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DEVELOPMENT AND CHARACTERIZATION OF CONDUCTIVE GRAPHENE-COATED PREPREG COMPOSITES FOR AVIATION APPLICATION

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DEVELOPMENT AND CHARACTERIZATION OF CONDUCTIVE GRAPHENE-COATED PREPREG COMPOSITES FOR AVIATION APPLICATION

By

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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July 2020

Chairman : Mohd Roshdi Hassan, PhD
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Developing a conductive composite using graphene is very important in manufacturing aircraft's structures toward enhancing their electrical and thermal properties for future aviation application. Consequently, this study was conducted to develop a method to coat carbon fiber reinforced epoxy composite (CFRE) with reduced graphene oxide (rGO) and functionalized graphene nanoplates (FGNP) to improve and enhance the electrical conductivity and self-heating properties of CFRE in order to obtain an outstanding conductive composite . Hence, this study started with identifying the gab in evaluating the available carbon fiber reinforced epoxy composite, then painting method was used in this study to coat CFRE with rGO, while inkjet printing method was used to coat CFRE with FGNP, after that the conductive composites (CFRE/rGO and CFRE/FGNP) was fabricated using hot press molding method, then many experiments were conducted on conductive composites to evaluate it's electrical and thermal properties. Firstly, atomic force microscopy, morphological characterization and tensile tests were conducted to evaluate the quality of conductive composites. Secondly, four point probe device was used to measure the electrical conductivity value for conductive composites. Finally, experimental work were conducted to study the self-heating of conductive composites.

The results showed that rGO and FGNP coating layers were uniformly and homogeneously distributed over the surface of CFRE, and there was no flaws or creation of voids between the coating layers and CFRE surface. It was found that the electrical conductivity of CFRE coated with rGO was enhanced and increased significantly by 81 times from 1.38 x 10^3 (S/m) to 1.12 x 10^5 (S/m) and with percentage of improvement of 8100 %, while, the electrical conductivity of CFRE coated with printed FGNP is enhanced and increased by 22 times from

1.38 x 10^3 (S/m) to 3.1 x 10^4 (S/m) with percentage of improvement of 2146 %. The experimental results showed that the self-heating of CFRE composite was improved and enhanced significantly after coating it with rGO and FGNP. The self-heating was homogeneous, effective and reached higher temperatures more than CFRE neat. It clear that the objectives of this study were achieved, where the conductive composites were developed using rGO and FGNP , hence the electrical and thermal properties were improved and enhanced significantly, which can contribute in aviation applications in the future .

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGEMBANGAN DAN PENCIRIAN KOMPOSIT PREPREG BERSALUT GRAPHENE KONDUKTIF BAGI KEGUNAAN PENERBANGAN

Oleh

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Membangunkan komposit yang bersifat pengalir dengan menggunakan graphene adalah sangat penting dalam pembuatan struktur pesawat untuk meningkatkan sifat elektrik dan haba mereka untuk aplikasi penerbangan di masa hadapan. Oleh yang demikian, kajian ini dilakukan untuk mengembangkan kaedah untuk melapisi komposit epoksi bertetulang gentian karbon (CFRE) dengan oksida graphene berkurang (rGO) dan nanoplate graphene berfungsi (FGNP) untuk memperbaiki dan meningkatkan pengaliran elektrik dan sifat pemanasan diri pada CFRE bagi menghasilkan komposit yang bersifat pengalir konduktif yang lebih tinggi. Oleh itu, kajian ini dimulakan dengan mengenal pasti jurang dalam menilai komposit berepoksi bertetulang serat karbon yang ada, kemudian kaedah melukis digunakan dalam kajian ini untuk melapisi CFRE dengan rGO, sementara kaedah percetakan secara inkjet digunakan untuk melapisi CFRE dengan FGNP, setelah itu komposit konduktif (CFRE / rGO dan CFRE / FGNP) dibuat menggunakan kaedah cetak panas, kemudian banyak eksperimen dilakukan pada komposit konduktif untuk menilai sifat elektrik dan haba. Pertama, mikroskopi daya atom, pencirian morfologi dan ujian tegangan dilakukan untuk menilai kualiti komposit konduktif. Kedua, alat pengesan empat titik digunakan untuk mengukur nilai pengaliran elektrik bagi komposit konduktif. Akhirnya, kerja eksperimen dilakukan untuk mengkaji pemanasan sendiri komposit konduktif.

Hasil kajian menunjukkan bahawa lapisan salutan rGO dan FGNP disalut secara seragam dan sekata ke atas permukaan CFRE, dan tidak ada kekurangan atau terhasilnya rongga antara lapisan dan permukaan CFRE. Didapati bahawa pengaliran elektrik pada CFRE yang dilapisi dengan rGO dapat ditingkatkan dan meningkat dengan ketara sebanyak 81 kali ganda dari 1.38×10^3 (S / m) kepada 1.12×10^5 (S / m). Sementara, pengaliran elektrik CFRE yang dilapisi dengan

FGNP yang bercetak dapat ditingkatkan dan meningkat sebanyak 22 kali dari 1.38×10^3 (S / m) kepada 3.1×10^4 (S / m) . Hasil eksperimen menunjukkan bahawa pemanasan sendiri pada komposit CFRE dapat diperbaiki dan ditingkatkan dengan ketara setelah disalut dengan rGO dan FGNP. Pemanasan sendiri adalah sekata, berkesan dan mencapai suhu yang lebih tinggi jika dibandingkan dengan CFRE sahaja. Jelas bahawa objektif kajian ini tercapai, di mana komposit konduktif dapat ditingkatkan dengan menggunakan rGO dan FGNP, oleh itu sifat pengaliran elektrik dan haba dapat diperbaiki dan ditingkatkan dengan ketara, yang dapat menyumbang dalam aplikasi penerbangan di masa depan.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CB	Carbon Black
CF	Carbon Fiber
CNTs	Carbon Nanotubes
GO	Graphene Oxide
CVD	Chemical Vapour Deposition
GNPs	Graphene Nano Plates
rGO	Reduced Graphene Oxide
GNFs	Graphene Nano Flakes
FGO	Few Layers Graphene Oxide
Ag	Silver
CFRP	Carbon Fiber Reinforced Plastic Materials
CFRE	Carbon Fiber Reinforced Epoxy Composite
LSP	Lightning Strikes Protection Systems
GTF	Graphene Thin Film
GrF	Graphene Foam
MWCNT	Multi Wall Carbon Nanotube
FGNs	Foliated Graphite Nanosheets
Gr	Graphite
EGr	Expanded Graphite
FGNP	Functionalized Graphene Nanoplates
GNR	Graphene Nanoribbons
AgnP	Silver Nanoparticles
FGMs	Functionally Graded Materials
AFM	Atomic Force Microscopy
V	Voltage
R	Electrical Resistance
σ	Electrical Conductivity
SEM	Scanning Electron Microscopy
σ_{max}	Ultimate Tensile Strength
E	Stiffness
IPS	Ice Protection System

CHAPTER 1

INTRODUCTION

1.1 Background

Metals have been used in aircraft manufacturing for decades. For example, four decades ago, aluminium was the dominant material in the aircraft industry, because it has many advantages such as lightweight, cheap and resistant to corrosion. Steel is also used in the aircraft industry, but at a much lower rate than aluminium, it has a higher value of tensile strength and elasticity, therefore, the steel used in the manufacturing of parts that need more strength in the aircrafts such as landing gear. In the 1970s. However. Metals are heavy, costly, need expensive maintenance, and prone to corrosion. Therefore, aircraft manufacturers recently turned to using composite materials in their aircraft industry, due to its distinctive advantages. Composite materials have lightweight, where the use of these lightweight materials in the manufacture of aircraft structures, leads to improve the fuel efficiency and performance of the aircraft, in addition to reducing operating costs in the long run. Composites also provide structural strength comparable to metallic alloys (stronger than aluminium or steel). Composites are five times lighter and seven times stronger than aluminium. Therefore, they are important to the aviation industry. Composite materials can resist the damage and fatigue; where it can absorb impacts caused by external sudden force. Composites also may be moulded into complex shapes more easily than other materials, this makes the designers to create nearly any shape or form freely (Nayak, 2014 and Sardiwal, 2014). In addition to that, composites can resist the corrosion caused by the weather and the harsh chemicals.

Common composite materials used on airplanes include fiberglass, carbon fiber reinforced epoxy composites. Glass fiber is one of the composite materials used in the manufacture of aircraft since the fifties of the last century, it is used in the manufacture of a Boeing 707 structure, with percentage of 2%, while the structure of Boeing 787 is comprised of more than fifty percent carbon fiber composite. Figure 1.1 shows the materials used in the structure. The weight of aircraft components made of composite materials is reduced by approximately 20%, such as in the case of the 787 Dreamliner. Airbus Company also used composites in primary airplane structures in the early 1980s. In the late 1990s, constructed the first carbon fiber keel beam for a large commercial airplane A340; composites are used throughout the new A380 Airbus (Peters, 2013, Baker, 2004, Jia-xiang, 2003, and Brick, 1988).

With the increased risks facing the aircraft in the air, such as lightning strike and ice accumulation, which leads to a significant decrease in its performance. It was necessary to find a new conductive composite material to solve these problems, because carbon fiber composite used in manufacturing the structure of aircraft does not conduct electricity well, therefore, they are subjected to repeated destruction by weather conditions. Nano-sized reinforcements like: Carbon nanotubes (CNTs), nanofiber and graphene are considered as key elements of the next-generation reinforced composites. With the incorporation of these nano-reinforcements into polymeric matrices, the mechanical, electrical and thermal properties can be enhanced without additional fillers. Therefore, the use of these conductive composites in the aircraft industry will solve these problems effectively; where conductive composite materials have allowed engineers to overcome difficulties and now they are frequently used in the aircraft industry. (Katunin, 2017 and Peters, 2013).

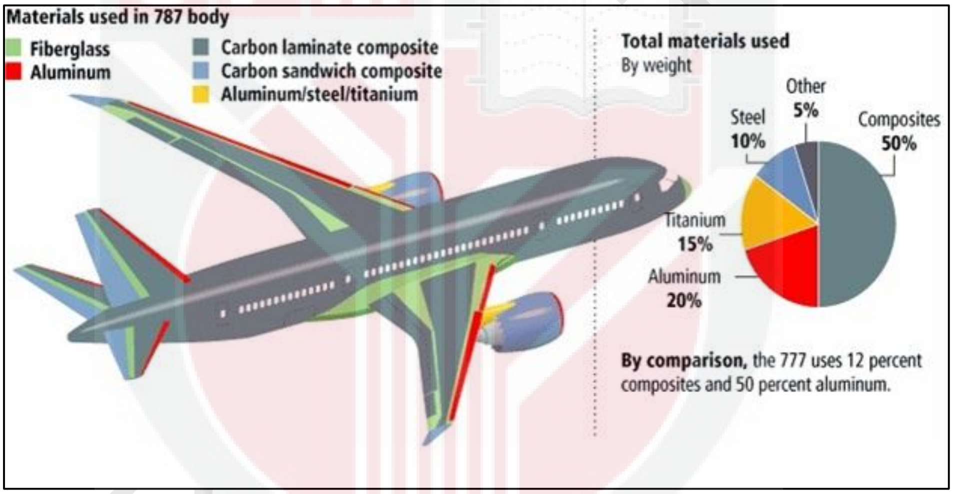

Figure 1.1 : Usage of various materials in the Boeing 787 Dreamliner (Jia-xiang, 2003, and Brick, 1988)

1.2 Problem Statement

Lightning can affect airplanes; lead to sparks, such sparking would present a danger of ignition of materials inside the aircraft skin. Lightning protection systems must provide a continuous conductive path of low resistance over the entire aircraft exterior, with additional protection in zones where lightning is most likely to attach. (Du, 2008 and Nayak, 2014). Ice accumulation on aircraft's surfaces represents also a major threat to safe and efficient operation. It has been an issue addressed by the aerospace industry since the dawn of flight. Ice accumulation on the leading edge of aircraft such as: wings and tails or within the aircraft's engine, is a serious problem that reduces the performance of the aircraft (Sørensen, 2015). Carbon fiber reinforced epoxy composite was used with high percentage in manufacturing the structures of aircrafts to reduce its weight and improve aircraft performance. Despite the advantages of carbon fiber reinforced epoxy composite, it is still unable to protect the aircraft from hitting lightning and de-icing its surfaces efficiently, Because carbon fiber reinforced epoxy composite does not have high electrical conductivity that is sufficient to transfer the high currents coming from lightning strikes without damage , in addition to that , self-heating property of carbon fiber reinforced epoxy composite is not sufficient to de-ice the aircraft surfaces efficiently, therefore it is necessary to improve the electrical and thermal properties of carbon fiber reinforced epoxy composite , in order to be used efficiently in aviation applications .

Significant effort has been exerted to develop surfaces that simplify the removing of ice or preventing its formation, and has low electrical resistance to carry lightning currents without significant damage to the aircraft. The use of the conductive composite materials in the manufacture of aircraft surfaces helps in reducing the weight of the aircraft and thus increases its performance. Finally Conductive composite materials can be investigated as a method to provide electro-thermal anti-icing/de-icing to new generation of aircrafts, because they have high thermal conductivity.

1.3 The Project Objectives

The objectives of this research work are to:

1. Develop a method to coat carbon fiber reinforced epoxy composite with reduced graphene oxide and functionalized graphene nanoplates, and evaluate the quality of coating using characterization, atomic force microscopy and tensile tests.
2. Evaluate the electrical conductivity of carbon fiber reinforced epoxy composite after coating it with reduced graphene oxide and functionalized graphene nanoplates using electrical conductivity test.
3. Evaluate the self-heating property of carbon fiber reinforced epoxy composite after coating it with reduced graphene oxide and functionalized graphene nanoplates using experimental work of self-heating.

1.4 Scope and limitations

Although there are many types of carbon fiber reinforced polymer composite, this research is limited to study carbon fiber reinforced epoxy composite in the aviation. The considered fiber and matrix are carbon fiber and epoxy respectively, which are commonly used in manufacturing the aircrafts structure. Different conductive fillers were used to improve the properties of carbon fiber reinforced epoxy composite, one of these conductive fillers is carbon – based material like carbon nanotube, carbon black, and graphene, this research is limited to use graphene with carbon fiber reinforced epoxy composite to enhance its properties. For considered graphene, this research is limited to use reduced graphene oxide and functionalized graphene nameplates with carbon fiber reinforced epoxy composite. Although there are different methods to prepare carbon fiber reinforced epoxy composite with graphene, this research is limited to use hand- layup method using hot press machine, and ink jet printer to prepare the specimens. For considered carbon fiber reinforced epoxy composite with graphene limited number of properties were utilized in this study, the considered properties of carbon fiber reinforced epoxy composite with graphene were limited in this study to electrical conductivity, electrical resistance, and resistivity and self-heating. For considered composite, the tensile axial load were conducted on the specimen to evaluate its electrical conductivity.

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LIST OF PUBLICATIONS

Alemour, B., Yaacob, M. H., Lim, H. N., & Hassan, M. R. (2018). Review of Electrical Properties of Graphene Conductive Composites. **Published in *International Journal of Nanoelectronics & Materials*, *11*(4). Scopus Journal**

Alemour, B., Badran, O., & Hassan, M. R. (2018). Improving The Electrical Conductivity Of Carbon Fiber Reinforced Epoxy Composite For Aviation Applications. **Published in The Ninth Jordanian International Mechanical Engineering Conference (JIMEC 2018).**

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Alemour, B., Lim, H. N., Yaacob, M. H., Badran, O., & Hassan, M. R. (2020). Electrical Conductivity and Self-Heating improvement of Carbon Fiber Reinforced Epoxy Composite Using Functionalized Graphene Nanoplates. ***Submitted to Reviews On Advanced Materials Science.*** **The journal is Thomson ISI journal with impact factor of 2.172.**

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