



**UNIVERSITI PUTRA MALAYSIA**

**DEVELOPMENT OF NOVEL ALUMINUM MATRIX COMPOSITE  
REINFORCED WITH HYBRID FILLER ( $\text{Fe}_3\text{O}_4\text{-SiC}$ ) FOR ENHANCEMENT  
OF MAGNETIC, THERMAL, CORROSION AND MECHANICAL  
PROPERTIES**

**NEGIN ASHRAFI**

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By

**NEGIN ASHRAFI**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,  
in Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

**April 2021**

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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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**April 2021**

**Chairman : Associate Professor Azmah Hanim binti Mohamed Ariff, PhD**  
**Faculty : Engineering**

In the present study, the hybrid reinforced magnetite; silicon carbide (Fe<sub>3</sub>O<sub>4</sub>-SiC) novel composite has been successfully fabricated by powder metallurgy method in the aluminum matrix. There is a demand in developing permanent magnetic composite with lightweight, corrosion resistance to develop the magnetic properties of aluminum matrix composites and obtain synchronization between electrical and thermal properties without mechanical degradation. Various researchers confirmed that Aluminum matrix composite (AMC) is an excellent multifunctional lightweight material with remarkable properties. However, to improve the wear resistance in the high-performance tribological application, hardness, and developing the corrosion resistance, magnetic, and thermal conductivity, optimized hybrid reinforcement of particulates magnetite, silicon carbide (SiC-Fe<sub>3</sub>O<sub>4</sub>) into an aluminum matrix needs to be examined. This study investigated the effect of adding 10, 15, 20, 30, 35 wt.% Fe<sub>3</sub>O<sub>4</sub> and constant amount of 5 wt.% Mg saturate as a binder, and preparation and fabrication of hybrid metal matrix composite reinforced with constant amount of Fe<sub>3</sub>O<sub>4</sub> (15, 30) wt. % and various silicon carbide particulates (10, 15, 20, 30) wt.% and 5 wt.% Mg as a binder. Commercially available pure aluminum powder, SiC, and Fe<sub>3</sub>O<sub>4</sub> were used to fabricate through the powder metallurgy method. Mixed powders were mechanically milled for 2 h using a planetary ball mill, and compacted of blend powder at a load of 250 MPa, then sintered at a temperature of 600 °C. Field-emission scanning electron microscopy (FE-SEM), energy-dispersive x-ray (EDX), optical microscopy (OM), X-ray diffraction (XRD), VSM (Vibrating sample magnetometer) for magnetic properties, micro flash thermal conductivity, a four-probe system for measuring the electrical properties, mechanical properties (hardness, wear), potentiodynamic polarization measurements to investigate the corrosion behavior was used to evaluate hybrid reinforcements effects on aluminum.

Adding SiC from 10 to 30 wt. % into Al-15 Fe<sub>3</sub>O<sub>4</sub> slightly improved the saturation magnetization (Ms) from approximately 2 to 6 (emu/g). The addition of 30% wt. % ferrimagnetic Fe<sub>3</sub>O<sub>4</sub> and 10 to 20% SiC nanoparticles into aluminum resulted in Ms between 5 to 11.058 (emu/g). Moreover, increasing the SiC 30 wt. % has improved the thermal conductivity of aluminum by 37%, while the electrical resistivity of the Al-Fe<sub>3</sub>O<sub>4</sub>-SiC composites increased by adding Fe<sub>3</sub>O<sub>4</sub> and SiC. Hence, the addition of these reinforcements (Fe<sub>3</sub>O<sub>4</sub>-SiC) to the composite shows a positive outcome towards mechanical properties, corrosion resistance (low corrosion rate) to increase the durability and life span of material during operation. By comparing the magnetization, thermal conductivity, mechanical properties of all samples, the combination of Al-30Fe<sub>3</sub>O<sub>4</sub>-20SiC, can be selected as an optimization composite especially for magnetic applications, indicating 6.55 (emu/g) for saturation magnetization, and 10<sup>-5</sup> (Ωm) for electrical resistivity. The thermal conductivity, hardness, and corrosion protection efficiency values have improved by 20%, 109%, 99.83% respectively. Moreover, the coefficient of friction (COF) is decreased by adding Fe<sub>3</sub>O<sub>4</sub> and SiC hybrid composite in tribology behaviors, and the lowest COF is (0.412) for Al-30Fe<sub>3</sub>O<sub>4</sub>-20SiC which is developed by 31%.

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

**PEMBANGUNAN KOMPOSIT Matriks ALUMINIUM NOVEL DIPERKUAT  
DENGAN PENGISI Hibrid ( $Fe_3O_4$ -SiC) UNTUK PENINGKATAN Sifat  
Magnetik, Termal, Hakisan dan Mekanikal**

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Dalam kajian ini, komposit baharu diperkuat hibrid magnetit; silikon karbida ( $Fe_3O_4$ -SiC) telah berjaya dihasilkan menggunakan kaedah metalurgi serbuk terhadap matriks aluminium. Terdapat permintaan komposit magnet kekal yang bersifat ringan, tahan hakisan, dihasilkan menggunakan teknik metalurgi serbuk konvensional berkos rendah, untuk mengembangkan kebolehtelapan magnetik komposit matriks aluminium dan mendapatkan keseimbangan antara sifat elektrik dan terma tanpa degradasi mekanikal dalam persekitaran yang menghakis untuk menghasilkan bahan magnet yang boleh digunakan dalam aplikasi yang berpotensi. Beberapa penyelidik telah mengesahkan bahawa aluminium matrix komposit (AMC) adalah bahan ringan multifungsi terbaik dengan sifat luar biasa. Namun, untuk meningkatkan ketahanan haus dalam aplikasi tribologi berprestasi tinggi, kekerasan, dan pembentukan ketahanan hakisan, kekonduksian magnetik, dan termal, tetulang hibrid perlu dioptimumkan (SiC-  $Fe_3O_4$ ) ke dalam matriks aluminium. Kajian ini menyiasat kesan penambahan (10, 15, 20, 30, 35) wt.%  $Fe_3O_4$  dan jumlah malar tepu 5 wt.% Mg sebagai pengikat, dan penyediaan dan pembuatan komposit matriks logam hibrid diperkuat dengan jumlah tetap  $Fe_3O_4$  (15, 30) berat. % dan pelbagai partikulat silikon karbida (10, 15, 20, 30) % berat dan 5 wt.% Mg sebagai pengikat. Serbuk aluminium tulen yang tersedia secara komersial, SiC, dan  $Fe_3O_4$  digunakan untuk pembuatan melalui kaedah metalurgi serbuk. Serbuk campuran digiling secara mekanis selama 2 jam menggunakan mesin bola planet, dan serbuk campuran dipadatkan pada tekanan 250 MPa, kemudian dipanaskan pada suhu 600 ° C. Mikroskop elektron imbasan pancaran medan (FE-SEM), serakan tenaga sinar-x (EDX), mikroskopi optik (OM), difraksi sinar-X (XRD), magnetometer getaran sampel (VSM) untuk sifat magnet, kekonduksian terma mikro kilat, sistem empat-kuar untuk mengukur sifat elektrik, sifat mekanik (kekerasan, kehausan), pengukuran polarisasi potensiodinamik untuk menyiasat tingkah laku kakisan digunakan untuk menilai kesan tetulang hibrid pada aluminium.

Menambah SiC dari 10 hingga 30 wt. % ke dalam Al-15 Fe<sub>3</sub>O<sub>4</sub> meningkatkan magnetisasi tepu (Ms) dari kira-kira 2 hingga 6 (emu/g). Penambahan 30% wt. % ferrimagnetik Fe<sub>3</sub>O<sub>4</sub> dan 10 hingga 20% partikel nano SiC menjadikan aluminium menghasilkan Ms antara 5 hingga 11.058 (emu/g). Lebih-lebih lagi, peningkatan SiC ke 30 wt. % telah meningkatkan kekonduksian terma aluminium sebanyak 37%, sementara daya tahan elektrik komposit Al-Fe<sub>3</sub>O<sub>4</sub>-SiC meningkat dengan penambahan Fe<sub>3</sub>O<sub>4</sub> dan SiC. Oleh itu, penambahan tetulang ini (Fe<sub>3</sub>O<sub>4</sub>-SiC) pada komposit menunjukkan hasil positif terhadap sifat mekanik, ketahanan kakisan (kadar kakisan rendah) untuk meningkatkan daya tahan dan jangka hayat bahan semasa operasi. Dengan membandingkan kemagnetan, kekonduksian terma, sifat mekanik semua sampel, gabungan Al-30Fe<sub>3</sub>O<sub>4</sub>-20SiC, dapat dipilih sebagai komposit pengoptimuman terutama untuk aplikasi magnet, menunjukkan 6.55 (emu/g) untuk magnetisasi tepu, dan 10<sup>-5</sup> (Ωm) untuk ketahanan elektrik. Nilai kekonduksian terma, kekerasan, dan kecekapan perlindungan kakisan telah meningkat masing-masing sebanyak 20%, 109%, 99.83%. Lebih-lebih lagi, pekali geseran (COF) diturunkan dengan penambahan komposit hibrid Fe<sub>3</sub>O<sub>4</sub> dan SiC dalam tingkah laku tribologi, dan COF terendah adalah (0.412) untuk Al-30Fe<sub>3</sub>O<sub>4</sub>-20SiC yang diorak sebanyak 31%.

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*Negin Ashrafi*



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## LIST OF ABBREVIATIONS

AMC	Aluminum Matrix composite
HAMC	Hybrid Aluminum Matrix Composite
MMCs	Metal Matrix Composite
PAMCs	Particle-reinforced AMCs
Al <sub>4</sub> C <sub>3</sub>	Aluminum carbide
BBC	Body-centered cubic
CTE	Coefficient of thermal expansion
EDS	Energy dispersive spectroscopy
Fe <sub>3</sub> O <sub>4</sub>	Magnetite
ROM	Rule of mixture
SEM	Scanning electron microscopy
SiC	Silicon Carbide
Wt %	Weight percentage
$\alpha$	Coefficient of thermal expansion
M <sub>s</sub>	Saturation magnetization
VSM	Vibrating sample magnetometer
H <sub>c</sub>	Coercivity
COF	Coefficient of friction
$\rho$ (X m)	Electrical resistivity
$\partial$ (S m <sup>-1</sup> )	Electrical conductivity
OM	Optical microscopy

# CHAPTER 1

## INTRODUCTION

### 1.1 Research background

Recently in various applications, composite materials are considered as more interesting materials compare to conventional materials since they create attractive properties in thermal, electromagnetic, electrical, and mechanical strength (Jaswinder et al, 2016; Xuan-hui et al., 2011). Among them, a broad variety of automotive, aerospace, defense, and electrical applications can be considered from aluminum matrix composites (AMC) due to attractive properties such as excellent corrosion resistance, high strength to weight ratio, and lower coefficient of thermal expansion (Francis et al., 2019; Sulaiman, et al., 2017).

Moreover, the new generation of composites focuses on two or three materials reinforcing aluminum or any other matrix. Previous research on reinforcement of SiC particles with carbon nanotube (CNT) or (Al<sub>2</sub>O<sub>3</sub>) reinforced hybrid aluminum matrix composites (HAMC) were focused more on the investigation of hardness, strength, wear, and thermal properties. All of these properties depend highly on the reinforcement weight percentage, chemical reaction with matrix, the grain size of reinforcement, and the production method. HAMC can be produced by squeeze casting, infiltration, semi-solid, and stir casting, where most of them involved high-temperature processes.

Only few investigations have been done on AMCs focusing on magnetic properties. Bayraktar (2016) developed an aluminum matrix composite with nano iron oxide (Fe<sub>3</sub>O<sub>4</sub>) to improve the magnetic properties of aluminum by powder metallurgy method. It is identified in previous studies that nanoparticles Fe<sub>3</sub>O<sub>4</sub> resulted in an enhancement in soft magnetic properties (Mahmoud, 2016). However, there is no systematic investigation on different compositions of Al-Fe<sub>3</sub>O<sub>4</sub>-SiC.

AMCs have a distinctive usage in multifunctional electronic packaging, optoelectronic devices, telecommunications industry, medical equipment, access control systems, and so forth (Ozben et al., 2008). Moreover, in today's world, permanent magnetic materials improvement has been focused by the requirement to supply more magnetic energy in eternally smaller volumes for combination in the variety of applications that comprise hybrid motors, transportation components, and clean energy technologies such as wind turbine generators (Goldman, 1999). It is believed that the use of modern permanent magnets is beneficial as they can withstand high temperatures due to their thermal stability. It is assumed that permanent magnet variable flux sources are more advanced than electromagnets for their small size and not requiring big power supplies or cooling criteria (Elizabeth et al., 2008; Jiles, 2003). Furthermore, there is a demand

to design advanced magnetic materials in the electrical machines of aircraft and automobiles to endure high speeds or high torque, heavy loads, and high temperatures (Laura et al., 2012). It is necessary to increase the demand for more efficient electromagnetic devices and electronic motors (Katie, 2017). These materials should be able to withstand temperatures up to 600 °C (Elizabeth, 2008). However, many researchers affirmed that (AMC) is an interesting composite for the extensive diversity of applications, excellent multifunctional lightweight material with applications in electrical, aeronautical, and automotive. However, only a few investigations have been done on Aluminum composite characteristics focusing on magnetic properties. It is identified in previous studies that nanoparticles  $\text{Fe}_3\text{O}_4$  resulted in an enhancement in soft magnetic properties (Fathy, 2015).

In addition, thermal management has become one of the most important aspects in the field of electronic devices, the energy-related areas such as the electronics and aeronautics industry have limitations in their application because of designing materials capable of removing heat and, at the same time, maintaining their dimensional stability in high temperatures in corrosive environments, while several studies addressed the thermal properties of AMCs (Xuan-hui et al., 2011; Clyne, 2003). However, there is no research focusing on the thermal properties of Al- $\text{Fe}_3\text{O}_4$ -SiC and Al- $\text{Fe}_3\text{O}_4$  by the powder metallurgy method. Furthermore, the coefficient of thermal expansion “ $\alpha$ ” of Aluminum is large; the significant changes in dimensions with temperature can lead to problems with metallic components with close tolerances. In contrast, the thermal expansion of ceramics such as SiC and metal oxide is much lower. The intrinsic thermal conductivity of SiC particles is high (Devaraju et al., 2013). In this regard, one of the effective ways to improve the thermal conductivity of aluminum is to add reinforcement particles with higher thermal conductivity.

Hence, in this present project the optimum amount of  $\text{Fe}_3\text{O}_4$  nanoparticles addition into Al-SiC hybrid composite (HAMC), and  $\text{Fe}_3\text{O}_4$  addition into the aluminum to fulfill the magnetic and electrical requirements were studied, the focus is to study if there are any mechanical degradation with regard to a high volume of reinforcement addition into the Aluminum matrix, while it's magnetic, electrical, thermal properties, and other physical characteristics were discussed with relation to finding optimum amount of nanoparticles filler with processing operational steps.

## **1.2 Problem statement**

Aluminum is an interesting element for the extensive diversity of applications because of its low weight, durability, high impact strength, and ductility, but it consistently required improvement in terms of mechanical properties such as resistance, strength, hardness, and corrosion. Aluminum is classified as paramagnetic a material alloy which means they have poor magnetic properties compared to ferrous materials such as alloy steel, titanium, cast iron, and carbon steel. Aluminum has the potential to be developed in fulfilling certain engineering properties required for the magnetic application environment.

Also, aluminum composites in electronic applications require thermal management. Based on that, the optimum amount of  $\text{Fe}_3\text{O}_4$ -SiC nanofiller adding into composite will be examined. The constant development of magnetic composites is fundamental for potential electromagnetic devices application (Kong, et al., 2013; Bayraktar et al., 2010). It was identified in literature studies that nanoparticles  $\text{Fe}_3\text{O}_4$  resulted in enhancement of soft magnetic properties. In addition, another goal is to improve mechanical properties including hardness and wear of composite. Silicon carbide was considered as the second filler to add into the metal matrix composite. The target is not only to develop its mechanical properties but also to develop the thermal conductivity, and corrosion of the composites. The intrinsic thermal conductivity of SiC particles is very high; hence it is one of the effective ways to improve the thermal conductivity of aluminum by adding reinforcement particles with higher thermal conductivity. Additionally, SiC is considered to be one of the most important microwave absorbing materials. In addition, it is expected that adding hybrid reinforcements improve the magnetic, thermal, hardness, wear, and corrosion of the composite.

### **1.3 Objective of the study**

This study is carried out with objectives as follows:

- I. To develop aluminum matrix composite reinforced with hybrid filler  $\text{Fe}_3\text{O}_4$ -SiC by using powder metallurgy technique, analyzing density, and microstructure.
- II. To analyze the magnetic, thermal and electrical properties of Al- $\text{Fe}_3\text{O}_4$ -SiC hybrid aluminum matrix composite.
- III. To evaluate the mechanical properties & corrosion behavior of Al- $\text{Fe}_3\text{O}_4$ -SiC hybrid aluminum matrix composite in order to assess the suitability of the developed composites for electromagnetic application.

### **1.4 Scope of the study**

This research was carried out within the scope of finding a suitable aluminum matrix composite reinforced by magnetite-silicon carbide that could be used in the aeronautic and electrical industries by powder metallurgy method. This novel hybrid composite can be used in electromagnetic shielding or absorption, microelectronic applications by providing better properties at higher strength and low weight, higher wear resistance, better corrosion resistance with magnetic and electrical properties. The excellent combination of good mechanical properties and low weight is the prime advantage for such a new class of lightweight materials in the electrical field, aerospace, and naval industries.

In order to accomplish the objectives of this experiment, the scope of the study, the scope of the study can be explained briefly as below:

- Fabrication of two types of composites Aluminum-magnetite, and Aluminum-magnetite-silicon carbide. The weight percentage of magnetite nano particles such as 10%, 15%, 20%, 30%, 35% ,and the second series were fabricating two series of composites by adding different ratio of 10%, 20 %, 30% silicon carbide to Al-15Fe<sub>3</sub>O<sub>4</sub> composite, and Al-30Fe<sub>3</sub>O<sub>4</sub>.
- The microstructure characterization and phase identification of the composite have been done by scanning electron microscope Field-emission scanning electron microscopy (FE-SEM), Energy-dispersive X-ray (EDX), optical microscopy (OM), and X-ray diffraction (XRD) to identify the microstructure modification after adding reinforcements. Density measurement has been done by Archimedes method.
- To investigate the magnetic properties by VSM (Vibrating sample magnetometer), micro flash for thermal conductivity measurement system, four-probe system for measuring the electrical conductivity and resistivity,
- Mechanical properties have been measured by vickers microhardness, and wear by utilizing a pin-on-disc configuration, in dry-sliding condition.
- Potentiodynamic polarization measurements method to investigate the corrosion properties of the composite in sea water environment.

There are few limitations in the fabrication of these composites with magnetic properties as below:

- Controlling all the fabrication parameters within ball milling, and controlling reactions between matrix and reinforcements is relatively difficult.
- Each test required a specific sample size which demands different mold or cutting samples into standard dimensions. Cutting by a CNC machine or precious saw might affect sample properties and surface of them.
- Agglomeration can influence negatively materials properties. Proper mixing is very essential in powder metallurgy to get a homogeneous batch of powders to avoid agglomeration. Although there is an exothermic reaction between aluminum and magnetite, so the time and speed of ball milling should be chosen carefully.



## 1.5 Importance of the study

The importance of the study can be classified as below:

- Developing a permanent magnetic composite with lightweight, corrosion resistance, and better mechanical properties by adding  $\text{Fe}_3\text{O}_4$  nanoparticles into aluminum.
- To fabricate novel hybrid filler reinforcements composite ( $\text{SiC-Fe}_3\text{O}_4$ ) by powder metallurgy method, to develop the magnetic properties of aluminum matrix composites and obtain synchronization between electrical and thermal properties without mechanical degradation in a corrosive environment.
- Focusing on thermal properties of the hybrid composite which is an essential factor in field of electronic and aeronautic industry.
- To produce magnetic materials by low-cost conventional powder metallurgy that can be used in electromagnetic and microelectronic packaging applications.

## 1.6 Outline of thesis

The thesis arrangement is designed as follows.

**Chapter 1** describes an introduction of composites and reinforced by hybrid fillers, the problem statement, the objective, and the scopes of the study, limitations, and the importance of this project. The explanation of the magnetic materials background, powder metallurgy process, chemical reactions between matrix and reinforcement, magnetic, thermal, electrical, and corrosion, mechanical, properties of the AMCs, HAMCs composites, reinforcements selection, magnetic composite application in various industries are explained in **Chapter 2**. The methodology of fabrication of the composites by conventional powder metallurgy method and the procedure of each experimental, and characterization have been described in **Chapter 3**. The results and discussion concerning the influence of added various weight percentages (Silicon carbide, Magnetite) to the aluminum matrix, on the magnetic, thermal, electrical, corrosion, density, and mechanical properties of AMCs, HAMCs composites were analyzed and mentioned in **Chapter 4**. Hence, the conclusion and suggestions for future works were mentioned in **Chapter 5**.

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## LIST OF PUBLICATIONS

Ashrafi, N., Azmah Hanim, M. A., Sarraf, M., Sulaiman, S., & Hong, T. S. 2020. Microstructural, Tribology and Corrosion Properties of Optimized Fe<sub>3</sub>O<sub>4</sub>-SiC Reinforced Aluminum Matrix Hybrid Nano Filler Composite Fabricated through Powder Metallurgy Method. *Materials*, 13(18), 4090

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### Book chapter

Negin Ashrafi, M.A. Azmah Hanim, S. Sulaiman, Tang Sai Hong and Masoud Sarraf. 2019. Mechanical Properties of Silicon Carbide Reinforced Aluminum Matrix Composites by Powder Metallurgy. Chapter in Case Study on Material Engineering and Applied Sciences: UPM and KU 2019. Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia. Malaysia. 187-192. (eISBN: 978-983-2408-68-0)



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