

SUPERVISOR DECLARATION

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

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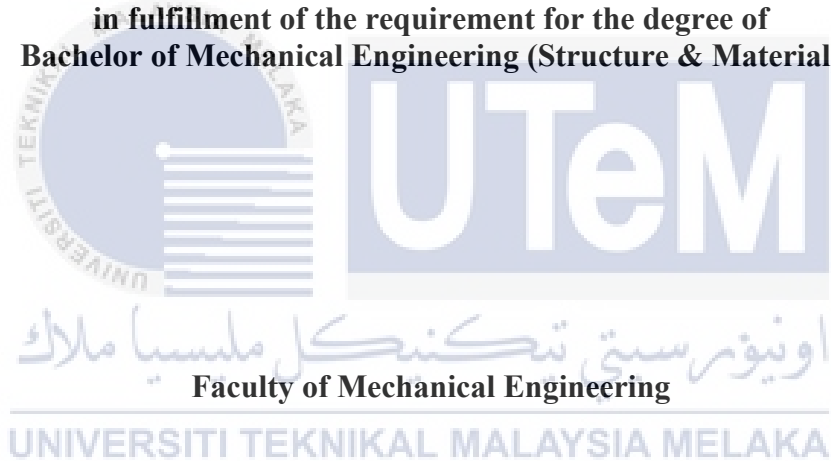
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**EFFECT OF STANNUM ON THE PROPERTIES OF
GRAPHITE/STANNUM/POLYPROPYLENE COMPOSITE FOR BIPOLAR
PLATE**

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**This report is submitted
in fulfillment of the requirement for the degree of
Bachelor of Mechanical Engineering (Structure & Material)**



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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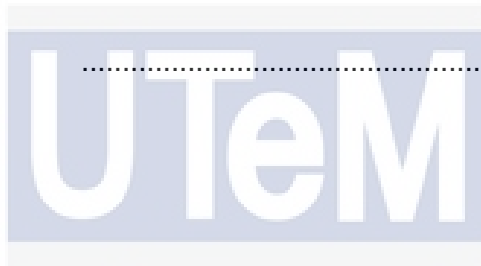
DECLARATION

I declare that this project report entitled “Effect of Stannum on the Properties of Gr/Sn/PP Composite of Bipolar Plate” is the result of my own work except as cited in the references

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DEDICATION

Expecially for my father, Mohd Nasir Bin Abd Kadir and my mother, Kasmawati Binti



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ABSTRACT

In this research, the high performance bipolar plate were fabricated by using compression molding. In polymer electrolyte membrane fuel cell (PEMFC), bipolar plate is considered one of the most important components which takes a large portion of stack stock. It has a multi-functional in the fuel cell stack. The fuel cells are the most promising power source in the future because it can provide good energy efficiency, little pollution and little noise. In this project, the raw materials used to fabricate the high performance bipolar plate are Graphite (Gr), Stannum (Sn) and Polypropylene (PP). The Gr and Sn will be used as fillers and PP will be used as binder (Resin). The composition ratio of fillers: binder will be fixed 80%; 20%. But for main-conductive filler ratio of Gr will vary from 70%, 65% and 60%. Meanwhile, the second conductive filler, Sn will vary from 10%, 15% and 20% from the total percentages of fillers (80%). The Polypropylene (PP) will be pulveriser and sieve in order to get 500 μ sizes. The fillers material will be mixed with binder material by using the ball mill machine before shaped by using the hydraulic compression molding machine with three different temperature such as 165°C, 170°C and 175°C. The effect of Sn loading and temperatures on the electrical conductivity and mechanical properties of GR/Sn/PP composite have been evaluated. The result shows that density and hardness have meet DoE standard and just electrical property and flexure strength did not meet the DoE, thus due to agglomeration complication and PP does not distribute well in bipolar plate. The maximum value of all testing result are electrical conductivity is 49.65 S/cm, flexural strength is 15.46 MPa, bulk density is 1.960 g/cm³ and shore hardness is 61.

ABSTRAK

Dalam kajian ini, prestasi tinggi plat dwikutub telah direka dengan menggunakan acuan mampatan. Dalam polimer sel bahan api membran elektrolit (PEMFC), plat dwikutub adalah merupakan sebagai salah satu komponen yang paling penting yang menyumbang sebahagian besar berat tindanan. Ia mempunyai pelbagai fungsi dalam tindanan sel bahan api. Sel bahan api adalah sumber kuasa yang paling utama di masa akan datang kerana ia boleh memberikan kecekapan tenaga yang baik, pencemaran sedikit dan bunyi sedikit. Dalam projek ini, bahan-bahan mentah yang digunakan untuk fabrikasi plat dwikutub berprestasi tinggi adalah grafit (Gr), Stannum (Sn) dan Polypropylene (PP). Gr dan Sn akan digunakan sebagai pengisi dan PP akan digunakan sebagai pengikat (Resin). Nisbah komposisi pengisi: pengikat ditetapkan pada 80%; 20%. Tetapi untuk nisbah pengisi konduktif utama Gr adalah 70%, 65% dan 60%. Sementara itu, pengisi konduktif kedua Sn adalah 10%, 15% dan 20% daripada jumlah peratusan total pengisi (80%). Polypropylene (PP) dikisar dan ayak untuk mendapatkan 500 mikron saiz. Bahan pengisi dicampurkan dengan bahan pengikat dengan menggunakan mesin kempa bola sebelum dibentuk dengan menggunakan mesin pengacuan mampatan hidraulik dengan tiga suhu yang berbeza iaitu 165°C, 170°C dan 175°C. Kesan bebanan Sn dan suhu terhadap kekonduksian elektrik dan sifat mekanikal Gr / Sn / PP komposit telah dinilai. Hasil kajian menunjukkan bahawa sifat ketumpatan dan kekerasan telah memenuhi piawaian DoE dan hanya elektrik dan kekuatan lenturan tidak memenuhi DoE, itu disebabkan oleh penumpuan komplikasi dan Sn tidak tertabur dengan baik dalam plat dwikutub. Nilai maksimum sampa hasil ujian adalah kekonduksian elektrik adalah 49.65 S/cm, kekuatan lenturan adalah 15.46 Mpa, ketumpatan pukal adalah 1,960 g/cm³ dan pantai kekerasan adalah 61.

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LIST OF ABBEREVATIONS

PEMFC	Polymer Electron Membrane Fuel Cell
CPCs	Conductive Polymer Composites
US-DoE	United State Department of Energy
Gr	Graphite
Sn	Stannum
PP	Polypropylene
ICP	Impact Copolymer
HPP	Homo-polymer Polypropylene
ASTM	American Society for Testing and Materials
S/cm	Siemen per Centimeter
Mpa	Mega Pascal
g/cm^3	Gram per <i>centimeter</i> ³
μm	Micron Meter
μA	Micron Ampere

CHAPTER 1

INTRODUCTION

1.1 FUEL CELL

A fuel cell is a device that uses an electrochemical reaction to directly convert the chemical energy of a fuel into useable electricity. It converts hydrogen and oxygen into water and this process also creates the electricity. Some other hydrocarbon such as natural gas and alcohol are also used as the fuel for fuel cell. In the fuel cell, the hydrogen gas is reacted with the oxidizing agent or oxygen to form water. It requires a constant sources of fuel and oxygen to produce the electricity continually as long as the sources are supplied constantly. Beside that, producing energy with water is an attractive concept behind fuel cell. The function is by combining hydrogen with oxygen as the simple reaction (Elizabeth and Condcliffe, 2002).

The fuel cell are also known as the promising power source in future for residential, mobile and automotive application. It can provide good energy efficiency over 40%, little pollution and little noise (Lee et al, 2007). Fuel cells are extremely attractive from an environmental standpoint because electrical energy is generated without combusting fuel. Fuel cells are known as great potential to be low emission power generation sources in the future due to its attractive characteristics such as high energy conversion efficiency (Antunes et al, 2011) very low chemical and acoustical pollution (Kuan et al, 2004).

Fuel cells works much like a battery, except it did not require electrical recharging. A battery stores all of its chemicals inside and converts the chemicals into electricity. Once those chemicals run out, the battery dies. On the other had, fuel cell gets the chemicals it utilizes from the outside in this way, it did not run out. Fuel cells can generate power almost indefinitely, as long as they have fuel to use. In the future, fuel cells are known as the most

potential power sources for residential, mobile and automotive applications. Fuel cells have the potential to replace the internal combustion engine in vehicles and provide power in stationary and portable power applications because they are energy efficient, clean, and fuel flexible (Muller et al, 2006).

By the same principle of producing hydrogen with oxygen to form water, Table 1.1 below has shown details each five types different of fuel cells have developed including typical size, efficiency, advantages and disadvantages.

Fuel Cell Type	Common Electrolyte	Operating Temperature	Typical Stack Size	Efficiency	Applications	Advantages	Disadvantages
Polymer Electrolyte Membrane (PEM)	Perfluoro-sulfonic acid	50-100°C 122-212°F typically 80°C	1 kW-100 kW	60% Transportation 35% stationary	<ul style="list-style-type: none"> • Backup power • Portable power • Distributed generation • Transportation • Specialty vehicles 	<ul style="list-style-type: none"> • Solid electrolyte reduces corrosion & electrolyte management problems • Low temperature • Quick start-up 	<ul style="list-style-type: none"> • Expensive catalysts • Sensitive to fuel impurities • Low temperature waste heat
Alkaline (AFC)	Aqueous solution of potassium hydroxide soaked in a matrix	90-100°C 194-212°F	10-100 kW	60%	<ul style="list-style-type: none"> • Military • Space 	<ul style="list-style-type: none"> • Cathode reaction faster in alkaline electrolyte leads to high performance • Low cost components 	<ul style="list-style-type: none"> • Sensitive to CO₂ in fuel and air • Electrolyte management
Phosphoric Acid (PAFC)	Phosphoric acid soaked in a matrix	150-200°C 302-392°F	400 kW 100 kW module	40%	<ul style="list-style-type: none"> • Distributed generation 	<ul style="list-style-type: none"> • Higher temperature enables CHP • Increased tolerance to fuel impurities 	<ul style="list-style-type: none"> • Pt catalyst • Long start up time • Low current and power
Molten Carbonate (MCFC)	Solution of lithium, sodium, and/or potassium carbonates soaked in a matrix	600-700°C 1112-1292°F	300 1 kW-3 MW 300 kW module	45-50%	<ul style="list-style-type: none"> • Electric utility • Distributed generation 	<ul style="list-style-type: none"> • High efficiency • Fuel flexibility • Can use a variety of catalysts • Suitable for CHP 	<ul style="list-style-type: none"> • High temperature corrosion and breakdown of cell components • Long start up time • Low power density
Solid Oxide (SOFC)	Yttria stabilized zirconia	700-1000°C 1292-1832°F	1 kW-2 MW	60%	<ul style="list-style-type: none"> • Auxiliary power • Electric utility • Distributed generation 	<ul style="list-style-type: none"> • High efficiency • Fuel flexibility • Can use a variety of catalysts • Solid electrolyte • Suitable for CHP & CHHP • Hybrid/ST cycle 	<ul style="list-style-type: none"> • High temperature corrosion and breakdown of cell components • High temperature operation requires long start up time and limits

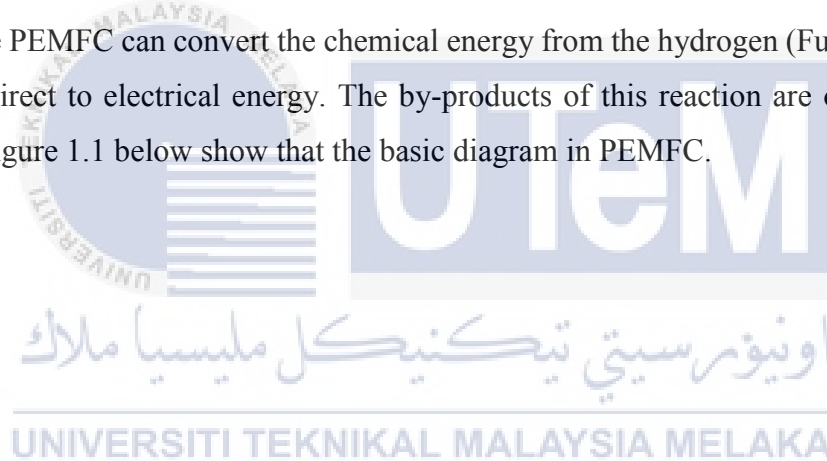
Table 1.1: Details on each type of Fuel Cell (Olsen, 2013)

1.1.1 Polymer Electrolyte Membrane Fuel Cell

Polymer Electrolyte Membrane Fuel Cells (PEMFC) are power generating devices that use an electrochemical reaction to change the energy from hydrogen fuel into usable power. It also known as fuel cell where electrolyte is made of an organic polymer that has the characteristic of a good proton carrier when in presence of a water solution (Costal et al, 2006).

The PEMFC are considered as for commercialization for portable and transportation applications because of their high energy conversion efficiency, low temperature, high power density and low pollutant emission. Besides that, cost and durability of PEMFCs are the two main challenges that must to be addressed to enable their commercialization (Maiyalagan and Pasupathi, 2010).

The PEMFC can convert the chemical energy from the hydrogen (Fuel) and oxygen (Oxidant) direct to electrical energy. The by-products of this reaction are only water and heat. The Figure 1.1 below show that the basic diagram in PEMFC.



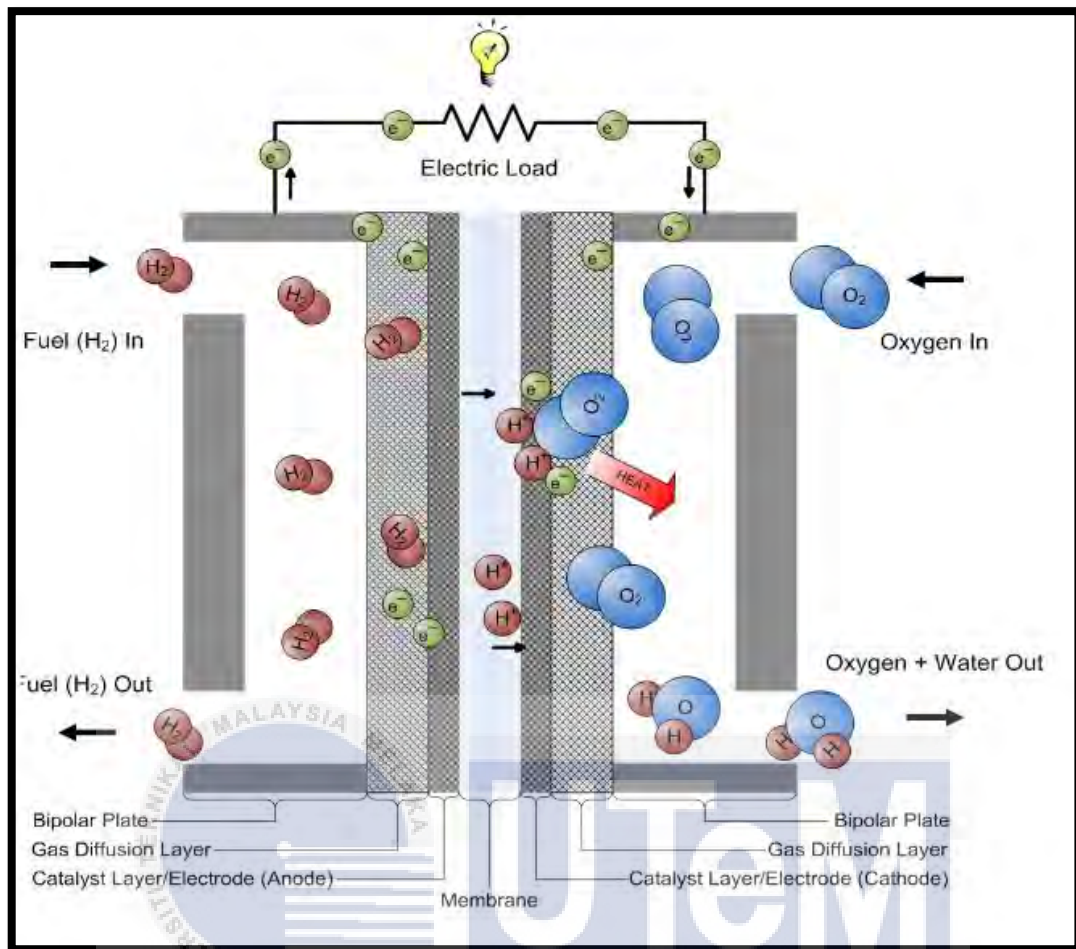


Figure 1.1: Basic Diagram of a PEMFC (Yan et al, 2015)

1.1.2 Components of Fuel Cell

The major components of the PEMFC are the Membrane Electrolyte Assembly (MEA), gasket, gas diffusion layer, bipolar plate, current collectors and endplates. The Figures 1.2 below shows the major components in PEMFC. Membrane Electrodes Assembly (MEA), bipolar plate, current collector and end plate are 4 main components of PEMFC.

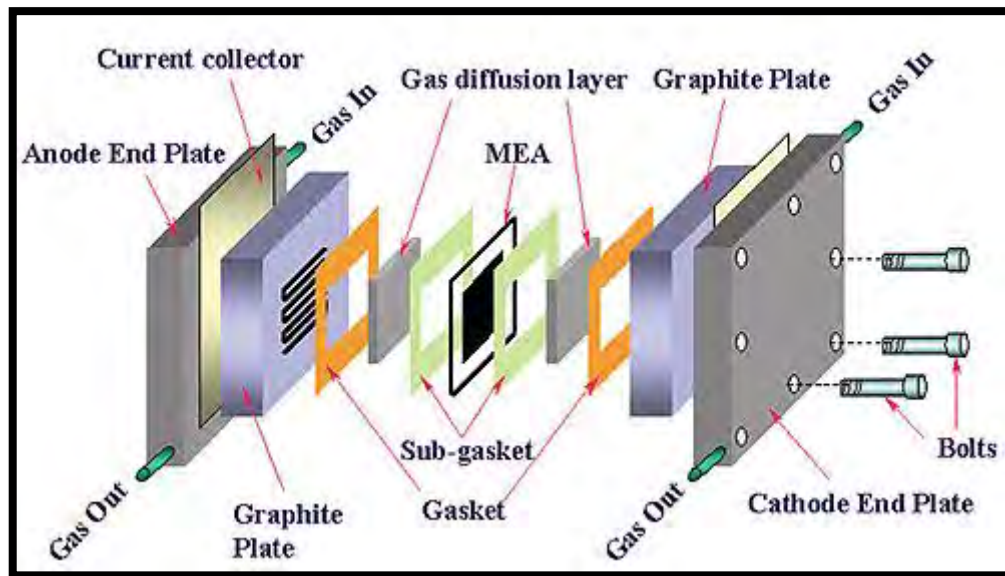


Figure 1.2: Major Components in the PEMFC

1.2 Problem Statement

Generally, in current fabrication of bipolar plate carbon based Gr, CB and CF, Gr and metal based Ferum are used as conductive fillers. Meanwhile PP is used as the binder. The main problem of metal is high tendency to corrosion, for pure graphite are too brittle and low in electrical conductivity, PP is non-conductive material and have a low strength. But through composite approach, the high performance bipolar plate can be produce. The composite approach is the use more than one conductive fillers to produce bipolar plate. But in fabrication of bipolar plate, not much research has been done to use the carbon based conductive fillers combined with metal based conductive fillers.

In this research, powder of PP is used as the binder and Gr and Sn as conductive fillers. Through composite approach, carbon based material Gr and Sn which this metal will be combine with PP to produce composite of Gr/Sn/PP. So the uses of PP can improved the brittle of graphite. However the combination of Gr/Sn/PP can increase the electrical of conductivity. The size of PP will be used is 500 μ m. To determine the effected of Sn loading, several related test such as conductivity, flexure test, density test, hardness and microstructure analysis will be performed.

1.3 Objective

The main objectives of this research are to study the effect of the Sn on the properties of Gr/Sn/PP composite for bipolar plates to be used in fuel cell. In this research is use as a main filler and powder of PP is used as the binder. The main objectives of the research are:

1. Study the effect of Sn loading on the properties of Gr/Sn/PP composite.
2. Determine the suitable temperature for Gr/Sn/PP composite through hot compression molding.

1.4 Scope of Project

This research will study the effect of Sn on the electrical and mechanical properties of Gr/Sn/PP composite. The ratio of filler (Gr/Sn) and binder (PP) is fixed at 80:20. The adding of small amount of SS in to Gr/Sn/PP composite thus will give synergy effect on electrical conductivity and mechanical properties. The small amount of Sn which is 10% is up to 20% (from the total weight of filler 80%) will be added into Gr/Sn/PP composite. Before the fabrication process using the hot press with the temperature up to 175°C, the mixture of Gr and Sn will be mixed used ball mill. In order to determine the effects of Sn, several size of PP powder (500µm) will be used in Gr/Sn/PP composite, the test such as electrical conductivity, flexure test, density test, hardness and microstructure analysis.

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CHAPTER 2

LITERATURE REVIEW

2.1 CONDUCTIVE POLYMER COMPOSITE

Conductive polymer composites (CPC) are formed by combination of conventional polymer with conductive polymers or filler allow creating new polymeric materials with unique electrical properties. Carbon black, graphite or other conductive filler are commonly used as a components of conductive polymer composites. A proper balance between electrical conductivity, mechanical properties and processing characteristic is an important requirement for a design of electroconductive thermoplastic composites (Omastova and Chodaka, 1999).

Futhermore, certain polymer are naturally conductive because they have conjugated chain structure and polymer like polyethlyne and polyproplene are not conductive because they do not have conjugated structure. They can become conductive by adding the conductive fillers like carbon black, graphite, metal and other conductive fillers. The factor that affecting the properties of a composites material are type of the fillers, filler size, filler dispersion, orientation of fillers in matrix, polymer matrix and some other factors. The most important characteristic to improve the properties of the composites materials is the interaction of fillers and polymer chain (Jianhua and Huang, 2005). The interaction takes place by attraction between the polymer chain and filler or chemical bonding between the polymers matrix and filler material. Chemical bonding between polymers matrix and filler materials can give the charge carriers move along polymer chain (Ahmad and Narissa, 2012).

2.1.1 Percolation Theory

The percolation theory explain about the composite electrical conductivity near the percolation rhesold value or critical volume Flory (1941) and Stockmayer (1943) have produced the percolation process to explain how small branching molecules react to form very big macromolecules (Stauffer, 1985). In an observation of electrical conduction in a polymer matrix, electron are able to move through conductive filler particles. When the fillere particles is in contact with one another for the electron to travel through. A continuous path can form through the polymer matrix and this path is known as conductive network. The materials with conductive network can turns to conductinbg material as shown in Figure 2.1

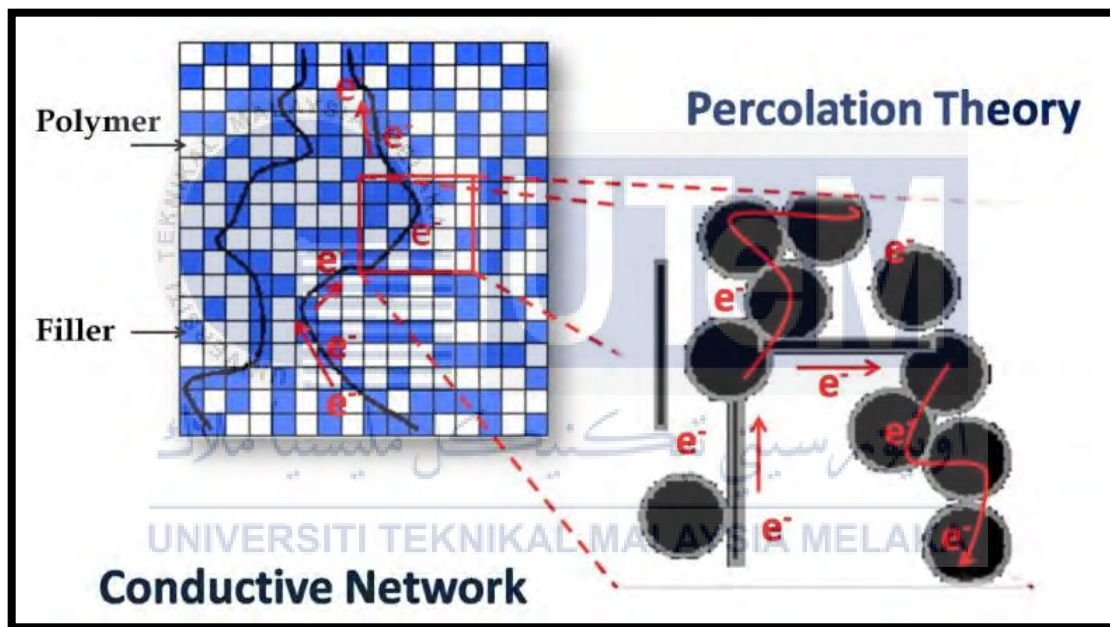


Figure 2.1 :Schematic of Percolation Pathway (Yeetsorn, 2010)

For the increment of conductive filler loading, as shown in Figure 2.2 the graph can divided into three main region to describe the relationship to the conductivity of conductive filled polymer composite. At region A which is a low filler loading, there are no path occurs for electron to transport and the electrical conductivity value is equal to zero. The composites is still like a pure polymer matrix. At a certain critical loading is known as the percolation threshold. Sufficient filler has been put so that the continuous conductive network is formed through the composite. Next to percolation threshold is a region with very slight increase in filler amount will produce a large increase in conductivity, as displayed by region B. After the region of accelerates increase, the conductivity decelerates its increase, and method that

of filler material as increase happens because the conductive network through the sample is complete. This is illustrated in area C. Finally, P_{max} is reached, at which point the electron movement do not increase with the addition of filler. The addition of more filler will not increase the conductivity to any significant amount (Renato et al, 2011).

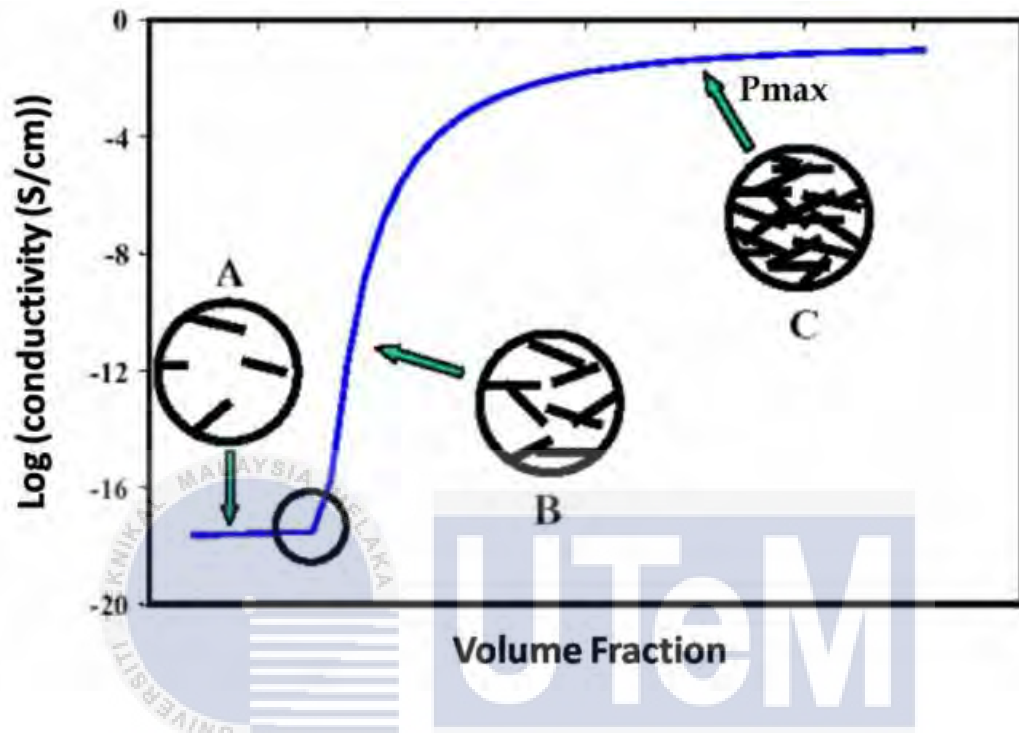


Figure 2.2: Percolation S-Curve (Mali, 2006)

At low filler loading, the conductivity remains very near to the conductivity of the pure polymer because the filler only occurs individually or in small clusters throughout the matrix. When the filler reaches the critical volume fraction or called percolation threshold, the conductivity of the composites will increase extremely with little increase of conductive filler. This is because the filler is enough to form inter-particles contact and create a percolating system. At high filler loading, the electrical conductivity will be plateau with increase filler concentration. The percolation value for every conductive composite system varies but share the same characteristic curve (Percolation s-Curve) (Clingerman et al, 2003).

2.2 BIPOLAR PLATE

The bipolar plate is a multi-functional component within a PEM fuel cell (Yuana and Zi, 2005). The Figure 2.3 show the graphite bipolar plate. Bipolar plates can be made from various materials with the most common being graphite, metal, carbon/carbon, and polymer composites. Each type of material has its strengths and weaknesses. (Baird et al, 2006) . These cells are composed of a sequence of units which each unit is assembled from membrane and bipolar plates. The main functionalities of these plates are uniform distribution of oxygen and hydrogen all over the effective surface of cell, conduction of electricity from one unit to another and transfer of produced heat from cells. In hydrogen fuel cells, hydrogen and oxygen are used to produce electricity, thus the plates must be properly sealed to prevent leakage of the gases to environment . There are some function of bipolar plates such as: (Zarmehri and Ehsan, 2013)

1. It connect and separate the individual fuel cells in series to form a fuel cell stack.
2. It distribute fuel gas and oxygen over the whole active surface area of the membrane-electrode assemblies
3. It conduct electrical current from the anode of one cell to the cathode of the next and also carry current away from the cell.
4. It facilitate water management within the cell
5. It support thin membrane and electrodes and clamping forces for the stack assembly

Bipolar plates are key components of PEM fuel cells. They are responsible for functions of vital importance to the long-term operation of these electrochemical devices. They play major roles in water and gas management, mechanical stability and electrical performance of fuel cells. Several types of materials are currently used in bipolar plates, including non-porous graphite plates, metallic plates with or without coating and a number of composite plates (Włodarczyk et al, 2012). The United States of America Department of Energy (US-DOE) has given a benchmark for the suitability of the bipolar plate in PEMFC. Based (US-DOE) show the requirement for composite must be meet the target (Antunes and Renato, 2011) as shown in Table 2.1

Table 2.1: Requirement for composite must meet the target (US-DOE)

Property	VALUE
Weight	$<0.4 \text{ kg kW}^{-1}$
Flexure Strength	$>25 \text{ MPa}$
Flexibility	3–5% deflection at mid-span
Electrical Conductivity	$>100 \text{ S cm}^{-1}$
Thermal Conductivity	$>10 \text{ W (m K)}^{-1}$
Gas permeability	$<2 \times 10^{-6} \text{ cm}^3 \text{ cm}^{-2} \text{ s}^{-1}$ at 80°C and 3 atm
Corrosion Resistance	$<1 \mu\text{A cm}^{-2}$
Low Bulk Density	$<5 \text{ g/cm}^3$
Shore Hardness	$>50 \text{ Mpa}$



Figure 2.3 : Bipolar Plate

Bipolar Plate make up about 70-80% of the stack weight and up to 45% of the production cost so it is the most important component of the fuel cell (Kakati, 2009). Bipolar plate has multi function in the fuel cell. It is used to distribute the oxygen to the cathode and the hydrogen to the anode, removing the water and heat from the reaction. Provides electrical contact between the plate to carry current from cell to cell and keep reactants separates. Many different types of material like graphite, metal or polymer composites with carbon or metal conductive filler can be used to make bipolar plate. (Huang and Jianhua, 2005). The Table 2.2 below shown the type of materials used to fabricate bipolar plate with its characteristic.

The thickness of the plate is another criteria need to be taken into consideration of design bipolar plate for the PEMFC because to minimize the stack volume and electrical resistance. The bipolar plate should be impermeable to reduce mixing of the oxidant and the fuel. The material must be corrosion resist due to the acidic environment present in fuel cell. It should also to be thermally conductive to remove the heat and electrically conductive to minimize Ohmic losses (Nunnerey, 1998). The bipolar plate should offer good mechanical strength and stiffness to support the electrolyte membrane and withstand the fastening force (Borup and Vanderbough, 1995). In order to meet design criteria, function and cost target for fabrication of the bipolar plate. The researcher are concentrating on the material selection to produce high performance bipolar plate.

Table 2.2: The type of Material Used To Fabricate Bipolar Plate with its Characteristic

Types of Material	Advantages	Disadvantages
Graphite	<ul style="list-style-type: none"> • Good electrical and thermal conductivity • Excellent chemical compatibility • Good corrosion resistance 	<ul style="list-style-type: none"> • Low mechanical strength • Difficult to machining the flow channel

Metal	<ul style="list-style-type: none"> • Good electrical and thermal conductivity • Good mechanical strength • Easy to fabricate and machining 	<ul style="list-style-type: none"> • Low resistance to corrosion in acidic condition • Aluminium and titanium and nickel bipolar plate need to be coated with protective layer to resist corrosion (high cost)
Polymer	<ul style="list-style-type: none"> • Low cost (compression molding) • Easy to create the flow channels • Better precision • Higher filler loading 	<ul style="list-style-type: none"> • Low electrical and thermal conductivity

2.2.1 Types of Bipolar Plates

There are many materials and methods for manufacture of bipolar plates. The most promising types and manufacturing methods of bipolar plates are described below. (Hermann et al, 2005) reviewed and discussed the different types of materials for the bipolar plate. Bipolar plate materials are broadly divided into metallic and carbon-based. Initially, carbon-based bipolar plates, particularly high-density graphite, dominated the R&D activities and other applications (Sheppard et al, 2001).

2.2.1.1 Graphite Bipolar Plate

The most commonly used bipolar plate material is graphite. Graphite plates exhibit excellent resistance to corrosion and low bulk resistivity (Chung, 2002). According to (Metha, 2003) Gr is the material has been usually in the manufacture of bipolar plates in the PEMFC because graphite has very good chemical stability to retain the fuel cell environment. Bipolar plates in the PEMFC have usually been made from Gr , since Gr has excellent chemical stability to survive the fuel cell environment. From the Table 2.3 shows the advantages and disadvantages of Gr plate (Yeetsorn and Fowler, 2011).

Advantages	Disadvantages
<ul style="list-style-type: none">• Good resistance	<ul style="list-style-type: none">• High costs
<ul style="list-style-type: none">• Low bulk resistivity	<ul style="list-style-type: none">• Difficult of machining
<ul style="list-style-type: none">• Low specific density	<ul style="list-style-type: none">• porosity
<ul style="list-style-type: none">• Low electrical contact resistance with electrode backing materials	<ul style="list-style-type: none">• Low mechanical strength• (brittleness)

Table 2.3: The advantages and disadvantages of graphite plate

2.2.1.2 Metallic Bipolar Plate

Metal sheets are preferable in industry due to strong mechanical properties, high electrical and thermal conductivity, and easy manufacturability into desired shapes. Various types of non-coated metal, metal alloy, and metal foams have been investigated for possible replacement of electro-graphite bipolar plates. They include aluminum, stainless steel, titanium, nickel, copper, and their alloys. In recent years, metallic bipolar plates have been attracting the attention of the research community because of their desirable characteristics,

such as high electrical conductivity, formability and manufacturability, gas impermeability, and superior mechanical properties (Hung et al, 2009). Metal plates offer higher strength, toughness and shock resistance than graphite plates, and their unique mechanical properties allow for fabrication of thinner plates. Although metals offer many advantages, they are, however, more susceptible to corrosion, which can adversely affect their performance and durability (Cheng et al, 2010). Table 2.4 below shown the advantages and disadvantages of metallic bipolar plate (Tawfik and Hung, 2012).

Table 2.4: Advantages and Disadvantage of Metallic Bipolar Plate

Advantages	Disadvantages
<ul style="list-style-type: none"> • Not porous • Good electrical conductivity and thermal conductivity • Good mechinability • Lighter than graphite bipolar plate • Durable 	<ul style="list-style-type: none"> • Easy to corrosion • Oxidising environment of the cathode compartment.

2.2.1.3 Polymer Composite Bipolar Plate

According to (Mahajan et al, 2007) Another material can offer more efficient in weight, corrosion resistant and cost in a machining is composite bipolar plate compared to the other bipolar plate. It consits of a polymer matrix with electrically conductive fillers. There are 2 types of polymers are usually used for polymer composite bipolar plate are thermoset and thermoplastic. Currently 70 to 90 wt% of a single type of graphite power in a thermosetting resin is ussualy used in fabricate of bipolar plate (Dweiri and Sahari, 2007).

The major advantages are that these are lower cost and lightweight, and are easily machined, with good corrosion resistance, relatively good mechanical properties, and good gas tightness. The major disadvantage is that polymers have extremely low electrical conductivity, so excessive conductive filler has to be incorporated. In this context, one needs

to remember that it is difficult to get high conductivity and sufficient mechanical properties simultaneously. Figure 2.4 below show the polymer composite bipolar plate.



Figure 2.4 : Polymer Composite Bipolar Plate

2.3 MATERIAL

The material that have been salected to investigate is Gr and Sn as conductive filler while the PP as the binder for bonding agent.

2.3.1 Graphite

According to (Plane, 2012) Gr as shown in Figure 2.5 is the most crystalline form of carbon, apart from diamond and fullerenes. It exhibits the properties both of metal such as thermal and electrical conductivity and of a non-metal such as inertness, high thermal resistance and lubricity.

According to (Chung, 2002) Gr is anisotropic, being a good electrical and thermal conductor within the layers (due to the in-plane metallic bonding) and a poor electrical and thermal conductor perpendicular to the layers (due to the weak van der Waals forces between the layers). The electrical conductivity enables graphite to be used as electrochemical electrodes and as electric brushes for (due to the in-plane covalent bonding) and weak perpendicular to the layers. As a result of this anisotropy, the carbon layers can slide with respect to one another quite easily, thus making graphite a good lubricant and pencil material.

According to (Howel, 1952), The structure, thermal properties, electrical characteristics, and mechanical behavior of graphite are reviewed and the highly anisotropic character of graphite crystals is emphasized. The interesting properties of graphite are due in large measure to the strong semimetallic bonds between carbon atoms in plane layers and to the relatively weak binding between planes. Details of its structure are due in part to the Frank dislocation mechanism for growth of crystals. Thus Gr has a low vapor pressure and approximately two-dimensional electrical and thermal conductivity. It is easily deformed in slip on the basal plane, but in polycrystalline form it is relatively strong at high temperature. Its lamellar nature is emphasized by its reaction with alkali metals and bromine.

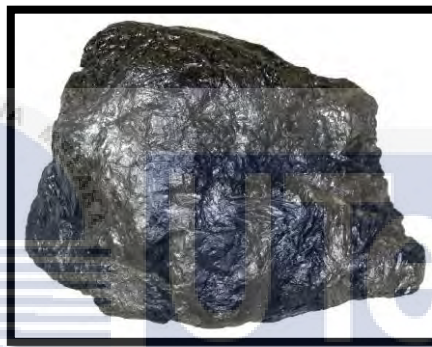


Figure 2.5: Graphite

2.3.2 Stannum

Tin or Stannum (Sn) shown in Figure 2.6 is an ancient metal. It is obtained chiefly from the mineral cassiterite, SnO_2 . Sn has long been used as a solder in the form of an alloy with lead, tin accounting for 5–70%. Such solders are used for joining pipes or electric circuits. Sn bonds readily to iron and is used for coating steel to prevent corrosion. Sn is a malleable, ductile silvery-white metal. It is nontoxic but certain organotin compounds are highly toxic (Habashi and Fathi, 2012).

Sn is silvery and malleable post transition metal. It is not easily oxidized in air and is used to prevent corrosion by coating other metal. Currently tin is used in many alloys most particularly tin/lead soft solder, normally containing 60% or more tin. Another large application of tin is corrosion-resistant tin plating of steel. Sn plated metal is used for packing because of its low toxicity and name by Sn can which made typically steel (Schwartz, 2002). Table 2.5 below shows the characteristics of Sn.

Table 2.5: The characteristics of Sn

Symbol	Sn
Atomic number	50
Group	14
Oxidation state	+2 – more stable +4
Abundant element	49 th
Stable isotopes	10

The physical properties of Stannum or Tin are shown in Table 2.6. Alloying elements like copper, antimony, bismuth, cadmium and silver to improve hardness. Tin tends rather easily to form hard, brittle intermetallic phase which are often unwanted. It does not form wide solid solution ranges in other metals in general and there are few elements that have appreciable solid solubility in tin (Molodets et al, 2000).

Element	Physical Properties
<ul style="list-style-type: none"> • Stannum or Tin(Sn) 	<ul style="list-style-type: none"> • Ductile • Malleable • Melting Temperature (232°C/449°F)

Table 2.6:Physical Properties of Stannum

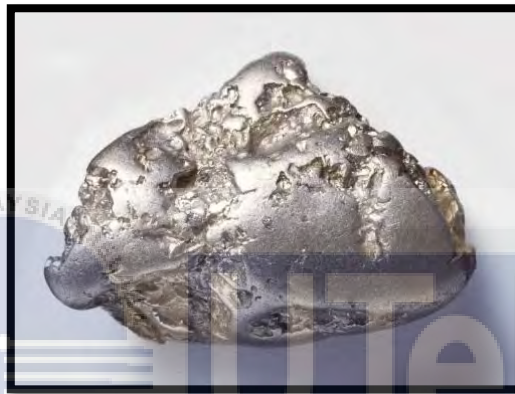


Figure 2.6: Stannum

2.3.3 Polypropylene

There is history on when the PP was found. The polymer is found around years 1954 and after discover the PP,grew a strong popularity in a fast time. PP is the commonly used as commercial polymer. The synthesis of the polypropylene is made by propylene gas at temperature between 50°C and 80°C. It was discovered in 1954 and gained a strong popularity very quickly due to the fact that PP has the lowest density among commodity plastics. PP is usually used polymer materials because of its physical,mechanical and thermal properties. It has very high melting point, low density and good impact resistance. It is used in many field examples like medicals, automotive and as electromagnetic interference (EMI) absorber material. The mechanical and electrical properties of PP can improve for other application.

PP has a relatively slippery surface which can make it a possible substitute for plastics like Acetal (POM) in low friction applications like gears or for use as a contact point for furniture. Perhaps a negative aspect of this quality is that it can be difficult to bond PP to

other surfaces (it does not adhere well to certain glues that work fine with other plastics and sometimes has to be welded in the event that forming a joint is required). Although PP is slippery at the molecular level, it does have a relatively high coefficient of friction - which is why acetal, nylon, or PTFE would be used instead. PP also has a low density relative to other common plastics which translates to weight savings for manufacturers and distributors of injection molded PP parts. It has exceptional resistance at room temperature to organic solvents like fats but is subject to oxidation at higher temperatures (a potential issue during injection molding).

PP has a crystalline structure with a high level of stiffness and a high melting point compared to other commercial thermoplastics. The Hardness resulted from the methyl groups in its molecular chain structure. PP is a lightweight polymer with a density of 0.90 g/cm^3 that makes it suitable in many industrial applications. Still, PP is not suitable to be used at temperatures below 0°C . Experiments proved that PP has excellent and desirable physical, mechanical, and thermal properties when used in room temperature applications. It is relatively stiff and has a high melting point, low density and relatively good resistance to impacts. Typical crystallinity of PP is between 40-60%. PP is a low-cost thermoplastic polymer with excellent properties like flame resistance, transparency, high heat distortion temperature, dimensional stability and recyclability making it ideal for a wide range of applications (Shubhra et al, 2011).

PP as shown in Figure 2.7 is a popular polymer due to its industrial interest, good availability, low cost, and wide range of physico-chemical properties which have been extensively utilized. It needs to be modified to improve its resistance to shock at low temperature, resistance to heat and especially its miscibility with other polymers (Weihua and Jingyuan, 1996).

It is a semicrystalline polymer that exhibits very attractive mechanical properties, like ductility and strength at room temperature or under moderate rates of deformation. However, under severe conditions it becomes brittle. This behavior makes it interesting for commercial and scientific fields to study methods for toughening these materials.(Eiras and Pessan, 2009).

In other hand, PP is semi-rigid, translucent, a good chemical resistance, tough, a good fatigue resistance and a good heat resistance. Moreover, PP has high softening or glass-transition point, high resistance to flexing stress, low water absorption, good electrical

resistance, a lightweight, dimensional stability, high impact strength and a non-toxicity property

Besides that, PP has an excellent chemical resistance and can be processed through many converting methods such as injection molding and extrusion. Generally, there are three different types of PP. First, PP containing only propylene monomer in a semi-crystalline solid form which is called a homo-polymer PP (HPP). Second, PP containing ethylene as a co-monomer in the PP chains at levels in the range of 1-8% and this is referred to as a random copolymer (RCP). Third, HPP containing a co-mixed RCP phase that has an ethylene content of 45-65% is referred to as an impact copolymer (ICP). PP chemical resistance can be described such as excellent resistance to dilute and concentrated acids, alcohols and bases, good resistance to aldehydes, esters, aliphatic hydrocarbons, ketones and limited resistance to aromatic and halogenated hydrocarbons and oxidizing agents (Maddah, 2016).



Figure 2.7: Polypropylene

2.4 FABRICATION METHOD

2.4.1 Compression Molding

Compression molding is a forming process in which a plastic material is placed directly into a heated metal mold, then is softened by the heat, and forced to deform to the shape of the mold as the mold closes (Todd et al, 1993). The Figure 2.8 below shows about the compression moulding.

The compression molding is favoured for both thermoplastic and thermoset matrix composite. There is different in the processing, the thermoset is cured by a chemical reaction where the thermoplastic material has to been procesed by heating to the melt phase and cooled in the mold. The thermoset has a shorter cycle time than thermoplastic material (Stubler et al, 2013).

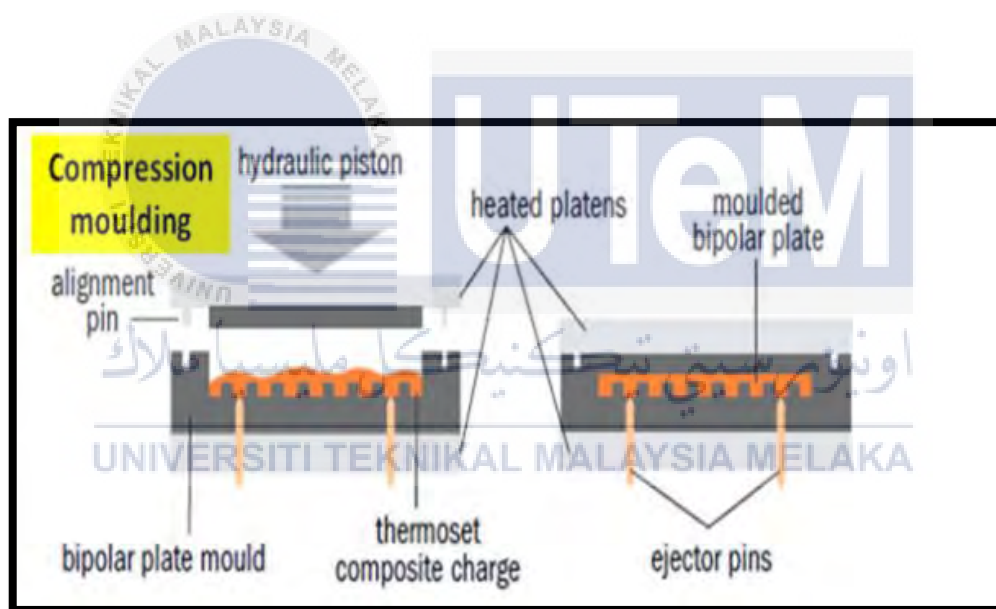


Figure 2.8: Compression Moulding

2.4.2 Injection Molding

The injection moulding process is one of the most important operations involved in polymer processing. The Figure 2.9 shows about the injection moulding. Higher viscosity of material has become the main concern for composite bipolar plate manufacturing. Injection molding of polypropylene has been recognized as one of most encouraging and low cost production process. Extrusion with appropriated die, rolling and thermoforming are other method to fabricating composite bipolar plate but injection molding is still the lower cost and most efficiency method (Hermann et al, 2005).

The mold regularly contains of two halves which are carried together, clamped into position and kept at a constant temperature. After that, hot molten plastic is then forced under pressure into the cooler mould. When the plastic has solidified, the clamps are released, the moulded object is ejected and the cycle is repeated. In this way, objects reaching in size from toy building blocks to car bonnets are produced (Whale et al, 1992).

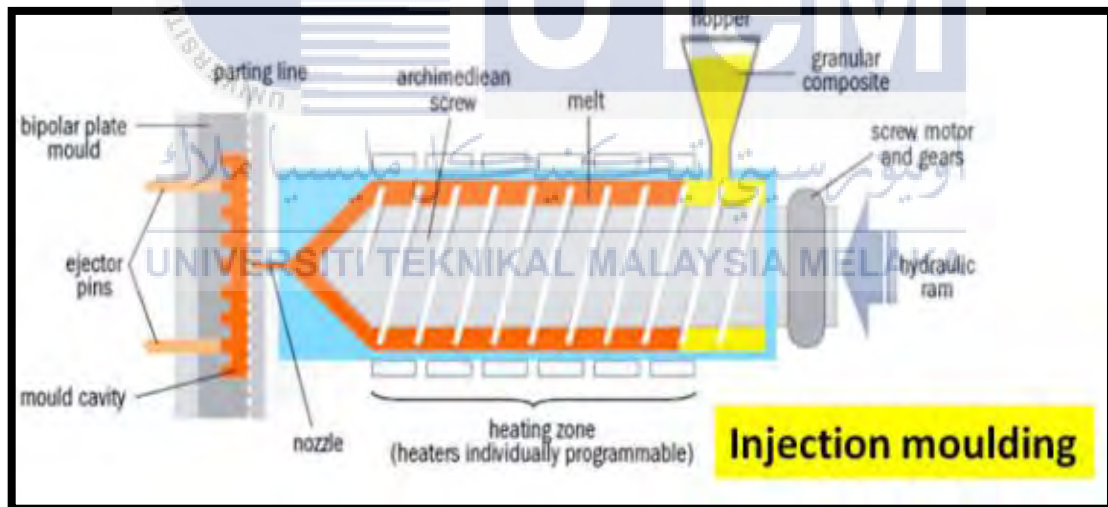


Figure 2.9: Injection Moulding

2.5 TESTING METHOD

2.5.1 Electrical Conductivity

Electrical conductivity is measured from resistance measurement of the composite plates. There are 2 types of resistance measurement generally performed for bipolar plates are in plane or bulk, example like along surface, through plane perpendicular to surface. Although much more difficult to develop on the technical point of view through planes resistant is directly related to the operating condition and should be preferred (Planes, 2012). Electrical conductivity of the sample was measured as per the ASTM C611 method using conventional four probe technique at a constant current supply ranging from 100-500 mA (Kakati et al, 2009).

2.5.2 Bulk Density

Density is a property of matter that is unique to each substance. It is a measure of the mass of the substance in a standard unit of volume. The density is also defined as its weight per unit volume although this quantity is more properly called specific weight. Different material have different densities. Density can observe easily. If two object have exactly the same size and shape, the denser one may feel heavier. But if their densities are very near, it will be very difficult to difference (Kakati, 2009).

2.5.3 Hardness

Shore hardness was measured with the help of a scleroscopic hardness tester (Imai Testing Machine Manufacturing from Japan,; model: Hardscope) as per the ASTM C886. It is a dynamic indentation type hardness test in which a diamond tipped hammer fall vertically from a fixed height over the composite bipolar plate. The hardness of the bipolar plate was measured with the help of the rebound height of the hammer. (Kakati et al, 2009).

The flexure was measured by 3 point method using Instron Universal Testing machine (Model 4411 Series Automated Material Testing System 1.38) according to ASTM D790. The support span was kept 40 mm and the sample dimension was 50 mm x 11.2 mm x 2.88 mm. The cross head speed was kept 0.1 mm/min. (Reza and Teherian, 2011).

2.5.4 MICROSTRUCTURE ANALYSIS

Microstructure analysis was made by Dino-Lite AM3111 handled digital microscope camera. It has natural colour technology providing colour true to their original appearance. It also has enhanced low light capability where it can see object clearly when there is minimal light available. It has a refresh rate up to 30 fps at 640 x 480 resolution and can magnify object up to 230x depending on working distance.



CHAPTER 3

METHODOLOGY

3.1 Experiment Overview

In this chapter, the details of the materials selection, specimen preparation on the method of fabrication until the testing method are discussed. The flow chart of the project in Figure 3.1 will clarify all the procedures in order to achieve project objective.

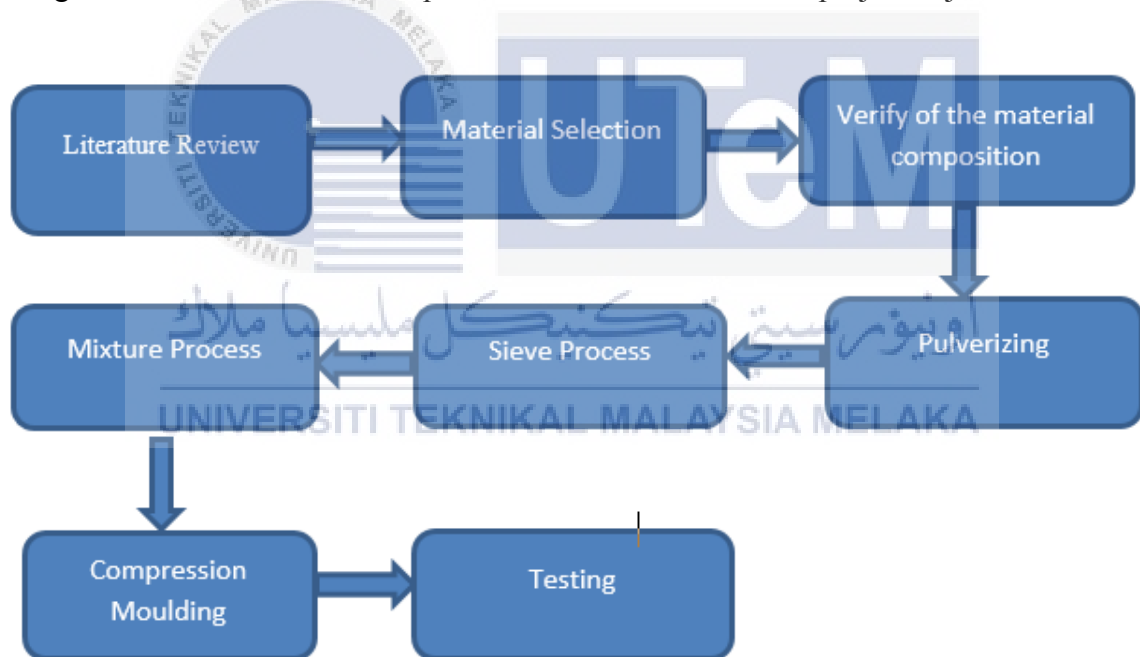


Figure 3.1 Flow chart of the process of fabrication Gr/Sn/PP composite for bipolar plate

3.2 Material Selection

The material selection is the most critical part for this research because the material selection selected will influence the properties of product and the method of fabrication. Material selection is the foundation of all engineering applications and design. The material selected should be studied correctly to achieved desired specification and properties to meet our objectives

For this research, the material used to fabricate the composite bipolar plate is conductive filler material and binder material. For this research, the material used to fabricate the composite bipolar plate is conductive filler material as a Gr/Sn and binder material as a PP.

3.3 FABRICATION METHOD

The fabrication method is process to manufacture the composite bipolar plate. There are several parameter and process undergo to get the high performace composite bipolar plate.

3.3.1 Characterization of Raw Material

The characterization of raw material were been evaluated are classified into six main parts which are grade, density, thermal stability, size, shape and resistivity for each material. The Table 3.1 below has shown the characteristic of the material that has been studied

Material	Graphite (Gr)	Stannum (Sn)	Polypropylene (PP)
Grade	3243	Aldrich	Titan (660)
Density	1.74	7.31	0.91-0.92
Thermal Stability °C	3500-4000	210-240	175-220
Size μm	65.901	33.82	-
Shape	Flake	Particulate	Flake
Resistivity $\Omega\text{ cm}$	1295 (10^{-8})	115(10^{-9})	10^{14}

Table 3.1:The Characteristics of the material

3.3.2 The Composition of Raw Material

After the materials have been selected, the composition ratio of filler: binder will be fixed 80:20. The binder in this experiment will be fixed to 20%. Each nine numbers of samples are made in different composition of Gr/Sn/PP based on weight percentage with different temperature. The Gr composition was decrease from 70%, 65% and 60% while the Sn is increase from 10%, 15% and 20%. The Table 3.2 below show the composition of composite of Sn/Gr/PP based on weight percentage (wt. %) in different temperature.

Sample	Filler		Binder	Temperature (°C)
	Sn (wt. %)	Gr (wt.%)	PP (wt.%)	
1	10	70	20	165
2	15	65	20	165
3	20	60	20	165
4	10	70	20	170
5	15	65	20	170
6	20	60	20	170
7	10	70	20	175
8	15	65	20	175
9	20	60	20	175

Table 3.2: Composition of composite of Gr/Sn/PP based on weight percentage wt. % in different temperature

3.3.3 Pulverizing

The pulverizing process to change PP crystal size to finer particles by using the Centrifugal Mill. The Figure 3.3 below shows the centrifugal Mill used in the pulverizing process. This machine is set about 2000 seconds to pulverize the PP. There are only 20 gram of PP in crystal shape are grinding into 230 mm x 198 mm x 59 mm pulveriser steel manganese grinding bowl.



Figure 3.2: Centrifugal Mill

3.3.4 Sieve process

After that, the sieve process is carried out using sieve shaker machine as shown in Figure 3.4 in order to get the desired size of 500 μm of PP. This process is used to separate the PP of 500 μm sizes from the PP powder that already get from pulveriser process. The sieve shaker machine is set vibrate in 5 minute to separately the PP powder in order to get 500 μm size particles of PP.



Figure 3.3: Sieve Shaker

3.3.5 Mixing process.

Mixing is a process that mix all three material of filler and binder like Gr, Sn and PP by using the Ball Mill Machine shown in Figure 3.2. This mixing process are conducted to mix all the filler and binder material using Ball Mill machine. The various different sized of steel ball are put inside in steel jar in order to get uniform mixing between three materials. The speed of the rotor is set to 900 rpm in 1 hour and half for three material is completely mixed equivalently.



Figure 3.4: Ball Mill Machine

3.3.6 Compression Molding

The samples of Gr/Sn/PP that was already mixed are placed in the steel mould. The mould of bipolar plate is a square shape with dimension of 100 mm x 100 mm. The three different temperature during hot compression is set to 165°C, 170°C, and 175°C based on Tables 3.2 and the pressure is set to 45 tonnes with pre-heated in 5 minutes and 10 tonnes. The machine used for this process is 200 Ton High Speed Hot Press Machine as shown in Figure 3.5. After hot compacting process was done, the molding was placed at cool place to make the molding cold faster. The samples surface of composite Gr/Sn/PP bipolar plate that have been fabricate as shown in Tables 3.3.



Figure 3.5: 200 Ton High Speed Hot Press Machine

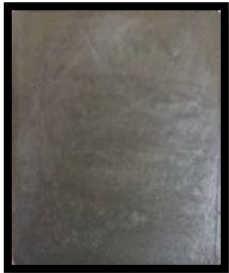
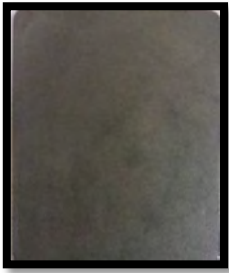
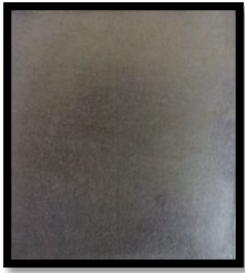



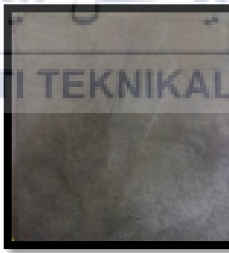
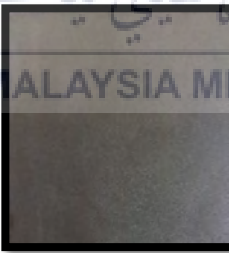
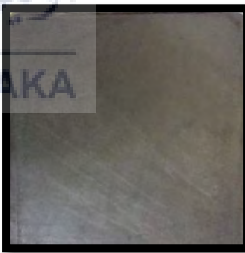
COMPOSITION Temperature	Gr/Sn/PP (70/10/20)	Gr/Sn/PP (65/15/20)	Gr/Sn/PP (60/20/20)
165 °C			
170 °C			
175 °C			

Table 3.3: Sample of composite bipolar plate Gr/Sn/PP after fabrication

3.3.7 Cutting Process

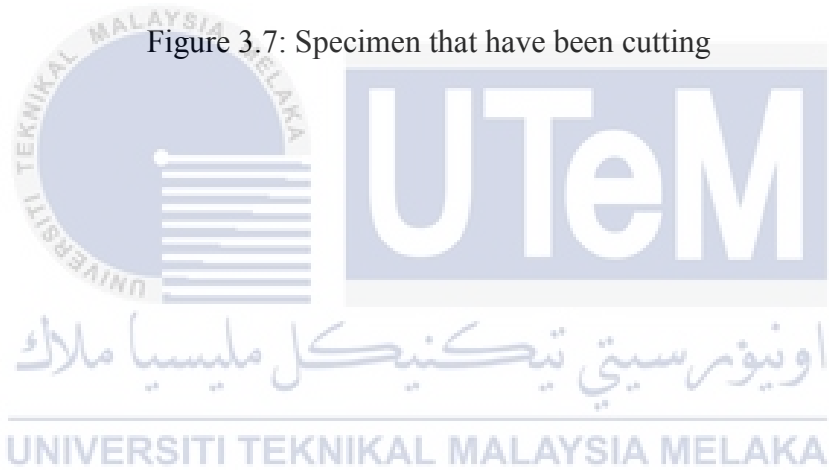
The sample of Gr/Sn/PP composite specimen that has been fabricate will be divide and cutting to the specified dimension to go through mechanical testing. The dimension that has been apply are according to ASTM D790-03 standard. Hence, the Proxxon table saw machine shown in Figure 3.6 will be use to cutting the rectangular shape specimen in dimension 100 mm x 13 mm x 3 mm shown in Figure 3.7.



Figure 3.6: Proxxon Table Saw Machine



Figure 3.7: Specimen that have been cutting



3.4 Testing Method

Testing method are conducted by five different test experiment such as electrical conductivity, flexure strength, shore hardness, bulk density and microstructure analysis.

3.4.1 Electrical Conductivity

The electrical conductivity of the sample was measured by using Jandel Multi Four Point Probe technique as shown Figure 3.8 at a constant current supply of 1mA. The test will show the value of voltage current in nine point at front and back surface sample. In this method, outer two probes are used to apply current to the sample and the inner two probes are used to measure the voltage. This test is done according to ASTM C611 standard.

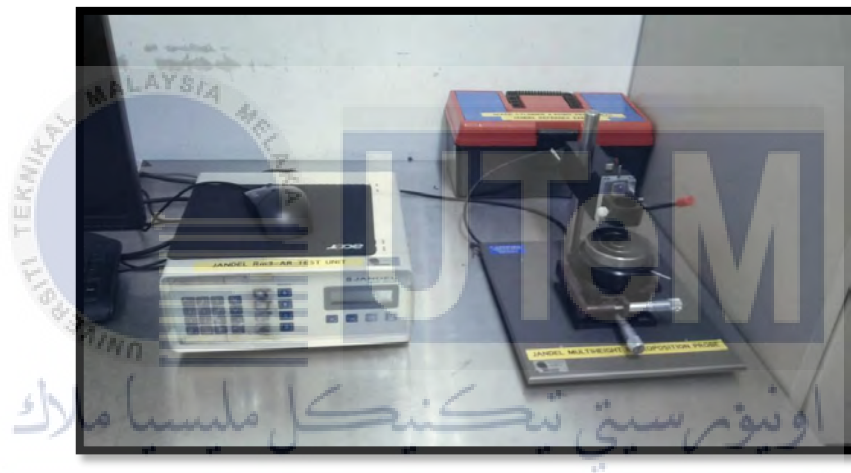


Figure 3.8: Jandel Multi Four Point Probe

3.4.2 Flexure Test

The flexure strength was measured using Instron Universal Testing Machine shown in Figure 3.9 by using standard ASTM D790-03. Flexural strength is one of the most important properties of bipolar plate as it may undergoes a high bending pressure during stacking of the cells. Three point flexural strength of the composite bipolar plate was evaluated using universal testing machine. Flexural testing is used to determine the bending properties of a material. Maximum stress and strain are calculated on the incremental load applied.

The sample of Gr/Sn/PP was cut by using proxxon table saw before doing the tensile test. The tensile are started until the specimen was fracture. The length of the sample is 100

mm and the width is 13mm. The maximum load that applied on the specimen is 30 KN. The span length is 50 mm and the speed is 0.1 mm/per second.

All the parameter such as total length, span length, width, thickness and speed are set in the software provided. Figure 3.9 shows the specimen that have been test using Universal Instron Testing Machine



Figure 3.9: Specimen that have been test using Instron Universal Testing Machine

3.4.3 Density Test

The density of the bipolar plate may be measured using Bulk Density as shown in Figure 3.10. It is measured of the mass of the substances in a standard unit of volume. This test was conducted according to ASTM D792 standard.



Figure 3.10: Electronic Densimeter

3.4.4 Shore Hardness

The shore hardness test are make according to ASTM D2240D standard. The hardness of the composite bipolar plate was measure that involves dropping a diamond tipped hammer, which falls inside a composite plate under the force of its own weight from a fixed height onto the test specimen as shown in Figure 3.12. The three reading of hardness each sample are take to make the average. The Figure 3.11 shows the shore tester device that used to measured the shore hardness. The strength and fracture toughness of the bipolar plate material were evaluated using the shore hardness shown in Figure 3.11



Figure 3.11: Shore Hardness Tester



Figure 3.12: Process of Shore Tester.

3.4.5 Microstructure Analysis

The behaviour of surface specimen can be analysed by using Dino Lite Microstructure as shown in Figure 3.13. Firstly the microscope Dino Lite must be plugin direct to computer. Then the specimen was located under the lens of microscope. After that, by adjusting the suitable magnification of lens, the surface picture of sample was scan and can see from the computer. The picture that have been scan was snap and save to the computer.



Figure 3.13: Dino Lite Microstructure

CHAPTER 4

RESULTS AND ANALYSIS

This chapter shows all the experiment result from all the testing that have been conduct such as electrical conductivity, flexural strength, bulk density, shore hardness and microstructure analysis. The result are analysed from the five tests that has been carried out in this project for the Gr/Sn/PP polymer composite bipolar plate.



4.1 ELECTRICAL CONDUCTIVITY

The electrical conductivity test of Gr/Sn/PP bipolar plate was measured by using Jandel Multi Height Four Point Probe. The average result of nine sample electrical conductivity testing is shown in Table 4.1. Based on the DoE target electrical conductivity is more than 100 S/cm.

Temperature ($^{\circ}\text{C}$)	Sample 10% Sn	Average (S/cm)	Sample 15 %Sn	Average (S/cm)	Sample 20% Sn	Average (S/cm)
165	1	49.65	4	10.10	7	8.97
170	2	28.62	5	7.335	8	9.83
175	3	11.70	6	10.62	9	8.98

Table 4.1: Average Result of the Electrical Conductivity of Specimens.

Based on table above, all the composition sample did not meet the requirement target of DoE 100 S/cm. The higher value of electrical conductivity is only 49.65 S/cm which is far different to the requirement target while the lowest is 8.97 S/cm.

4.2 FLEXURE STRENGTH

The flexure strength by 3-point bending test was carried out by using the Intron Universal Testing Machine. Table 4.2 below show the nine average value of maximum Flexure Strength that have been collected. The average value of maximum flexure strength is determined by repeated three times for each specimen. Based on the DoE target the flexural strength is more than 25 Mpa.

Temperature ($^{\circ}\text{C}$)	Sample 10% Sn	Average (Mpa)	Sample 15% Sn	Average (Mpa)	Sample 20%Sn	Average (Mpa)
165	1	10.65	4	12.81	7	15.46
170	2	10.21	5	10.42	8	14.86
175	3	8.01	6	9.35	9	10.9

Table 4.2: Average Result of the Maximum Flexure Strength

From the Table data 4.2 above, all the specimen not achieve the DoE target which the flexure strength must above to 25 Mpa. From the table above, the maximum flexure strength that have obtained is 15.46 Mpa that far away from DoE target standard. While the minimum flexure strength is 8.01 Mpa.

4.3 Bulk Density

The bulk density for each specimen is measured by using Digital Electronic Densimeter. Each specimen was tested three times to obtain the average value. Table 4.3 below show the nine specimen average result that has been obtained. Based on DoE target, the bulk density of the bipolar plate must be less than 2g/cm^3 . The highest value of bulk density is 1.960 g/cm^3 while the lowest is 1.690 g/cm^3 .

Temperature ($^{\circ}\text{C}$)	Sample 10%Sn	Average (g/cm^3)	Sample 15%Sn	Average (g/cm^3)	Sample 20%Sn	Average (g/cm^3)
165	1	1.663	4	1.750	7	1.812
170	2	1.720	5	1.841	8	1.960
175	3	1.690	6	1.810	9	1.871

Table 4.3: Average Result of the Bulk Density

4.4 Shore Hardness

The shore hardness of specimen is measured by using analog shore tester. Shore hardness is the resistance of the materials towards indentation (Selamat et al, 2016). The Table 4.4 below presented the variation average result of shore hardness of bipolar plate based on each composition. All the data was taken after the hardness test was done. Each specimen was tested five times to obtain the average value of shore hardness. Based on DoE target, the shore hardness of bipolar plate must be more than 50.

From the data in Table 4.4 the highest value of shore hardness is sample with 10 wt. % Sn at molding temperature of 170°C. From other side, the lowest shore harness is also 10 wt. % Sn but different temperature with 175°C.

Temperature	Sample	Average	Sample	Average	Sample	Average
(C°)	10%Sn		15%Sn		20%Sn	
165	1	57.3	4	57.6	7	58.6
170	2	61.0	5	62.3	8	59.8
175	3	53.0	6	56.6	9	58.0

Table 4.4: Average Result of the Shore Hardness

4.5 MICROSTRUCTURE ANALYSIS

The Table 4.5 below show the microstructure view of sample composite Gr/Sn/PP bipolar plate that has been prepared. It shows that the microstructure of surface and cross section with 200 mm and 100 mm magnification lens using Dinolite Digital Microscope. The surface that have scan can show any presence of porosity, crack or the voids on the sample based on the composition. From the Figure 4.1 below shown distribution of Gr/Sn/PP that shown Gr is black colour and Sn is white colour.

Figure 4.1: The Distribution of Gr and Sn in Bipolar Plate

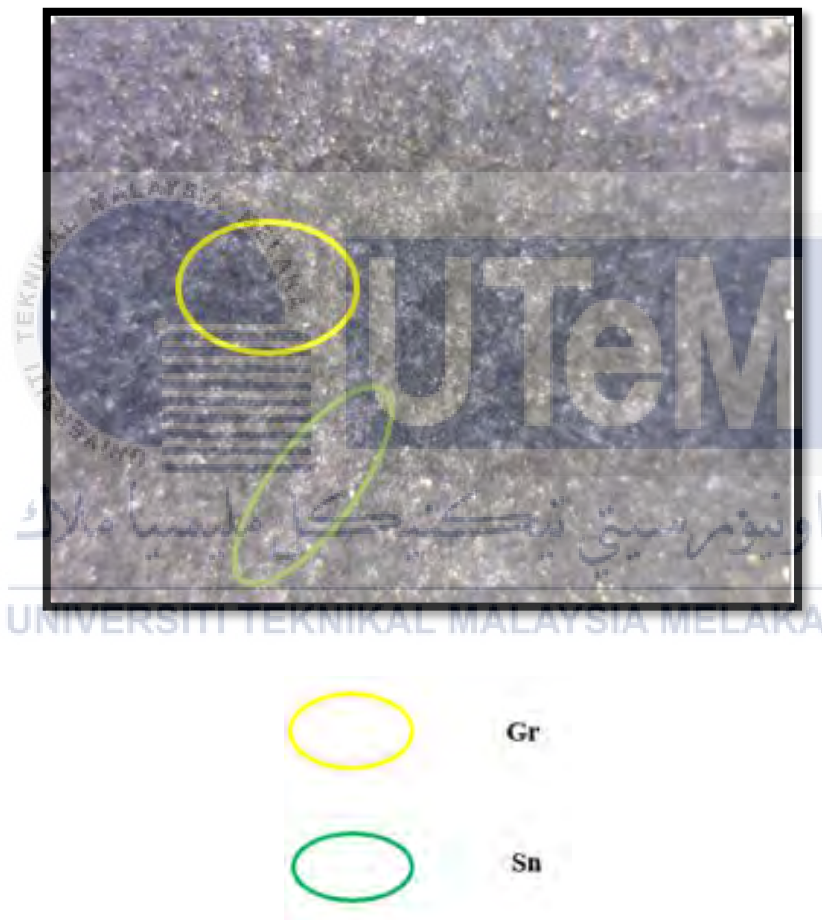





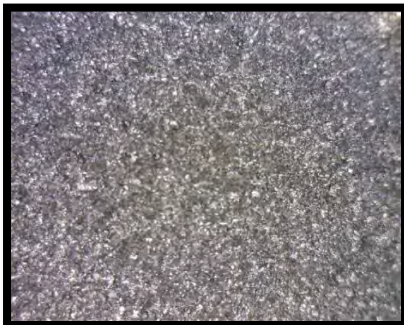
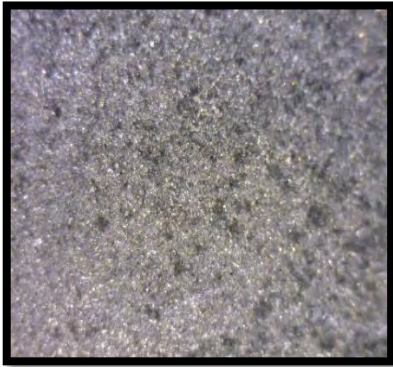
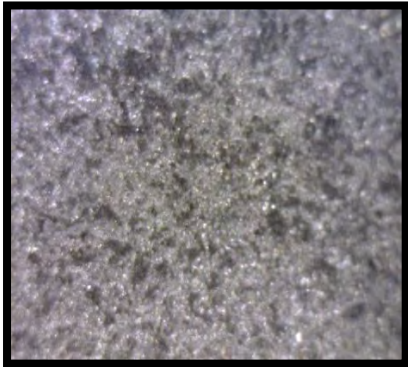
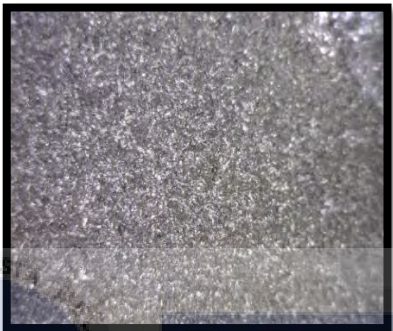
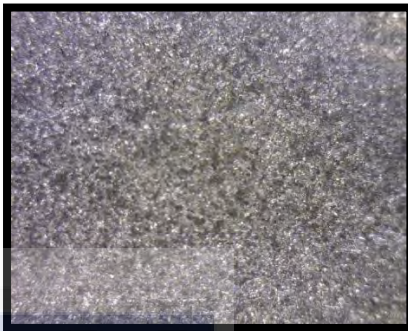




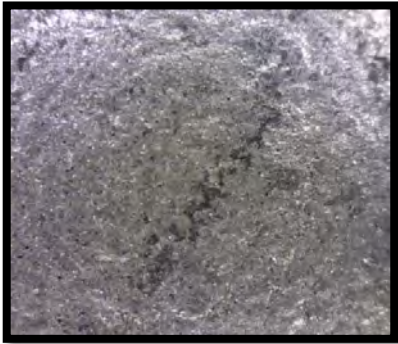
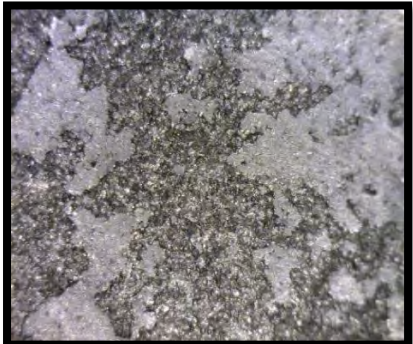
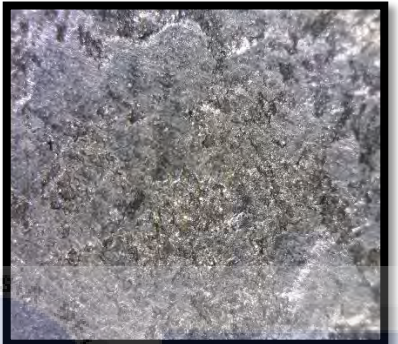



Table 4.5: Microstructure view surface and cross section on the composition with 200 mm and 100 mm Magnification

Sample	Composition (Gr/Sn/PP) (T) °C	Microstructure View 200 mm	Microstructure Cross Section 100 mm
1	70/10/20 (165°C)		
2	65/15/20 (165°C)		
3	60/20/20 (165°C)		

4	70/10/20 (170°C)		
5	65/15/20 (170°C)		
6	60/20/20 (170 °C)		
7	70/10/20 (175 °C)		

8	65/15/20 (175 °C)		
9	60/20/20 (175 °C)		

CHAPTER 5

DISCUSSION

In this chapter all the result mechanical testing obtained in chapter 4 will be discussed. These result are electrical conductivity, flexural strength, bulk density, shore hardness and microstructure analysis.



5.1 Effect of Sn wt.% with Different Temperature on Electrical Conductivity

The results of electrical conductivity testing have been presented and illustrated below in Figure 5.1 and 5.2, bar chart and graph of average electrical conductivity against percentages weight of Sn.

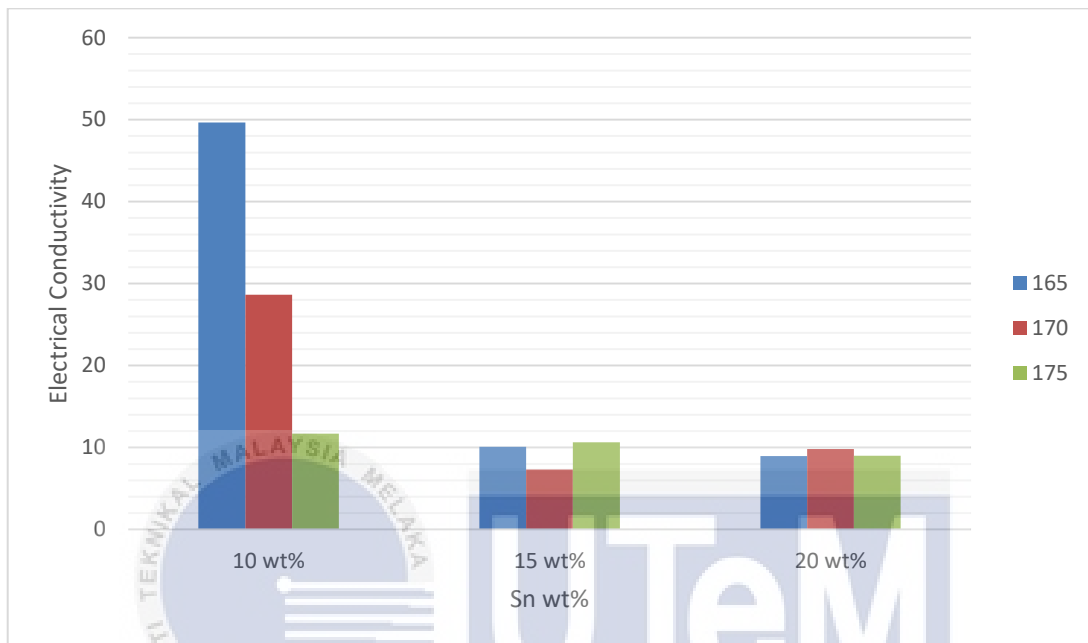


Figure 5.1: Electrical Conductivity (S/cm) versus Sn wt%

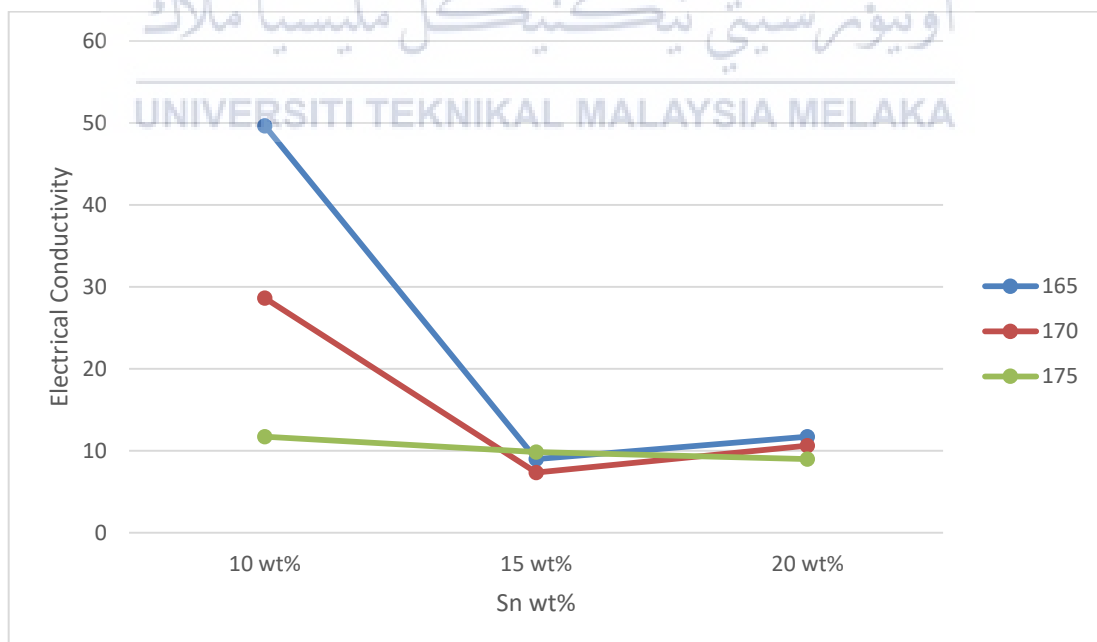


Figure 5.2: Graph of Average Result of Electrical Conductivity (S/cm) against Sn wt. %

In this research, the fabrication of bipolar plate must have good in electrical conductivity. It is most important requirement for commercialized of bipolar plate in the industry. The maximum requirement of electrical conductivity to achieve DoE target must be higher than 100 g/sm. Moreover, there are three factor that influenced the electrical conductivity for bipolar plate which is size, shape and orientation of conductive filler of the composite (Kakati et al, 2009). But there are some problem in this experiment because the electrical conductivity of sample did not meet the target of DoE.

The value of electrical conductivity, low are due to over melted of PP as binder and at this stage PP is only become binder between filler particle but covered all particles. This will affect the conducting tunnel among the Gr and Sn and gives more barrier to conducting the network (Selamat et al, 2016). It also may be due to fabrication process when the sample are not mix well during mixer process.

From the Figure 5.1 and 5.2 above, the maximum of electrical conductivity test is sample with 10 % wt. Sn that give electrical conductivity of 49.65 S/cm while the minimum value of electrical conductivity is sample with 15 % wt. Sn that gives electrical conductivity 7.335 S/cm. The Figure 5.1 and Figure 5.2 also shows that the electrical conductivity of Gr/Sn/PP composite of bipolar plate have not increased with the increment of Sn % wt. So, the value of electrical conductivity will be increased if the weight percentages of Sn is decreased. Besides that, for 15% wt. and 20% wt. of Sn, the electrical conductivity of this range are not different based on Sn wt. % and temperature molding that give value around 10 S/cm and 7 S/cm.

In this research, it found that the temperature molding was also can affect the electrical conductivity. At the 10 Sn % wt., the highest temperature molding will be decreased the electrical conductivity of specimen. Furthermore, the electrical conductivity each specimen with 15% and 20% of Sn was only change slightly at different temperature molding.

5.2 Effect of Sn wt. % with Different Temperature on Flexure Strength.

The results of flexure strength testing have been presented and illustrated below in Figure 5.3 and 5.4, bar chart and graph of average flexure strength against percentages weight of Sn were been plotted.



Figure 5.3: Flexure Strength (Mpa) versus Sn wt. %

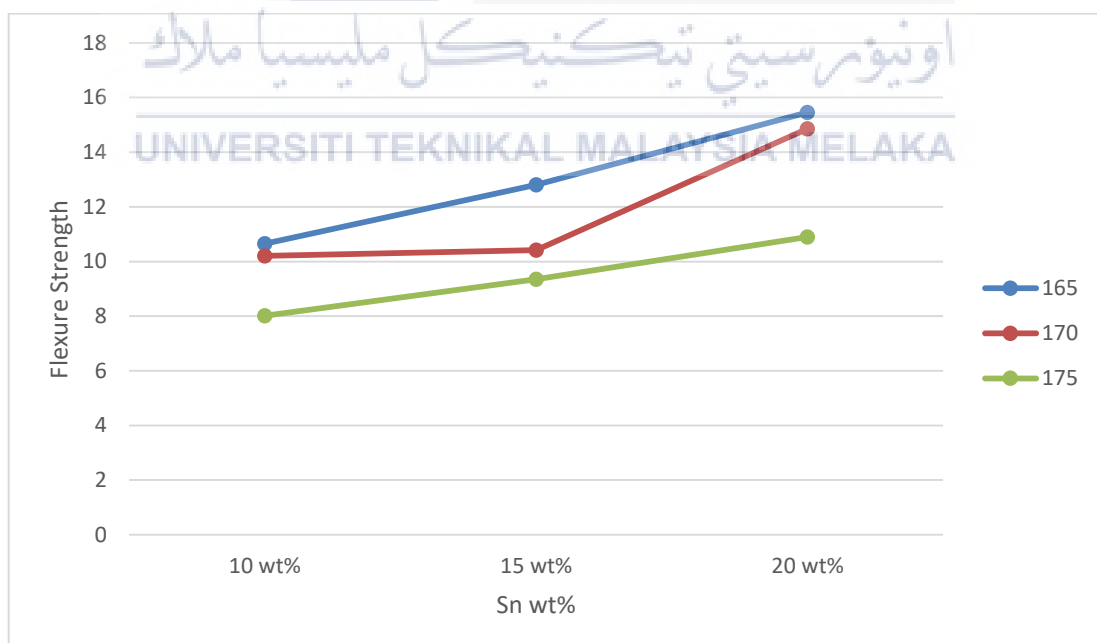


Figure 5.4: Graph of Average Result of Maximum Flexure Strength (Mpa) against Sn wt. %

Flexural Strength is defined as the ability to resist deformation under load. It is measured at the highest stress experienced within the material at the moment of rupture (Cheng and Hwa, 2014). The bipolar plate requires good mechanical strength to support structure of PEMFC. The bipolar plate require good mechanical strength because it also need to withstand the high clamping forces of good stacking process and vibration during application of vehicular (Szantes, 2010). It shows that PP as a binder is to provide strength and improve the brittleness of Gr. From the research, all specimen composite Gr/Sn/PP did not achieve the DoE target due to the Gr particles will not be wetted well with PP and its will ruptured because of incomplete compaction (Selamat et al, 2010).

From the Figure 5.3 and 5.4 above, the sample composition 70% Gr have smaller flexure strength than sample composition 60% Gr .It shows that flexure strength will reduce as the graphite is increased because the brittleness of Gr. This also is due to adhesion between resin and Gr become weaker as the graphite content increased. Small-size graphite powder possesses more surface area than longer Gr powder. Hence, small-size Gr powder with 70 wt. % Gr content has a stronger absorbing ability than that of longer Gr powder. The porosity and number of voids increases with decreasing graphite size (Kuan et al , 2004).

From the research, the flexure strength of bipolar composite were decreased as graphite is increased, the bar chart show Gr content is 70%, the flexure strength is decreased and Gr content is 60%, the flexure strength is increased. Besides that, the size of Sn also can affect the flexure strength of samples. From the Figure 5.3 and 5.4, it shows the increment of Sn loading can increase the flexure strength. From the other side, the decrease of Sn wt% can reduce the flexure strength. The maximum of flexure strength is 15.46 Mpa with 20% wt of Sn while the minimum is 8.01 Mpa with 10 Sn %wt. From the Figure 5.3 and 5.4 above, the temperature molding can affect the flexure strength with slightly different. For composition 10 %wt. Sn the flexure strength 10.65 Mpa at temperature molding 165°C will decreased to 10.21 Mpa at temperature molding 170° C and continually decreased to 8.01 Mpa at temperature molding 175°C. This trend was same in rest composition which the flexure strength was reduced constantly when the temperature was increased.

5.3 Effect of Sn wt. % with Different Temperature on Bulk Density

The results of bulk density testing have been presented and illustrated below in Figure 5.5 and 5.6, bar chart and graph of average bulk density against percentages weight of Sn were been plotted.

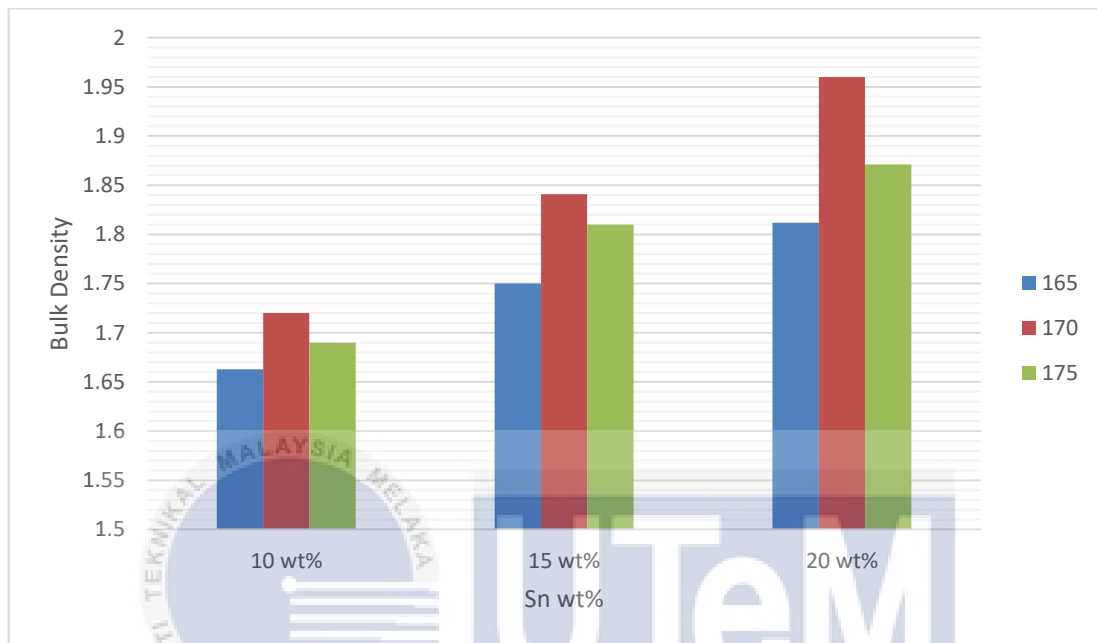


Figure 5.5: Bulk Density (g/cm^3) versus Sn wt. %

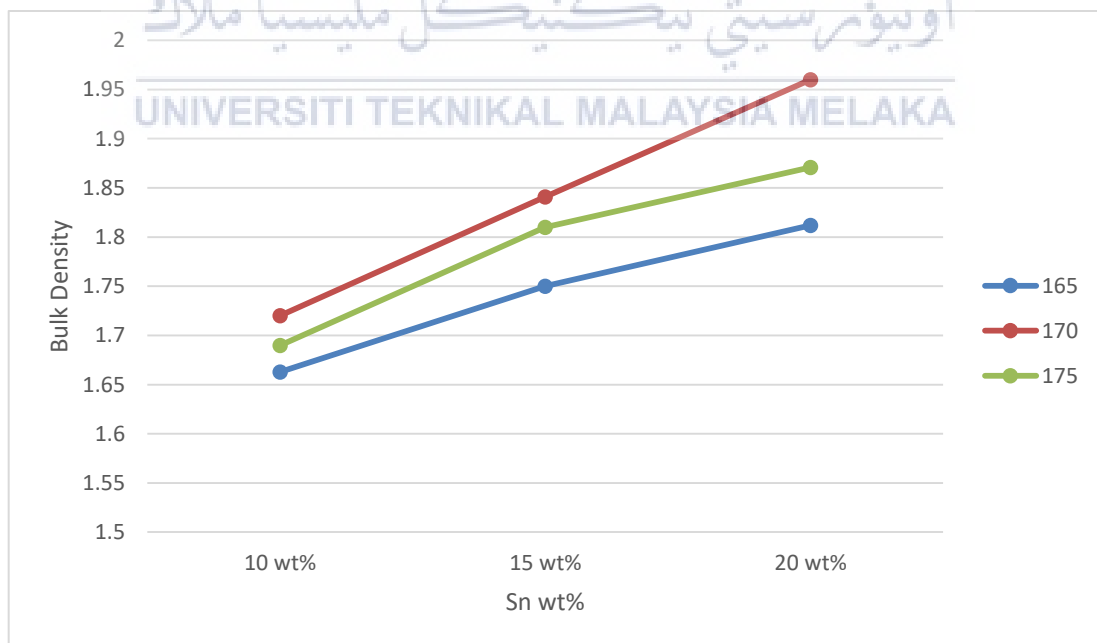


Figure 5.6: Graph of Average Result of Bulk Density against Sn wt. %

Bulk density of specimen normally is affected by the weight and size of particles. The less weight and particles size of the filler materials, the lower the value of bulk density (Selamat et al, 2013). Figure 5.5 and 5.6 show that the bulk density of the Gr/Sn/PP composite bipolar plate was increased with the increments of Sn%. The density of all composition of Gr/Sn/PP sample was reach the DoE target which all bulk density was smaller than 2g/cm^3 . It shows the minimum of bulk density was 1.663 g/cm^3 with of 10 wt. % Sn while the maximum bulk density was 1.960 g/cm^3 with 20 Sn wt. %, this is due to the higher composition and density of Sn. The bulk density is an important property for the Gr/Sn/PP composite for bipolar plate in PEMFC. Bulk density of specimens normally affected by the weight and size of particles. The lesser the weight of the filler materials, the lower the bulk density (Selamat et al, 2016).

From the Figure 5.7 and 5.6, it shows the increments of Sn loading will be increase the bulk density of the specimen. Initially at 10 wt. % of Sn, the bulk density increased with the increments of Sn wt. % from 15 wt. % and 20 wt. %. Besides that, the temperature molding also will affect the bulk density of specimen. The temperature molding of 170°C will give higher value of bulk density compared to other while 165°C will give lowest value of bulk density.

5.4 Effect of Sn wt. % with Different Temperature on Shore Hardness

The results of shore hardness testing have been presented and illustrated below in Figure 5.7 and 5.8, bar chart and graph of average shore hardness against percentages weight of Sn were been plotted.

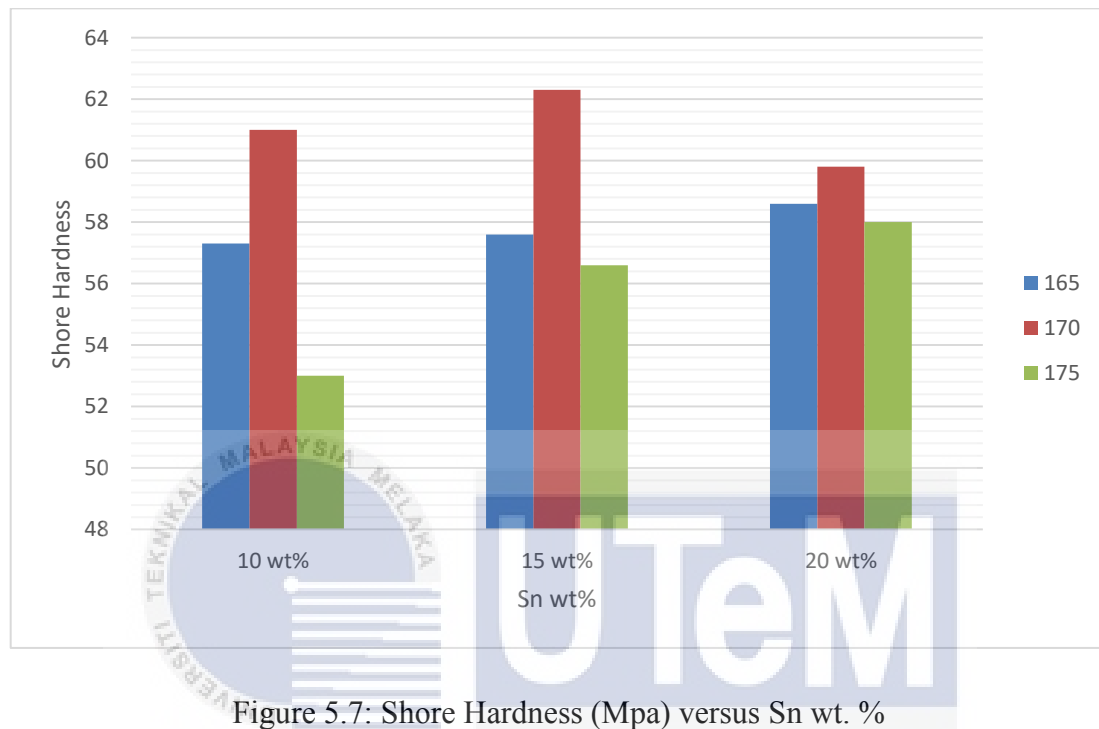


Figure 5.7: Shore Hardness (Mpa) versus Sn wt. %

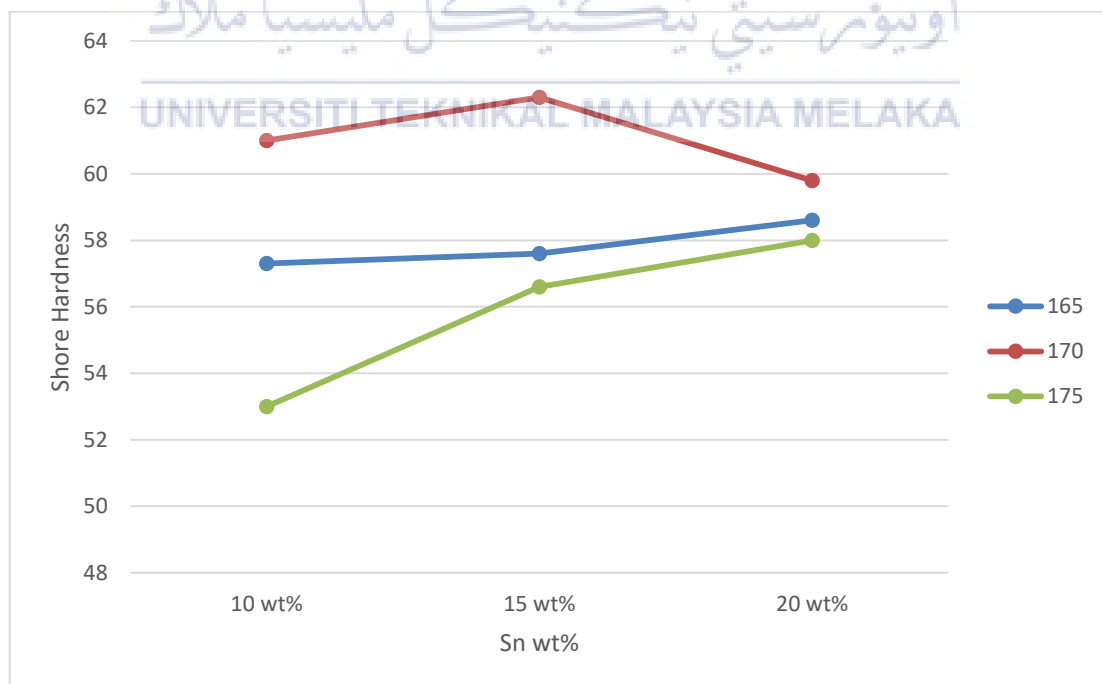
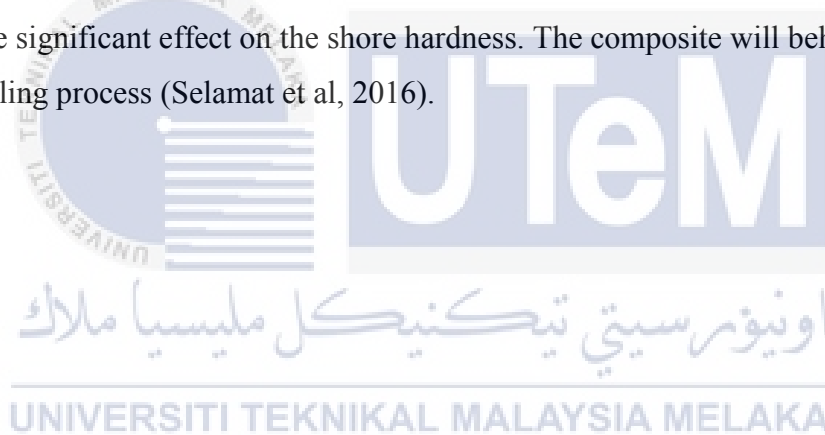


Figure 5.8: Graph of Average Result of Shore Hardness against Sn wt. %

The Figure 5.7 and 5.8 show that the highest shore hardness at composition 15% wt. Sn with 62.3.Mpa while lowest shore hardness is sample with 10% wt. Sn with shore hardness is 53.0 Mpa. From the result data Table 4.4 that have obtain, the range of shore hardness off all specimen is from 53 to 62.3 Mpa which has exceeding DoE target. The shore hardness of samples show a constant trend with the increment of Sn wt. %. Initially 10 % wt. and 15 % wt. the shore hardness is increase with the increment of Sn. But at 20 % wt Sn and temperature molding 170°C the shore hardness is decrease.

The data also present that each composition with temperature molding 170°C give higher shore hardness compared to other. Hence, it shows that the shore hardness at 170°C is suitable temperature for making bipolar plate in highest of hardness. The average value of shore hardness of all samples achieve the minimum requirement target from US Doe. It shows that the shore hardness was affected by the compaction of the materials and the voids that formed during fabrication process. The duration of cooling process of the samples will also give the significant effect on the shore hardness. The composite will behave brittle due to rapid cooling process (Selamat et al, 2016).



5.5 MICROSTRUCTURE ANALYSIS

From the picture that has captured in Table 4.5 using Dinolite Microscope, the sample has been mixture with Gr/Sn/PP but there are some void in cross section microstructure in certain space due to the material sample that are not mixed well during the fabrication process using ball mill mixer. After that, the particles also were found agglomerate at certain area as shown in Figure 5.9 due to Sn are not spread well in the plate. So the electrical conductivity not perform well because the Sn are not filled perfectly at the void of bipolar plate. After that, the effect of temperature molding also can make void to the sample composite bipolar plate.

After that, the PP also concentrate in gather at certain space only that will be effect the strength of the sample. It did not hold the conductive filler like Gr and Sn perfectly. So the sample of bipolar plate was easy to rupture. Besides that, the porosity or micro crack formed in the structure of samples is due to shearing forces occurred between particles Gr and PP as binder. This shearing force has incited during the deformation of press. The electrical properties of samples has affected because this porosity or micro crack around graphite particles becomes barriers to electrical conductivity and resulting lower value of electrical conductivity than the unpress sample (Selamat et al, 2011).

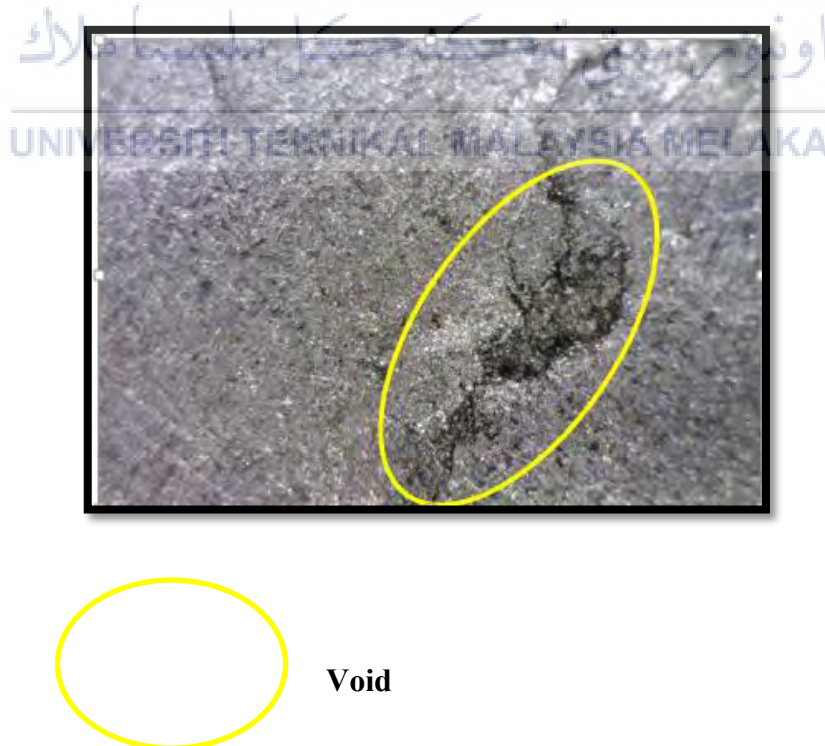


Figure 5.9: Void that have been detect on specimen.

Table 5.1: The microstructure surface of 10, 15 and 20 % wt. Sn with temperature 165°C

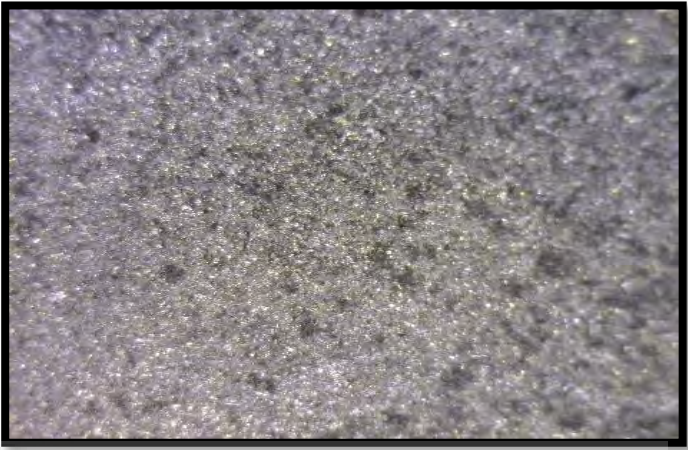


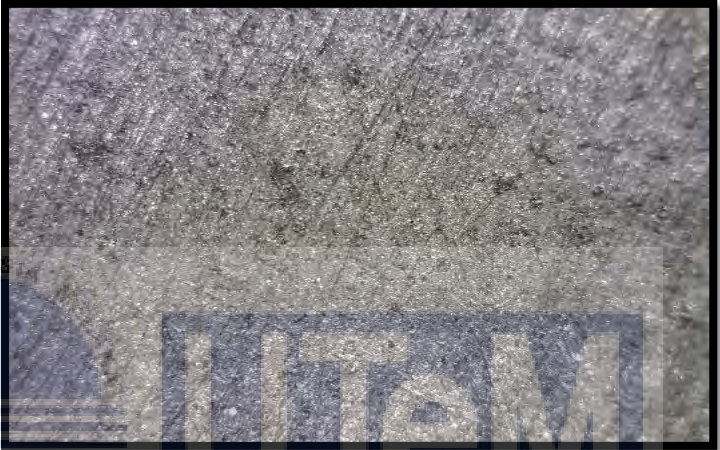
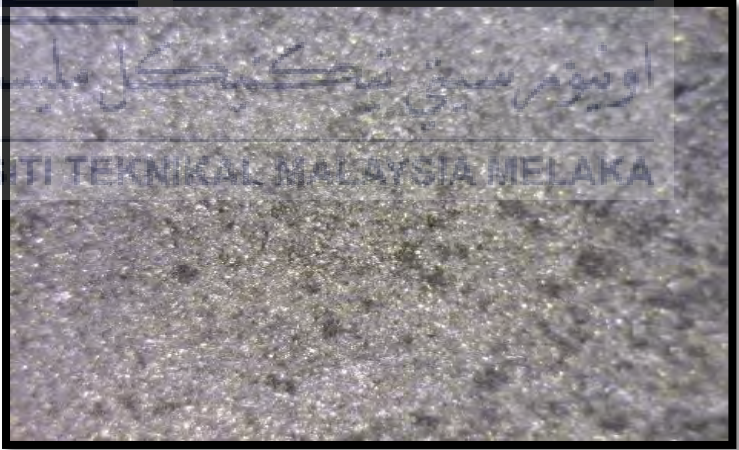
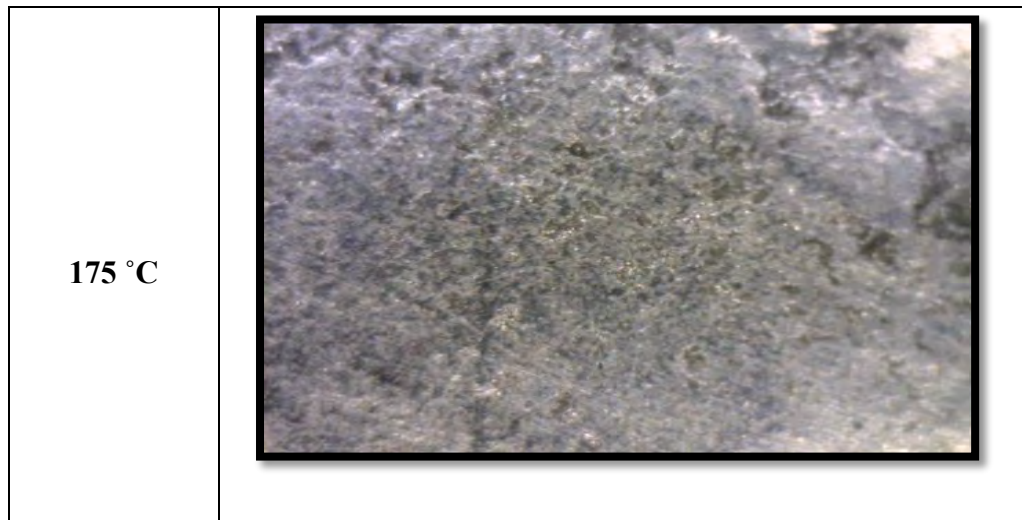
Sn wt. %	Temperature 165 °C
10 wt. %	
15 wt. %	
20 wt. %	

Table 5.2: The microstructure surface of temperature 165°C, 170°C and 175°C with 10% Sn

Temperature (°C)	10% wt. Sn
165 °C	
170 °C	



The Table 5.1 above show the surface appearance with Sn wt. % loading with temperature 165 °C. The distribution of Sn particles in each particles composite has significant effects on the properties of Gr/Sn/PP composite. From the Table 5.1 it can conclude that the increment of Sn wt. % with same temperature 165 °C can increase the saiz of Sn. Besides that, it can conclude the higher size of Sn will be decrease the electrical conductivity while the lower size of Sn can increase the electrical conductivity. There show the size particles of Sn will be affect the electrical conductivity. Meanwhile, shows that the higher value of electrical conductivity is 10 wt. % Sn with lower size of Sn while the lowest electrical conductivity is 20 wt. % Sn with bigger size of Sn. After that, the larger size of Sn also can make more agglomerate, so it can reduce the electrical conductivity of the sample.

The Table 5.2 above show the microstructure analysis appearance with Sn 10 wt. % loading with temperature 165°C, 170°C and 175°C. From the Table 5.2, shows the microstructure of surface with three different temperature at 10% wt. Sn loading. The temperature molding is set to 165°C, 170°C and 175°C in order to analyse the microstructure each sample. It shows clearly that the lower temperature can give good microstructure and less void of sample. At temperature molding 170°C the microstructure of sample have some agglomerate and at 175°C there are more void detect at the sample. So the higher temperature can give more agglomerate and void that can affect the electrical conductivity and flexure strength of the sample.

CHAPTER 6

CONCLUSION AND RECOMMENDATION

6.1 CONCLUSION

In this research, the main objectives of investigation is to study the effect of Sn on the properties of Gr/Sn/PP composite for bipolar plate and to determine the suitable temperature of Gr/Sn/PP through compression molding. The conductive filler like Gr and Sn will fixed to 70%/10% wt., 65%/15% wt. and 60%/20% wt. while the PP as the binder will fixed 20%. The total nine sample was fabricated with increment of Sn (10%wt, 15%wt and 15%wt) in three different temperature such as 165°C, 170°C and 175°C. As the conclusion that can made from this research, the effect of temperature molding can influenced the decreasing of electrical conductivity of bipolar composite Gr/Sn/PP. But at Sn loading 15%wt and 20% wt. there not much different in electrical conductivity. After that, the temperature also will be influenced the properties of composite bipolar plate. From this research that have been made, the higher temperature molding can increased the void of the bipolar plate. It will be effect the low electrical conductivity and flexure toughness of the bipolar plate.

The effect of Sn on the properties of Gr/Sn/PP composites and temperature of compression molding for bipolar plate were studied. Based on the result obtained, the following conclusion were made:

1. The electrical conductivity and flexure strength does not achieve DoE target due to void and agglomeration complication.
2. The shore hardness and bulk density of composite bipolar plate achieve the DoE target.
3. The molding temperature during compaction process can affect the properties of composite bipolar plate.
4. The distribution of Sn will give low electrical conductivity of composite bipolar plate.
5. PP can improve the mechanical properties of Gr/Sn/PP composite bipolar plate because it can be function as bonding agent, increase strength and improve the brittleness of Gr.

6.2 RECOMMENDATION

At the end of study, there are some recommendation for further research to improve the results of properties Gr/Sn/PP composite bipolar plate. The recommendation given as below:

1. The period of mixing process should be longer to ensure that the Gr/Sn/PP are mix perfectly.
2. The size of chamber for mixer should be bigger size and the movement must be 360° rotation to assure the material must mix perfectly and equivalent.
3. The size of particles of PP are changed to small sized such as 50µm, 100µm and 250 µm.
4. The Mylar paper is used to prevent the sticky material at the mold plate during the compressing process.
5. The Scanning Electron Microscope (SEM) must be used to analyse the microstructure of composite Gr/Sn/PP in order to get accurate and clear image.

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APPENDIX

APPENDIX I GANTT CHART PSM I

No	TASK	WEEK													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	CONFORMATION PROJECT TITLE														
2	OBJECTIVE, SCOPE AND PROBLEM STATEMENT														
3	LITERATURE REVIEW														
4	PROPOSAL PSM 1 WRITING														
5	FABRICATION -Pulverizer Process														
6	REPORT PSM 1 WRITING														

APPENDIX II GANTT CHART PSM II

No	TASK	WEEK													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	FABRICATION -Sieve Process -Premixing Process -Compression Process														
2	TESTING -Electrical Conductivity -Flexure Strength -Bulk Density -Shore Hardness -Microstructure Analysis														
3	LITERATURE REVIEW														
4	PROGRESS REPORT PSM 2														
5	RESULT AND ANALYSIS														
6	REPORT PSM 2 WRITING														