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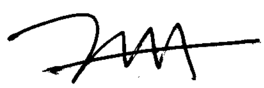
Signature : 

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Date : 30 JUNE 2015

THE EFFECT OF CARBON NANOTUBE ON THE PROPERTIES OF GRAPHITE, CARBON BLACK AND EPOXY COMPOSITE FOR THE BIPOLAR PLATE

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This report is submitted to Faculty of Mechanical Engineering as a requirement to get award of Degree of Mechanical Engineering (Structure & Material)

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JUNE 2015

DECLARATION

"I hereby declare that the work in this report is my own except for summarise and quotations which have been duly acknowledgement."

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ABSTRACT

Nowadays, conductive polymer composite (CPCs) bipolar plate had been the target to replace the pure-graphite or metal based bipolar plate. This is due to the low manufacturing cost and light of total mass of bipolar plate in Polymer Electrolyte Membrane Fuel Cells (PEMFCs). PEMFCs are able to efficiently generate high power densities, thereby making the technology potentially attractive for certain automobile industry and portable applications. Meanwhile, single filler in polymer-graphite composites have shown low electrical conductivity and mechanical properties. Therefore, the aim of this research is to study the effect of Carbon Nanotube (CNTs) as third filler on the electrical and mechanical properties of Graphite (Gr), Carbon Black (CB) and Epoxy (EP) composite. The second aim of this research is to determine the critical loading of CNTs in Gr, CB and EP composite. The ratio of fillers (Gr/CB /CNTs) and binder (EP) is fixed at 80: 20. The adding of small amount of CNTs into Gr/CB/EP composite thus will gives synergy effects in electrical conductivity and mechanical properties. The small amount of CNTs which is 0, 5, 10, 15 wt% (from the total weight of fillers 80%) will be added into Gr/CB/EP composite. Before the fabrication process using the hot press, the mixture of Gr, CB and CNTs will be mixed used ball mill. Therefore, it will mix with EP by using the blender. In order to determine the effect of CNTs content in Gr/CB/EP composite, the tests such as electrical conductivity, flexure strength test, bulk density test and hardness had been carried out. The used of CNTs as third filler of 10 wt% in Gr/CB/EP composites shown the best result in the in-plane electrical conductivity, flexural strength, density and hardness being 616.22 S/cm, 12.80 MPa, 1.200 g/cm^3 and 50.3 respectively.

ABSTRAK

Pada masa kini, konduktif polimer komposit (CPC) plat bipolar telah disasarkan untuk menggantikan plat bipolar tulen - grafit atau berasaskan logam. Hal ini adalah kerana kos pembuatan yang rendah dan jumlah jisim plat bipolar yang ringan di Sel Polimer Elektrolit Membran Bahan Api (PEMFCs). PEMFCs berupaya menjana secara cekap ketumpatan kuasa yang tinggi, sekali gus menjadikan teknologi ini berpotensi untuk aplikasi dalam industri automotif dan penjana kuasa mudah alih. Sementara itu, pengisi tunggal dalam komposit polimer - grafit telah menunjukkan kekonduksian elektrik dan sifat-sifat mekanik yang rendah. Jadi, tujuan utama kajian ini adalah untuk mengkaji kesan tiub nano karbon (CNTs) sebagai pengisi ketiga terhadap sifat-sifat elektrik dan mekanik pada Grafit (Gr) / Karbon Hitam (CB) / epoksi (EP) komposit. Tujuan ke dua, kajian ini adalah untuk menentukan muatan kritikal CNTs dalam Gr, CB dan EP komposit. Nisbah pengisi (Gr/CB/CNTs) dan pengikat (EP) ditetapkan pada 80:20. Penambahan sedikit CNTs ke Gr/CB/EP komposit dengan itu akan memberi kesan sinergi dalam kekonduksian elektrik dan sifat-sifat mekanik. Penambahan CNTs seperti 0, 5, 10, 15 wt.% (dari jumlah berat pengisi 80%) telah ditambahkan dalam Gr /CB/ EP komposit. Sebelum proses fabrikasi dengan menggunakan penekanan panas, campuran Gr, CB dan CNTs perlu dicampur dengan mengunakan pengisar bola. Selepas itu, campuran disebatikan dengan EP dengan menggunakan mesin pengisar. Dalam usaha untuk menentukan kesan kandungan CNTs dalam Gr/CB /EP komposit, ujian seperti kekonduksian elektrik, ujian kekuatan lenturan, ujian ketumpatan pukal dan kekerasan telah dijalankan. Kandungan CNTs yang digunakan sebagai pengisi ketiga sebanyak 10 wt% dalam komposit Gr/CB/EP menunjukkan keputusan terbaik bagi satah kekonduksian elektrik, kekuatan lenturan, kepadatan dan kekerasan menjadi 616,22 S/cm, 12.80 MPa, 1.200 g/cm^3 dan 50.3.

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LIST OF ABBREVIATION AND SYMBOL

CNTs	=	Carbon Nanotubes
CB	=	Carbon Black
Gr	=	Graphite
EP	=	Epoxy
PEMFC	=	Polymer Electrolyte Membrane Fuel Cell
H_2	=	Hydrogen
O_2	=	Oxygen
DC	=	Direct Current
ΔGr	=	Gibbs Energy
°C	=	Degree Celsius
°F	=	Fahrenheit
AFC	=	Alkaline Fuel Cell
PAFC	=	Phosphoric Acid Fuel Cell
MCFC	=	Molten Carbonate Fuel Cell
SOFC	=	Solid Oxide Fuel Cell
MEA	=	Membrane Electrolyte Assembly
GDL	=	Gas Diffusion Layer
S/cm	=	Siemen per centimetre
mK	=	Mili Kelvin
MPa	=	Mega Pascal
g/cm^3	=	Gram per centimetre cube
wt %	=	Weight Percentage
CPCs	=	Conductive Polymer Composites
MWNT	=	Multi-Walled Nanotubes
SWNT	=	Single-Walled Nanotubes

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CHAPTER 1

INTRODUCTION

1.1 OVERVIEW

Fuel cells are playing a very important role in nowadays. Fuel cells are fundamentally much more energy-efficient than conventional power sources. At the same time, it can achieve as high as 80% system efficiency in integrated units with cogeneration of heat utilization (Ling Du, 2008). In this century, it cannot be denied that fuel cells are expected to play a major role in the economy. In order to reduce the impact of our energy consumption on the environment, fuel cells represent an attractive solution, for instance addressing the issue of intermittent behaviour of renewable sources (Planes et al., 2012).

In research of Shimshon and Tom (2008), the proton exchange membrane fuel cells (PEMFCs) convert hydrogen and oxygen (or air) into electricity at low operation temperature (< 100 ◦*C*). The PEMFC fuel cell is also sometimes called a polymer electrolyte membrane fuel cell (PEMFC). Meanwhile, in order to improve their commercial liability, Planes et al. (2012) found out some applications as for automotive application may require better volumetric and gravimetric power density (>1kW/L and 1kW/kg). Among various types of fuel cells, Ling Du (2008) state that PEMFCs are considered a promising candidate for a zero-emission power source required in environmentally friendly transportation and stationary applications. The PEMFCs is recognized by the U.S. Department of Energy (DOE) as the main candidate to replace the internal combustion engine in transportation applications (Ling Du, 2008). In the recent years, the PEMFCs has received intensive researches from both alternative energy and environmental consideration owing to their attractive features of high power density, low operating temperature and converting fuel to water as the only by product of fuel cells (Selamat. et al., 2013). Hence, it especially for bipolar plate which accounts for nearly 38% in a fuel cell stack cost (Selamat, et al., 2013), it is one of the most costly components in PEMFCs. Therefore, the study on performance materials of bipolar plates (BPs) has become a very hot and critical research issue nowadays. On the material stand point, BPs application should thus present a balance of various physical and chemical properties. The BPs materials or composite materials properties must be considered for achievable design/performance for a fuel cell application, specifically, electrical and thermal conductivity, gas permeability, mechanical strength, corrosion resistance and low weight (Mathur, et al., 2008). The ideal bipolar plate must meet the target properties specified by DOE. The properties requirements shown in Table 1.1 should be satisfied for the fabrication of a bipolar plate.

Table 1.1: Requirement properties for the bipolar plate which is DOE target (Source: Yeetsorn et al., 2012)

Properties	**Value**
Electrical conductivity	> 100 [S/cm]
Thermal conductivity	>10 [W/(mK)]
Shore Hardness	> 50
Flexural strength	> 25 [MPa]
Bulk density	<5 [g/cm3]

The conventional materials for fabricating bipolar plate are based on either pure graphite materials or metals. Graphite is the most commonly used as material for bipolar plate, but the disadvantages of pure graphite are its brittleness and difficulty in machining process. Thus, require thick bipolar, which that the fuel cell stack that become large and heavy (Suherman,. et al. 2013). However, conductive polymer composites (CPCs) as bipolar plate's materials are become an attractive option for PEMFCs. CPCs is fabricate from the mixed of conductive fillers such as Graphite (Gr), Carbon Black (CB) and Carbon Nanotube (CNT) had been incorporated in Epoxy (EP) as matrix for fabrication of electrical conductive polymer composite plate. Hence, in order to obtain the better electrical conductivity of this composite, the combinations of multi fillers have been used as bipolar plate materials (Selamat, et al., 2013). Meanwhile the reinforced fillers commonly used including Gr, CB and CNTs which have been incorporated into composites materials to enhance the overall performance. Thus composite of bipolar plates had been prepared by conventional polymer processing technique (Selamat, et al., 2013).

1.2 PROBLEM STATEMENT

The performance PEMFC in producing energy is depending on the bipolar plate materials has been used (Ahmad, et al., 2013). In research of Suherman, et al. (2013), the single filler in polymer-graphite composites have shown low electrical conductivities and mechanical properties. While, the additional of CB until 30% as second filler, will not be wetted well with polypropylene (PP) resin and its will deteriorate the conductivity of the composite because of incomplete compaction (Selamat, et al., 2013). At the same time, it seems that the PP resin cannot wetted well all the fillers and this caused the electrical conductivity decreases as the CNTs contents increases. The weight percentage of CNTs may increases to get better electrical conductivities and mechanical properties (Suherman, et al., 2013). While, the weight percentage of CNTs can be increased up to 15 wt% and this may the maximum weight percentage that it can be dispersed into other materials while doing the fabrication of composites bipolar plate (Bairan, 2013). Therefore, PP as matrix materials may need to change to others materials. EP have low-viscosity may be selected to replace the PP due to epoxy have better wetting conditions with conductive fillers. Therefore, the research will determine the effect of CNT on the properties of Gr,CB and EP composites for bipolar plate. It will also determine the critical loading of CNTs on electrical conductivities and mechanical properties of Gr, CB and EP composites as bipolar plate.

1.3 OBJECTIVES

The main objective of this research is to study the effect of Carbon Nanotube (CNTs) as third filler on the mechanical and electric properties of Graphite (Gr), Carbon Black (CB) and Epoxy (EP) composite. The second objective of this research is to determine the critical loading of CNTs in Gr, CB and EP composite.

1.4 SCOPE PROJECT

This research will study the effect of CNTs loading on the electrical and mechanical properties of Gr, CB and EP composite. The ratio of fillers (Gr , CB and CNTs) and binder (EP) is fixed at 80 : 20. The adding of small amount of CNTs into Gr, CB and EP composite thus will gives synergy effects in electrical conductivity and mechanical properties. The small amount of CNTs which is 0, 5, 10, 15 wt.% (from the total weight of fillers 80%) will be added into Gr, CB and EP composite. Before the fabrication process using the hot press, the mixture of Gr, CB and CNTs will be mixed used ball mill. In order to determine the effect of CNTs content in Gr, CB and EP composite, the tests such as electrical conductivity, flexure test, density test, and hardness will be carry out.

CHAPTER 2

LITERATURE REVIEW

2.1 FUEL CELL

Fuel cell is a device of electrochemically which convert the chemical free energy of gaseous or liquid reactants into direct current (DC) electricity. In the simplest case of fuel cell, it was operating with hydrogen (fuel) and oxygen (air) as reacting gases. Figure 2.1 was shows the basic fuel cell operation. In current research (Kreuer, 2013), a proton or oxide ion was current equivalent to the electrolyte and parts of the heterogeneous electrode structures. The driving force of this process is the Gibbs energy (ΔG_r) of the reaction:

$$H_2 + \frac{1}{2}O_2 \rightarrow H_2O$$

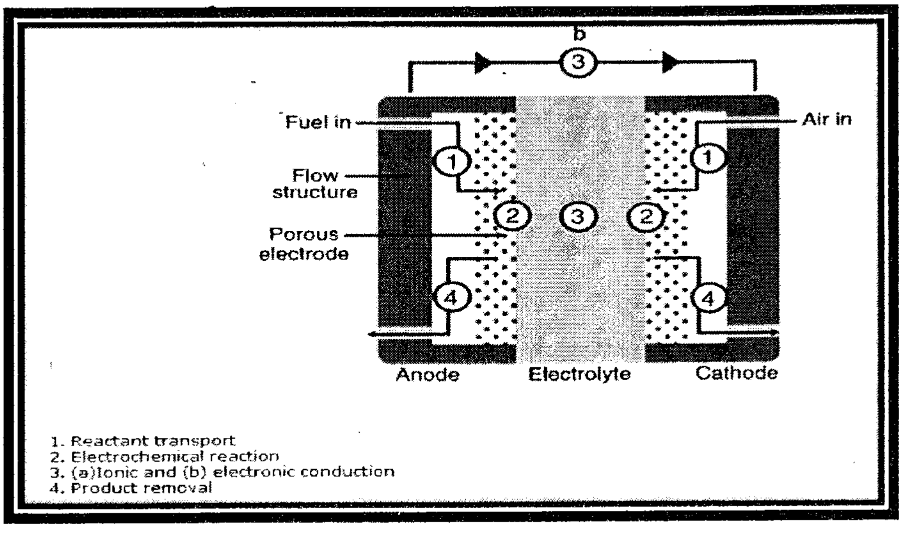

Figure 2.1: Basic Fuel cell operation

(Source: E-learning course of IITS & IISC. NPTE, 2013)

In the Table 2.1 shows five different types of fuel cells with its characteristics like common electrolyte, operating temperature, typical construction, internal reforming, oxidant, efficiency and its application.

Table 2.1: Different type of fuel cell
(Source: Barbir, 2013)

Fuel cell type	**Alkaline fuel cell (AFCs)**	**Polymer electrolyte membrane fuel cell (PEMFCs)**	**Phosphoric acid fuel cell (PAFCs)**	**Molten Carbonate fuel cell (MCFCs)**	**Solid Oxide fuel cell (SOFCs)**
Common electrolyte	OH^- ions (typically aqueous KOH solution)	H^+ ions (with anions bound in polymer membrane)	H^+ ions (H_3PO_4 solution)	$CO_3^=$ ions (typically molten $LiKaCO_3$ eutectics)	$O^=$ ions (stabilized ceramic matrix with free oxide ions)
Operating temperature	190-500°F (90-260°C)	150-180°F (65-85°C)	370-410°F (190-210°C)	1200-1300°F (650-700°C)	1350-1850°F (750-1000°C)
Typical construction	Plastic ,metal	Plastic, metal or carbon	Carbon , porous ceramic	High temperature metal , porous ceramic	Ceramic , high temperature metals
Internal reforming	No	No	No	Yes , good temperature match	Yes , good temperature match
Oxidant	Purified air to O_2	Air to O_2	Air to enriched air	Air	Air
Efficiency	60%	60% transportation 35% stationary	40%	45-50%	60%
Application	-military -space	- backup power - portable power - distributed generation - transportation	- distributed generation	- electric utility - distributed generation	- distributed generation - auxiliary power - electric utility

2.2 POLYMER ELECTROLYTE MEMBRANE FUEL CELLS (PEMFCs)

Polymer electrolyte membrane fuel cells (PEMFCs) are able to efficiently generate high power densities, thereby making the technology potentially attractive for certain mobile and portable applications. Despite the fact that fuel cells have many advantages, such as high conversion efficiency at partial load, clean exhaust gases, modular design and low noise production (Barbir, 2013). Meanwhile, their marketability will depend heavily on whether these fuel cells can compete with the incumbent technologies on performance, cost, and reliability in specific application. Due to the cell separator is a polymer film and the cell operates at relatively low temperatures, issues such as sealing, assembly, and handling are less complex than most other fuel cells. Wang, et al. (2013) had mentioned that in their about PEMFCs technology differentiates itself from other fuel cell technologies in that a solid phase polymer membrane is used as the cell separator/electrolyte.

The PEMFCs is seen as the main fuel cell candidate technology for light-duty transportation applications. While PEMFC are particularly suitable for operation on pure hydrogen, fuel processors have been developed that will allow the use of conventional fuels such as natural gas or gasoline (Wang, et al., 2013). Figure 2.2 shows the schematic of PEMFCs.

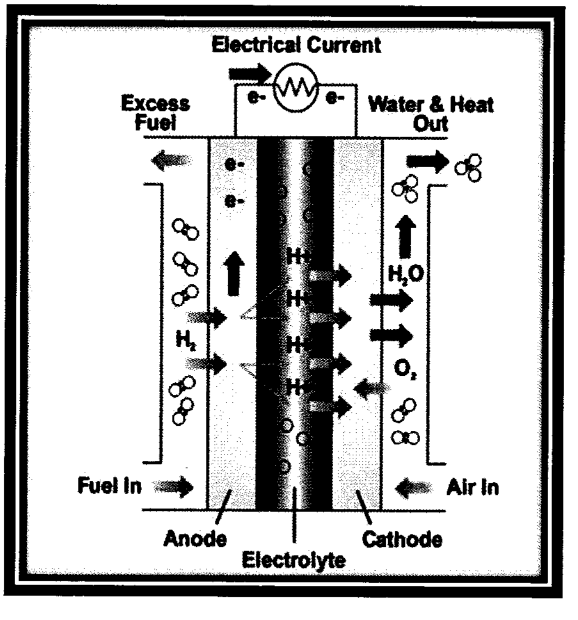

Figure 2.2: Schematic of PEMFCs

(Source: E-leTE, 2013)

2.3 MAIN COMPONENTS OF FUEL CELL

Figure 2.3 shows the structure diagram of PEMFC. There are four main components of PEMFCs which are Membrane Electrode Assembly (MEA), Bipolar plate (BPs), End plate and Current Collector. The MEA is the heart of the PEMFCs and much work is currently being done to find cheaper and thinner membranes whilst maintaining durability. Table 2.2 shows the primary components of PEMFCs with its function.

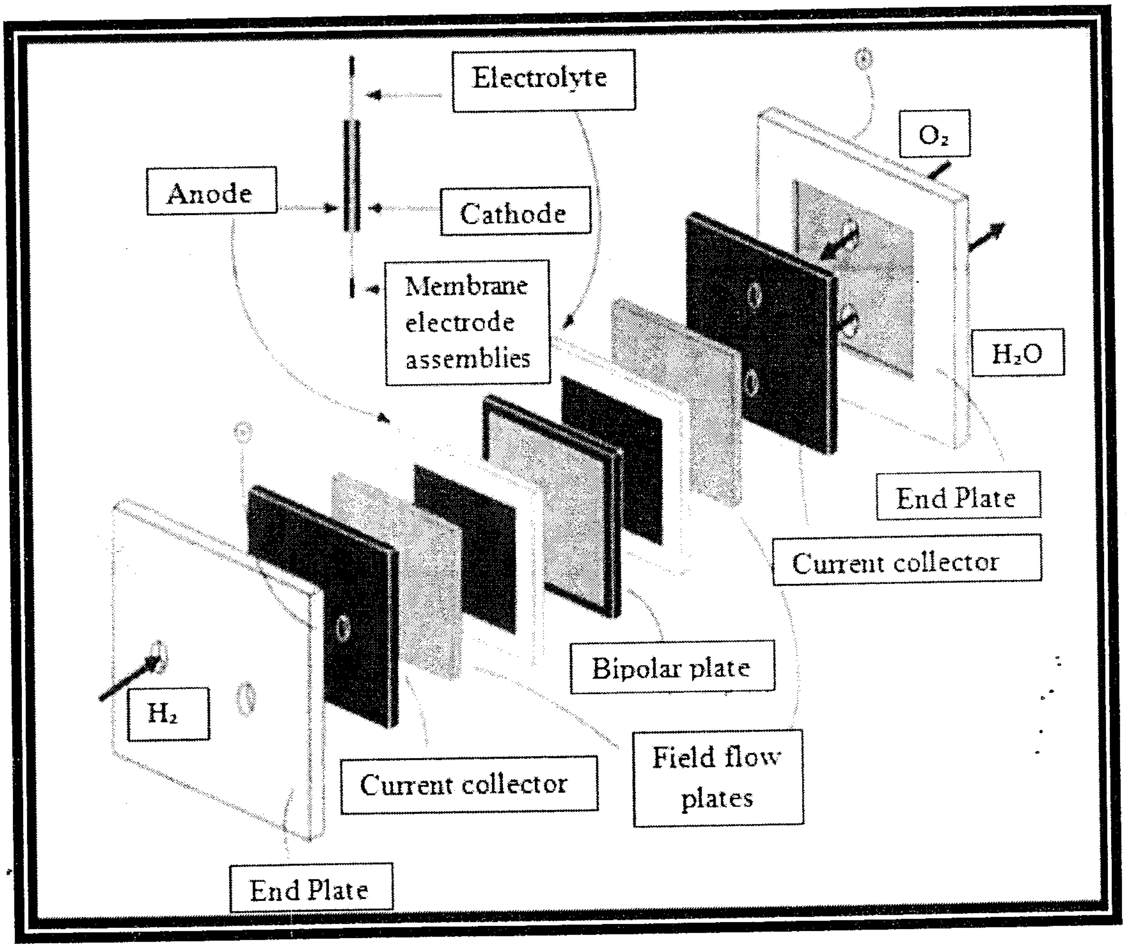

Figure 2.3: Structure diagram of PEMFCs
(Source: E-learning course of IITS & IISC. NPTE, 2013)

Table 2.2: Primary components of a PEMFC
(Source: Wang, 2006)

Components	Material	Functionality
Membrane Electrode Assembly (MEA)	Solid polymer electrolyte impregnated with catalyst layers for the anode and cathode Porous carbon paper or cloth gas diffusion layer (GDL)	Consists of two electrodes, a membrane electrolyte and two GDLs. The membrane separates (with gas barrier) the two half-cell reactions and allows protons to pass through from anode to cathode. The dispersed catalyst layers on the electrodes promote each half reaction. The GDL evenly distributes gases to the catalyst on the membrane, conducts electrons from the active area to the bipolar plates and assists in water management.
Bipolar Plate (BPs)	Graphite , stainless steel , or thermoplastics materials	Distributes gases over the active area of the membrane. Conducts electrons from the anode of one electrode pair to the cathode of next electrode pair. Carries water away from each cell.
End Plate	Material with good mechanical strength (normally steel or aluminium)	Provides integrated assembly for the entire fuel cell stack.
Current Collector	Metal material with good electric contact and conductivity (normally copper)	Collects and transfers the current from the stack to an external circuit.