DECLARATION

"I hereby declare that the work in this thesis is my own except for summaries and quotations which have been duly acknowledged."

Signature : ……………………………………………

Author : TIO KOK WEI

Date : 27 MAY 2015

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“I hereby declare that the work in this thesis is my own except for summaries and quotations which have been duly acknowledged.”

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Date : 30 JUNE 2015

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ABSTRACT

Bipolar plates are key components of Proton Exchange Membrane (PEM) fuel cells. They carry current away from the cell and withstand the clamping force of the stack assembly. Therefore, PEM fuel cell bipolar plates must have high electrical conductivity and adequate mechanical strength, in addition to being light weight and low cost in terms of both applicable materials and production methods. In this research, the raw materials used to fabricate the high performance bipolar plate are Graphite (Gr), Stannum (Sn) and Polypropylene (PP). The Gr and Sn act as fillers and the PP acts as binder. The ratio of fillers (Gr/Sn) and binder (PP) is fixed at 80:20. For the mutli-conductive filler, small amount of Sn which is 10 up to 20wt% (from the total weight of fillers 80%) have been added into Gr/Sn/PP composite. The fillers were mixed by using the ball mill machine. The second stage of mixing process between the filler and binder is also carried out by using ball mill machine before the compaction process by the high speed hot press hydraulic machine. The effect of formation temperatures (160^{o}C-170^{o}C) on the properties of Gr/Sn/PP composite need to be study in more detail especially the electrical and mechanical properties of Gr/Sn/PP composite. The result shows that there are significant improvement in the electrical conductivity and bulk density which are exceeding the US-DoE target with the maximum value of 265.35S/cm and 1.682g/cm^3 respectively. For the mechanical properties, flexural strength and shore hardness does not show any significant improvement which was not achieving the US-DoE target with the maximum value of 8.68 MPa and 28.2 respectively. The flexural strength and shore hardness of the Gr/Sn/PP composite do not achieved the target of DoE due to the different mixing process with previous researchers. The mixing of the filler and binder for this research was mixed using the ball mill machine while the mixing process for the previous researcher is using the internal mixer.

ABSTRAK

Plat bipolar adalah komponen utama dalam “Proton Exchange Membrane Fuel Cell” (PEMFC). Ia membawa sumber elektrik keluar dari sel dan menahan daya pengapitan himpunan tindanan. Oleh itu, plat bipolar mesti mempunyai kekonduksian elektrik yang tinggi dan kekuatan mekanikal yang mencukupi. Selain itu, plat bipolar mestilah ringan dan kos rendah dari segi kedua-dua bahan berkenaan dan kaedah pengeluaran. Dalam kajian ini, bahan-bahan mentah yang digunakan untuk menghasilkan plat bipolar yang berprestasi tinggi adalah “Graphite” (Gr), “Stannum” (Sn) dan “Polypropylene” (PP). Gr dan Sn bertindak sebagai pengisi dan PP bertindak sebagai pengikat. Nisbah pengisi (Gr/Sn) dan pengikat (PP) ditetapkan pada 80:20. Untuk pengisi mutli-konduktif, jumlah yang sedikit Sn iaitu 10 sehingga 20wt% (dari jumlah berat pengisi 80%) akan ditambahkan dalam Gr/Sn/PP komposit. Pengisi akan dicampurkan dengan menggunakan mesin kempa bola (ball mill machine). Peringkat kedua proses pencampuran antara pengisi dan pengikat juga dilakukan dengan menggunakan mesin kempa bola sebelum proses pemadatan oleh mesin hidraulik pengacuan mampatan. Kesan suhu pembentukan (160 hingga 170 ^{o}C) terhadap sifat-sifat Gr/Sn/PP komposit perlu dikaji dengan lebih terperinci terutamanya sifat-sifat elektrik dan mekanikal Gr/Sn/PP komposit. Hasil kajian menunjukkan bahawa terdapat peningkatan yang ketara dalam kekonduksian elektrick dan ketumpatan yang melebihi sasaran DoE, masing-masing dengan nilai maksimum 265.35 S/cm dan 1.682 g/cm^3. Bagi sifat mekanikal, kekuatan lenturan dan kekerasan tidak menunjukkan sebarang peningkatan yang ketara kerana tidak mencapai sasaran DoE, masing-masing dengan nilai maksimum 8.68 MPa dan 28.2. Kekuatan lenturan dan kekerasan komposit Gr/Sn/PP tidak mencapai sasaran DoE kerana proses pencampuran yang berbeza dengan penyelidik sebelumnya. Pencampuran pengisi dan pengikat untuk kajian ini adalah bercampur dengan menggunakan mesin kempa bola manakala proses pencampuran untuk penyelidik terdahulu menggunakan mesin pengaduk dalaman ‘internal mixer’.

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LIST OF ABBREVIATION AND SYMBOL

PEMFC	=	Polymer Electron Membrane Fuel Cell
AFC	=	Alkaline Fuel Cell
PAFC	=	Phosphoric Acid Fuel Cells
MCFC	=	Molten Carbonate Fuel Cells
SOFC	=	Solid Oxide Fuel Cells
MEA	=	Membrane Electrolyte Assembly
GDL	=	Gas Diffusion Layer
CPCs	=	Conductive Polymer Composites
HCC	=	Hybrid Conductive Composite
PANi	=	Polyaniline
US-DoE	=	United State Department of Energy
ASTM	=	American Society for Testing and Materials
Gr	=	Graphite
Sn	=	Stannum
PP	=	Polypropylene
S/cm	=	Siemen per Centimeter
MPa	=	Mega Pascal
g/cm^3	=	Gram per $Centimeter^3$
µA	=	Micron Ampere
µm	=	Micron Meter

CHAPTER 1

INTRODUCTION

1.1 FUEL CELL

Fuel cell is an electrochemical energy conversion device which can convert the chemical energy from hydrogen and oxygen into water, heat and in the process it will produces electricity. Hydrogen is commonly acts as fuel and sometimes hydrocarbons such as natural gas and alcohols like methanol are used. Fuel cells are different with batteries because a battery will eventually exhausted when the chemicals stored inside the cell has been used up. For the fuel cells, they can produce electricity as long as the inputs constant source of fuel and oxygen has been supply continuously. The electrolytic process will occurs and electricity will be generated. This process will never end up with the continuous supply of inputs.

Fuel Cells generate electricity through an electrochemical process in which the energy stored in a fuel is converted directly into electricity. Fuel cells are extremely attractive from an environmental stand point 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. 2010), very low chemical and acoustical pollution (Selamat et al. 2013). Unlike power sources that use fossil fuels, the by-products from an operating fuel cell are heat and water. In 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.

1.2 PROBLEM STATEMENT

According to Ahmad et al. (2013), the performance of Polymer Exchange Membrane Fuel Cell (PEMFC) in producing energy is depending on the bipolar plate materials used. The problem that faced by researchers is the properties of carbon-based materials which have poor electrical and thermal conductivity, fragile structure and low mechanical strength (Zi et al. 2006). However, other materials such as metal has high tendency to corrosion, costly and time consuming for machining (Selamat et al. 2013). In order to tackle the weaknesses of pure Gr as bipolar plate, the hybrid conductive composite (HCC) material was chosen as bipolar plate becomes preferable. The Gr/Sn/PP composite bipolar plate can be easily manufactured by hot compression molding method (Antunes et al. 2011). Gr/Sn/PP composite were studied in previous research that can be applied as bipolar plate in PEMFC. The ratio of fillers (Gr/Sn) and binder (PP) when it is 80:20 will gives the best result in the test such as flexure test, density test, hardness and microstructure analysis but the problem faced by the researchers is the electrical conductivity of the bipolar plate is too low and can't achieve the US-DoE target as PEMFC bipolar plate. The effect of formation temperature on the properties of Gr/Sn/PP composite need to be study and improved. From the previous researchers, the formation temperature for compounding process of Gr/Sn/PP composites is set around 200°C and hot pressing around 175°C. Thus affect the electrical conductivity was low due to agglomeration complication and effect of the particles size (Lim, 2014). This experiment need to be done in lower formation temperatures (160 up to 170 °C) and to know the effect on the properties of Gr/Sn/PP composite, several testing need to be conducted such as electrical conductivity, flexure test, density test, hardness and microstructure analysis will be analysed to produce high performance bipolar plate.

1.3 OBJECTIVES

1. Study the effect of formation temperature on the properties of Gr/Sn/PP composite.
2. Determine the suitable process parameter for Gr/Sn/PP composite.

1.4 SCOPE

This research will study the effect of Sn loading on the electrical and mechanical properties of Gr/Sn/PP composite. The ratio of fillers (Gr/Sn) and binder (PP) is fixed at 80:20. The adding of small amount of Sn into Gr/Sn/PP composite thus will gives synergy effects on electrical conductivity and mechanical properties. The small amount of Sn which is 10 up to 20wt% (from the total weight of fillers 80%) will be added into Gr/Sn/PP composite. Before the fabrication process using the hot press at the temperatures of 160 up to 170°C, the mixture of fillers (Gr and Sn) will be mixed used ball mill. The second stage of mixing process between fillers and binder (PP powder) is also carried out by using ball mill machine followed by compaction process using the high speed hot press hydraulic machineand the temperatures will be set at 160 up to 170°C. In order to determine the effect of formation temperatures in Gr/Sn/PP composite, the tests such as electrical conductivity, flexure test, density test, hardness and microstructure analysis will be performed.

CHAPTER 2

LITERATURE REVIEW

2.1 TYPES OF FUEL CELL

There are various types of fuel cells had been developed nowadays and they are classified according to the types of electrolyte used. There are classified as five types, which are polymer exchange membrane fuel cell (PEMFC), alkaline fuel cells (AFC), phosphoric acid fuel cells (PAFC), molten carbonate fuel cells (MCFC) and solid oxide fuel cells (SOFC). Table 2.1 shows the characteristics on each type of fuel cells.

Table 2.1: Characteristics on Each Type of Fuel Cell
(Source: Wee, 2007)

Fuel Cell Type	**Polymer Exchange Membrane (PEMFC)**	**Alkaline (AFC)**	**Phosphoric Acid (PAFC)**	**Molten Carbonate (MCFC)**	**Solid Oxide (SOFC)**
Common Electrolyte	Perfluoro Sulfonic acid	Aqueous solution of potassium hydroxide soaked in a matrix	Phosphoric acid soaked in a matrix	Solution of lithium, sodium, and potassium carbonates, soaked in a matrix	Yttria stabilized zirconia

Fuel Cell Type	Polymer Exchange Membrane (PEMFC)	Alkaline (AFC)	Phosphoric Acid (PAFC)	Molten Carbonate (MCFC)	Solid Oxide (SOFC)
Operating Temperature	50-100 °C 122-212°F typically 80°C	90-100°C 194-212°F	150-200°C 302-392°F	600-700°C 1112-1292°F	700-1000°C 1202-1832°F
Typical Stack Size	<1kW-100kW	10-100kW	400kW 100kW module	300kW-3MW 300kW module	1kW-2MW
Efficiency	60% transporta-tion 35% stationary	60%	40%	45-50%	60%
Applications	Backup power, portable power, distributed generation, transporta-tionand speciality vehicles	Military and space	Distributed generation	Electric utility, distributed generation	Auxiliary power, electric utility and distributed generation

Fuel Cell Type	Polymer Exchange Membrane (PEMFC)	Alkaline (AFC)	Phosphoric Acid (PAFC)	Molten Carbonate (MCFC)	Solid Oxide (SOFC)
Advantages	-Solid electrolyte reduces corrosion & electrolyte management problems -Low temperature -Quick startup	-Cathode reaction faster in alkaline electrolyte, leads to high performance -Low cost components	-Higher temperature enables CHP -Increased tolerance to fuel impurities	-High efficiency -Fuel flexibility -Can use a variety of catalysts -Suitable for CHP	-High efficiency -Fuel flexibility -Can use a variety of catalysts -Solid electrolyte -Suitable for CHP & CHHP
Disadvantages	-Expensive catalysts -Sensitive to fuel impurities -Low temperature waste heat	-Sensitive to CO_2 in fuel and air -Electrolyte management	-Pt catalyst -Long start up time -Low current and power	-High temperature corrosion and breakdown of cell component -Long start up time -Low power density	-High tempera- ture corrosion and breakdown of cell component -High tempera- ture operation requires long start up time and limits.

2.1.1 Polymer Electrolyte Membrane Fuel Cell

Polymer electrolyte membrane fuel cells (PEMFC) have emerged as the most common type of fuel cell under development today. It is also known as the proton exchange membrane fuel cells based on the key characteristics of the solid electrolyte membrane to transfer protons from the anode to the cathode. The solid electrolyte avoids problems caused by liquid electrolytes used in other systems, and the temperature range of <100°C enable rapid start-up under low temperature operation, with operation possible down to subfreezing temperatures. The lower temperature also allows a wider range of materials to be used and enables relatively easy stack design in terms of sealing issues and material selection (Cifrain et al. 2003). This type of fuel cell is the most feasible for use under transportation applications. The highly acidic membrane necessitates the use of highly reactive catalysts, with platinum being the only one in use today sufficiently active to achieve required performances (Wee, 2007). The fuel used can either be pure hydrogen or a hydrogen containing stream, typically produced from a reformed fuel, such as natural gas, methanol or other fuels like kerosene and propane. Figure 2.1 shows the basic diagram of PEMFC.

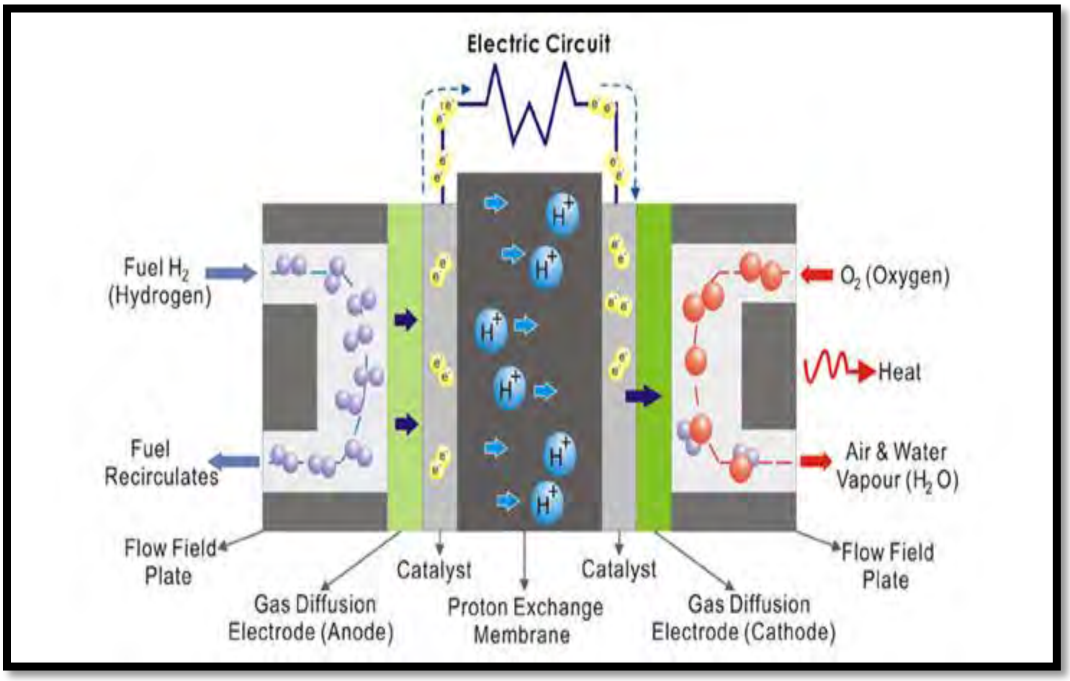

Figure 2.1: Basic Diagram of PEMFC

(Source: Wee, 2007)

2.1.2 Components of PEMFC

Polymer fuel cells (PEMFC) are formed by the membrane electrolyte assembly (MEA), gaskets, gas diffusion layers, bipolar plates, current collectors and endplates. Figure 2.2 shows the majors components in the PEMFC and Table 2.2 shows the materials and function of main components in PEMFC. There are 4 main components of PEMFC:

1. Membrane Electrode Assembly (MEA)
2. Bipolar Plate
3. Current Collector
4. Endplate

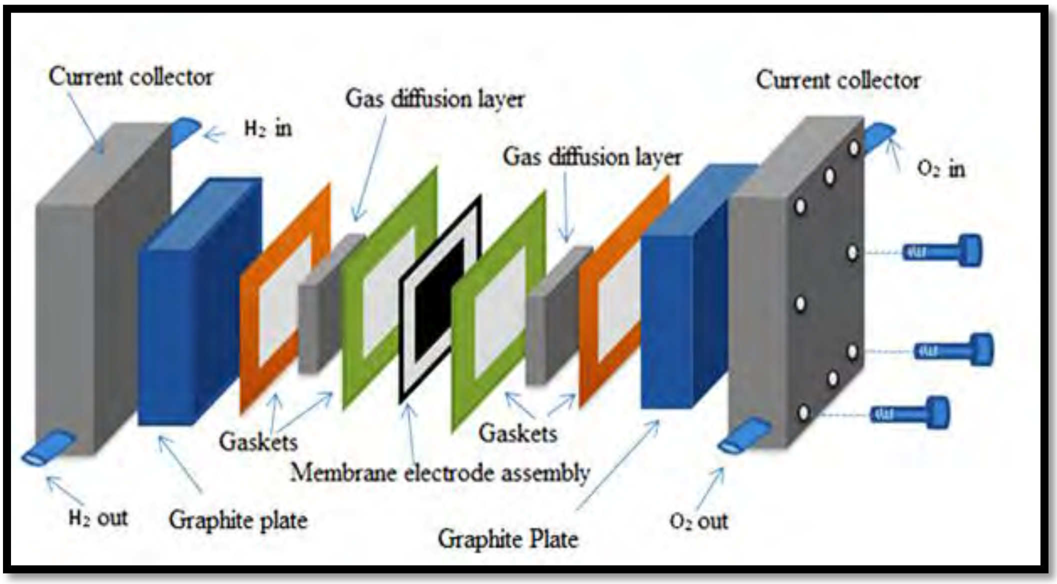

Figure 2.2: Major Components in the PEMFC

(Source: Wee, 2007)

Table 2.2: Materials and Function of Main Components in PEMFC
(Source: Wang, 2006)

Component	Material	Function
Bipolar Plate	Graphite Stainless Steel Thermoplastic Materials	-To transport the incoming gases to the gas diffusion layer (GDL) surface. -To remove the exhaust gases and produce water. -Transfer electrons and heat. -Provide mechanical structure.
Membrane Electrode Assembly (MEA)	Solid polymer electrolyte impregnated with catalyst layers for the anode and cathode. Porous carbon paper or cloth for gas diffusion layer (GDL)	-Transport of the reactant gases to the catalyst layers, allowing the gases to diffuse under the flow-field landings. -Removal of product water. -Transport of electrons from the catalyst layer to the flow-field landings. -Heat conduction. -Mechanical support membrane and catalyst layers to span flow-field channels.
Current Collector	Metal with good electrical conductivity (Copper)	-Accumulate and transfers the current from the stack to the external circuit.
Endplate	Material with good mechanical strength (Steel and aluminium)	-Provides integrated assembly for the entire fuel cell stack.

2.1.3 Bipolar Plate

Bipolar plates (also known as flow-field plates) are as the most crucial component for the Polymer Electrolyte Membrane Fuel Cell (PEMFC) which is assembled on either side of the GDLs, the entire unit comprising the unit cell of the fuel cell stack. The plates are typically made out of carbon or graphite-polymer composite materials or metals. The bipolar plates act an important role due to its multi-functional in the fuel cell stack. According to Wang (2006), the functions of the bipolar plate in PEMFC are shown in Table 2.3.

Table 2.3: Functions of the Bipolar Plate in PEMFC
(Source: Wang, 2006)

No.	Functions of Bipolar Plate
1	Conducting electrons to complete the circuit. -collect and transport electrons from the anode and cathode. -connect individual fuel cells in series to form a fuel cell stack of the required voltage (i.e., fuel cells are typically arranged in a bipolar configuration).
2	A flow path for gas transport was provided to distribute the gases over the entire electrode area uniformly.
3	Separating oxidant and fuel gases by feeding hydrogen to the anode and oxygen to the cathode, while removing product water and un-reacted gases.
4	Providing mechanical strength and rigidity to support the thin membrane and electrodes and clamping forces for the stack assembly.
5	Providing thermal conduction to help regulate fuel cell temperature and removing heat from the electrode to the cooling channels.

According to the United State Department of Energy (US-DoE), there are some basic requirements that need to be achieved by a standard bipolar plate due to its multiple responsibilities and the challenging environment in which the fuel cell operates (Wee, 2007). Table 2.4 shows the requirement of US-DoE for bipolar plate.

Table 2.4: Requirement of US-DoE for Bipolar Plate

Electrical Conductivity	>100 Scm^{-1}
Chemical Stability and Corrosion Resistant	<1 μAcm^{-2}
Thermal Conductivity	>10W $(mK)^{-1}$
Bulk Density	<5 g/cm^{-3}
Flexural Strength	>25MPa
Shore hardness	>50

For the design criteria, the thickness of the bipolar plate is the important criterion that needs to be taken into consideration when designing the bipolar plate for the PEMFC because the stack volume and electrical resistance can be minimized by changing the thickness of bipolar plate. The bipolar plate must be impermeable to prevent the mixing of the oxidant and the fuel. Due to the acidic environment present in the fuel cell, the material must be corrosion resistant. As a bipolar plate, it should also be thermally conductive to remove the heat from fuel cell, electrically conductive to minimize Ohmic losses, offer good mechanical strength and rigidity to support the electrolyte membrane and withstand the fastening forces (Antunes et al. 2011). In order to fulfil the design criteria, function and cost targets for fabrication of the bipolar plate, the researchers are doing improvement and researches on the material selection to produce the high performance bipolar plate with lower cost.