineering, 2008).
- 6. If the reading not in the range mentioned try multiple places until get the data in the

range. If there no reading in the range, that mean the 4 point probe having problem.





Figure 3.26 : Calibrating the 4 point probe using resistance tester glass (Step 6 & 7).
- 7. If the data of resistance sheet is around the range, proceed with the sample.
- 8. The sample was placed on the 4 point probe.
- 9. Press the 4 point probe, make sure all the 4 pins touch the conductive ink. If the pins not touching the conductive ink there will be no reading shown in the 4 point probe.



- 10. Store the reading by press the enter button on the 4 point probe, if hold the enter button it will delete all the stored data (refer Figure 3.21). This step will be repeated until the sample obtain 15 data for 5 points marked on the sample, where each point having 3 data to obtain the average value.
- 11. Repeat step 8-10 until all of the samples being tested/ measured.
- 12. The data can be download in the computer through the 4 point probe software.

### 3.3.1 CASE 1 : Room Temperature Against Resistance Test

The first case is to measure the samples in 32°C which is the room temperature (RT) and without applying any strain on it. The resistivity will be tested and measured by 4 point probe, the unit of resistivity measured by the test is ohm/square. The samples that already prepared will be measured. Both of the samples (glass slide and TPU) were tested for this.

The basic equation can be relate with the data and the resistance is :



The value of the resistance, voltage and current can be found by using the Jandel RM3000+ software that link with the 4 point probe.

The way to conduct the experiment are similar as the 4 point probe steps that are mentioned before.

This measurement are used to comparing the value of the resistivity when the thickness of the conductive ink were changes. The result will be presented in the next chapter.

### **3.3.2** CASE 2 : Designed Temperature Against Resistance Test (Thermal Test)

The second case also will comparing the value of the resistivity between each samples which having difference conductive ink thickness (2, 4, 6, 8 layers). But the test also will related to the thermal performance of the stretchable printed ink.

The steps and method are similar as 'CASE 1' but after calibrating the 4 point probe, the samples will be heated by the hair dryer (40, 60 and 100 °C) and the temperature will be measured using thermal imaging camera. All of the 4 samples have to be tested for the thermal performance (2, 4, 6, 8 layers). The temperature measurement of the samples will be focused on the temperature applied on the ink.

After the required temperature achieve, the samples will be tested and the measurement will be read by the 4 point probe.



# 3.3.3 CASE 3 : Strain Against Resistance (Mechanical Test)

The third case will find out the effect of the strain on the samples either the strain applied by the Vernier caliper will affecting the resistivity of the sample. The strain applied to the samples are 20, 40, 60, 80 %. The purpose of the test is to know the mechanical performance of the samples under strain.

The steps and method used similar as 'CASE 1' but there was no need to use hair dryer since in this case the temperature used is the room temperature and the variable that changes in this case are the thickness of the ink (2, 4, 6, 8 layers) and the strain applied (20, 40, 60, 80 %) on the samples.

The samples will be place on the Vernier caliper using cellophane tape and will be stretch to certain distance, and the 4 point probe will be used to measure the resistivity of the samples under strain. All of samples will be undergo all of the strain required which is 20, 40, 60, 80 %. The step to use the 4 point probe also same as mentioned before.



# 3.4 Safety and Precaution

In handling chemical or doing experiment there must be safety and precaution need to be taken in order to prevent accident and unwanted injuries. So before doing any experiment it is safer to know about the material and the apparatus that being used, because preventing is better than cure.

The 'Bare' carbon conductive ink is safe to be used based on the Material Safety Data Sheet (MSDS). As been mention in the literature review, the conductive ink are made up from 3 main component which is filler, binder/resin and solvent, but in the Bare carbon conductive ink all of three component are marked as company secret (BareConductive, 2015b).

The conductive ink is safe to be use since it is made up as water-based conductive ink, so the apparatus used can be cleaned easily by using water which preventing the apparatus accidentally having chemical reaction when being used in other experiment and it is also easily dissolve in the water which not clogging the drain when cleaning process. It is also non-toxic, so there will be no problem if accidentally inhaling the smell of the conductive ink (BareConductive, 2015b).

As for the precaution and first aid measures if accidentally inhaling the chemical, having skin contact or eye contact and ingestion. The steps need to be taken are as following (BareConductive, 2015b) :

- Inhalation Change to different location from the exposure. Provide oxygen if needed or applying artificial respiration if necessary. Obtain medical treatment if ill effect occur.
   Skin Contact Wash the chemical with plenty of soap and water. Remove the contaminated cloth and wash it before reusing. Obtain medical treatment if ill effect occur.
- **3.** Eye Contact Wash it with plenty of water for at least 15 minutes. Obtain medical treatment if ill effect occur.
- Ingestion Rinse the mouth with water, don't ever induce vomiting. Obtain medical treatment if ill effect occur.

#### **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### 4.1 Introduction

A good stretchable printed ink must have a good physical or mechanical characteristic and also having good thermal performance. It is mentioned that the carbon ink and the TPU substrate are really compatible partner since carbon ink providing good adhesion to the substrate such as TPU. Besides that the carbon ink also compatible with the screen printing method or in this research the screen printing method used is razor straight line that following the ASTM standard (ASTM, 2017; Mosallaei et al., 2017).

In this chapter the result of the measurement will be discussed, either the thickness of **UNIVERSITI TEKNIKAL MALAYSIA MELAKA** the ink affecting the resistivity reading of the samples, either the temperature applied (40, 60, 100 °C) to the samples will affecting the resistivity reading on the samples and the last one is either the strain applied (20, 40, 60, 80 %) to the samples affecting the value of the resistivity of the samples.

The result will be divided into three topics which is CASE 1, CASE 2 and CASE 3. The CASE 1 will be divided into two sub-topics because of the usage of two different substrates which are glass slide and TPU. Next, CASE 2 and CASE 3 will having only one sub-topic since only one substrate being used which is TPU.

# 4.2 CASE 1 : Room Temperature Versus Resistance Test

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The concept of the electricity flow actually similar as the flow of water in the pipe, whereas the bigger the diameter of the pipe, the higher the volume of water can flow. So, logically if the concept of electricity flow and water flow in pipe are similar, then the thicker the ink will causing lower resistivity because more current can flow in the thicker ink. It is mentioned that the reduction of the resistivity is due to the increasing contact area between the electrical particles hence increasing the conductivity of the conductive ink track (Khirotdin et al., 2016).

So in the CASE 1 the samples will be measured in the room temperature and without any strain applied to them. There are ten samples which five samples from glass slide and 5 samples from TPUs. The data of the glass slide will be discussed first and then the data gather from the TPUs.

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## 4.2.1 Glass Slide

The substrate use for this samples is glass slide which is a rigid body non-flexible and non-stretchable substrate. The purpose printing the conductive ink on the glass slides are to focusing the result only on the effect of the different thickness of the conductive ink to the resistivity of the conductive ink measured by the 4 point probe. So this are the result obtain from 5 samples of glass slides by the 4 point probe.

EKUIRA	Number of Layer	Total Average R <sub>s</sub> (ohm/sq)
TIT	2	494.93
200	4	289.33
也	کل ملتسبا ما	او نوم سنة بيكند
LINIP	- 8 UEDRITI TEVA	350.48
UNI	PERSI10	201.98

Table 4.1 : Glass slide data



Figure 4.1 : Effect of sheet resistance of different layers of ink on glass slide

As can be seen the lowest sheet resistance is 201.98 ohm/sq which is the glass slide with 10 layers and the highest sheet resistance is 494.93 ohm/sq which is the glass slide with 2 layers. The result same as expected and mentioned before in which is the thicker the ink, the lower the resistivity or the higher the conductivity of the ink track (Khirotdin et al., 2016).

But the pattern of the graph of the sample with 6 and 8 layers suddenly rising even the thickness of the ink increase, the resistivity also increase. This phenomena can be due the printing technique, since the glass slide were the first sample been printed which mean lack of experience and basic technique of screen printing. Lack of experience can make the printing process of the ink not consistent and affecting the measurement of resistivity.

It also can be due to void occur on the ink, the probability of the void become higher when the layer of the ink is too thin which making the electrical flow not smooth and affecting the measurement of the data (Khirotdin et al., 2016).

### 4.2.2 Thermal Polyurethane (TPU)

Thermal polyurethane is a substrate that stretchable and flexible which completely opposite from the glass slide. But for CASE 1 the temperature used was only room temperature which is 32 °C and no strain applied to the TPUs. Since the CASE 2 and CASE 3 will be tested on the TPUs, so the CASE 1 is to test and see the pattern of the resistivity of the samples against the different thickness of the ink from 4 samples. The data measured by the 4 point probe are as shown on Table 4.2 and Figure 4.2.

Table 4.2 : TPUs data

+ <sup>4</sup>				
Sample	Number of Layer	Total Average R <sub>s</sub> (ohm/sq)		
6	2	465.92		
7	4	392.94		
8	6	297.55		
9	8	119.89		



are quite similar and as expected which is the thicker the ink, the lower the resistivity reading or the higher the conductivity of the ink track.

The pattern of the graph is constantly decreasing as the thickness increase. This result pattern is similar as past studies conducted by other researcher (Happonen et al., 2016).



Figure 4.3 : Resistance versus Line width versus thickness by layer (Happonen et al., 2016)

As can be seen in Figure 4.3 that each time the thickness of the sample increasing the resistance will be decrease. The resistance also can be reduce by increase the width of the conductive ink if referring to the pattern of the graph above.

The reason when increasing the thickness or the width of the conductive ink will reduce the resistivity is due to the increase of the cross-sectional area which allowing more electric current can be flow through the conductive ink (Happonen et al., 2016).

## 4.3 CASE 2 : Designed Temperature Against Resistance Test (Thermal Test)

In the CASE 2 the effect of ink thickness and temperature against the resistivity will be discussed in detail. There were sixteen results since there are four temperature which are the room temperature (32°C), 40°C, 60°C and 100°C each temperature having four different thicknesses (2, 4, 6, 8 layers). The figure below is the example of the method used to measure the temperature applied on the samples using Thermal Imaging Camera. The temperature applied to the samples until the ink achieve the designed temperature.



Figure 4.4 : Temperature measured using Thermal Imaging Camera

Table 4.3 shows the sheet resistance obtain in the unit of  $\Omega$ /sq from each sample from various thicknesses (2, 4, 6 and 8 layers) and temperature (Room temperature (RT=32 °C), 40 °C, 60 °C and 100 °C) and was represented in the form of graph in Figure 4.4.

Number of	Thickness	Sheet Resistance, R <sub>s</sub> (Ω/sq)			Ω/sq)
layers	(mm)				
		RT	40 °C	60 °C	100 °C
ANLAYS/A					
2	0.08	302.58	189.62	150.73	111.69
No.	L. R.				
	0.16	192.62	164.81	104.9	64.27
6 6 0,0 1,110	0.24	130.59	110.45	87.14	50.02
ليسيا مملاك	0.32	107.08	83.25	62.3	36.97

Table 4.3 : CASE 2 : Thermal test data

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Figure 4.5 : CASE 2 : Effect of sheet resistance with different temperature and ink thickness

Referring to the Figure 1 it can be seen that every time the number of layers increasing the resistivity will be decrease which mean the conductive ink become more conductive. For the room temperature the highest resistivity is the two layers and the lowest resistivity is the eight layer with 382.58  $\Omega$ /sq and 107.0 8  $\Omega$ /sq respectively. If comparing the room temperature result in Figure 4.2 and Figure 4.4 it can be seen that the result from each thickness not similar.

The reason of the different value even in same thickness is due to the human error. In this case the printing was done manually so there was possibility to make the dimension of the conductive not perfectly similar and the dimension of the conductive ink can affect the reading of the samples. But the pattern of the result still similar which as the thickness of the conductive ink increase the resistivity will be decrease. The reason of reduction in resistivity is as mentioned in CASE 1 before where the reason is due to the increase of the cross-sectional area of the conductive ink which allowing more electricity can be flow through the conductive ink (Happonen et al., 2016).

As for the thermal test, it can be seen that each time the temperature increase, the resistivity will be decrease for the same thickness samples. Referring to the Figure 4.4 when the thickness of the samples two layers at the room temperature obtain 302.58  $\Omega$ /sq meanwhile for the same thickness but in 100 °C the resistivity drop to 111.69  $\Omega$ /sq. The pattern at all thickness is similar for example at the thickness of eight layers for the room temperature and 100 °C reading of resistivity are 107.08  $\Omega$ /sq and 36.97  $\Omega$ /sq respectively. The thermal test result is consistent with the study conducted by Khairilhijra, Tan and Khairul which is when the temperature increasing, the resistivity will be decrease (Khirotdin et al., 2016).

The resistivity of the sample decreasing as the temperature increase is due to the increasing of the contact area between the particle in the conductive ink which allow more current to flow through the ink track (Khirotdin et al., 2016).

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# 4.4 CASE 3 : Strain Against Resistance Test (Mechanical Test)

The result of CASE 3 will be discussed on the effect of strain applied to the sample against the resistivity of the sample. The samples used in this case are five samples where each sample was stretch using stretchable jig with 0, 20, 40, 60 and 80 % of strain applied on the sample.

The original dimension of the conductive ink was  $2.0 \times 1.0$  cm and based from the original length of the conductive ink which is 2.0 cm was used as the datum or the 0 % strain applied on the samples. The length of the conductive ink based on the % strain applied on the sample was represented in the table below.

	2.9	
	% Strain applied	Length of the conductive ink (mm)
1	MILL.	
-	فل مليدهيا ملاد	ويومرسيني20يكي
U	VIVER <sup>20</sup> TI TEK	NIKAL MALA <sup>24</sup> SIA MELAKA
	40	28
	60	32
	80	36

Table 4.4 : The length of the conductive ink based on the % strain applied

In Table 4.5 shows the sheet resistance obtain by the 4 point probe from the each sample from various thicknesses (2, 4, 6, 8 layers) and various strain applied (0, 20, 40, 60, 80 %). The data from the table was tabulated in form of graph in Figure 4.5.

Number	Thickness	Percentage of Strain, %				
of Layer	( <b>mm</b> )	0	20	40	60	80
2	0.08	463.50	1151.43	2815.86	3592.95	-
4	0.16	263.60	520.01	738.19	1955.68	2652.33
6	0.24	194.57	458.75	732.25	1151.01	1537.93
8	0.32	155.74	332.47	567.92	1176.15	2430.61

Table 4.5 : CASE 3 : Mechanical test data



Figure 4.6 : CASE 3 : Effect of strain on sheet resistance with different layers of ink

From the observation it can be seen that the pattern of the graph is as the higher percentage of strain applied to the sample, the higher the resistivity obtain. Referring to the 2 layers graph line when there is no strain applied to the sample the value is 463.50  $\Omega$ /sq but when 20 % of the strain applied on it the resistivity increase tremendously to 1151.43  $\Omega$ /sq and the strain applied to the 2 layers sample was increased until 60 % strain applied with the reading of 3592.95  $\Omega$ /sq. The resistivity keep increase because the surface of the conductive ink begin to showing small crack on it. This crack on the conductive ink will disturb the flow of the electricity along the path which increase the resistivity. But there is no problem for the TPU to handle such strain because the TPU can be stretched 300 % from its original length (Mosallaei et al., 2017).

The 2 layers sample begin to be unreadable when 80 % strain applied to it. There was no data obtained on the sample, this is due to the conductive ink breaking apart from each other forming separated straight line which making the 4 point probe not be able to obtain any reading from the sample. Referring to diagram below it can be seen that the conductive ink separated into few straight line that not allowing the 4 point probe to take reading on the conductive ink.

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Figure 4.7 : 80 % strain applied on 2 layers sample

As the thickness increase the resistance is reducing but when the strain applied to the sample the resistivity will increase rapidly in all thickness. Referring to 8 layers sample which is the yellow line in the graph, the sheet resistance when there is no strain applied on the sample is 155.74  $\Omega$ /sq and when the strain increase to 20 % the resistivity drop to 332.47  $\Omega$ /sq. The resistivity of the sample with 8 layers steadily increasing each time until 60 % strain applied on it with the reading of 1176.15  $\Omega$ /sq the sample resistivity rapidly increase and producing higher resistivity than 6 layer sample when it is 60 % strain applied.

The reason is due to the method in handling the Vernier caliper when stretching the sample which is categories in human error. But basically the pattern of the result can be explain that the thicker the conductive ink, the higher strain can be applied on the sample since in this study only 2 layers sample obtained unreadable data when 80 % strain applied. As for the pattern

of the resistivity against the percentage of strain applied it can be said that the higher strain applied to the sample, the higher resistivity obtained by the sample.



Figure 4.8 : (A) 60 % strain applied on 6 layers sample and (B) 60 % strain applied on 8 layers

sample.

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Based on the observation of the figure above it can be said that the condition of the conductive ink in the 8 layers sample was far better compared to the 6 layers sample, yet the resistivity obtained from the 8 layers sample far greater than the 6 layers. This result can be due to unobservable micro crack occuring on the surface of the 8 layers conductive ink. The crack is too small that can't be seen through naked eyes. But the micro crack greatly affecting the flow of the electricity on the conductive ink that resulting increase of resistivity reading (Merilampi et al., 2010).

The result is acceptable since the pattern of the graph is similar as the result obtained from other researcher which is when the higher strain applied, the resistivity will also increase. The Figure 4.7 is the result obtained by Merilampi and his friends in his study



#### **CHAPTER 5**

### CONCLUSION AND RECOMMENDATION

### 5.1 Conclusion

In conclusion the effect of line thickness cross-sectional geometry to stretchable printed circuit under thermal performance was investigated. From the data obtain can be concluded that there were three method to control the resistivity of the SPC besides changing the conductive ink used. The first method to control the resistivity is change the thickness of the ink. As the thickness of the ink increasing, the resistivity will decrease. The reason of the resistivity drop is due to the increase of cross-sectional area of the ink that allowing more electricity to flow inside the ink which reducing the resistivity. The second method to control the resistivity is by controlling the temperature applied on the samples. When a higher temperature applied on the sample or more accurate on the conductive ink, the resistivity will become lower. The reduction in the resistivity is because of the increase of contact area between particle inside the conductive ink which allowing more current to flow through the ink track and made the current flow more smoothly. The last method to control the resistivity is by controlling the strain applied to the sample. When the sample applied on a strain, the higher the strain applied on it, the higher resistivity obtain by the sample. This is due to the cross-sectional area of the ink reduce since the original length of the conductive ink increase which will reducing the width and the thickness of the ink when more strain applied to the sample. The second reason is due to the breakage and crack produce on the conductive track. When higher strain applied to the sample, the conductive ink begun to produce crack on the surface of the ink which disturbing the flow of the current on the conductive ink that causing the resistivity reading of the sample increase.

### 5.2 **Recommendation for Future Works**

The study of the conductive ink quite active nowadays since the demand of conductive ink increasing day by day. The conductive ink only widely used for research purpose so there are several new finding about the conductive ink. So the potential of the conductive ink not been fully explored yet due to certain reason such as the availability of machine needed and limited resources. For example in this study, the preparation of mechanical test, there is no proper stretchable jig for stretching the sample. Instead using proper equipment such as the stretchable jig, we use Vernier caliper to stretch the sample. The use of proper equipment can produce more accurate result and much more trusted. So in the future the usage of the proper equipment is a must. Hence the fabrication of stretchable jig is needed in order to continue and further exploring the capabilities of the conductive ink in proper and more accurate way.

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This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: F390 - 11

# Standard Test Method for Sheet Resistance of Thin Metallic Films With a Collinear Four-Probe Array<sup>1</sup>

This standard is issued under the fixed designation F390; 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 ( $\epsilon$ ) 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  $\mu$ m 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<sup>-2</sup> to 10<sup>-4</sup>  $\Omega/\Box$  (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.

#### Referenced Documents 2.1 ASTM Standards:<sup>2</sup> VERSITI TEKNIKAI

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

F388 Method for Measurement of Oxide Thickness on Silicon Wafers and Metallization Thickness by Multiple-Beam Interference (Tolansky Method) (Withdrawn 1993)<sup>3</sup>

#### 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  $10^{-3}$   $\Omega$ -cm.

3.1.3 sheet resistance,  $R_s[\Omega/\Box]$  — 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  $\mu$ m. In all cases all of

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<sup>&</sup>lt;sup>1</sup>This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputter Metallization.

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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Withdrawn. THe last approved version of this historical standard is referenced on www.astm.org.

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the four paths from the electrical measurement equipment inputs to the film surface must be identical.

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  $\pm 10$  % 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  $\pm 0.1$  % 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  $\pm 0.1$  % 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 E2251.

#### 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  $\mu$ m and less than 100  $\mu$ m. Thickness variation shall be less than  $\pm 10 \%$  of the nominal thickness for thickness from 0.01  $\mu$ m to 0.1  $\mu$ m, inclusive; for greater thicknesses, the variation shall be less than  $\pm 5 \%$  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 F388.

#### 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:  $S_{ij} = [(C_j + D_j)/2] + [(A_j + B_j)/2],$ 

$$\begin{split} S_{2j} &= \big[ (E_j + F_j)/2 \big] - \big[ (C_j + D_j)/2 \big], \text{ and} \\ S_{3j} &= \big[ (G_j + H_j)/2 \big] - \big[ (E_j + F_j)/2 \big] \end{split}$$

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  $S_{ii}$  calculated above and the equation:

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



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

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

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

7.1.2.4 Calculate the average probe spacing S as follows:

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

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

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

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  $S_i$ . 7.1.3.2 The average values of the separations  $(S_1, S_2, \text{ and } S_3)$  shall not differ by more than 5 % of  $S_i$ .

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:

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

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:

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

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

1.2.3.2 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  $(23\pm 2^{\circ}C)$  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  $\pm 2^{\circ}$ . Lower the probe onto the test specimen ensuring that the center of the probe array is centered on the specimen within  $\pm 10 \%$  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:

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

9.3 Calculate the sample standard deviation as follows:

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

9.3.1 Requirement—For acceptance of the resistance, the sample standard deviation s shall be less than 1 % of R.

9.4 Calculate the ratio of the specimen width w (see 6.2) to the average probe separation 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/S	ℓ /w = 1	ℓ /w = 2	ℓ /w = 3	ℓ /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
00	4.5324	4.5324	4.5324	4.5324

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9.5 Calculation the ratio of the film thickness *t* (see 6.3) to the average probe separation  $\overline{S}$  (see 7.1.2.4). Find the correlation factor F(t/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

#### TABLE 2 Thickness Correction Factor for Thin Films

t/S	F(t/S)
0.4000	0.0005
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

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.

#### 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  $\pm 0.44$  % of the average value for sheet resistance values in the range from 25 to 40  $\Omega/^2$  and  $\pm 1.7$  % 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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### APPENDIX B Razor Straight Line Printing Method Standard



# Standard Test Method for Volume Resistivity of Conductive Adhesives<sup>1</sup>

This standard is issued under the fixed designation D2739; 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 (a) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 This test method covers the determination of the volume resistivity of resin-based conductive adhesives in the cured condition. The test is made on a thin adhesive layer as prepared in a bonded specimen. This test method is used for conductive adhesives that are cured either at room temperature or at elevated temperatures.

1.2 The values stated in either SI or other units shall be regarded separately as standard. SI equivalents to screw threads are shown in the figures.

1.3 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

2.1 ASTM Standards;<sup>2</sup>
D618 Practice for Conditioning Plastics for Testing KAL
D907 Terminology of Adhesives
2.2 Federal Specification:
QQ-B-626 Composition 22<sup>3</sup>
2.3 ASTM Adjuncts:
Assembly Jig<sup>4</sup>

#### 3. Terminology

#### 3.1 Definitions:

3.1.1 Many terms in this test method are defined in Terminology D907.

3.1.2 conductivity, n—the ratio of the current density carried through a specimen to the potential gradient paralleling the current. This is numerically equal to the conductance between opposite faces of a unit cube of liquid. It is the reciprocal of resistivity. **D2864, D27** 

3.1.3 resistivity, volume, n—the ratio of the electric potential gradient to the current density when the gradient is parallel to the current in the material. D1566, D11

#### 4. Summary of Test Method

4.1 The volume resistivity of adhesive layers cured between metal adherends is measured on a resistance bridge. Tensile adhesion plugs (Fig. 1)<sup>5</sup> are described in this test method. Any other test specimens and materials can be used as long as similar precautions (see Section 7) are observed regarding preparation and tolerances.

#### 5. Significance and Use - and 9

5.1 Accurate measurement of the volume resistivity of conductive adhesives is important, particularly with respect to applications in electronic packaging techniques. This method measures the resistance of conductive adhesives used in thin films as part of a bonded assembly. This does not imply that the measured results are applicable to different configurations with different metals. This method may be used for acceptance testing and for screening materials.

#### 6. Apparatus

6.1 Kelvin (Resistance) Bridge, calibrated to 1 % accuracy.6

6.2 With the agreement of the interested parties, any metal tensile adhesion plugs (Fig. 1) can be used to prepare the tensile adhesion specimens.

Note 1-Different metals will inherently provide different resistance

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<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

Current edition approved Nov. 1, 2017. Published November 2017. Originally approved in 1968. Last previous edition approved in 2010 as D2739 – 97 (2010). DOI: 10.1520/D2739-97R17.

<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from DLA Document Services, Building 4/D, 700 Robbins Ave., Philadelphia, PA 19111-5094, http://quicksearch.dla.mil.

<sup>&</sup>lt;sup>4</sup> Detailed drawings of the assembly jig are available from ASTM International Headquarters. Order Adjunct No. ADJD2739. Original adjunct produced in 1987.

<sup>&</sup>lt;sup>5</sup> Plugs to accommodate banana plug-No. 192, Herman H. Smith, Inc., or equivalent.

<sup>&</sup>lt;sup>6</sup> Satisfactory resistance bridges are made by: Leads and Northup Co. Bridge Catalog No. 4306, Minneapolis Honeywell Division Catalog No. 1622, and Biddle Instruments Catalog No. 603282.
## APPENDIX B Razor Straight Line Printing Method Standard



FIG. 1 Brass Tensile Adhesion Specimens with Electrical Connections

values. The measured resistance is dependent on resistance at the adhesive-adherend interface due to metal oxide formation. The extent of oxide formation varies with locality and laboratory conditions. Brass, conforming to Federal Specification QQ-B-626 Composition 22, is a convenient metal. However, in order to minimize oxide formation, especially where measurements are critical, as in referee measurements, it is recommended that the metal plugs be plated with either gold or silver to a thickness of not less than 1 µm (0.000040 in.). Any size plug up to 30 mm in diameter can be used with the aid of an alignment jig.<sup>4</sup>

## 7. Test Specimen

7.1 Thoroughly abrade the face of the specimen to be bonded (other than gold- or silver-plated) with crocus cloth 452<sup>7</sup> or equivalent, and wipe with clean solvent, such as reagent-grade methyl ethyl ketone, immediately prior to bonding.

7.2 Mix the adhesive to be tested in accordance with the manufacturer's instructions, taking care to mix in as little air as possible.

7.3 Coat sufficient adhesive on the surface to be bonded to ensure uniform squeeze-out around the edge of the bonded area. Remove excess adhesive, controlling the bond thickness as shown on an assembly jig.<sup>4</sup>

7.4 Prepare five specimens each for the following two thicknesses: (1)  $0.13 \pm 0.02$  mm, and (2)  $0.51 \pm 0.01$  mm.

7.5 Cure the adhesives in accordance with the manufacturer's instructions. Curing under other conditions of time and temperature is acceptable by an agreement of the interested parties.

#### 8. Preparation of Apparatus

8.1 Before application of the adhesive, set the adhesive thickness by tightening plugs in place, using a feeler gauge to determine the clearance between plugs.

8.2 Bottom out the top plate on the guide post before and after application of the adhesive.

8.3 After the adhesive has been cured, remove or loosen (at a temperature close to the highest temperature of cure) any remaining shims, screws, or devices of this nature, so that the adhesive layer cools in an unrestrained condition.

## 9. Conditioning

9.1 Condition the cured test specimens at  $23 \pm 1^{\circ}$ C (73.4  $\pm$  1.8°F) and 50  $\pm$  5 % relative humidity for at least 88 h prior to the test.

9.2 Test at the conditions specified in 8.1 above.

9.3 If conditions other than those given herein are needed, it is suggested that they be selected from those given in Practice D618.

### 10. Procedure

10.1 Make contacts (banana-plug type) so that when the specimen is in the measuring circuit, the potential leads are inside the current leads (for example, potential lead connections are closer to the adhesive bond) (Fig. 1). Make the measurements with the specimen in a stress-free position, since semirigid and flexible formulations are very sensitive to stress change during measurements.

<sup>&</sup>lt;sup>7</sup> Crocus cloth 452 is available from the Carborundum Co., Niagara Falls, NY.

## **APPENDIX B Razor Straight Line Printing Method Standard**

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#### 10.2 Measure and record the resistance.8

Note 2-If the resistance value of the adhesive layer is greater than  $10^{-4} \Omega$ , the resistance of the plugs can be ignored and the total resistance taken as the resistance of the adhesive layer. The resistivity of copper, silver, and brass are listed for comparison.9 Metal

#### Volume Resistivity at Temperature

Copper	1.729 × 10 <sup>-6</sup> Ω⋅cm at 20°C
Silver	1.629 × 10 <sup>-6</sup> Ω⋅cm at 20°C
Brass	3.65 × 10 <sup>−6</sup> Ω.cm at 0°C

#### 11. Calculation

11.1 Calculate the volume resistivity of the test specimen as follows:

$$V = R_0 A / L \tag{1}$$

where:

V = volume resistivity,  $\Omega \cdot m$ ,

- $R_0$  = observed resistance of the test specimen,  $\Omega$  (as measured with the bridge), corrected for any error found in the calibration of the bridge, including the resistance metallic plugs cleaned as specified in Section 7,
- = cross-sectional area of the test specimen, m<sup>2</sup>, and A
- = thickness of adhesive layer, m. L

8 A general description of the Kelvin resistance bridge is found in Stout, M. B., Basic Electrical Measurements, Par. 4.21 to 4.26, Prentice Hall, Inc., April, 1961. 9 American Institute of Physics Handbook, Second Edition, Gray, E. H., Ph.D., ed., 1957.

#### 12. Report

12.1 Report the following information:

12.1.1 Complete identification of the adherend and adhesive materials tested, including adhesive type, source, and manufacturer's code number,

12.1.2 Curing conditions used,

12.1.3 Conditioning environment and test environment,

12.1.4 Number of specimens tested per sample,

12.1.5 Dimensions of each test specimen including adhesive line thickness,

12.1.6 Corrected resistances of each test specimen,

12.1.7 Volume resistivity, in ohm-centimeters, of each test specimen,

12.1.8 Average volume resistivity of the sample, when more than one test specimen per sample is used, and

12.1.9 Standard deviation of the volume resistivity measurements when more than one test specimen per sample is used.

#### 13. Precision and Bias

13.1 Precision and bias have not been determined for this test method.

## 14. Keywords

14.1 adhesive; conductive adhesive; conductivity; resistivity; volume resistivity

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# APPENDIX C Bare Carbon Conductive Ink Datasheet



#### 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.



<ul> <li>High resistivity</li> </ul>	
Nontoxic	
Water-soluble	
<ul> <li>Can be used to create cap</li> </ul>	acitive touch and proximity sensors
<ul> <li>Can be used as a potentio</li> </ul>	meter or resistive circuit element
<ul> <li>Compatible with many sta</li> </ul>	ndard printing processes
TYPICAL PROPERTIES	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 minut Drying time can be reduced by placing Electric Paint under a warm lamp or c

#### See below summary table of typical properties. UNIVERSITI TEKNIKAL MALAYSIA MELAKA 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 /	$32\Omega/Sq$ 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.

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