



Faculty Of Engineering

**Investigation of Hardness and Corrosion Properties on Graphene
Nanoplatelets Reinforced Magnesium Composite**

Abang Mohamad Harith Bin Abang Beniamin

**Master of Engineering
2023**

Investigation of Hardness and Corrosion Properties on Graphene
Nanoplatelets Reinforced Magnesium Composite

Abang Mohamad Harith Bin Abang Beniamin

A thesis submitted

In fulfillment of the requirements for the degree of Master of Engineering

(Mechanical Engineering)

Faculty of Engineering
UNIVERSITI MALAYSIA SARAWAK
2023

DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



.....

Signature

Name: Abang Mohamad Harith Bin Abang Beniamin

Matric No.: 20020099

Faculty of Engineering

Universiti Malaysia Sarawak

Date: 5 August 2023

ACKNOWLEDGEMENT

Firstly, my sincere appreciation is given to my supervisor, Dr Noor Hisyam Bin Noor Mohamed, for giving me a golden opportunity to do this research for my master's degree. His contribution in providing me with guidance and valuable suggestions helped me to coordinate my research project and produce this thesis. Besides that, I am grateful to my co-supervisor, Dr. Mahsuri Binti Yusof, for her dedication to her ideas and suggestions to improve my research study process.

Furthermore, I would like to thank all the lecturers of the Faculty of Engineering UNIMAS, the technical staff of the Faculty of Engineering UNIMAS, and all my colleagues from the postgraduate research program for their knowledge-sharing, guidance, and advice throughout the research project executed. They are always there when help is needed; therefore, their support is appreciated in completing the research and thesis writing. I want to thank UNIMAS for providing all the required resources and facilities throughout my study.

I appreciate and regard all the moral support and enthusiastic encouragement from my parents, wife, brother, relatives, and friends. Lastly, many thanks to those who have helped me directly or indirectly to complete my research.

ABSTRACT

Magnesium (Mg) has been used for many decades in various applications. This metal has attractive mechanical properties such as strength and hardness. Although Mg is lightweight and has high strength, one of the major limiting factors in using Mg includes its corrosion resistance. In the natural environment, the poor corrosion resistance of Mg and Mg alloys in saline and physiological environments remains a constant threat for Mg and Mg alloys as potential automotive parts, aerospace materials or biomedical implants. This research is done to study the hardness and corrosion properties of Mg and Mg-based composites. The hardness was determined using the Vickers microhardness test. Based on the results, Mg0.2%Mn%6%Zn-1% GNPs had the highest hardness at 48 HV, followed by Mg0.2%Mn%6%Zn-0.5% GNPs at 45 HV, Mg0.2%Mn%6%Zn at 44 HV, and HP Mg at 34 HV. Increasing GNP content with zinc and manganese reinforcement is important for improving hardness in sintered Mg-based GNPs composites. An immersion test on GNP-reinforced Mg composites showed that Mg0.2%Mn%6%Zn-1% GNPs had the highest corrosion resistance (PW=17.546mm/y), followed by Mg0.2%Mn%6%Zn-0.5% GNPs (PW=19.770mm/y), Mg0.2%Mn%6%Zn (PW=22.958mm/y), and HP Mg (PW=90.3146mm/y). GNPs also lowered the corrosion rate in saline solution. Corrosion morphology and type were determined by 3D optical microscope, SEM, and EDX. Overall, this study has managed to fabricate Mg-based composites using the powder metallurgy method. Adding GNP as reinforcement has increased hardness and corrosion properties significantly, thus proving the strengthening effect of corrosion resistance by GNPs in Mg-based composites.

Keywords: Magnesium-based composites; graphene nanoplatelets; powder metallurgy; microhardness test; corrosion immersion test

***Penyiasatan Tentang Sifat Kekerasan Dan Pengaratan Bagi Komposit Magnesium
Dengan Nanoplatelet Graphene***

ABSTRAK

Magnesium (Mg) telah digunakan selama beberapa dekad dalam pelbagai aplikasi. Logam ini mempunyai sifat mekanikal yang menarik seperti kekuatan dan kekerasan. Walaupun Mg ringan dan mempunyai kekuatan tinggi, salah satu faktor penghad utama dalam menggunakan Mg termasuk rintangan pengaratannya. Dalam persekitaran semula jadi, rintangan pengaratan yang lemah bagi aloi Mg dan Mg dalam persekitaran masin dan fisiologi kekal sebagai ancaman berterusan bagi aloi Mg dan Mg sebagai bahagian automotif yang berpotensi, bahan aeroangkasa atau implan bioperubatan. Penyelidikan ini dilakukan untuk mengkaji kekerasan dan sifat pengaratan komposit berasaskan Mg dan Mg. Kekerasan ditentukan menggunakan ujian microhardness Vickers. Berdasarkan kajian, Mg0.2%Mn%6%Zn-1% GNP mempunyai kekerasan tertinggi pada 48 HV, diikuti oleh Mg0.2%Mn%6%Zn-0.5% GNP pada 45 HV, Mg0.2%Mn %6%Zn pada 44 HV dan HP Mg pada 34 HV. Peningkatan % GNP dengan tetulang zink dan mangan adalah penting untuk meningkatkan kekerasan dalam komposit GNP berasaskan Mg tersinter. Ujian rendaman ke atas komposit Mg bertetulang GNP menunjukkan bahawa Mg0.2%Mn%6%Zn-1% GNP mempunyai rintangan kakisan tertinggi (PW=17.546mm/y), diikuti oleh Mg0.2%Mn%6%Zn-0.5% GNP (PW=19.770mm/y), Mg0.2%Mn%6%Zn (PW=22.958mm/y) dan HP Mg (PW=90.3146mm/y). GNP juga menurunkan kadar kakisan dalam larutan garam. Morfologi dan jenis pengaratan ditentukan oleh mikroskop optik 3D, SEM, dan EDX. Secara keseluruhannya, kajian ini telah berjaya menghasilkan komposit berasaskan Mg menggunakan kaedah metalurgi serbuk. GNP sebagai tetulang telah

meningkatkan kekerasan dan sifat pengaratan dengan ketara, sekali gus membuktikan kesan pengukuhan rintangan pengaratan oleh GNP dalam komposit berasaskan Mg.

Kata kunci: *Komposit berasaskan magnesium; graphene nanoplatelet; metalurgi serbuk; ujian mikro kekerasan; ujian pengaratan rendaman*

TABLE OF CONTENTS

	Page
DECLARATION	i
ACKNOWLEDGEMENT	ii
ABSTRACT	iii
ABSTRAK	iv
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF ABBREVIATIONS	xii
CHAPTER 1 INTRODUCTION	1
1.1 Study Background	1
1.2 Problem Statement	4
1.3 Objectives	4
1.4 Scope of Project	5
1.5 Chapter Summary	5
CHAPTER 2 LITERATURE REVIEW	7
2.1 Magnesium and its Alloys	7
2.1.1 Magnesium and its Alloys in Industry	9
2.1.2 Alloying of Magnesium	16

2.2	Heat treatment of Magnesium-based Alloy	21
2.3	Metal Matrix Composites for Magnesium	22
2.3.1	Titanium-based Particles Reinforced Mg Composite	24
2.3.2	Carbon Nanotubes Reinforced Mg Composite	25
2.3.3	Graphene Nanoplatelets Reinforced Mg Composite	27
2.4	Corrosion of Magnesium	30
2.4.1	Mechanism of Mg Corrosion	31
2.4.2	Types of Mg Corrosion	33
2.4.3	Factors Affecting Mg Corrosion Rate	37
2.5	Powder Metallurgy Technique for Mg-Based Composites	40
2.6	Chapter Summary	42
CHAPTER 3 METHODOLOGY		43
3.1	Introduction	43
3.2	Materials	44
3.3	Equipment	44
3.4	Fabrication of Composites	46
3.5	Microstructural Characterisation	48
3.6	Hardness Test	49
3.7	Corrosion Immersion Test	51
3.8	Corroded Surface Evaluation	54

3.9	Chapter Summary	54
CHAPTER 4	RESULTS AND DISCUSSIONS	56
4.1	Introduction	56
4.2	Microstructure Characterisation	56
4.3	Vickers Hardness Test	65
4.4	Corrosion Immersion Test and Corrosion Rate Measurements	68
4.5	Surface Evaluation After Immersion Test	70
4.6	Chapter Summary	79
CHAPTER 5	CONCLUSION AND RECOMMENDATIONS	81
5.1	Conclusion	81
5.2	Recommendations	82
	REFERENCES	84
	APPENDICES	105

LIST OF TABLES

	Page
Table 2.1: Comparison between some properties of Mg, Ti, Al, and Fe (Kulekci, 2008)	9
Table 2.2: Magnesium alloys applications in car components (Gaines, Cuenca, et al. 1996; Kulekci, 2008)	11
Table 2.3: Advantages of Mg-based alloys for biomedical implants	15
Table 2.4: Alloying elements and the effects on Mg-based alloy	17
Table 2.5: Mg cast alloys and their specific properties (Bruce Abbott et al., 2001)	20
Table 2.6: Some properties of graphene nanoplatelets (Ghosh et al., 2008; Stankovich et al., 2006; Yung et al., 2013)	28
Table 3.1: Type of sample for Mg-based composite fabrication	43
Table 3.2: Several equipments with models and their functions	45
Table 4.1: Characterisation of elements on samples using EDX	62
Table 4.2: Hardness results on samples	65
Table 4.3: Corrosion rate obtained using hydrogen evolution and weight loss after 5 days	69
Table 4.4: Elements characterisation of samples after immersion test before corrosion product removal by using EDX	75

LIST OF FIGURES

	Page
Figure 2.1: Magnesium metals in various forms available commercially. (Courtesy from Press Metal Berhad)	8
Figure 2.2: Some of the car parts that use Mg alloys (Kulekci, 2008)	10
Figure 2.3: Application of magnesium for aircraft parts (Gwyne & Lyon, 2007)	13
Figure 2.4: Some of the Magnesium-based implants for the human body (Y. Sun et al., 2019)	15
Figure 2.5: Graphene model structure (Retrieved December 04, 2017, from https://www.themanufacturer.com/articles/talga-resources-first-delivery-of-a-grapheneproduct/)	28
Figure 3.1: Steps on powder metallurgy process of Mg-based composites	47
Figure 3.2: Fabricated HP Mg sample before polish and HP Mg samples after polish	49
Figure 3.3: Vickers hardness test diagram	51
Figure 3.4: Setup for the corrosion immersion test	53
Figure 4.1: Microstructure of 1) HP Mg, 350× 2) HP Mg, 1000× 3) Mg6%Zn0.2%Mn, 350× 4) Mg6%Zn0.2%Mn, 1000× 5) Mg0.2%Mn6.0%Zn-0.5% GNPs, 350× 6) Mg0.2%Mn6.0%Zn-0.5% GNPs, 1000× 7) Mg0.2%Mn6.0%Zn-1% GNPs, 350× 8) Mg0.2%Mn6.0%Zn-1% GNPs, 1000× observed under a 3D optical microscope.	58
Figure 4.2: Microstructure of 1) HP Mg, 1000× 2) HP Mg, 2000× 3) Mg-6%Zn-0.2%Mn, 1000× 4) Mg-6%Zn-0.2%Mn, 2000× 5) Mg0.2%Mn6.0%Zn-0.5% GNPs, 2000× 6) Mg0.2%Mn6.0%Zn-0.5% GNPs, 2000× 7) Mg0.2%Mn6.0%Zn-1% GNPs, 1000× 8) Mg0.2%Mn6.0%Zn-1% GNPs, 2000× observed under SEM	61
Figure 4.3: Bar chart of average Vickers hardness of the samples	66
Figure 4.4: Graphs of hydrogen evolution volume against time for all samples	68
Figure 4.5: The surface appearance of the corroded surface before and after product removal using an optical microscope with 50× magnification	71

Figure 4.6: The surface appearance of the corroded surface after immersion before corrosion product removal using SEM	74
Figure 4.7: Schematic model on the corrosion mechanism of Mg-based composites (Rashad et al., 2017)	78

LIST OF ABBREVIATIONS

AgNO ₃	Silver Nitrate
Al	Aluminium
BCC	Body Centred Cubic
CNT	Carbon Nanotubes
CO ₂	Carbon Dioxide Gas
Cr ₂ O ₃	Chromium (III) Oxide
Cu	Copper
EDX	Energy Dispersive X-ray Spectroscopy
FCC	Faced Centred Cubic
GNPs	Graphene Nanoplatelets
H	Hydrogen
H ₂	Hydrogen Gas
HCP	Hexagonal Closed Packed
HP Mg	High Purity Magnesium
Mg	Magnesium
Mg(OH) ₂	Magnesium Hydroxide
MgCl	Magnesium Chloride

MMC	Metal Matrix Composites
Mn	Manganese
NaCl	Sodium Chloride
Ni	Nickel
O	Oxygen
O ₂	Oxygen Gas
RE	Rare Earths Metals
SEM	Scanning Electron Microscope
Ti	Titanium
VHN	Vickers Hardness Number
Zn	Zinc
Zr	Zirconium

CHAPTER 1

INTRODUCTION

1.1 Study Background

Magnesium (Mg) is widely used in industrial applications due to its lightweight properties. This lightweight property of Mg in industrial applications, mainly automotive, can increase the power-to-weight ratio, reducing fuel consumption and increasing fuel efficiency (Romanowski, 2019). Meanwhile, Mg is used for medical applications that are less dense for implants (Witte, 2010). Despite having some exclusive properties in terms of mechanical properties, Mg has a critical limitation on its corrosion resistance. Mg is one of the most reactive metals towards corrosion in most metals. The corrosion of Mg can be happened either by generalised corrosion, localised corrosion, galvanic corrosion, or environmental cracking (Atrens et al., 2011).

Mg-based composites are one of the interests of researchers for automotive applications other than Mg-based alloys. This development is made to enhance the mechanical properties and the resistance toward corrosion of Mg. One of the latest developments of Mg in engineering applications is fabricating magnesium reinforced with graphene nanoplatelets composite. Graphene nanoplatelets (GNPs) is a thin graphite sheets with a thickness of less than 100 nm, made from nanosized graphene particles. Graphene is a single freestanding monolayer of graphite that originates from carbon. It is the first 2D material ever manufactured by humankind, having a thickness of one atom (0.34 nm) and lateral size orders of magnitudes larger than graphite (Cataldi et al., 2018).

Recent research studies by Rashad, Tang, et al. (2015) and Rashad, Asif, et al. (2014) show that magnesium-reinforced graphene nanoplatelets composites have superior properties compared to magnesium alloys and other magnesium-based composites. Rashad et al., 2015 reported that the magnesium composite with the addition of graphene nanoplatelets helps maintain the lightweight property as the density of magnesium is not influenced. Therefore, Mg-reinforced GNPs composite gives identical mass value with Mg.

Another study by Arab and Marashi (2019) stated that GNPs could help increase the hardness by 14% by applying friction stir processing and by 41% by adding GNPs due to grain refinement, dynamic recrystallisation and pinning effect of GNPs. Besides that, other mechanical properties such as universal tensile strength, elongation, Vickers hardness and porosity have improved on Mg-reinforced GNPs composites using the thixomoulding process found by Chen et al. (2019). They also reported that the GNPs' mechanical properties significantly improved on AZ91D-GNPs composites compared with AZ91D Mg alloy. The thixomoulding process is well known for common industry manufacturing, promising the production of Mg-based composites. This is due to the shortened process time to show that this method can produce large-scale production in the future.

Under a corrosive environment, Mg reacts in an aqueous solution to form magnesium hydroxide ($\text{Mg}(\text{OH})_2$) as a corrosion product at the end of the reaction. It protects Mg from further corrosion by forming a thin layer that is formed on the surface. After all, these $\text{Mg}(\text{OH})_2$ films are only limited to alkaline solutions as a protective layer. They can break through immediately in non-alkaline, which are neutral, acidic, or saline solutions. These solutions can cause catastrophic corrosion to occur. In a saline environment, especially in

sodium chloride solution, the $\text{Mg}(\text{OH})_2$ layer is removed as the chloride ions convert $\text{Mg}(\text{OH})_2$ to form magnesium chloride (MgCl) soluble salt (Pourbaix, 1974).

Many researchers investigated the corrosion behaviour of Mg and its alloys, including the heat treatment of Mg alloys. The most prevalent type of corrosion that happens in Mg is galvanic corrosion. Galvanic corrosion occurs when Mg is coupled with other more cathodic metals than magnesium. Alloying of Mg can also induce galvanic corrosion when alloyed with more anodic alloying elements such as aluminium and accelerates the corrosion rate faster when the iron is the impurity of magnesium alloys (Guangling Song, Hapugoda et al. 2004). Comprehensive and detailed research on the corrosion of Mg and its alloys demonstrates that high-purity magnesium has a lower corrosion rate than Mg alloys. Several methods, such as weight loss measurement, hydrogen evolution and electrochemical measurement, such as Tafel extrapolation, were used to determine the corrosion rate (Qiao, Hort, Zainal Abidin, et al., 2012; Shi, Liu, & Atrens, 2010).

The mechanical properties and corrosion resistance are much better in Mg-based composites than in non-reinforced Mg for future applications in automotive and biomedical (Rashad, Pan, Tang, et al., 2015). Some properties, such as strength and hardness, are better with 0.3% GNPs with the semi-powder metallurgy method. Further research was also done by Rashad, Pan, et al. (2017) to study the corrosion resistance of Mg-reinforced GNPs using the stir casting method. Although their results reveal that the presence of GNPs in different matrices decreases the corrosion resistance of composites, this finding might be improved by adjusting the parameters of the experimental methods, such as using the powder metallurgy technique and adding other metals in synthesising the composite.

1.2 Problem Statement

In the automotive, aerospace, and medical industry specifically, the challenge nowadays is to fabricate materials which have high hardness with better corrosion resistance. Magnesium and its alloys are the material that fulfil requirements which attract many engineers and technologists for applications in automotive and medical. Despite magnesium-based alloys having superior mechanical properties and hardness to pure magnesium, the corrosion resistance of magnesium alloys is worse than pure magnesium as the reactions with other alloying elements influence the corrosion properties. On the other hand, pure Mg gives better corrosion resistance compared with some Mg-based alloys with the limitation on the mechanical properties' superiority. Thus, several improvements have to be made for Mg-based alloys limitations by the researchers.

1.3 Objectives

The objectives of this research are:

1. To investigate the effect of different GNPs compositions towards the hardness properties of GNPs reinforced Mg composites.
2. To investigate the effect of different GNPs compositions towards the corrosion behaviour of GNPs reinforced Mg composites.
3. To study the microstructure and composition of fabricated GNPs reinforced Mg composites.

1.4 Scope of Project

In this research project, the composition of alloying elements in the Mg sample is the primary concern. This investigation is to observe how the difference in the design of magnesium composites can affect the mechanical properties and corrosion behaviour. This research focused on Mg composites with 6% of zinc and 0.2% of manganese compositions of the metal matrix. Meanwhile, the percentage of the reinforcement used GNPs ranged from 0%, 0.5% and 1%. Besides that, the fabrication method used on GNPs reinforced Mg composites that utilise the powder metallurgy method. The materials characterisation of the area using the 3D optical microscope, scanning electron microscope and energy dispersive X-ray spectroscopy. Furthermore, the hardness testing uses the Vickers microhardness test, and the corrosion test is conducted by the immersion test. The corrosion rate was determined using hydrogen evolution and weight loss measurement.

1.5 Chapter Summary

This thesis outline explains the summary of each chapter from Chapter 1 to Chapter 5.

Firstly, Chapter 1 is an introduction which describes the background of the research topic, problem statement, objectives, and the scope of the research project.

Chapter 2 describes the literature review, which focuses more on the importance of the proposed research topic with previous relevant research that the researchers had done. This chapter first describes magnesium and its alloys. Further description is focused more on magnesium and its alloys in automotive applications and alloying of magnesium. The heat treatment is described further to know the limitation of the heat treatment toward

magnesium-based alloys. Meanwhile, magnesium-based composites mainly focus on the type of magnesium-based composites with the explanation of mechanical properties and corrosion resistance. Other than that, the corrosion behaviour of magnesium is discussed further. Lastly, this chapter also describes magnesium's powder metallurgy in fabrication.

Next, Chapter 3 is the methodology part of the research project. This chapter describes the research's materials, equipment, and step-by-step procedures. This chapter also highlights the mechanical and corrosion test of the composites conducted on the project.

Chapter 4 is the part where all the results and observations of the test conducted on the samples were obtained. The results and observations include the data in figures, tables, graphs and more. The discussion of the results obtained is also portrayed in this chapter.

Lastly, the last chapter is Chapter 5. This chapter concludes with the hardness and corrosion properties of graphene nanoplatelets-based reinforced magnesium composites. Besides that, the recommendation for the research project is also included in this chapter.

CHAPTER 2

LITERATURE REVIEW

2.1 Magnesium and its Alloys

Magnesium (Mg) is one of the most abundant metals present in the Earth's crust. It can be found in seawater at approximately 2.8% and other ore forms such as dolomite ($\text{CaMg}(\text{CO}_3)_2$), magnesite (MgCO_3) and carnallite ($\text{KMgCl}_3 \cdot 6\text{H}_2\text{O}$). In the periodic table, Mg is in group 2, period 3 element known as alkaline earth metals. Mg is the lightest of all the engineering metals, having a 1.74 g/cm³ density. It is 35% lighter than aluminium (2.7 g/cm³), 60% lighter than titanium and over four times lighter than steel (7.86 g/cm³) (Mordike & Ebert, 2001). Mg is also categorised as light metal, apart from aluminium and titanium. Mg has proven to become desirable engineering material in many applications, especially in the automotive, aerospace, and biomedical industries, with its low density, adequate specific strength, stiffness, and excellent damping capacity.

Mg is an excellent engineering metal widely used due to its significant properties and is readily available commercially. Mg is readily available for industries in various primary foundry forms such as bar, billet, foiled, rolled, and more, as shown in Figure 2.1. However, the applications of pure Mg in these industries still need to be improved due to poor corrosion resistance, relatively low strength, and low ductility, as mentioned by (Blawert, Hort, & Kainer, 2004).



Figure 2.1: Magnesium metals in various forms available commercially. (Courtesy from Press Metal Berhad)

In most applications, Mg is stronger per unit than aluminium (Al), titanium (Ti) or iron (Fe). Mechanical properties such as strength and stiffness are important for automotive applications. Some properties that are important for Mg compared with other metals for scientific and engineering research and applications are shown in Table 2.1.

Table 2.1: Comparison between properties of Mg, Ti, Al, and Fe (Kulekci, 2008)

Property	Magnesium	Titanium	Aluminium	Iron
Crystal Structure	HCP	HCP	FCC	BCC
Density at 20°C (g/cm ³)	1.74	4.54	2.70	7.86
Coefficient of thermal expansion 20–100°C (×10 ⁶ /C)	25.2	8.6	23.6	11.7
Young's modulus of elasticity (MPa)	44.126	113.8	68.947	206.842
Tensile strength (MPa)	240 (for AZ91D)	880 (for Ti-6Al-4V)	320 (for A380)	350
Melting point (°C)	650	1668	660	1536

2.1.1 Magnesium and its Alloys in Industry

Nowadays, many industries, such as the automotive industry, lead engineers and technologists from various car manufacturers. They realised the demand for cars with superior properties, strong, stiff, and high corrosion resistance. Mg has been widely used as a suitable metal for the automotive industry because of its lightweight property. Besides, Mg and its alloys are preferred and commonly used in the automotive industry due to their high specific strength and excellent cast ability (Rashad et al., 2017). The weight reduction in automobiles saves energy required for the car to accelerate and thus reduces greenhouse gas and carbon dioxide CO₂ emissions that can pollute the environment. Reducing the automotive weights will result in a significant percentage of improvement in fuel economy and efficiency (Atrens et al., 2015). For high-performance cars, such as racing cars, the body framework must be light to accelerate faster within an abbreviated period (Easton et al., 2008). Light weighting provides up to a 7% improvement in fuel economy for each 10%

reduction in vehicle weight when included with a suitable-sized powertrain (Joost & Krajewski, 2017). Figure 2.2 shows parts of the car that use Mg alloys as their primary materials. Specific car parts that use Mg are also shown in Table 2.2.

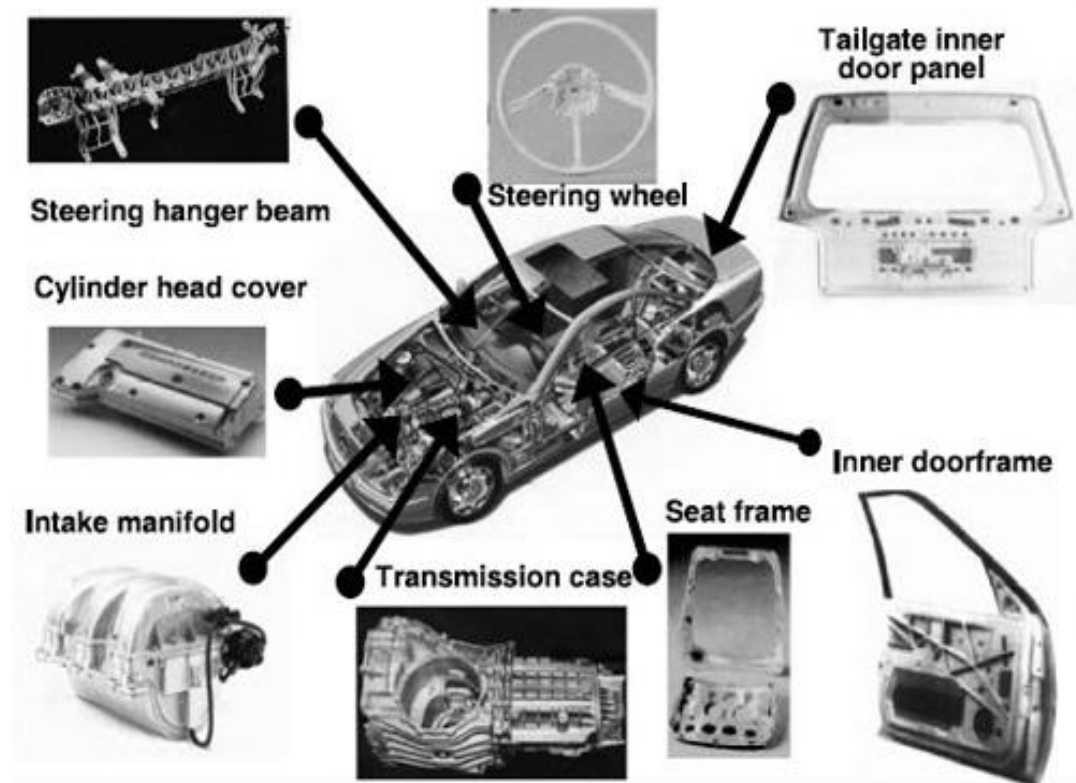


Figure 2.2: Some of the car parts that use Mg alloys (Kulekci, 2008)

Table 2.2: Magnesium alloys applications in car components (Gaines, Cuenca, et al. 1996; Kulekci, 2008)

Engine and parts transmission	Engine block, gearbox, crankcase, oil pump housing, cylinder head cover, transfer case, covers, cams, bed plate, engine cradle, clutch, and engine parts
Interior parts	Steering wheel covers, seat components, instrument panel, brake and clutch pedal, airbag retainer, door inner.
Chassis components	Wheels, suspension arms, engine cradle, rear support, tailgate, bumper, brake system, fuel tank system
Body components	Cast components, radiator support, sheet components, extruded parts, exterior and interior components, seats, instruments, and controls

The aviation sector is one of the fastest-growing sectors globally in terms of both passenger and cargo traffic. Modern aerospace transportation has been crucial to the global economy until now, but it also contributes significantly to gaseous pollutants. About 13% of overall greenhouse gases and 25% of CO₂ emissions come from fossil fuel combustion in transportation. The global aviation industry generates around 2 wt% of all human-induced emissions and 12 wt% of CO₂ emissions from all transport sources. The Air Transport Action Group has established some ambitious goals in order to minimise the negative impact. They aim to achieve carbon neutrality by 2020, increase fuel efficiency by 1.5 wt% annually, and reduce carbon emissions by 50% by 2050 in comparison to 2005 levels (Czerwinski, 2014).

To fulfil environmental obligations, innovative technologies for aircraft body and propulsion systems must be adopted. Additionally, the utilisation of advanced materials and cooling technologies can lead to substantial enhancements in fuel efficiency and reduced emissions per passenger kilometre. An alternative way to reduce jet fuel consumption is the application of lighter materials in aircraft construction (Wendt et al., 2016). Modern aircraft

require structural metals in addition to fibre composites and polymers. While aluminium is the traditional aerospace material, its properties have already been optimised, and its potential for increased strength and the accompanying reduction in component size and weight is limited. Therefore, magnesium is considered the primary candidate for significant weight reduction (Kainer & John Wiley & Sons., 2000).

There are several reasons Mg is chosen as a material used for aerospace industries. Magnesium is a highly beneficial metallic material due to its superior castability, ductility, and machinability when compared to conventional metals. Additionally, replacing heavier metals with magnesium can increase fuel efficiency and decrease emissions for aircraft, resulting in cost savings on fuel and overall operation (Czerwinski, 2014; Hughes, 2011). To illustrate the savings potential, a standard Boeing 747 passenger model is used as an example. If all the aluminium alloys used in an aircraft were replaced with magnesium alloys, the overall weight of the aircraft could be reduced by about 60.4 tonnes (28% of its "operating empty weight"). Over time, there have been numerous experiments conducted in the aerospace industry where aircraft were built entirely using magnesium or magnesium as the base material due to the potential benefits it offers (Gupta, 2017).

The use of magnesium alloys plays a key role in the aerospace industry due to their unique properties. Magnesium alloys are used in aerospace parts such as leading and trailing edge flaps, control surfaces, actuators, door frames, wheels, engine gearboxes, power generation components, and structural items (Dziubińska et al., 2016; A. A. Luo, 2013). It has been reported that aluminium alloys make up 40% of the total weight of an average economy-class aircraft seat. However, a weight savings of about 30% can be achieved by redesigning the seat with magnesium alloys. This means a reduction of 360 kg in weight for

a typical narrow-body Airbus with 117 seats A318 and up to 4200kg for a wide-body Airbus with 700 seats A380 (Czerwinski, 2014; Stachel & KG, 2010).



Figure 2.3: Application of magnesium for aircraft parts (Gwyne & Lyon, 2007)

Conversely, magnesium and magnesium-based alloys are also used for biomedical applications. Interest in magnesium alloys as biodegradable metals has strongly grown over the last decades. Using Mg as an implant material reduces the risk of long-term incompatible implant interaction with tissues and eliminates the second surgical procedure to remove the implant and minimise size complications. Magnesium alloys are of interest mainly because of their biocompatibility, moderate corrosion rate, and appropriate mechanical properties when using the right alloying system and process parameters. Therefore, Mg alloys are also known as engineering biomaterials.

Biomaterials are used for diagnosing, treating, repairing, or replacing damaged tissues and further enhancing the function of organisms (X. Liu et al., 2022). Biomaterials are important in medical applications, especially for use in orthopaedic surgery, such as bone and joint replacements. Bone implant materials are an important part of biomedical materials, and about 70%–80% of implants are made of biomedical alloys (Hafeez et al., 2019). Magnesium (Mg) with biodegradability has gradually attracted extensive attention in the biomedical field (Dong et al., 2022). It exhibits mechanical performances similar to human bone (density of 1.74 g/cm³ and elastic modulus of 41–45 GPa) and good biocompatibility (J. Chen et al., 2018; Dong et al., 2022).

Mg and Mg-based alloys have mechanical and physical properties compatible with human bone, making them different from other biomaterials. They have similar densities and elastic modulus, which eliminates any elastic mismatches between implants and the bone (Y. Chen et al., 2014; Dorozhkin, 2014). Additionally, Mg is naturally present in the composition of bone and is an essential metal for metabolism (Saris et al., 2000). However, the primary challenge with Mg-based implants is their low corrosion resistance, which leads to unexpected and rapid degradation within a living system. Researchers have been exploring various strategies to enhance corrosion resistance and offer industrially applicable Mg-based biodegradable implants. These strategies include developing magnesium alloys with elements that slow down the corrosion process, surface treatments that form protective layers on the implant, and using biodegradable coatings that gradually degrade and release protective ions or drugs to inhibit the corrosion process. The introduction of Mg-based biodegradable implants into the market is expected to revolutionise the medical industry (Sezer et al., 2018).

Representative Magnesium-based Implants

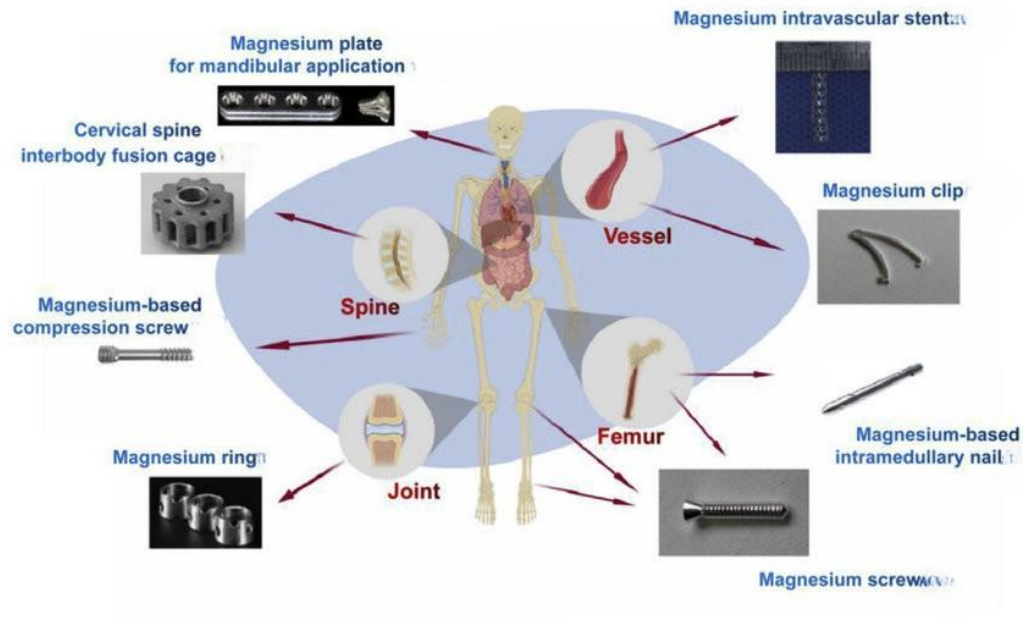


Figure 2.4: Some of the Magnesium-based implants for the human body (Y. Sun et al., 2019)

Table 2.3: Advantages of Mg-based alloys for biomedical implants

Advantages	Description
Low density and elastic modulus	Density and elastic modulus are similar to those of cortical bone (Seetharaman et al., 2022)
High specific strength	The strength-to-weight ratio is approximately 35–260 kNm/kg (Seetharaman et al., 2022)
Machinability	Mg has excellent machinability, is easy to achieve stable dimensions and can be easily processed into complex shapes (Kirkland et al., 2011)
Stress shielding effect	The elastic modulus of Mg is very close to that of bone, and many problems associated with implant stress shielding can be greatly reduced (Amukarimi & Mozafari, 2021)
Biocompatibility	Mg is biocompatible and has been shown to have osteogenic functions (Seetharaman et al., 2022)
Degradability	Mg eventually degrades completely in the body, which is beneficial to the patient (Kumar et al., 2018)

In automotive manufacturing, aerospace constructions and biomedical applications, the usage of commercially pure Mg alone is insufficient to be used without any metallurgy treatment. Mg should undergo specific treatment to make it stronger, stiffer, and more corrosion resistant. To overcome these limitations, Mg could be alloyed with other alloying elements (Yamada et al., 1999), strengthened with heat treatment (Čížek, Greger, Pawlica, et al., 2004) or made into new materials such as composites (Jiang Li, & Wang, 2003).

2.1.2 Alloying of Magnesium

Alloying of Mg is an alloy that primarily consists of pure Mg mixed with different elements that give rise to an entire range of materials, each of which is designed to maximise a particular characteristic such as strength, ductility, formability, machine-ability, or electrical conductivity (Polmear, 1994). Mg alloys can be classified into two categories; wrought and cast alloys. A wrought alloy is an alloy that is produced by plastic deformation by cold working such as rolling, forging and much more. Slooff, Zhou, Duszczyk, et al. (2007) has explained that the wrought magnesium alloy application is restricted. This reason is caused by low formability due to the small number of slip systems available for deformation, as Mg has an HCP crystal structure. Meanwhile, cast magnesium alloy is the Mg is alloyed, then heated from a molten state to be poured into the desired shape and allowed to solidify. The casting of magnesium alloys can be done either by sand casting, permanent mould casting or die casting. Table 2.3 shows the effects of alloying metals on the property of Mg based on several published research works.

Table 2.4: Alloying elements and the effects on Mg-based alloy

Alloying elements	Effects in microstructure	Effects on mechanical properties	Effects on corrosion resistance
Aluminium, Al	Solid solution treatment produces non-coherent, coarse precipitates of equilibrium $Mg_{17}Al_{12}$ (lying on the basal plane of the matrix) (Tamura et al., 2008)	Increase and enhance stiffness, hardness, tensile strength and castability (Prasad et al., 2022). It gives superior ductility and less dense alloys (Dobrzański & Tański, 2009) By using laser melting of AZ91, ultimate tensile strength can be achieved up to 274MPa (Niu, Shen, & Fu, 2018)	Increase corrosion resistance with a network distribution of $Mg_{17}Al_{12}$ in Mg alloys (Ding, Wen, Hodgson, et al., 2014) For Mg-Al alloys, the secondary phases are discretely distributed along the grain boundaries, leading to severe galvanic corrosion (Mingo et al., 2018)
Zinc, Zn	Grain structures produced are large and not amendable for grain refining. Susceptible for microporosity effect (Budinski & Budinski, 1999) With the increase of Zn content from 0 to 1.8 wt. %, the grain size of the Mg alloys gradually decreases from 7.1 to 2.7 μm (Wang et al., 2019)	Improves the yield strength, excellent solid solution strengthening and ageing strengthening (Ding et al., 2014)	Zinc can reduce hydrogen gas evolution when corrosion happens (Mordike & Ebert, 2001) Zinc surpasses Ni and iron impurities to improve the corrosion resistance of Mg-based alloys (Gupta & Ling, 2011)
Manganese, Mn	The addition of manganese helps to reduce the iron impurities, even without the presence of aluminium (Tamura et al., 2008)	Increase strength-to-weight ratio to Mg and slightly increase ultimate tensile strength (Kulekci, 2008) Strength, yield anisotropy and ductility have been improved (Yu et al., 2018)	Increase the corrosion rate of Mg without adding other alloying elements by increasing cathodic kinetics (Gandel, Birbilis, Easton, et al., 2010) With 0.5% Mn, the Mg-based alloy possesses the best corrosion resistance, and the tendency of intergranular corrosion and galvanic corrosion increase with the continuous addition of Mn (T. Zhao et al., 2019)

Table 2.4 continued

Copper, Cu	Produces lamellar structure when alloyed with zinc (Polmear, 1994) Cu has a proper effect on refining Mg-2%Zn alloy grain size in the as-cast condition. The average grain size of Mg-2%Zn alloy reduced from about 1000 to 200 μm in the case of Mg-2%Zn-5%Cu (Lotfpour, Emamy, Dehghanian, et al., 2017)	Improves ductility of the magnesium alloys by about 3-8% (Polmear, 1994) The incorporation of 0.25 wt% Cu in Mg-1Al alloy resulted in more excellent compression and yield strength than pure Mg (Safari, Toroghinejad, & Kharaziha, 2019)	Incorporation of 0.25 wt% Cu in Mg-1Al alloy resulted in a significantly reduced degradation rate than pure Mg after immersion in simulated body fluid (SBF) solution (Safari et al., 2019)
Rare earth metals, RE	Mg-Ag-RE alloys Mg_3R compound is produced at grain boundaries of Mg solids solution embedded with a fine precipitate of $\text{Mg}_{12}\text{Nd}_2\text{Ag}$ precipitates (Budinski & Budinski, 1999)	Mg-Y-RE alloys give good strength and creep resistance up to 300°C (Budinski & Budinski, 1999) Mg-Al-Ce alloys prove that they can improve creep properties at elevated temperatures (Pettersella & Westengen, 1996)	Mg-Y-RE alloys give a high affinity of oxygen that prone to corrosion of the alloy (Budinski & Budinski, 1999)
Zirconium, Zr	Promotes grain refinement when alloying with zinc (Gandel et al., 2010)	Improve the strength and stiffness, significantly enhancing ductility, elongation, and ultimate yield strength of Mg alloy with a small amount of addition for binary Mg-Zr alloy (Ding et al., 2014)	Increase corrosion resistance to Mg alloy with a small amount (2% composition) of Zr (Ding et al., 2014)
Silicon, Si	The Mg_2Si particles are coarse and not granular in shape because of dendritic solidification (Bamberger, 2001) The average grain size of the Mg_2Si phase decreases with an increase in the weight percentage of silicon act as a grain growth inhibitor (Seth, Singh, Singh et al., 2020)	Improve the hardness of magnesium alloys up to 139 Vickers hardness (Mining, Higashi, & Prefecture, 1996) The hardness increases with increasing Si addition gradually from 4 wt% to 6 wt% of Si (Seth et al., 2020)	Reduces corrosion resistance oxidation tendency (Kulekci, 2008)

In most automotive, aerospace, and biomedical industries from various countries, Mg cast alloy is widely used, as studied by (Easton et al., 2008; Luo, and Nakatsugawa, et al., 1995). Mg can be easily cast by either the hot-chamber or cold-chamber die-casting process. Casting rates for magnesium are higher than for other die-cast metals because magnesium's lower heat content produces fast chilling of molten metal. Some parts that are made from Mg can be die-cast up to 50% faster than similar parts in aluminium, depending on their size and configuration. Table 2.5 shows examples of some Mg cast alloys and their specific applications.

Table 2.5: Mg cast alloys and their specific properties (Bruce Abbott et al., 2001)

Alloy designation	Alloying elements	Uses	Basic properties
AZ91	9.0% Al, 0.7%Zn 0.13%Mn	General casting alloy	Good castability, good mechanical properties at T<150°C
AM60	6.0% Al, 0.15%Mn	High-pressure die-casting alloy	More excellent toughness and ductility than AZ91, with slightly lower strength.
AE44	Mg-Al-rare earth system	General casting alloy	Better creep behaviour and castability than AE42
AS41	4.2%Al, 1.0%Si	General casting alloy	Better creep resistance than AZ91 at elevated temperatures but lower strength
ZE41	4.2%Zn, 1.2%RE, 0.7% Zr	Specialised casting alloy	Rare earth addition improves creep strength at elevated temperatures.

2.2 Heat treatment of Magnesium-based Alloy

Post-treatment of metals, such as heat treatment, is done to Mg-based alloy to increase the mechanical properties. Heat treatment is an operation that involves heating and cooling metal alloys in a solid state. Heat treatment can be classified into solution heat treatment and precipitation hardening. Solid solution strengthening involves the formation of a single-phase solid solution of metals via quenching. In some cases, solid solution strengthening can be achieved by introducing foreign atoms that decrease dislocation mobility, effectively hardening the material. Meanwhile, precipitation heat treatment involves the addition of impurity particles to form fine precipitates during heat treatment. These precipitates act as obstacles to dislocation motion, providing additional strengthening. A suitable heat treatment process will improve machinability, mechanical properties, and corrosion resistance and produce a homogenous microstructure.

Homogenisation and subsequent ageing treatment of AZ91D alloy improve mechanical properties considerably. Early studies on Mg-based alloys ageing showed improved strength with the increasing refinement of precipitates. Finer precipitates were produced at lower ageing temperatures, a shorter ageing time, and lower Al content (Čížek et al., 2004; K. C. Wu, Yeh, & Chang, 2015). Other than AZ91D alloy, Mg with RE alloys Mg–2Gd–2Nd–2Y–1Ho–1Er–0.5Zn–0.4Zr (wt%) prepared by direct-chill semi-continuous casting technology has been made by Li et al. (2018). Their research has proven that solid solution heat treatment with RE addition of 8 wt% exhibits a remarkable age-hardening response in terms of hardness in hardness value, HV ($\Delta HV = 35.7$), which demonstrates that the multiple RE (RE which consists of Gd (Gadolinium), Nd (Neodymium), Y (Yttrium), Ho (Holmium), Er (Erbium)). Mg alloying instead of single Gd can effectively improve the age-hardening response. Other mechanical properties have a relatively good combination of

high strength/hardness (UTS (ultimate tensile strength) > 300 MPa; TYS (tensile yield strength) > 210 MPa; hardness 115.3 HV) with proper ductility ($\epsilon \approx 6\%$), which are contributed mainly involving ageing precipitation of $\beta\epsilon$ and β'' phases of Mg alloy. Meanwhile, the result was a moderate thermal conductivity (52.5 W/(mK)) for thermal properties.

Unfortunately, such heat treatment may significantly impact corrosion behaviour due to modifying microstructures on Mg-based alloys. Ageing treatment of homogenised AZ91D Mg alloy leads to the precipitation of the beta phase along the grain boundaries. The ageing temperature, time, and specific processing route influence the size and morphology of the residues. As the beta phase may act either as an effective cathode or a physical barrier to corrosion, depending on its volume fraction, it is crucial to study the influence of this phase on corrosion and electrochemical behaviour (Gu, Lian, et al., 2006).

Heat treatment of Mg-based alloys improves mechanical properties such as tensile strength and hardness. However, some properties, such as toughness and corrosion resistance, are being sacrificed. The preferable solution to overcome the limitation of heat treatment of Mg-based alloys is to produce Mg-based composites.

2.3 Metal Matrix Composites for Magnesium

Metals and alloys are generally produced and shaped in bulk form, but they can also be intimately combined with another material to improve their performance. In this modern world, composite materials have started to be applied in automotive applications. One of the most common composites is metal matrix composites (MMCs). In their most generic form,

metal matrix composites consist of metals as the main constituent of the matrix, while the other constituent is a reinforcement that can be either ceramics, metals, non-metals, or organic compounds. The matrix acts as a binder and provides the bulk properties of the material, while the reinforcement enhances certain properties, such as strength, stiffness, thermal conductivity, or wear resistance.

The metal matrix composite industry is still in the early development stage for typical applications and is less widely developed compared to polymer matrix composites. However, MMCs have shown better properties than pure metals themselves. It has a higher specific modulus, higher specific strength, better properties at elevated temperatures and corrosion resistance. With suitable MMC fabrication, the composite will promise to give better properties and last a longer lifetime than even alloyed metals (Zhou et al., 2020). Besides, MMCs give advantages in physical properties such as moisture absorption resistance, non-inflammability, high electrical and thermal conductivities, and resistance to most radiations (Matthews & Rawlings, 1999).

In MMC fabrication, the metal matrix should be more robust than monolithic metals. However, this property may be limited due to the anisotropic properties of the composites based on the specific fabrication. To overcome the anisotropic property, the suitable fabrication of composite is the critical factor. Fabrication of reinforcement by particulate form gives the composite uniform mechanical strength in all directions (Jones, 2018).

A study by Cai et al. (2012) found that adding Zn beneficially refines the grain size of the Mg matrix, and grain refinement is widely known to be preferable for reducing the corrosion behaviour of Mg. The smaller size of grains induced by alloying Zn into Mg would increase the grain boundary areas, which are known as the least susceptible area to be

attacked by corrosion medium. The study was followed by Mohamad Rodzi et al. (2019) study as magnesium zinc reinforced with hydroxyapatite, a biodegradable material. Their fabrication process using single-step processing in a planetary mill shows mechanical properties such as hardness, while another method using a single-step processing ball mill shows a better degradation rate.

2.3.1 Titanium-based Particles Reinforced Mg Composite

One research conducted by Hassan and Gupta (2002) proposed that magnesium can be made into MMC reinforced with titanium by melt deposition technique followed by hot extrusion. The results show that in the microstructure of Mg composites, there are no macrostructural defects such as the presence of micropores and shrinkages and microstructure, minimal porosity presence in the composite and titanium phosphate-magnesium (TiP –Mg) interfacial integrity with the distribution of titanium particulates. Mechanical results show the yield strength of composite increases for about 53 - 64 MPa difference compared with pure Mg. The ultimate tensile strength is decreased by 10 MPa, which is affected by the presence and the increasing amount of Ti reinforcement. Other studies by Kondoh et al. (2003) revealed that Magnesium reinforced (Mg_2Si/Mg) composite alloys show fine microstructures and superior mechanical properties: ultimate tensile strength is about 350 MPa while yield strength is over 300 MPa. Increasing the ultimate tensile strength and yield strength are by the microstructure of fine Mg_2Si dispersoids with a mean particle size less than 5 μm and a refined matrix grain of 2 – 5 μm .

Another research was conducted by Tacikowski et al. (2014) to study the corrosion resistance of Mg reinforced with Ti. Their research fabricated the AZ91 magnesium with aluminium and zinc alloy reinforced with tightened titanium nitride (TiN) layers composite by a hybrid method. The corrosion test was conducted using electrochemical methods with 0.5 M NaCl solution. The results have shown that using the hybrid method, the AZ91 Mg alloy reinforced with TiN highly gives corrosion resistance. This is because the tightened layers composed of a TiN surface layer and an aluminium sub-layer diffusively bonded with the substrate, separated by a thin transition titanium sub-layer, ensure outstanding corrosion resistance in a 0.5 M NaCl solution.

Further research was made by Xiao et al. (2020) that Mg-based composite reinforced with a specific spatial arrangement of in-situ titanium boron TiB_2 nanoparticles. This nanocomposite is fabricated using an in-situ casting method assisted by semi-solid mechanical stirring and conventional hot extrusion. Their research found that the mechanical properties Compared with AZ91 matrix Mg alloy, the designed nanocomposite exhibits enhanced strength without sacrificing ductility and achieves simultaneous strengthening and toughening effects due to the specific spatial arrangement of TiB_2 nanoparticles. The improved strength of the nanocomposite is due to the grain refinement, dislocation strengthening from geometrically necessary dislocations and the activation of the Orowan mechanism and load transfer effect.

2.3.2 Carbon Nanotubes Reinforced Mg Composite

Previous research findings by Goh et al. (2006) have proposed that the Mg should be made into composites reinforced by 0.3% carbon nanotubes (CNT). Carbon nanotubes

(CNTs) are one type of nanomaterial, which are made of carbon atoms arranged in a cylindrical structure. The structure is akin to graphite sheets (a hexagonal lattice of carbon) rolled up into seamless cylinders. In this study, the Mg composites were created through the powder metallurgy technique, with their physical and mechanical properties were examined.

This study showed that the porosity of Mg composites is higher than pure Mg. With the results porosity level of the nanocomposites increases with increasing weight percentages of CNT. Up to 1.15 vol% percentage of porosity was found when 0.3 wt.% of CNT was incorporated. Meanwhile, in mechanical properties, it is found that the yield strength of Mg CNT composites gives better results, up to 20 MPa, as CNT composition increases up to 0.3 than pure Mg. The ultimate tensile strength of Mg composites increases significantly to 5 MPa in difference. Goh et al. (2006) also compared the previous research that uses Mg reinforced with silicon carbide (SiC) that the yield strength is 18 MPa lower and ultimate tensile strength is 34 MPa lower than Mg CNT composites. Therefore, the Mg CNT composites have better mechanical properties than conventional Mg–SiC composites when synthesised with powder metallurgy.

Another research by Say et al. (2020) had found the effect of corrosion on Mg CNT composites. In their study, Mg-CNT was immersed in 3.5 wt.% NaCl solution using evolved hydrogen gas, weight loss and potentiodynamic polarisation measurements. The results show that micro galvanic corrosion was the factor in the corrosion mechanism of the composites. The corrosion rate of the Mg–CNT microstructure increased drastically with an increase in the wt.% CNT from 0.3 to 1.3. The preamble results suggest that the CNT acted as effective cathodes to accelerate corrosion. In short, Mg-based carbon nanotubes with high CNT led to more corrosion.

2.3.3 Graphene Nanoplatelets Reinforced Mg Composite

Graphene nanoplatelets, known as GNPs (thin graphite sheets with a thickness of less than 100 nm), are nanoparticles made from graphene. Graphene is a type of carbon allotropes with a two-dimensional form factor, atomic size and hexagonal or honeycomb-like shape lattice, as shown in Figure 2.3 (Yung, Pierpoint, et al., 2013). GNPs is a diminutive size of graphene measured between sub-micrometre to 100 micrometres. GNPs are available commercially in powder form. Since its discovery, the single-layered atom-thick flatbed monolayer structure has revolutionised the nanotechnology platform. Graphene is one of the thinnest materials, founded in 2004 by Geim and Novoselov (Dhand, Rhee, Kim, et al., 2017).

The mechanical properties of a single graphene layer were measured by nanoindentation, exhibiting Young's modulus of about 1 TPa, and strength of 130 GPa, making graphene one of the most robust materials available today (Bizao, Botari & Galvao, 2014). The thermal conductivity of a graphene layer is exceptionally high and measured to be approximately 3080–5150 W/mK (Ghosh et al., 2008). GNPs exhibit exciting properties such as lightweight, high aspect ratio, electrical and thermal conductivity, mechanical toughness, low cost, and planar structure (Cataldi et al., 2018). Further properties of GNPs are summarised in Table 2.5. Recent studies showed the possibility of large-scale graphene production for different applications (Zheng & Kim, 2015).

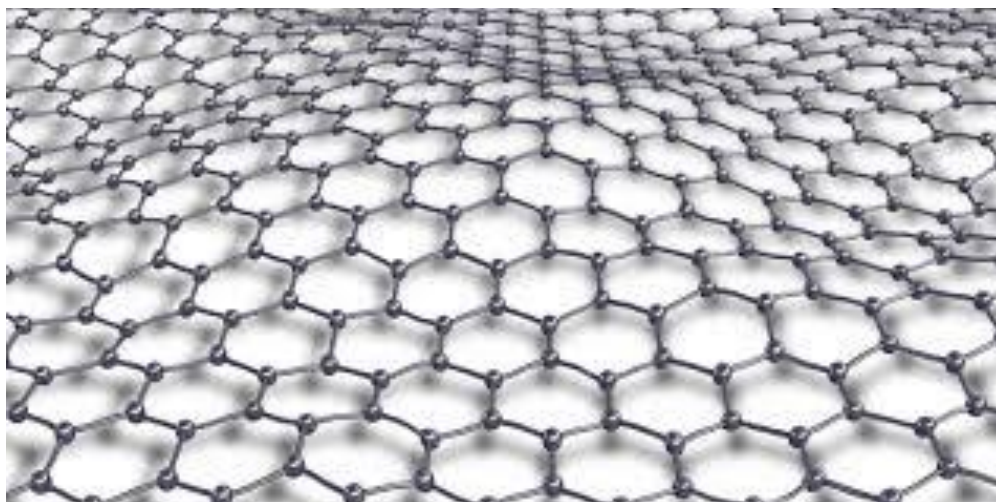


Figure 2.5: Graphene model structure (Retrieved December 04, 2017, from <https://www.themanufacturer.com/articles/talga-resources-first-delivery-of-a-grapheneproduct/>)

Table 2.6: Some properties of graphene nanoplatelets (Ghosh et al., 2008; Stankovich et al., 2006; Yung et al., 2013)

Properties	Value parallel to Surface
Density (g/cm ³)	2.25
Thermal Conductivity (watts/m-K)	3,000
Thermal Expansion (m/m/dg-K)	4-6 x 10 ⁻⁶
Tensile Modulus (GPa)	1,000
Young Modulus (GPa)	1,060
Tensile Strength (GPa)	5

A study conducted by Rashad, Pan, Tang, and others (2015a) explored the use of graphene in creating an Mg-based composite to enhance the mechanical properties of Mg,

specifically its strength and stiffness. However, another study by Rashad et al. (2017) states that the corrosion potential of AZ31 and AZ61 Mg composites was less noble than their monolithic alloys, which may be due to the galvanic effects of GNPs when immersed in NaCl solutions. Adding GNPs into AZ31 and AZ61 alloy matrices has increased the corrosion currents due to dislocation densities at the interface of composites, creating crevice attacks and leading to a high corrosion rate of composites compared to unreinforced alloys.

The fabrication of Mg reinforced with GNPs composite was made to improve the strength, hardness, and other mechanical properties. One research conducted by L. Y. Chen et al. (2012) have stated that GNPs act as a corrosion barrier in the metal and hence lower the corrosion rate of the metal. Graphene properties that are lightweight, stiff, and excellent electrical conductivity have made it attractive to researchers to explore Mg-based composites, as the graphene can also act as a corrosion barrier to the Mg matrix.

The strength of Mg reinforced with GNPs composite in terms of improving mechanical properties has been studied by Du et al. (2019). Their remarkable research that GNPs introduce defects such as carbon vacancies, broken carbon bonds, oxygen-containing functional groups, and wrinkles with high specific surface area to ZK60 Mg alloy. The comparison between ZK60 Mg alloy, ZK60 Mg reinforced GNPs without defects, and ZK60 Mg reinforced GNPs with defects proved that GNPs defects can also improve the mechanical properties such as tensile yield strength at 263 MPa, 63% enhancement than ZK60 (161 MPa). However, defects on GNPs improve the elongation percentage of ZK60 alloy. The elongation from 15.6 to 22.5 with a 44% increase is much better than ZK60 GNPs without defects. These indicate that the unique interface microstructure in ZK60 reinforced GNPs with defects composite can vastly enhance plasticity without strength sacrifice (Guo et al., 2019).

The corrosion resistance of MMCs mainly depends on the protective passive film formation on the surface of the magnesium matrix. This protective layer of Mg can be produced from the gas-phase deposition process (Gray & Luan, 2002; Tjong, 2013). Besides that, several factors influence the formation of the protective layer. These factors include the usage of reinforcing particles, alloy composition, size and distribution in the matrix, fabrication processing method, and the interface between the matrix and reinforcement (Afifi, 2014). Furthermore, a minimal change in any of these factors can significantly affect the corrosion behaviour of the composites.

GNPs are known to withstand corrosive resistance; even in the outermost graphene layer under corrosion, GNPs with much more graphene layers retain their base graphitic structure (Cheng et al., 2015). The formation of a protective graphene layer is produced instead of magnesium because of the shifting electron due to the electrical conductivity of the graphene (Ghavanini, Theander, & Industriteknik, 2015).

2.4 Corrosion of Magnesium

Mg and its alloys have high strength-to-weight ratios. Still, poor corrosion resistance affects the viability of increased magnesium usage in industries, especially in automotive and other medical or engineering applications (Makar & Kruger, 1993). It has been recognised that the corrosion of Mg is the most anodic metal compared to the other metals (Guangling Song & Atrens, 2000). Mg is susceptible to the physiological environment due to all engineering metals' lowest standard potential $-2.38\text{V}_{\text{nhe}}$. Pure magnesium's Pilling-Bedworth ratio (P-B ratio) is 0.81 (T. Chen et al., 2019). Besides, Mg alloys often contain second phases, which significantly influence the corrosion behaviour of Mg alloys. On the one hand, the morphology and structure of the second phase substantially affect the corrosion

resistance of Mg alloys. Finely dispersed second-phase particles are beneficial to forming continuous corrosion product films; constant and net-distributed second phases can act as physical barriers to hinder the spread of corrosion and thus may improve corrosion (Zhang et al., 2020).

Despite all the corrosion phenomena, Mg's actual corrosion potential depends upon other factors such as environment and surface status. Mg is the most active metal used in engineering applications. It corrodes so readily in some environments that magnesium alloys often serve as sacrificial anodes on structures such as ships' hulls, buried pipelines, and steel piles (G. L. Song, Dudney et al., 2014).

2.4.1 Mechanism of Mg Corrosion

Magnesium dissolution in aqueous environments generally proceeds by electrochemical reaction with water to produce magnesium hydroxide ($Mg(OH)_2$) and hydrogen gas (H_2), a mechanism which is relatively insensitive to the oxygen concentration (Kekare & Kulkarni, 2021; Makar & Kruger, 1993). Equation 2.1 explains the overall probable response.



This net reaction can be expressed as the total of the following partial reactions:



The mechanism described involves some intermediate steps, most remarkably the initial production of monovalent magnesium ions having short lifetimes. Most of the studies of the kinetics of Equation 2.1 concluded that the rate of the attack that occurs at pH less than about 11 is controlled by the diffusion of reactants or products through the surface film, even under open-circuit conditions (James, 1943; Makar & Kruger, 1993). The results of newer studies in solutions buffered at pH 9-10 favour a dissolution rate controlled by charge transfer at the corrosion potential.

As the corrosion proceeds, the metal surface experiences a local pH increase because of the formation of magnesium hydroxide $\text{Mg}(\text{OH})_2$, whose equilibrium pH is about 11. The protection this film supplies is thus highly dependent on the exposure conditions. Mg is resistant to corrosion in small volumes of water, free of species aggressive to the film. In the atmosphere, Mg reacts further with carbon dioxide, forming magnesium carbonate as a sealer for the hydroxide film. High-purity Mg, therefore, has the potential to be highly corrosion resistant, and Mg alloys perform better in the atmosphere than iron (G. Song & Atrens, 2003). Problems appear when impurity elements are present at levels that boost micro galvanic cell action, when the metal or alloy is galvanically coupled to an effective cathode material, or when the nature of the environment prevents the formation or maintenance of a protective film, as mentioned by Makar and Kruger (1993).

In summary, Mg's corrosion in aqueous environments proceeds electrochemically according to Equation 2.1. Magnesium may form a surface film known as $\text{Mg}(\text{OH})_2$, which protects it in alkaline and poorly buffered environments (Ghali, Dietzel, & Kainer, 2013; Makar & Kruger, 1993).

2.4.2 Types of Mg Corrosion

In Mg, several types of corrosion are essential to be known in engineering applications. General corrosion, also known as uniform corrosion, occurs when a metal's entire surface degrades at a fairly uniform rate. It is the most common type of corrosion and is easy to predict and measure. For Mg and Mg-based alloys, general corrosion is a particular concern. In aqueous environments, especially those with a high pH, Mg is prone to rapid oxidation due to its position in the galvanic series and its standard potential. The reaction generally occurs as follows:



The magnesium (Mg) reacts with water (H₂O) to form magnesium hydroxide (Mg(OH)₂) and hydrogen gas (H₂). This reaction is quite rapid, and the resultant Mg(OH)₂ layer is often porous and does not effectively protect the underlying Mg from further corrosion, leading to a high corrosion rate. In addition, the high corrosion rate can result in the release of a large amount of hydrogen gas and high local pH, both of which can cause adverse biological responses (Dong et al., 2022; Lindström et al., 2004).

The second type of corrosion is galvanic corrosion. Galvanic corrosion (bimetallic corrosion) occurs when two different metals are in electrical contact in an electrolyte, where the more noble metal is protected. The more active metal tends to corrode. This corrosion can occur between Mg with other metals, especially the more noble metals than Mg (K. Xiao, Dong, et al., 2016). Different metals and alloys have their unique electrode potentials. One metal will act as a cathode and the other as an anode when in contact with an electrolyte. The more active a metal is, the more likely it will form an anode in an electrolytic environment. While the more noble a metal is, the more likely it will create a cathode in the

same environment. The electrolyte acts as a conduit for ion migration, transferring metal ions from the anode to the cathode. As a result, the anode metal corrodes faster than it otherwise would, while the cathode metal corrodes more slowly and, in rare cases, may not corrode (H. Liu et al., 2021; Pryor & Astley, 2013).

G. Song et al. (2004) investigated the galvanic corrosion of Mg alloys using AZ91 alloy coupled with aluminium A1380, zinc and steel 4150. Based on their finding, the galvanic corrosion rate of AZ91D in contact with zinc, aluminium alloy, or steel increases with time. The critical results of their research showed that steel is the worst of the three cathode metals studied in terms of the galvanic corrosion rate, the distribution of galvanic current density and the influence of the cathode/anode area ratio, followed by zinc. Lastly, aluminium causes the most miniature corrosive attack on the AZ91D alloy.

Another galvanic corrosion research conducted by Sekar et al. (2020) on ZE41 Mg alloy coupled with Al7075 alloy with three different NaCl solutions. The fabrication method was enhanced by equal channel angular pressing (ECAP). Based on their research, in a 0M NaCl solution, a composite oxide/hydroxide layer of magnesium and aluminium is formed. At the same time, the surface film consists only of a layer of magnesium oxide/hydroxide layer when tested in 0.1M and 1M NaCl due to the aggressive nature of chloride ions. Galvanic corrosion resistance diminished 26% after the fourth pass ECAP compared to as-received counterparts when tested in 0.1M NaCl solution. However, the as-cast sample pits were observed, while the fourth pass sample exhibited uniform corrosion, which is preferred in engineering applications. Their findings revealed that resistance to galvanic corrosion evinced a remarkable improvement of 58% and 54% after the fourth pass ECAP due to the

fine distribution of second-phase particles (T-phase) in interaction with 0 and 1M NaCl, respectively (Sekar et al., 2020).

Besides that, pitting corrosion can also happen in Mg. Pitting corrosion is a form of localised corrosion that forms small spots or pits in the metal. Pitting corrosion in Mg can be caused by localised chemical or mechanical damage to the protective oxide film. Pitting corrosion can also occur in non-passivating alloys with protective coatings or certain heterogeneous corrosive media (Ghali et al., 2013). The latest research by Boland et al. (2018) made a model on the WE43 Mg alloy to study more mechanical and corrosion for pitting corrosion model calibration. Their research found that pitting corrosion happened on WE43 alloy and showed the result by computational modelling. They found that the AZ31 and Mg WE43 alloys have similar corrosion characteristics and highlight the robustness of the pitting corrosion model (Boland et al., 2018).

Another type of corrosion in Mg is intergranular corrosion. Intergranular corrosion in Mg forms a localised attack of the grain boundaries caused by secondary phase precipitation. Intergranular corrosion has much sharper tips than pitting corrosion. It is a more drastic stress riser and has a more damaging contribution to corrosion fatigue. The grain boundaries are always the preferred sites for precipitation and segregation in alloys. It is generally regarded that alloys with intermetallic phases or compounds are highly susceptible to intergranular corrosion (Zeng et al., 2006).

A form of attack at narrow gaps, called 'crevices', appears to be crevice corrosion. Crevice corrosion in Mg can occur by attacking a metal surface between two joining surfaces. This corrosion happens on metal-to-metal joints or metal-to-non-metallic materials. Crevice corrosion can be initiated due to the hydrolysis reaction in the case of Mg and Mg-

based alloys, at least in certain conditions where oxygen does not play a significant role in the corrosion mechanism. The formation of Mg hydroxide should influence the properties of the interface between the Mg and the solution in the crevice (Ghali et al., 2013; H. Wu et al., 2019).

Crevice corrosion is generally not desirable to occur in Mg and Mg-based alloys, as the hydrogen evolution potential is lower than that of water, and the oxidation of Mg by dissolved oxygen is negligible. However, other factors, such as the pH gradient induced by the hydrolysis of corrosion products and the corrosion of Mg in the crevice, may be accelerated by the accumulation of chloride ions in $\text{Mg}(\text{OH})_2$ (B. Chen et al., 2020).

Stress corrosion cracking (SCC) is a severe form of corrosion damage that can lead to sudden and catastrophic failure of engineering equipment and structures. In the case of magnesium (Mg) and Mg alloys, SCC is a significant concern and has been the subject of extensive research.

SCC in Mg-based alloys can occur in various environments, including moist air, high-purity water, chloride-containing solutions (such as NaCl), sulfuric acid (H_2SO_4), nitric acid (HNO_3), and hydrogen chloride (HCl) solutions. Alloys containing aluminium (Al) are believed to be particularly susceptible to SCC in air, distilled water, and chloride-containing solutions (Zeng et al., 2006).

The mechanisms of SCC in Mg-based alloys can be broadly categorised into two groups: continuous crack propagation by anodic dissolution at the crack tip and discontinuous crack propagation by a series of mechanical fractures at the crack tip (J. Jiang et al., 2023).

1. **Dissolution Models:** These models propose that SCC occurs due to the preferential attack and dissolution of the metal at the crack tip. Some examples of dissolution models include the preferential attack model, film rupture model, and tunnelling theory.
2. **Brittle Fracture Models:** These models involve mechanical fractures at the crack tip and include cleavage processes and hydrogen embrittlement (HE) theory. Hydrogen embrittlement is a phenomenon where hydrogen atoms absorbed into the metal lattice can weaken the material, making it more susceptible to brittle fracture.

Finally is fatigue corrosion, where Mg is exposed simultaneously to cyclic stresses and corrosive attack, leading to Mg failure. This corrosion is caused when the mechanical degradation of stress increases and the number of cycles required to cause fracture decreases. As in the case of stress corrosion cracking, corrosion fatigue depends on the interactions among loading, environmental, and metallurgical factors. This phenomenon then causes the metal surface of Mg with a protective film to start to corrode by push-pull bending induced by cyclic stresses. If the Mg is simultaneously exposed to a corrosive environment, the failure can occur at even lower loads and after a shorter time (Unigovski, Eliezer et al., 2003; Zeng et al., 2006).

2.4.3 Factors Affecting Mg Corrosion Rate

There are several factors that can influence the corrosion of magnesium. One of the main factors is on metallurgical properties such as alloying elements. Alloying elements such as aluminium on Mg-based alloys decreases the corrosion rate, and further addition of Al percentage will increase the corrosion rate in aqueous solutions containing chloride. The atmospheric corrosion rate is decreased with increasing Al concentration (Jönsson & Persson, 2010; Merino et al., 2010). For example, the corrosion rate of AZ61 was 20% lower

than that of AZ31 in a continuous condensation atmosphere, attributed to the higher concentration of Al in AZ61 (Feliu et al., 2011). Alloying with rare earth elements (RE) also decreased corrosion rates of AZ-series and AM-series Mg alloys in aqueous chloride-containing solutions (Arrabal et al., 2015; G. Wu et al., 2005). In atmospheric environments, a considerable decrease in the corrosion rate occurs for Mg alloys containing RE elements (Arrabal et al., 2012, 2015). Arrabal et al. (2012) found that alloying with Nd or Gd decreased the corrosion rate of AM50. The Nd or Gd reduced the volume fraction of Al-Mn intermetallics because of the formation of Al-MnRE and Al₂RE intermetallic phases. The micro-galvanic corrosion became weaker because the Al-Mn-RE and Al₂RE intermetallic phases are less efficient cathodes. Similarly, Arrabal et al. (2015) found that alloying with Nd reduced the corrosion rate of AZ91 by 70% under real atmospheric corrosion conditions.

Another factor based on the metallurgical properties that affect the Mg corrosion rate is the microstructure. In the matrix phase of Mg-based alloys, grain refinement improves the mechanical properties and corrosion performance (Pu et al., 2011; Yamashita et al., 2001). Grain size can also influence the electrochemistry of Mg-based alloys (Cao et al., 2015; Pu et al., 2012). Liao et al. (2013) reported that the corrosion rate of AZ31B had an inversely linear relationship with $d^{1/2}$ (grain size) in a cyclic neutral-salt spray test. In marine and urban environments, the corrosion rate of the AZ31B with fine grains was lower than that of the AZ31B with coarser grains, especially in urban environments. Y. gang Li et al. (2013) found that the attributed lower corrosion rate of extruded AM60 to the smaller grain size and more homogeneous microstructure compared with the ingot AM60. Grain refinement by extrusion resulted in a uniformly distributed, more-protective corrosion film. Moreover, grain refinement may also reduce the possibility of surface film cracking because of the

lower residual stress in the surface film (Liao et al., 2012; Orlov et al., 2011; Ralston & Birbilis, 2010).

Another factor that influenced the Mg corrosion rate is the environmental factors. Firstly, the common environmental factor is the presence of chloride ions. Chloride is one of the most corrosive species, which can induce the breakdown of the Mg surface oxide film, resulting in rapid Mg corrosion. This has been widely investigated in aqueous solutions (Ardelean et al., 2013; Cao et al., 2013; Vilarigues et al., 2008). The increasing corrosion rate with increasing concentration of Cl^- was attributed to Cl^- ions penetrating and breaking down the somewhat protective corrosion product film. The partially protective layer formed on the AM50 was entirely penetrated by Cl^- , which significantly increased the corrosion rate (Danaie et al., 2013). The more negative corrosion potential could be attributed to the greater amount of actively corroding surfaces (Ben-Hamu et al., 2010).

Besides chloride ions, oxygen gas O_2 significantly contributes to cathodic depolarization and thus differentiates atmospheric corrosion from immersion corrosion. O_2 is present in atmospheric corrosion in a dissolved form in the surface water film. O_2 does not significantly contribute to the corrosion of Mg in distilled water or saline solutions because hydrogen evolution is the main cathodic reaction in this case (Baril et al., 2001; Ben-Hamu et al., 2010). For example, Beldjoudi et al. (1993) found that the cathodic curve for Mg in a NaCl solution saturated with $\text{Mg}(\text{OH})_2$ was the same with aeration and deaeration.

Other than that, dust particles commonly present in the atmosphere include the products of the natural weathering of rocks and soil, emission of combustion engines and other artificial sources. The dust particles can absorb moisture, conglomerate on metal surfaces, and accelerate corrosion. Dust particles can accelerate Mg corrosion by decreasing the critical relative humidity for forming a liquid water film. The size of dust particles

influences the pit size of Mg alloys (Y. Gang Li et al., 2013). J. Chen et al. (2008) found that dust particles stimulate micro-droplet formation. The hydrophilicity decreases in the following order: salt particle > dust particle > AlMn phase > other phases such as primary Mg and eutectic Mg phase. In a marine environment, airborne particles are mainly soluble, such as sea salt containing chlorides that accelerate Mg corrosion (Jönsson et al., 2011).

2.5 Powder Metallurgy Technique for Mg-Based Composites

Powder metallurgy is a technique used to fabricate individual or alloy powders from metal powders. It is one of the MMC synthesis methods by using solid-state fabrication other than diffusion bonding. Besides alloy powders, this technique can be used with admixing of non-metallic powders with metal powders. This technique consists of three basic steps: mixing, compacting, and sintering. Using powder metallurgy offers a homogeneous and uniform distribution of reinforcement particles in the matrix for composite synthesis (Rashad, Pan, et al., 2015). The powder metallurgy technique is a reasonable and easier process for producing an Mg matrix composite. There are many advantages occurred for composites. The manufacturing process temperature is low, and the distribution of reinforcement is high (Jayasathyakawin, Ravichandran et al., 2020). Mg plays a vital role in powder metallurgy in mechanical properties and corrosion resistance to develop Mg-based bio composite via degenerate melt deposition technique (Ali, Hussein, & Al-Aqeeli, 2019).

The powder metallurgy technology has facilitated the fabrication of more complex metal structures than other metal-forming methods, such as stir casting and hot extrusion. This technique has proven to give great precision to structural metal pieces, and extensive series of pieces with narrow tolerances can be made. Powder metallurgy can also help control

the metal porosity, perform well under stress, and increase mechanical properties such as hardness and wear resistance (Angelo & Ravisankar, 2021).

Previous research by Rashad et al. (2014) has fabricated Mg-GNPs using powder metallurgy instead of the ball milling technique. This is because ball milling was considered a significant problem when using Mg as the process produces heat which can burn Mg powder quickly. Other than that, powder metallurgy can be accomplished with a hot extrusion process, as researched by Goh et al. (2006). In their study, Mg reinforced with CNT can be synthesised to observe the physical and mechanical properties of the composites.

Moreover, research by Feng and Han (2010) fabricated calcium polyphosphate-reinforced ZK60A magnesium alloy composites using the powder metallurgy technique. Their study stated that the powder metallurgy technique was more suitable for preparing uniform MMCs than the stir casting technique. Their research proved that using the powder metallurgy technique gives better mechanical properties in terms of compressive strength and corrosion resistance by adding calcium polyphosphate to reinforce the Mg composite.

In another previous research by Rodzi and Hussain (2018) powder metallurgy method on Mg-based composites can improve the mechanical properties. In their research, the compressive strength obtained can be achieved by additional treatment of ball milling on the Mg-Zn reinforced with hydroxyapatite composite with parameters from 2, 4, 6, and 8 hours of milling 209 MPa with Vickers hardness on 70.47 HV. These results proved that the powder metallurgy technique with ball mill treatment might help increase strength due to the Mg-based composites' dispersion more evenly.

2.6 Chapter Summary

As aforementioned in Chapter 2, there are several limitations to thoroughly researching Mg-based GNPs on microstructure, mechanical properties and corrosion resistance using the powder metallurgy technique. Therefore, this chapter describes the fundamentals of Mg and its alloys, heat treatment of Mg, Mg-based composites with several reinforcements, corrosion on Mg and its alloys, and powder metallurgy technique for Mg-based composites. The study chose Mg as a base metal, giving superior properties for industrial applications and Mg-based alloys. Many Mg-based alloys have been found to provide advantages and disadvantages in mechanical properties and corrosion resistance. Besides, heat treatment of Mg-based alloys is discussed further. Whilst the heat treatment gives superior properties, the corrosion resistance of Mg-based alloys is sacrificed. Mg-based composites are discussed in several types of reinforcement, including Mg-based GNPs, by previous researchers on the properties. Other than that, the corrosion types and the mechanisms of Mg and Mg-based alloys were discussed. Lastly, Mg and Mg-based alloys and Mg-based composites can be fabricated using powder metallurgy methods besides other fabrication methods with some advantages to the samples.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter defines the methodology used in conducting this research study. These involve the synthesis of Mg-based composites along with Mg alloy matrix without reinforcement and pure Mg, microstructural characterisation, mechanical hardness testing and corrosion test conducted. This chapter presents all the materials and equipment used in the research, sample preparation, experiment, and analysis. In this research study, four types of samples were fabricated and investigated the relationship of % of GNPs affecting the hardness and corrosion resistance; High Purity (HP) Mg and three Mg-based composites (Mg-6wt.%Zn-0.2%wt.%Mn) consisting of 0% GNPs, 0.5% GNPs and 1% of GNPs.

Table 3.1: Type of sample for Mg-based composite fabrication

Sample	Compositions of GNPs
High Purity Mg	HP Mg
Mg-6wt.%Zn-0.2%wt.%Mn	0%
Mg-6wt.%Zn-0.2%wt.%Mn 0.5%GNPs	0.5%
Mg-6wt.%Zn-0.2%wt.%Mn 1%GNPs	1%

3.2 Materials

The chemical materials used for the experimental process include magnesium (Mg) powder by Merck (>99% purity, density 1.74 g/cm³), zinc (Zn) powder by Merck (325 mesh size, ≥99% trace metals basis), manganese (Mn) powder by Merck, graphene nanoplatelets (GNPs) powder by Sigma Aldrich (5 µm particle size, surface area 120-150 m²/g, density 2.0-2.25 g/cm³), sodium chloride (NaCl) solution by Merck, magnesium hydroxide (Mg(OH)₂) powder by Sigma Aldrich. Meanwhile, the consumables were ethanol by R&M Chemicals, filter paper, silica gel, argon gas for tube furnace, silicon carbide abrasive paper (consisting of 1000, 1200, 1500, and 2000grit), alumina (Al₂O₃) suspension, nitric acid (HNO₃) solution by Merck, chromium (III) oxide (Cr₂O₃) solution by R&M Chemicals, silver nitrate (AgNO₃) powder by Sigma Aldrich, fishing line was used in this research study.

3.3 Equipment

The laboratory equipment that was used for this experimental research study includes in Table 3.2.

Table 3.2: Several equipment with models and their function

Equipment	Function
Mechanical agitator with a magnetic stirrer <i>Model: Fisher Scientific Isotemp Stirrer</i>	For stirring and mixing Mg, Mn and Zn powder
Bath sonicator <i>Model: UC-45D-PRO-220</i>	To sonicate GNPs before mixing with Mg, Mn and Zn mixtures.
Hydraulic compressing machine <i>Model: Shimadzu SSP-10A</i>	For compressing powdered samples at specific pressure and time
Drying oven	To dry powdered samples before powder compaction.
Tube furnace <i>Model: Berkeley Scientific BSG-1200X</i>	Used for sinter samples under argon gas environment with specific temperature and heat rate
Grinding and polishing machine <i>Model: Dace Technologies Nano-2000S</i>	To grind and polish samples for microstructure observation.
Vickers microhardness testing machine <i>Model: Shimadzu HVM-G31S</i>	To determine the hardness using Vickers Hardness Number.
3D optical microscope <i>Model: Hirox RH-2000</i>	To observe the surface of the samples, especially the grain size.
Scanning Electron Microscope (SEM) <i>Model: Hitachi TM3030</i>	To observe the structure of the samples further for the distribution of microstructure.
Energy Dispersive X-ray Spectroscopy (EDX) <i>Model: JEOL JSM-7400F</i>	To determine the composition of the elements in the sample.
Weighing scale <i>Model: AND FX-500i</i>	To weigh the Mg, Mn and Zn powder and the samples for weight loss calculation after the corrosion test.
Burette	To measure the reading of hydrogen evolution trapped inside during the immersion test.
Retort stand	To support the burette during the immersion test.
Beaker	Hold the powdered mixture during sample preparation and the NaCl solution during the immersion test.
Desiccator	To dry the samples and prevent any moisture inside the container.

3.4 Fabrication of Composites

The powder metallurgy technique is chosen as fabricating technique because of its advantages: simple fabrication process, availability of raw materials, and low chances of formation of intermetallic compounds mainly in the metal matrix (Manohar et al., 2021). To fabricate a sample of 1 wt% of GNPs reinforced Mg-6wt.%Zn-0.2%wt.%Mn composite (Mg-6Zn-0.2%wt.%Mn-1% GNPs), 0.03 g of GNPs powder is mixed with 50 ml of ethanol in a beaker. The mixture is then sonicated using a bath sonicator for about 1 hour. 2.784 g of Mg powder, 0.18 g of Zn powder and 0.006 g of Mn powder are added to 50 ml of ethanol in a beaker, and the mixture is stirred on a magnetic stirrer for 2 hours. GNP's and Mg mixture solutions are mixed and stirred with a magnetic stirrer for another 1 hour. The mixed and stirred solution is then filtered using filter paper. The filtered mixture is replaced into a petri dish and then left to dry in an oven at 50°C for a day to ensure complete drying of the powder mixture.

After drying was done, 1 g of the dried powder mixture was taken and then compressed using a hydraulic compressor with a pressure of 50 kN for 20 minutes to produce a cylindrical pallet. Then, the obtained cylindrical pallet is sintered using the tube furnace at a set temperature of 500°C heating rate of 10°C/min for 2 hours with an argon gas environment to enhance the bonding between Mg mixtures and GNPs. As a precautionary purpose, the sintered pallet will be taken after the furnace has been cooled down completely. This sample fabrication method is then repeated to fabricate Mg6%Zn0.2%%Mn-0.5% GNPs, Mg-6%Zn-0.2%Mn and high purity (HP) Mg, respectively.

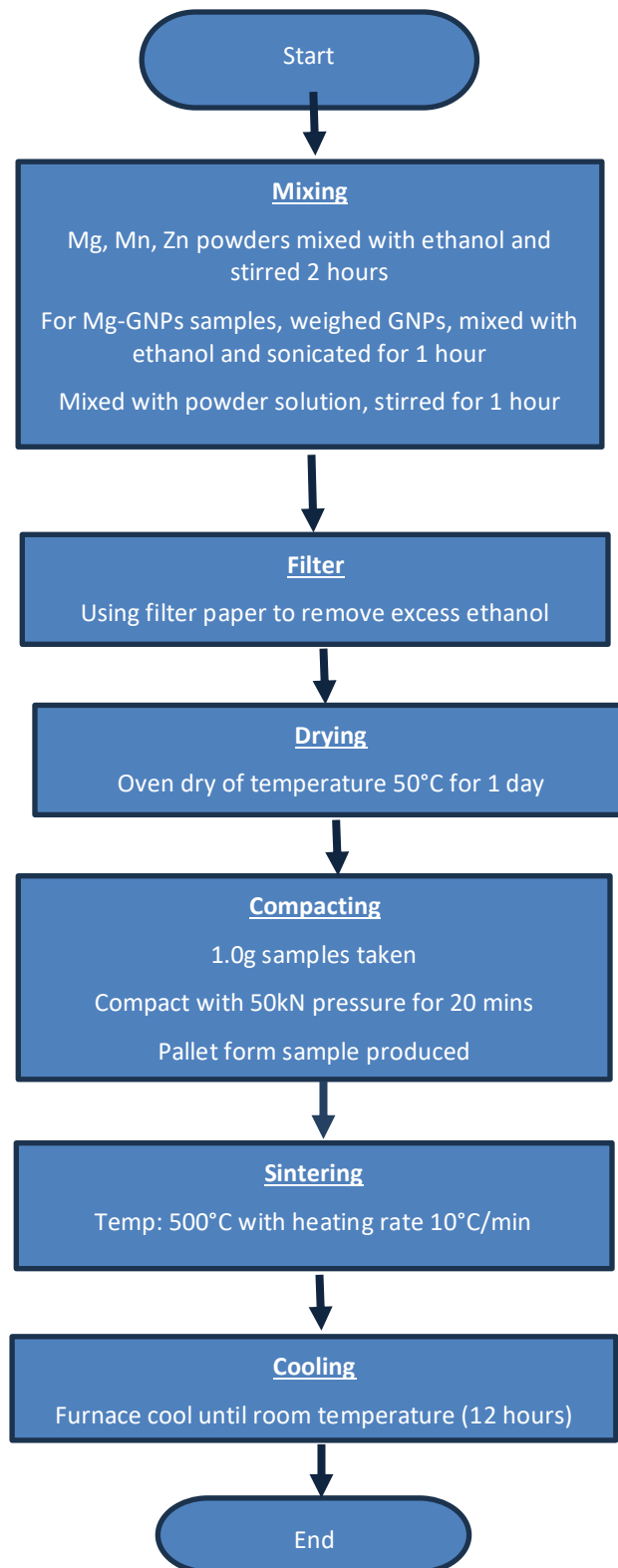


Figure 3.1: Steps on powder metallurgy process of Mg-based composites

3.5 Microstructural Characterisation

The microstructural characterisation is conducted to study the characteristic of the grain boundary, interface integrity between matrix and reinforcement, the presence of porosity and the grain size of the composite samples. Observing under a 3D optical microscope and a Scanning Electron Microscope (SEM) characterises this characterisation process. A 3D optical microscope was used as it gave higher resolution when the samples zoomed up to 1000× magnification, using high brightness light emitting diode (HB-LED) support and having a significantly high dynamic range (HDR) for the image produced. Mapping the composition of the composite was used with SEM. SEM provides topological and elemental information at magnifications from 10 to 100 000× with the virtual depth of field.

After the composite samples were fabricated, all the composites were ground with 1000 to 2000 grit of silicon carbide (SiC) abrasive paper and then polished with alumina suspension until mirrored-like surfaces were obtained. Then, the polished composites were washed with distilled water and allowed to dry using high-pressure air. After that, the samples are etched using a 3% nital (nitric acid mixed with ethanol) solution. The composite samples are only dipped into the etching solution for 2 seconds to prevent over-etching on the samples' surface.



Figure 3.2: Fabricated HP Mg sample before polish and HP Mg samples after polish

All the composite samples are characterised using a 3D optical microscope and SEM. The observed images and analysis of the microstructure of the composites are obtained.

3.6 Hardness Test

The hardness test is a measurement of the resistance of the material against plastic deformation. The hardness of a metal is measured by forcing an indenter onto the composite surface. A known load is applied to the indent slowly at 90° onto the tested sample's surface in hardness testing. It is based on the principle of indentation hardness, where a pyramidal diamond indenter is pressed into the surface of the material, and the size of the indentation is used to calculate the material's hardness. After indentation is done, the indenter is withdrawn from the surface. The hardness measurement is then calculated based on the cross-sectional area and depth of indentation depending on the type of hardness machine used.

In this study, the hardness test of the composite samples is done using the Vickers microhardness test. The Vickers hardness test (HV) was according to ISO 6507 standard for metallic materials. From this standard, depending on the test force and specimen material, the Vickers hardness value lies between 1 and 3,000 HV.

The fabricated samples are indented with a 5 kgf load for 10 seconds. The diamond shape indentation on the surface is then measured and recorded. The test is repeated with three readings recorded for each of the samples, and then the average of the readings is calculated as the hardness of the samples. The indentation results were then calculated based on the following formula:

$$\begin{aligned}
 \text{Vickers Hardness(HV)} &= \frac{\text{Applied Load}}{\text{Surface Area of Depression}} \\
 &= \frac{2P \sin\left(\frac{136^\circ}{2}\right)}{d^2} \\
 &= 1.854 \frac{P}{d^2}
 \end{aligned}$$

Where,

$$P = \text{Applied Load (kgf)}$$

$$d = \frac{d_1 + d_2}{2}$$

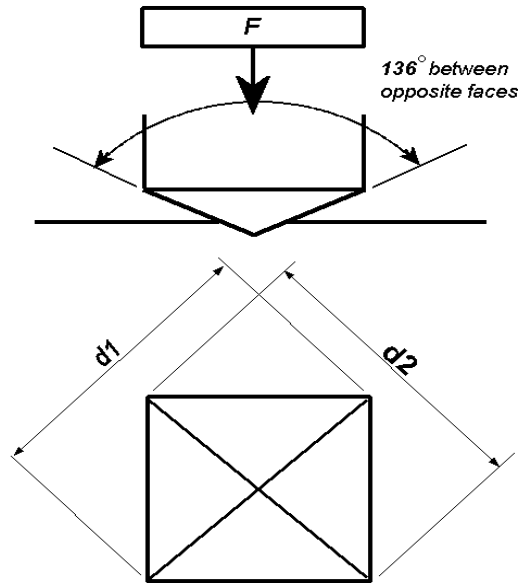


Figure 3.3: Vickers hardness test diagram

3.7 Corrosion Immersion Test

The corrosion immersion test is a common method for evaluating the corrosion resistance of a material. In this test, a sample of the material is immersed in a solution that is intended to simulate the environment to which the material will be exposed in service. The solution can be a simple saltwater solution, or it can be a more complex mixture of chemicals designed to replicate a specific industrial such as Hank's solution or natural environment.

The immersion test of the samples was carried out at room temperature according to ASTM-G31-72. All the fabricated Mg-based composite samples are immersed in a 3% aqueous NaCl solution saturated with Mg(OH)₂ at room temperature to determine their corrosion behaviour. The 3% NaCl was used as the corrosion medium to simulate the seawater solution. The 3 % NaCl solution is made by mixing NaCl powder with distilled

water in a beaker. To calculate the amount of sodium chloride needed, use the following formula:

Amount of NaCl (in grams) = 3% (desired concentration) × Volume of solution
(in mL or L)

To prepare 1 litre (1000 mL) of a 3% NaCl solution:

Amount of NaCl = 3% × 1000 mL = 0.03 × 1000 mL = 30 grams

The mixture was then stirred with a magnetic stirrer until the powder was dissolved. Before proceeding with the immersion in 3 % NaCl saturated with Mg(OH)₂, the samples are ground, polished and weighed. Each sample was then cleaned with distilled water, dried, and immersed in an agitating cleaning solution for 10 minutes. The cleaning solution is made from 200 g/L of chromium oxide (Cr₂O₃), 10 g/L of silver nitrate (AgNO₃), and 100 ml/L of distilled water to clean the samples from surface contaminations. These samples are hung inside a filter funnel and supported with a fishing line during the test, as shown in Figure 3.2, during immersion for five days.

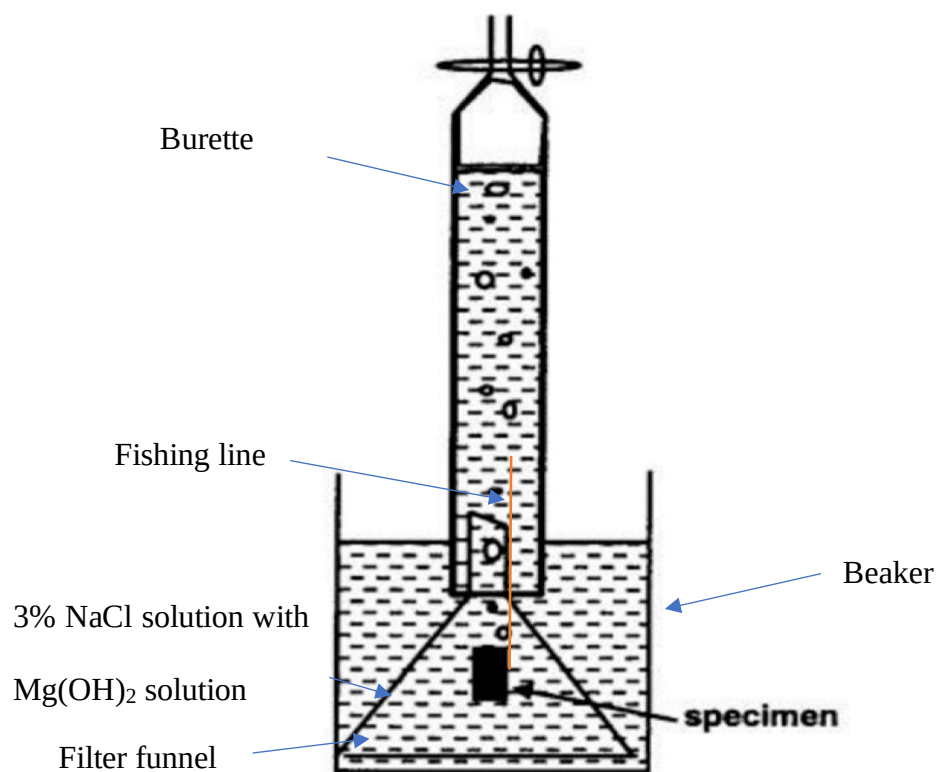


Figure 3.4: Setup for the corrosion immersion test

The corrosion behaviour is identified by collecting the evolved hydrogen gas corroded samples. The hydrogen gas evolved was collected in a filter funnel, and the reading was taken using a burette. The hydrogen gas with bubbles observed and collected

by the funnel went into the burette and gradually displaced the test solution. The burette reading of the NaCl solution during the immersion test is recorded every 3 hours.

After the immersion test, each sample was removed from the setup, washed with distilled water, dried with high-pressure air, and dried in a desiccator for a day. After that, the samples are rinsed in the chromic acid cleaning solution (200 g/L chromium (VI) oxide, CrO_3 + 10 g/L silver nitrate, AgNO_3) to remove the corrosion products on the samples. The samples were cleaned again using distilled water and pure ethanol, air-dried in high-pressure air and a desiccator for a day and then weighed. The test was then repeated with another batch of corrosion immersion tests twice, with the three readings of hydrogen evolution for each of the samples recorded.

3.8 Corroded Surface Evaluation

Corroded surface evaluation is conducted to analyse the samples' surface before and after removing corrosion products. This evaluation is analysed using a 3D optical microscope and the scanning electron microscope (SEM) on the surface morphology. Energy Dispersive X-ray spectroscopy (EDX) is used to determine and analyse the corrosion product compositions of the samples.

3.9 Chapter Summary

This study first experimented by fabricating Mg-based GNPs samples using the powder metallurgy mixing, compacting, and sintering technique at specific temperatures and times. After the pieces were fabricated, microstructural observations were done to gain more structure of grains of the composites fabricated and the chemical compositions.

The next step was mechanical testing on the microhardness Vickers test to analyse the hardness of the composite samples. Lastly, the corrosion immersion test was conducted, and its results were validated by hydrogen evaluation, weight loss and observation of the corroded surface morphology.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

In this chapter, the study's results are presented and discussed with reference to the aim of the study, which was to determine the microstructure of the fabricated GNP-reinforced Mg composite. The two other objectives are to determine the hardness properties of GNPs reinforced Mg composites and the second to compare the corrosion rate of GNPs reinforced Mg composites using hydrogen evolution and weight loss measurement. These aspects were described in the previous chapter, which presented the methodology used in the study.

4.2 Microstructure Characterisation

Figure 4.1 show the microstructure of HP Mg, Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs, and Mg0.2%Mn6.0%Zn-1% GNPs composite obtained from 3D optical microscope after sample preparation process including grinding, polishing, and etching, were done respectively.

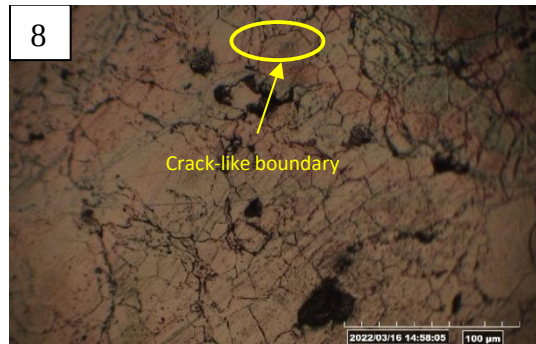
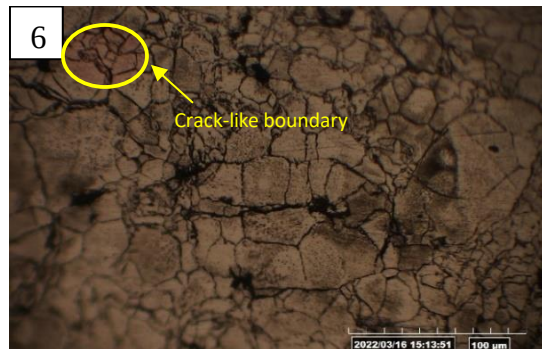
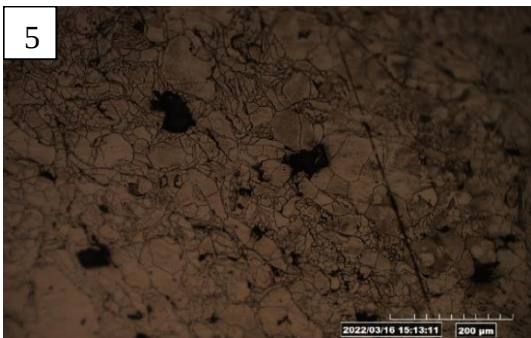
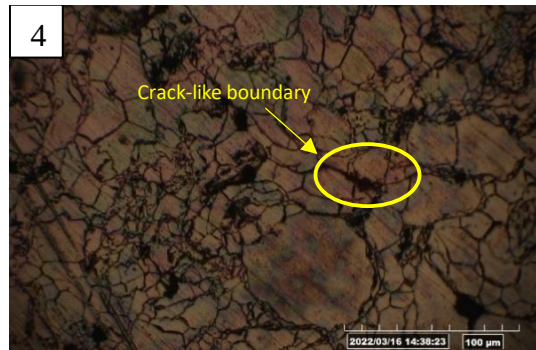
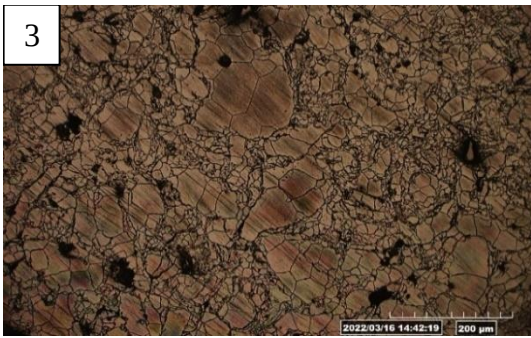
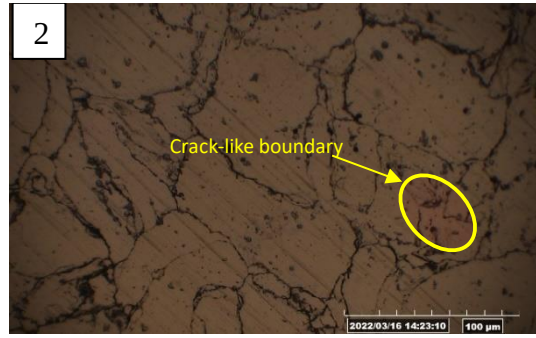
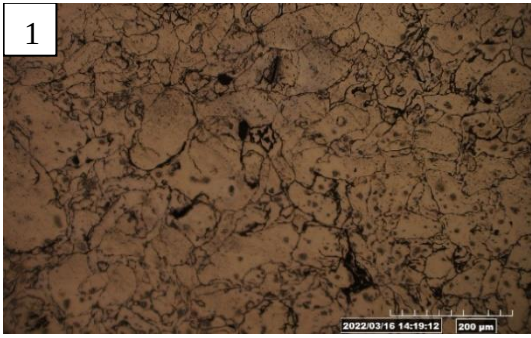


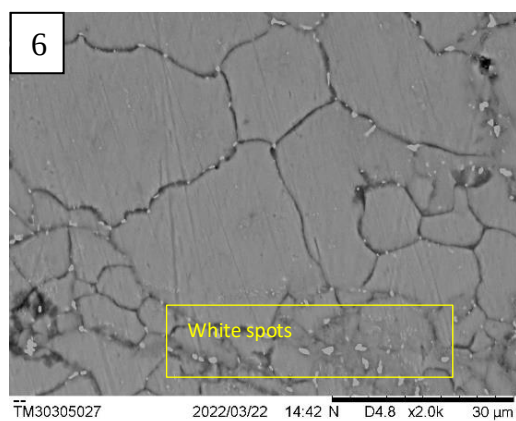
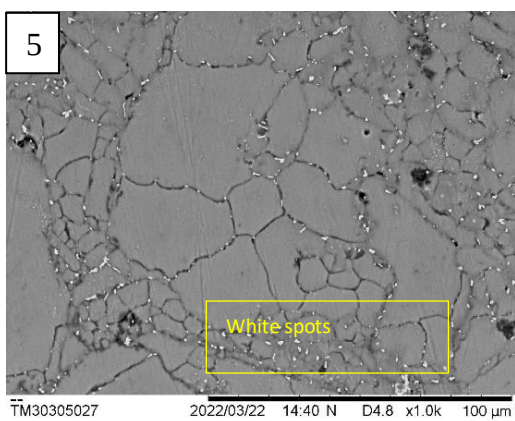
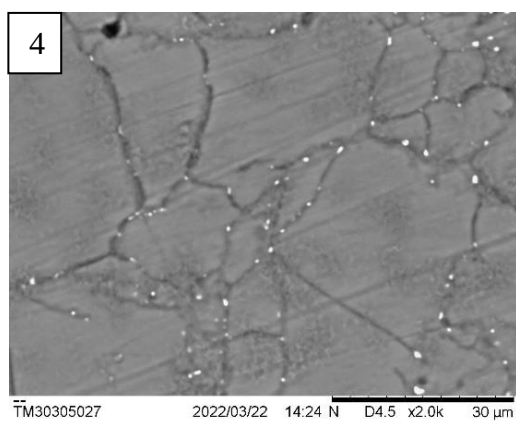
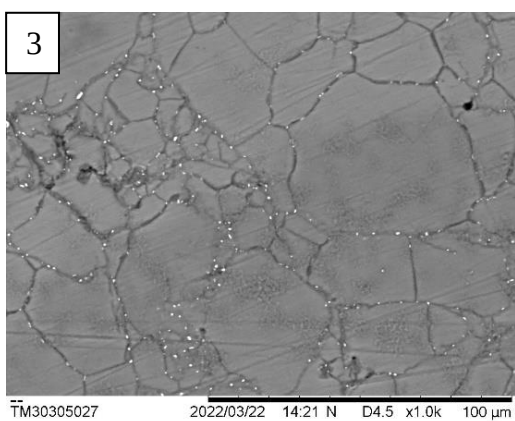
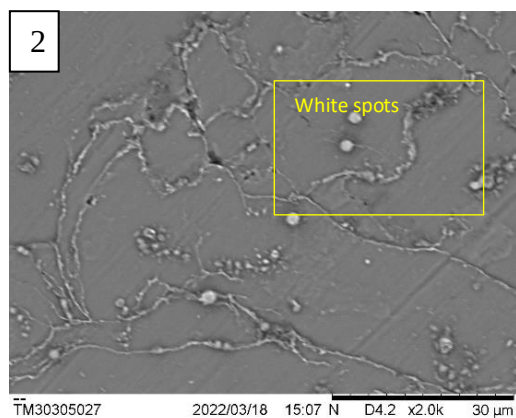
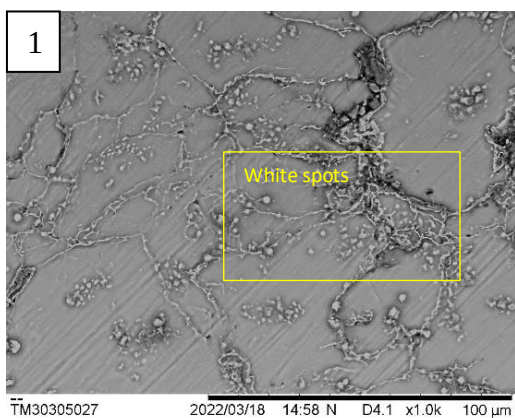
Figure 4.1: Microstructure of 1) HP Mg, 350× 2) HP Mg, 1000× 3) Mg6%Zn0.2%Mn, 350× 4) Mg6%Zn0.2%Mn, 1000× 5) Mg0.2%Mn6.0%Zn-0.5% GNPs, 350× 6) Mg0.2%Mn6.0%Zn-0.5% GNPs, 1000× 7) Mg0.2%Mn6.0%Zn-1% GNPs, 350× 8) Mg0.2%Mn6.0%Zn-1% GNPs, 1000× observed under a 3D optical microscope.

Based on the 3D optical microscope images in Figure 4.1, all the samples' grains are equal and irregular. It was observed that HP Mg has the largest grain size, followed by Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs. Adding Zn to the composite caused the grain size to coarsen (Budinski & Budinski, 1999). Besides Zn, the addition of Mn helps on the reducing the grain size. After adding 0.2 wt% Mn, the grain size is slightly reduced to about 25-35% compared with the sample without Mn. It is known that when the Mn content exceeds 1.3 wt%, the effect of Mn on the grain refinement is gradually weakened (T. Zhao et al., 2019). GNPs in the composite have successfully suppressed the grain size growth, resulting in more refined grains than Mg0.2%Mn6.0%Zn (Rashad, Pan, & Asif, 2016). Since grain boundaries contribute primarily to the diffusion channels of Mg0.2%Mn6.0%Zn alloy matrix and GNPs in the formation of nucleation sites, they also helped reduce the grain size.

The heterogeneous nucleation mode promoted the formation of a tight mechanical interfacial bonding between the GNPs and Mg0.2%Mn6.0%Zn matrix. With the increase in GNPs content, more heterogeneous nucleation substrates are provided, which leads to the formation of more crystal nuclei of Mg on the GNPs. Hence, the increase in nucleation rate can effectively refine the Mg crystal grains at a given Mg0.2%Mn6.0%Zn matrix (Sun et al., 2020). This implies that GNPs can act as nucleation sites and grain refiners in magnesium composites.

It is observed that the Mg0.2%Mn6.0%Zn-0.5% GNPs, and Mg0.2%Mn6.0%Zn-1% GNPs, produced almost equal the size of the grains with the help of GNPs to reinforce the Mg alloy matrix. Many crack-like boundaries were observed in all samples, highlighted in Figures 4.1 2), 4), 6), and 8), which differ from the grain boundaries. These crack-like boundaries were formed after the powder compaction that was low binding between the Mg0.2%Mn6.0%Zn powders when compaction was done. This may be developed due to the duration and pressure applied lowered to produce compact samples during the fabrication of the pallet sample. To overcome the crack-like boundaries, there is a need for more uniform pressure to be applied during the compaction before the sintering of samples is done.

On the other hand, further observation of the samples was also conducted using SEM based on Figure 4.2.



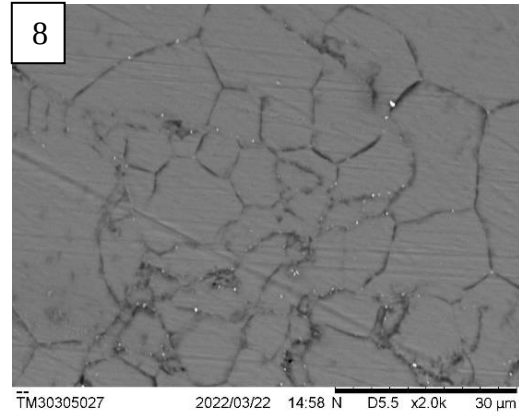
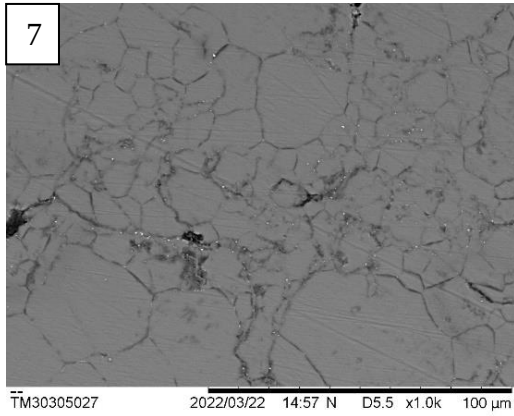


Figure 4.2: Microstructure of 1) HP Mg, 1000× 2) HP Mg, 2000× 3) Mg-6%Zn-0.2%Mn, 1000× 4) Mg-6%Zn-0.2%Mn, 2000× 5) Mg0.2%Mn6.0%Zn-0.5% GNPs, 2000× 6) Mg0.2%Mn6.0%Zn-0.5% GNPs, 2000× 7) Mg0.2%Mn6.0%Zn-1% GNPs, 1000× 8) Mg0.2%Mn6.0%Zn-1% GNPs, 2000× observed under SEM

Scanning Electron Microscope (SEM) was also used to investigate the morphology of magnesium graphene-reinforced composites. Figure 4.2(1-8) shows the micrographs of the samples, magnified at 1000× and 2000×. The electron power of the image produced was set at 15kV. Meanwhile, Figure 4.3 shows the elements present identified from the EDX analysis at spotted areas.

Table 4.1: Characterisation of elements on samples using EDX

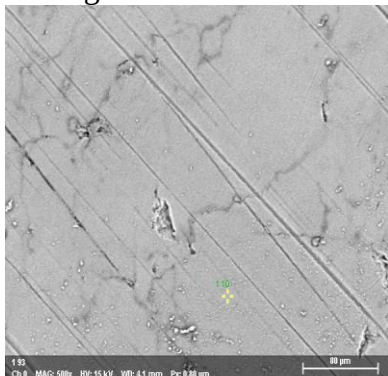
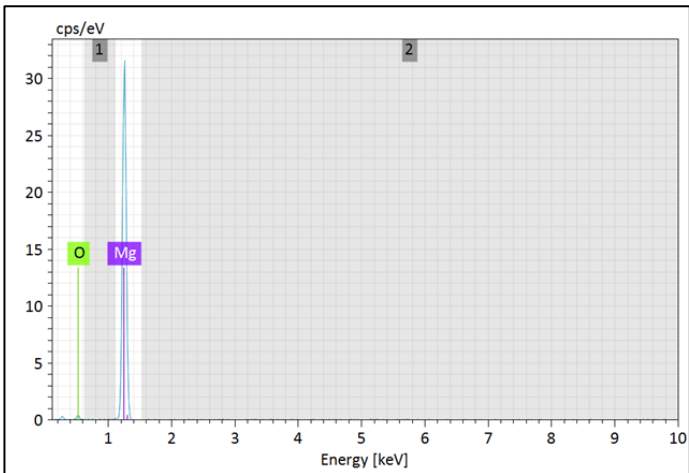
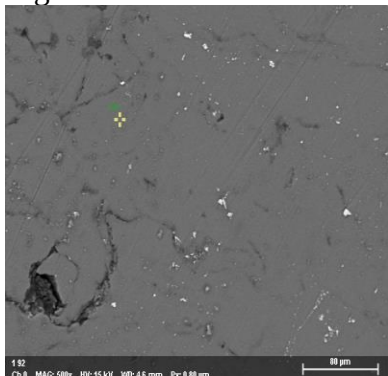
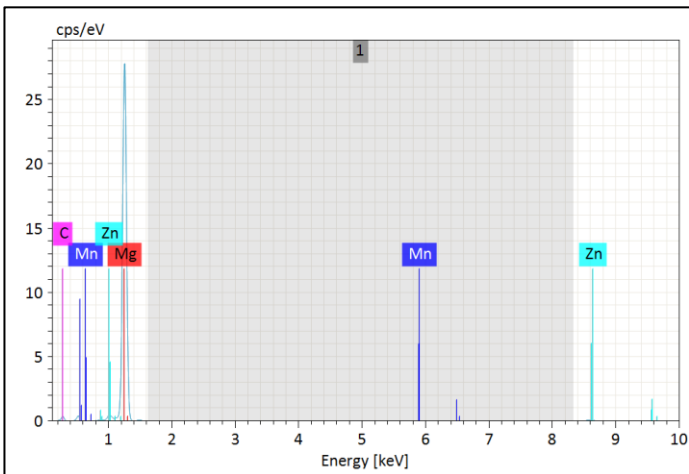
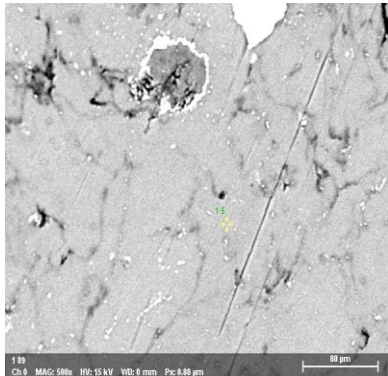
Samples	Compositions and Graph																								
HP Mg 	<table><tr><th>Element</th><th>At. No.</th><th>Netto</th><th>Atom %</th></tr><tr><td>O</td><td>8</td><td>51</td><td>3.72</td></tr><tr><td>Mg</td><td>12</td><td>9936</td><td>96.28</td></tr><tr><td colspan="2"></td><td>Sum</td><td>100</td></tr></table> 	Element	At. No.	Netto	Atom %	O	8	51	3.72	Mg	12	9936	96.28			Sum	100								
Element	At. No.	Netto	Atom %																						
O	8	51	3.72																						
Mg	12	9936	96.28																						
		Sum	100																						
Mg-6%Zn-0.2%Mn 	<table><tr><th>Element</th><th>At. No.</th><th>Netto</th><th>Atom %</th></tr><tr><td>C</td><td>6</td><td>35</td><td>0.51</td></tr><tr><td>Mg</td><td>12</td><td>24870</td><td>93.07</td></tr><tr><td>Mn</td><td>25</td><td>126</td><td>0.18</td></tr><tr><td>Zn</td><td>30</td><td>1802</td><td>6.24</td></tr><tr><td colspan="2"></td><td>Sum</td><td>100</td></tr></table> 	Element	At. No.	Netto	Atom %	C	6	35	0.51	Mg	12	24870	93.07	Mn	25	126	0.18	Zn	30	1802	6.24			Sum	100
Element	At. No.	Netto	Atom %																						
C	6	35	0.51																						
Mg	12	24870	93.07																						
Mn	25	126	0.18																						
Zn	30	1802	6.24																						
		Sum	100																						

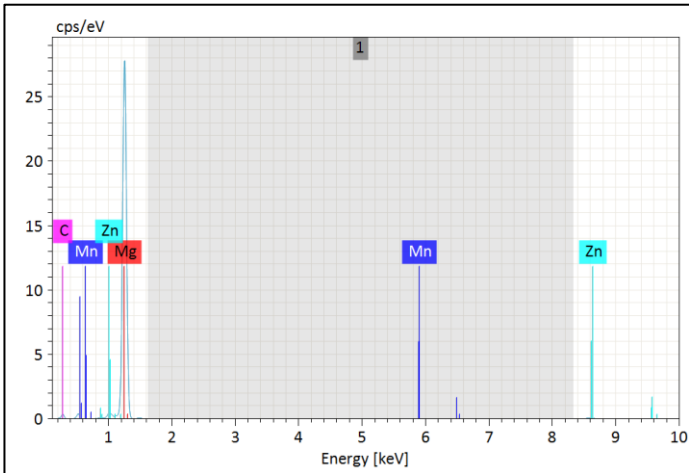
Table 4.1 continued

Mg-6%Zn-0.2%Mn-0.5%GNPs

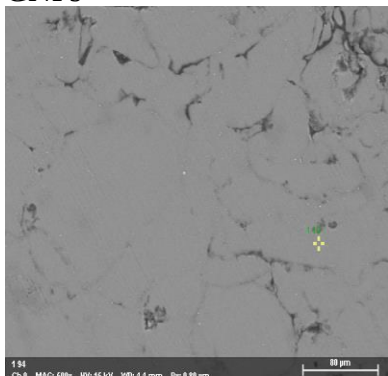


189
Ch 0 MAG: 500x E0: 15 kV WD: 8 mm Pz: 0.88 μm
Name Date Time HV Mag
189 11/04/2022 09:20:44 15.0 keV 500x

Element	At. No.	Netto	Atom %
C	6	35	0.51
Mg	12	24870	93.07
Mn	25	126	0.18
Zn	30	1802	6.24
		Sum	100

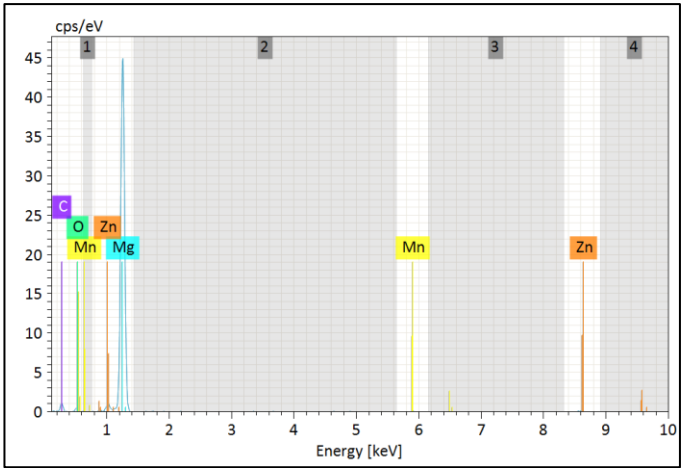


Mg0.2%Mn6.0%Zn-1%GNPs



194
Ch 0 MAG: 500x E0: 15 kV WD: 4.4 mm Pz: 0.89 μm
Name Date Time HV WD Mag
194 11/04/2022 10:16:43 15.0 keV 500x 4.4 mm

Element	At. No.	Netto	Atom %
O	8	297	3.01
Mg	12	79941	89.74
Mn	25	105	0.21
Zn	30	1369	6.05
C	6	24	0.99
		Sum	100



The grain boundaries are observed clearly on the surface for pure magnesium and composites, as shown in Figure 4.2. The micropores, highlighted in the white spots, can be seen on the surface due to oxidation during the sintering process. This is proved by EDX images where the O peak is significantly detected on the specimen. The other reason may be attributed to the poor dispersion of two-dimensional GNPs reinforcement in the matrix (L. Wu et al., 2018). Furthermore, the presence of graphene is obvious in the grain boundaries. Cluster formation can be seen in some places, and reinforcements exhibit binding characteristics. Figure 4.2 (5-8) shows the micrographs of Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs. There is an agglomeration on the grain boundaries since graphene has a high surface area and van der Waals bonding between carbon atoms. However intermetallic phase can only be seen as carbides for some samples (Turan et al., 2017).

An oxygen element was found in all samples. This is because an oxide layer which is the $Mg(OH)_2$, a protective layer, was formed on the surface of all models. In Mg0.2%Mn6.0%Zn and Mg0.2%Mn6.0%Zn-1% GNPs, almost 6 % Zn was detected. This is to confirm that the samples contained 6 % Zn added during the fabrication process. The Zn element from the EDX result was below 6 %, possibly due to some Zn; the powder remained in the mould. However, the Mn element was not detected during the EDX analysis, even though 0.2 % was added during fabrication. The low amount of Mg powder might have remained in the mould, or the EDX could not identify it. Carbon element was detected in Mg0.2%Mn6.0%Zn-0.5% GNPs, along with Mg0.2%Mn6.0%Zn-1% GNPs. Carbon presence on this validates that the GNPs were added during the composite fabrication. Carbon atoms also might be homogenously distributed in the matrix for composites because the carbon trends are generally constant at the point. In Table 4.1, the scratch lines present

on the images were because of the grinding effect of the samples. To overcome the effect, the grinding process should be added more SiC paper with finer grits and always sand in the direction of the metal's grain to avoid creating new scratches.

4.3 Vickers Hardness Test

The samples were tested using 5kgf of load for 10 seconds in this test. The d_1 and d_2 values of d_1 and d_2 were recorded, and the d_1 and d_2 were the diagonal lengths of the indentation left on the material's surface after load removal, as shown in Figure 3.1. The Vickers hardness for HP Mg, Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs samples were recorded, and the average Vickers Hardness Number (VHN), as well as standard deviation, were calculated as shown in Table 4.2.

Table 4.2: Hardness results on samples

Type of sample	VHN 1	VHN 2	VHN 3	Average VHN	Standard Deviation
HP Mg	34.40	34.50	34.25	34.38	0.1027
Mg0.2%Mn6.0%Zn	44.40	44.35	44.45	44.40	0.0408
Mg0.2%Mn6.0%Zn-0.5%GNPs	45.55	45.45	45.50	45.50	0.0408
Mg0.2%Mn6.0%Zn-1%GNPs	48.40	48.30	48.50	48.40	0.0816

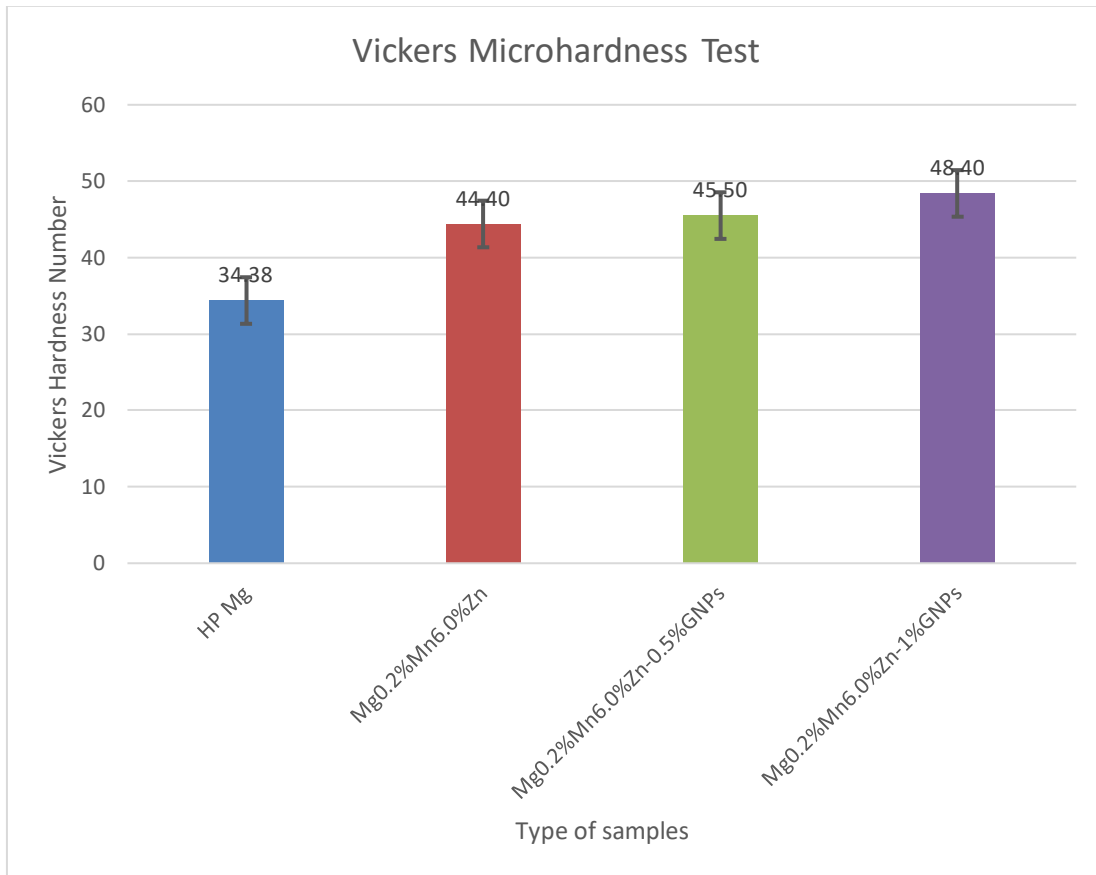


Figure 4.3: Bar chart of average Vickers hardness of the samples

Three hardness measurements were conducted for each composite type during the test, and the average values of each composite were presented in Table 4.2. From the result obtained, Mg0.2%Mn6.0%Zn-1% GNP showed the highest value of VHN, giving it the highest hardness sample of the other samples. For Mg0.2%Mn6.0%Zn-0.5% GNP, it offers higher hardness than Mg0.2%Mn6.0%Zn, and the least hardness is HP Mg. By comparison, Mg0.2%Mn6.0%Zn-1% GNP gives an increase of hardness by 34.15%, Mg0.2%Mn6.0%Zn-0.5% GNP hardness increased by 27.84%, and Mg0.2%Mn6.0%Zn gives rise of hardness by 25.64%, all compared with HP Mg.

Apart from that, the Mg0.2%Mn6.0%Zn-1% GNP exhibited a greater degree of hardness than Mg0.2%Mn6.0%Zn by 8.7%. This is because the inclusion of GNP has

marginally enhanced its mechanical properties, particularly its hardness. From the bar chart in Figure 4.3, the alloying of Mg with Mn and Zn can give better mechanical hardness properties than the HP Mg by 25.6%.

Proven by the results and compared to previous research, adding graphene nanoplatelets into the magnesium matrix increases the hardness. The reason is that the GNPs restrict the dislocation movement of the Mg matrix, and the constituents present as Zn and Mn as alloying elements. Zn and Mn improve the mechanical properties not only by reducing the grain size of Mg alloy but also introduced dislocations in the microstructure (Shuai et al., 2018; Wang et al., 2019). The presence of a more intricate reinforcement significantly enhances the hardness of the fabricated composites. The interfacial area between magnesium and reinforcement is larger due to the large specific surface area of graphene nanoplatelets. Therefore, the hardness is enhanced by the strong bonding between the matrix and the reinforcement. In this context, it was assumed that the diffusion of carbon atoms leads to high sintering temperatures, which would result in good bonding (Saber et al., 2020). Besides that, Liu et al. (2017) discussed that the mechanical properties of alloys and composites could change due to the high-temperature diffusion rate during the sintering process. Therefore, the reinforcement particles in the composite's matrix can bring the particles closer together through thermal diffusion on compressed powdered samples (C. Liu et al., 2017).

4.4 Corrosion Immersion Test and Corrosion Rate Measurements

The hydrogen gas H₂ evolution collected during the five days of immersion was tabulated in graphs as shown in Figure 4.4.

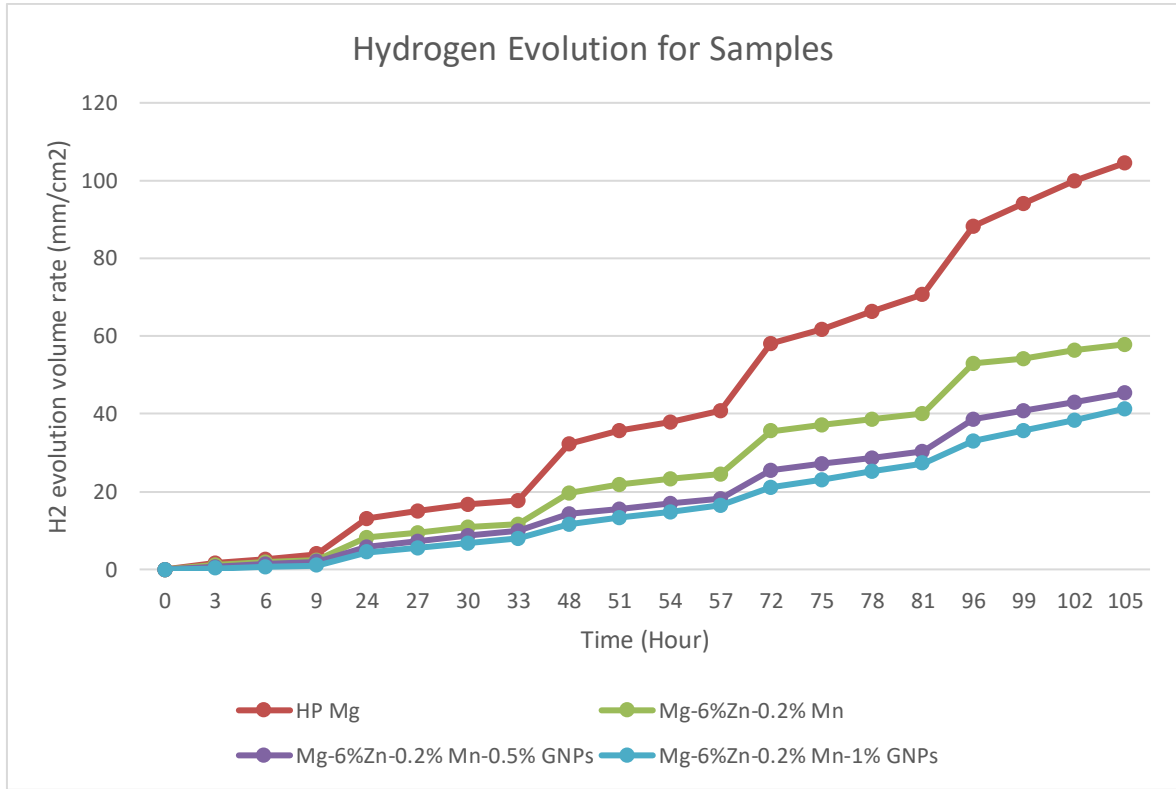


Figure 4.4: Graphs of hydrogen evolution volume against time for all samples

The hydrogen evolution volume (ml/cm²/h) from the graphs in Figure 4.4 was obtained from the slope of each graph, and these data, the average hydrogen evaluation rate in days, V_H (ml/cm²/day), were determined. Annual corrosion rates using average corrosion rate P_H (mm/y) were determined by converting V_H to P_H by using the conversion equation (M. C. Zhao et al., 2008b):

$$P_H = 2.279V_H$$

On the other hand, the average corrosion rate by weight loss P_w (mm/y) was calculated using the equation provided by (M. C. Zhao et al., 2008a):

$$P_w = 2.1\Delta W, \text{ where } \Delta W = \frac{W_b - W_a}{At_t}$$

Where:

W_b = weight of the sample before immersion test (g)

W_a = weight of the sample after removal of corrosion products after immersion test (g)

A = surface area of the sample (cm²)

t_t = immersion duration (days)

Table 4.3: Corrosion rate obtained using hydrogen evolution and weight loss after 5 days

Type of sample	Incubation period (h)	PH (mm/y)	PW (mm/y)
HP Mg	0	43.6362	99.4468
Mg6%Zn0.2%Mn	24	24.1706	55.0848
Mg6%Zn0.2%Mn-0.5%GNPs	24	18.9212	43.1215
Mg6%Zn0.2%Mn-1%GNPs	24	17.2642	39.3450

Based on the graphs in Figure 4.4, the hydrogen evolution rate for each of the different samples is shown with the presence of the incubation period. It is observed that the incubation period was determined in the samples for 24 hours in time except for HP Mg samples. The incubation period is known as when the hydrogen evolution rate becomes low, causing the $Mg(OH)_2$ protective layer. This can be explained by the partially stable protective layer $Mg(OH)_2$ helping as the corrosion protection film. The corrosion rate increases further when all the $Mg(OH)_2$ protective layer has been dissolved. These higher

hydrogen evolution rates on both samples were caused by the disintegration of the thin surface film of $\text{Mg}(\text{OH})_2$ and then replaced with a new film produced when immersed in a 3% NaCl solution with saturated $\text{Mg}(\text{OH})_2$. These samples show that the hydrogen evolution volume rate increases as the immersion time increases. Apart from that, it was observed that there was no incubation period in the HP Mg samples due to the experimental setup. While taking the readings, it was discovered that some of the filter funnels had been dropped and not properly attached to the burette on HP Mg. Therefore, this caused some errors where carbon dioxide from the external environment was collected instead of hydrogen gas in the burette.

In this Figure 4.4, the corrosion rate on HP Mg shows the highest, followed by $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}$, $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}-0.5\%$ GNPs and finally $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}-1\%$ GNPs. This also has been proven by referring to the calculations on the P_H and P_W from Table 4.2. The addition of Zn in the Mg-based composite proved that Zn could help the reduction of the corrosion rate by lowering the hydrogen evolution volume compared with HP Mg samples, as discussed by Mordike and Ebert (2001). The corrosion rate can be improved more on $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}$ by adding 0.5% and increasing to 1% of GNPs as the Mg composites compared with the $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}$ sample. GNPs addition has been revealed to prove that the GNPs will act as a corrosion barrier in Mg composites and hence lower the corrosion rate of the Mg composites (L. Y. Chen et al., 2012).

4.5 Surface Evaluation After Immersion Test

After five days of immersion of samples in the 3% NaCl solution, the morphology of the surface of the samples was investigated by optical microscope, SEM and EDX.

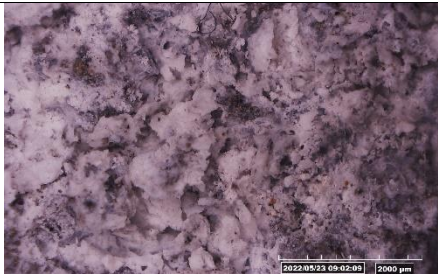
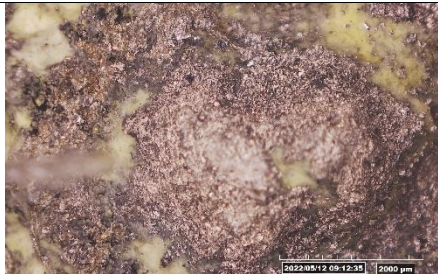
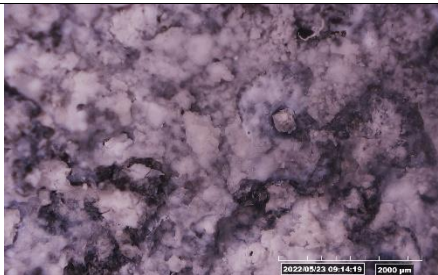
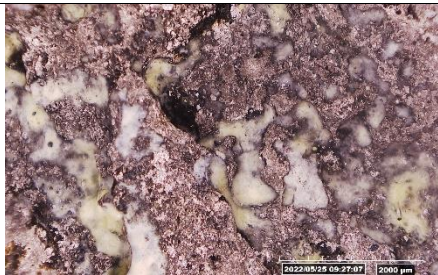
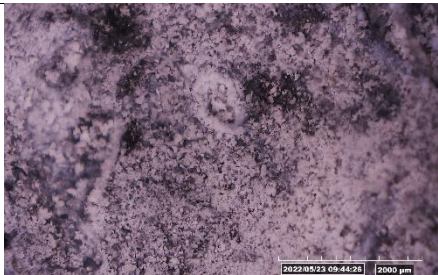
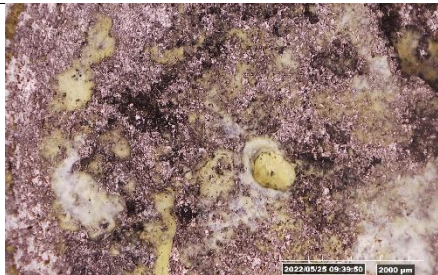
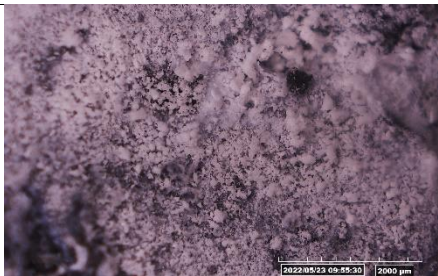
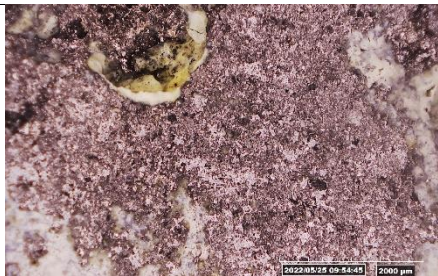
Sample	Before product removal	After product removal
HP Mg		
Mg0.2%Mn6.0% Zn		
Mg0.2%Mn6.0% Zn-0.5% GNPs		
Mg0.2%Mn6.0% Zn-1% GNPs		

Figure 4.5: The surface appearance of the corroded surface before and after product removal using an optical microscope with 50× magnification

Figure 4.5 shows the surface appearances of all samples taken before and after corrosion product removal observed under a 3D optical microscope. The corrosion product, white precipitates, was clearly observed on all samples known as magnesium chloride

(MgCl). MgCl presence is further revealed from the EDX results in Table 4.4. Besides, there were cracks seen in the corrosion products, which were attributed to the shrinkage of the corrosion products during the drying process at room temperature.

The image of HP Mg after the corrosion removal product was seen as unclear due to the uneven surface after the immersion test and the abnormal shape gained after the immersion test. The Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs, and Mg0.2%Mn6.0%Zn-1% GNPs samples can be seen clearly. Most of the specimen surface shows signs of corrosion, while some areas have experienced deep heterogenous corrosion. The following ranking indicates the corrosion for all samples from the observations of the corroded surfaces: HP Mg > Mg0.2%Mn6.0%Zn > Mg0.2%Mn6.0%Zn-0.5% GNPs > Mg0.2%Mn6.0%Zn-1% GNPs which is consistent with the ranking of corrosion rates evaluated from the hydrogen evolution and weight loss. This contradicts the previous research as the hydrogen evolution data of Mg0.2%Mn6.0%Zn reinforced 1 % GNPs was lower might be because some evolved hydrogen trapped under the surface was more severe. Other than that, it is because some hydrogen dissolves in the Mg metal rather than evolving as hydrogen gas (Nogita et al., 2009).

Surface morphology for the samples after immersion before corrosion product removal using SEM at 500× magnification and 15kV electron power are shown in Figure 4.6. In each case, there were corrosion products on the specimen surface. The cracks in the corrosion products on HP Mg and Mg0.2%Mn6.0%Zn are attributed to the shrinkage of the corrosion products during drying. EDX also characterised the corrosion products' elemental concentrations. Some adjustments to parameters, such as the magnification and electron

power, had to be made to prevent any mishaps during the characterisation process, such as the charging effect.

From the SEM morphology in Figure 4.6, it is known that the corrosion products formed on the samples indicated that all the samples had undergone corrosion when immersed in 3% NaCl with saturated $\text{Mg}(\text{OH})_2$. This figure shows that all the samples except $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}-0.5\%$ GNPs had a uniform surface attack of the corroded surface, which is suspected of a uniform corrosion phenomenon. Meanwhile, $\text{Mg}0.2\%\text{Mn}6.0\%\text{Zn}-0.5\%$ GNPs has some pit area highlighted at the bottom right besides uniformly corroded; hence, pitting corrosion happens at the samples. Pitting corrosion attacked some localised weaker surfaces as the surface layer of $\text{Mg}(\text{OH})_2$ was depleted during the immersion test. The elements contained revealed from the EDX analysis are shown in Table 4.4.

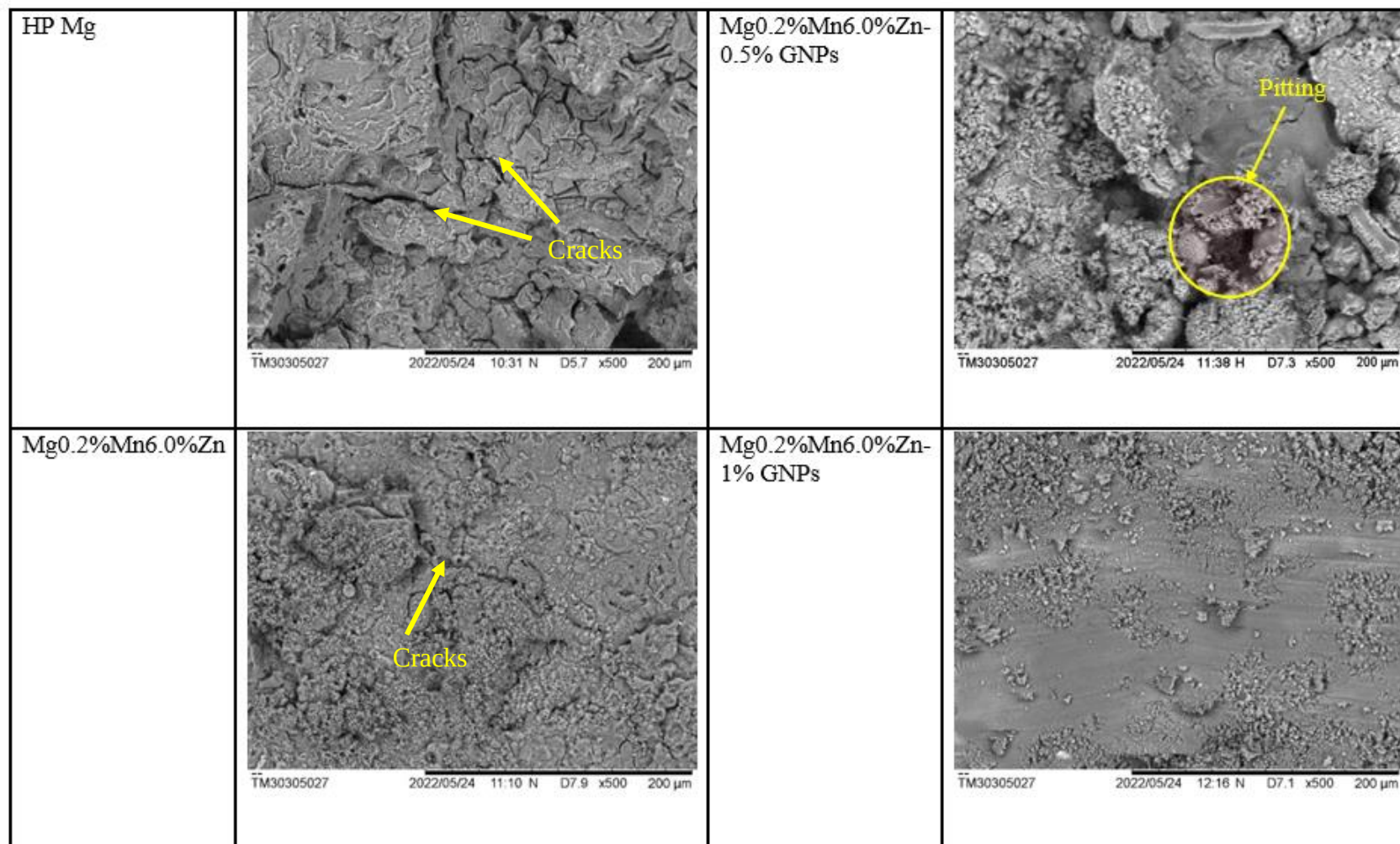


Figure 4.6: The surface appearance of the corroded surface after immersion before corrosion product removal using SEM

Table 4.4: Elements characterisation of samples after immersion test before corrosion product removal by using EDX

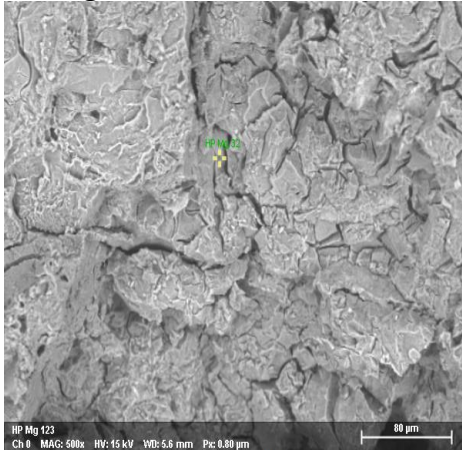
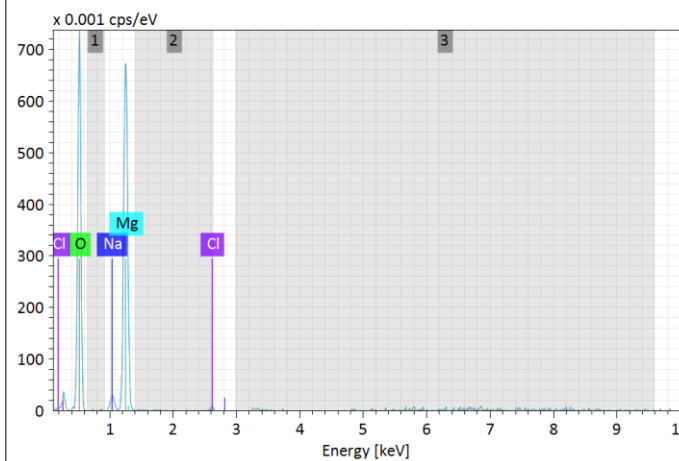
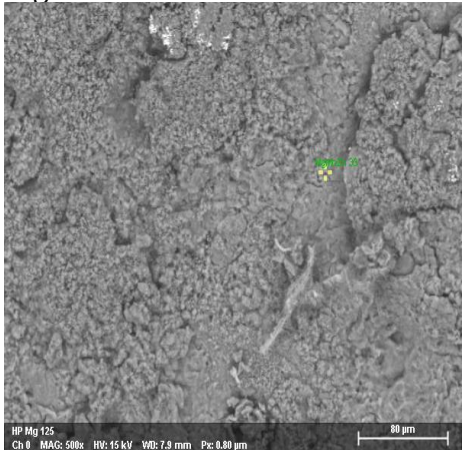
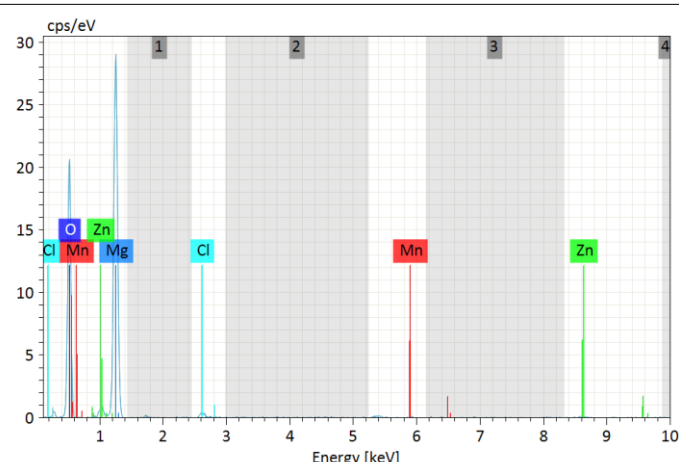
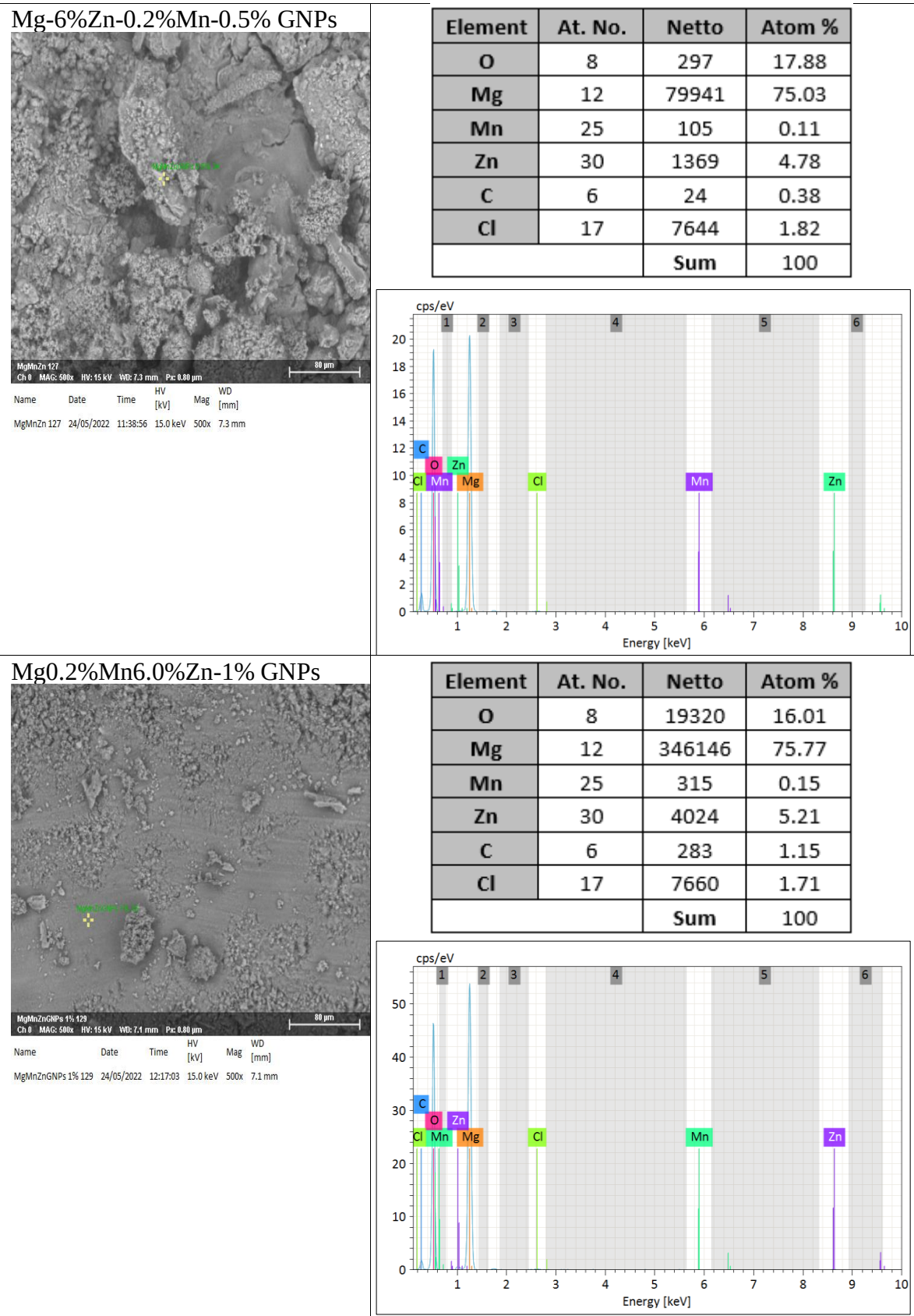
Samples	EDX Graph																																								
HP Mg <table><tr><th>Name</th><th>Date</th><th>Time</th><th>HV [kV]</th><th>Mag</th><th>WD [mm]</th></tr><tr><td>HP Mg 123</td><td>24/05/2022</td><td>10:40:59</td><td>15.0 keV</td><td>500x</td><td>5.6 mm</td></tr></table>	Name	Date	Time	HV [kV]	Mag	WD [mm]	HP Mg 123	24/05/2022	10:40:59	15.0 keV	500x	5.6 mm	<table><tr><th>Element</th><th>At. No.</th><th>Netto</th><th>Atom %</th></tr><tr><td>O</td><td>8</td><td>3055</td><td>26.26</td></tr><tr><td>Mg</td><td>12</td><td>26384</td><td>71.29</td></tr><tr><td>Na</td><td>11</td><td>350</td><td>1.12</td></tr><tr><td>Cl</td><td>17</td><td>533</td><td>1.33</td></tr><tr><td colspan="2"></td><td>Sum</td><td>100</td></tr></table> 	Element	At. No.	Netto	Atom %	O	8	3055	26.26	Mg	12	26384	71.29	Na	11	350	1.12	Cl	17	533	1.33			Sum	100				
Name	Date	Time	HV [kV]	Mag	WD [mm]																																				
HP Mg 123	24/05/2022	10:40:59	15.0 keV	500x	5.6 mm																																				
Element	At. No.	Netto	Atom %																																						
O	8	3055	26.26																																						
Mg	12	26384	71.29																																						
Na	11	350	1.12																																						
Cl	17	533	1.33																																						
		Sum	100																																						
Mg-6%Zn-0.2%Mn <table><tr><th>Name</th><th>Date</th><th>Time</th><th>HV [kV]</th><th>Mag</th><th>WD [mm]</th></tr><tr><td>HP Mg 125</td><td>24/05/2022</td><td>11:01:26</td><td>15.0 keV</td><td>500x</td><td>7.9 mm</td></tr></table>	Name	Date	Time	HV [kV]	Mag	WD [mm]	HP Mg 125	24/05/2022	11:01:26	15.0 keV	500x	7.9 mm	<table><tr><th>Element</th><th>At. No.</th><th>Netto</th><th>Atom %</th></tr><tr><td>O</td><td>8</td><td>4396</td><td>23.05</td></tr><tr><td>Mg</td><td>12</td><td>43333</td><td>71.34</td></tr><tr><td>Mn</td><td>25</td><td>57</td><td>0.08</td></tr><tr><td>Zn</td><td>30</td><td>945</td><td>3.75</td></tr><tr><td>Cl</td><td>17</td><td>189</td><td>1.78</td></tr><tr><td colspan="2"></td><td>Sum</td><td>100</td></tr></table> 	Element	At. No.	Netto	Atom %	O	8	4396	23.05	Mg	12	43333	71.34	Mn	25	57	0.08	Zn	30	945	3.75	Cl	17	189	1.78			Sum	100
Name	Date	Time	HV [kV]	Mag	WD [mm]																																				
HP Mg 125	24/05/2022	11:01:26	15.0 keV	500x	7.9 mm																																				
Element	At. No.	Netto	Atom %																																						
O	8	4396	23.05																																						
Mg	12	43333	71.34																																						
Mn	25	57	0.08																																						
Zn	30	945	3.75																																						
Cl	17	189	1.78																																						
		Sum	100																																						

Table 4.4 continued



From this EDX experiment data, all samples show the presence of Mg, O and Cl elements. These O and Cl elements were precipitated from the solution used for the immersion, and Mg was from the fabricated samples. Zn was identified in the Mg0.2%Mn6.0%Zn sample from the fabricated sample. For Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs sample, in addition to Mn and Zn, were detected. The Mn and Zn were attributed to the fabrication material used as an alloying element. Na was also detected from the HP Mg sample as it was present besides Cl and O from the NaCl solutions after immersion.

The reinforcements and microstructure strongly influence the stability of the hydroxide film. On the surface of the original Mg matrix, as a result of exposure to air, a MgO film is formed. The oxide film formed naturally on pure Mg as a result of exposure to air indicated that a mixture of Mg, MgO, and Mg(OH)₂ is formed. When this surface is exposed to the aqueous solution, Mg and/or MgO react with water forming a thick layer of Mg(OH)₂. As the corrosion process continues, a barrier of MgO/ Mg(OH)₂ layer, which is believed to control the corrosion performance, is formed. However, this layer is partially protective and occasionally destroyed, especially in the presence of Chloride ions. The destroyed points are nucleation sites for pitting (Nordlien et al., 1997).

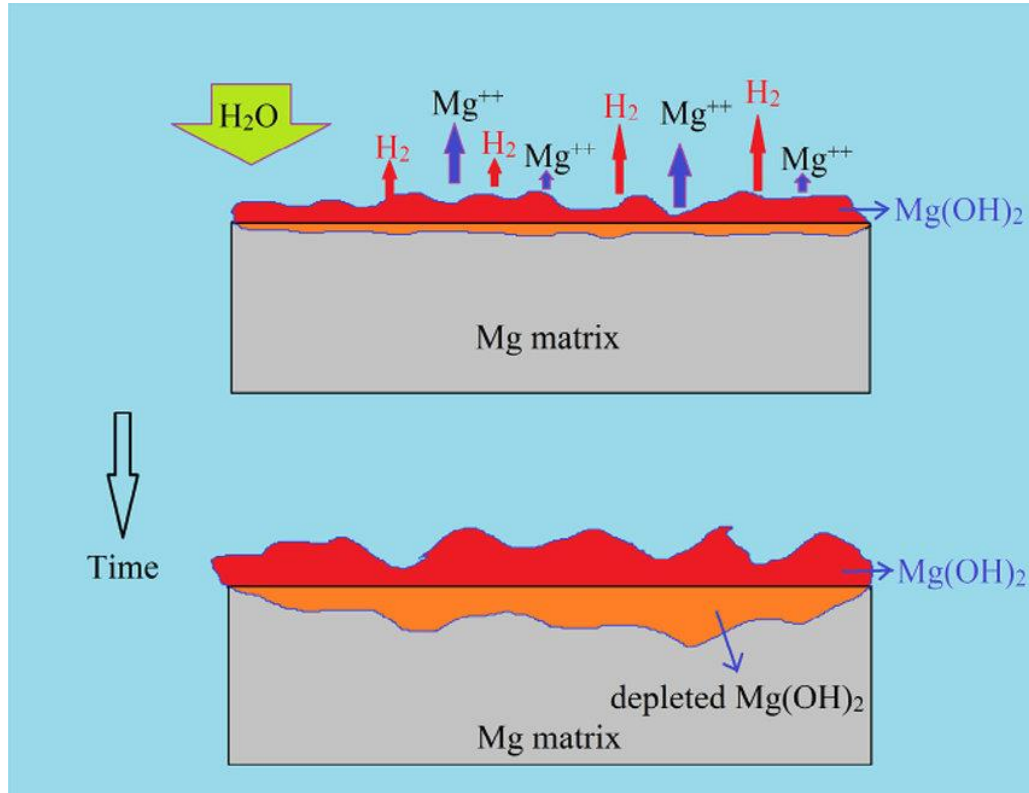


Figure 4.7: Schematic model on the corrosion mechanism of Mg-based composites (Rashad et al., 2017)

Figure 4.7 displays a schematic diagram illustrating the corrosion mechanism of Mg composite. When exposed to a corrosive environment, the Mg matrix behaves anodically compared to adjacent GNPs zones, which are believed to act as cathodic sites. Consequently, the composite's corrosion begins to occur preferentially on the matrix near GNPs zones, resulting in the formation of localised pits that deepen over time. The anodic reaction creates Mg^{2+} ions, which diffuse outward from the metal surface, while chloride ions migrate inward to maintain electro-neutrality. As corrosion progresses, the pit expands, undermines the partially protective film on the Mg matrix, and deepens at the expense of the Mg matrix, including the GNPs zones. The protective oxide-hydroxide layer's discontinuity at the GNPs

and matrix interface also renders sites near the graphene susceptible to pitting initiation (Rashad et al., 2017).

The presence of the Cl element in all samples showed that the corrosion products had formed into magnesium chloride (MgCl) precipitates. These MgCl precipitates were formed because of the depletion of the $\text{Mg}(\text{OH})_2$ layer that protects the samples from further corrosion. In a 3% NaCl solution, it was observed that the Cl ions were responsible for the samples' corrosion. If the corrosion immersion period increase, the NaCl solution will convert completely as the chloride ions convert $\text{Mg}(\text{OH})_2$ to form MgCl precipitates soluble salt (Pourbaix, 1974).

The corrosion of HP Mg, Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs immersed in the 3% NaCl solution were dominantly determined by the Cl ions where there is less tendency to form partially protective surface film formation. The higher Cl ion concentration will give a higher tendency for passive film breakdown (G. Song & Atrens, 2000, 2003).

4.6 Chapter Summary

Several observations can be made based on the results and discussions mentioned in Chapter 4. From the microstructural characterisation of the samples, adding GNPs with increasing wt.% and Mn and Zn provide grain refinement of the microstructure and thus provides grain boundaries that hinder the surface structure, which leads to improving the hardness. Besides, the hardness of the samples was improved, too, not only with the addition of GNPs but with a suitable fabrication method provided by the sintering on the powder metallurgy method. Corrosion rates for the samples were also determined as the GNPs

reinforced Mg composites provide better corrosion rate when immersed with NaCl solution with the condition of the surface attacked mostly due to uniform corrosion. Lastly, from the corrosion immersion test, MgCl is known to be the final corrosion product that precipitates on the surface of the samples.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The fabrication of GNPs reinforced Mg0.2%Mn6.0%Zn composites, Mg0.2%Mn6.0%Zn and HP Mg samples was successfully conducted, the microstructure characterisation, the microhardness test and corrosion behaviour of these samples were studied. The properties of Mg and its composites were also understood during this project.

In this study, the fabrication of a magnesium alloy matrix reinforced with GNPs composites using the powder metallurgy technique was accomplished. The HP Mg, Mg alloy and GNPs reinforced Mg composites can be fabricated using the powder metallurgy technique with the same parameters. The first objective was successfully achieved by studying the effect of GNPs on their mechanical properties, which is the hardness value. The hardness of Mg0.2%Mn6.0%Zn-1% GNPs was highest compared to Mg0.2%Mn6.0%Zn-0.5% GNPs, followed by Mg0.2%Mn6.0%Zn and finally HP Mg. By comparison, Mg0.2%Mn6.0%Zn-1% GNPs gives an increase of hardness by 34.15%, Mg0.2%Mn6.0%Zn-0.5% GNPs hardness increased by 27.84%, and Mg0.2%Mn6.0%Zn gives rise of hardness by 25.64%, compared with HP Mg respectively.

Other than that, the corrosion rate of the samples was determined by immersion test in 3% NaCl solution with saturated Mg(OH)₂. The corrosion behaviour on HP Mg, Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs were studied and analysed. Based on the results, as the immersion period increased, the hydrogen evolution rate also increased. The corrosion rates in P_H and P_W for HP Mg, Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs

samples were tabulated. The corrosion rate of HP Mg was the highest, followed by Mg0.2%Mn6.0%Zn, Mg0.2%Mn6.0%Zn-0.5% GNPs and Mg0.2%Mn6.0%Zn-1% GNPs. The surface morphology shown that HP Mg, Mg0.2%Mn6.0%Zn and Mg0.2%Mn6.0%Zn-1% GNPs were having a uniform corrosion while pitting corrosion happened on Mg0.2%Mn6.0%Zn-0.5% GNPs. From the corrosion rate results, these proved that the addition of GNPs could improve the corrosion resistance to Mg-based composites.

Finally, the third objective of this study to determine the microstructure of the fabricated GNP-reinforced Mg composite and the surface morphology of each sample after the corrosion immersion test was successfully conducted through 3D optical microscope and SEM. The identification of elements on the corrosion products, $\text{Mg}(\text{OH})_2$ and MgCl , was successfully determined through EDX analysis.

5.2 Recommendations

Some recommendations can be made to improve the research further in future works. These include:

1. The mechanical properties such as tensile strength, compression strength, and torsion and fatigue, are outside the project due to the focus on the properties such as hardness and corrosion behaviour. It is proposed that a tensile test or nanoindentation of GNPs reinforced Mg composites be conducted to further understand the composites' mechanical properties.
2. The compositions of GNPs should be added more for precise comparison of data on hardness and corrosion properties, such as 0.1% and 1.5% GNPs.

3. A research with different sintering temperatures of Mg-based composites is suggested to investigate further the relationship between the hardness effect and the composites' microstructure with specific sintering temperatures.
4. In order to make Mg-based composites commercially viable in the future, studies related to other fabrication methods, machining, welding, or other forms of deformation such as rolling, cold pressing, equi-channel angle pressing (ECAP) should be carried out in the research.

REFERENCES

- Afifi, M. A. (2014). Corrosion Behavior of Zinc-Graphite Metal Matrix Composite in 1 M of HCl. *ISRN Corrosion*, 2014, 1–8. <https://doi.org/10.1155/2014/279856>
- Ali, M., Hussein, M. A., & Al-Aqeeli, N. (2019). Magnesium-based composites and alloys for medical applications: A review of mechanical and corrosion properties. *Journal of Alloy and Compounds*, 792, 1162–1190. <https://doi.org/10.1016/j.jallcom.2019.04.080>
- Amukarimi, S., & Mozafari, M. (2021). Biodegradable magnesium-based biomaterials: an overview of challenges and opportunities. *MedComm*, 2(2), 123-144.
- Angelo, P. C., & Ravisankar, B. (2021). Micro and nano fabrication by powder metallurgy. *Micro and Nano Fabrication by Powder Metallurgy*, 1–40.
- Arab, M., & Marashi, S. P. H. (2019). Effect of graphene nanoplatelets (GNPs) content on improvement of mechanical and tribological properties of AZ31 Mg matrix nanocomposite. *Tribology International*, 132, 1–10.
- Ardelean, H., Seyeux, A., Zanna, S., Prima, F., Frateur, I., & Marcus, P. (2013). Corrosion processes of Mg–Y–Nd–Zr alloys in Na₂SO₄ electrolyte. *Corrosion Science*, 73, 196–207. <https://doi.org/10.1016/j.corsci.2013.03.036>
- Arrabal, R., Matykina, E., Pardo, A., Merino, M. C., Paucar, K., Mohedano, M., & Casajús, P. (2012). Corrosion behaviour of AZ91D and AM50 magnesium alloys with Nd and Gd additions in humid environments. *Corrosion Science*, 55, 351–362. <https://doi.org/10.1016/j.corsci.2011.10.038>

- Arrabal, R., Mingo, B., Pardo, A., Matykina, E., Mohedano, M., Merino, M. C., Rivas, A., & Maroto, A. (2015). Role of alloyed Nd in the microstructure and atmospheric corrosion of as-cast magnesium alloy AZ91. *Corrosion Science*, 97, 38–48.
- Atrens, A., Liu, M., & Zainal Abidin, N. I. (2011). Corrosion mechanism applicable to biodegradable magnesium implants. *Materials Science and Engineering B: Solid-State Materials for Advanced Technology*, 176(20), 1609–1636.
- Atrens, A., Song, G.-L., Liu, M., Shi, Z., Cao, F., & Dargusch, M. S. (2015). Review of Recent Developments in the Field of Magnesium Corrosion. *Advanced Engineering Materials*, 17(4), 400–453. <https://doi.org/10.1002/adem.201400434>
- Bamberger, M. (2001). Structural refinement of cast magnesium alloys. *Materials Science and Technology*, 17(1), 15–24.
- Baril, G., Blanc, C., & Pébère, N. (2001). AC Impedance Spectroscopy in Characterizing Time-Dependent Corrosion of AZ91 and AM50 Magnesium Alloys Characterization with Respect to Their Microstructures. *Journal of The Electrochemical Society*, 148(12), B489. <https://doi.org/10.1149/1.1415722>
- Beldjoudi, T., Fiaud, C., & Robbiola, L. (1993). Influence of homogenization and artificial aging heat treatments on corrosion behavior of Mg-Al alloys. *Corrosion*, 49(9), 738–745.
- Ben-Hamu, G., Eliezer, D., & Shin, K. S. (2010). Studies on the influence of chloride ion concentration on the corrosion behavior of ZSMX magnesium alloy. *Advanced Materials Research*, 95, 47–50. <https://doi.org/10.4028/www.scientific.net/amr.95.47>
- Bizao, R. A., Botari, T., & Galvao, D. S. (2014). Mechanical Properties of Graphene Nanowiggles. *MRS Proceedings*, 1658.

- Blawert, C., Hort, N., & Kainer, K. U. (2004). Automotive Applications of Magnesium and Its Alloys. *Transactions of the Indian Institute of Metals.*, 57(4), 397–408. <https://doi.org/10.4028/www.scientific.net/MSF.419-422.67>
- Boland, E. L., Shirazi, R. N., Grogan, J. A., & McHugh, P. E. (2018). Mechanical and Corrosion Testing of Magnesium WE43 Specimens for Pitting Corrosion Model Calibration. *Advanced Engineering Materials*, 20(10), 1–11.
- Bruce Abbott, T., Abbott, T., & Easton, M. (2001). Properties of Magnesium Die Castings for Structural Applications Statistical mechanical modelling View project Properties of Magnesium Die Castings for Structural Applications. In *Materials Forum* (Vol. 25). <https://www.researchgate.net/publication/272831620>
- Budinski, K. G., & Budinski, M. K. (1999). *Engineering Materials: Properties and Selection*. Prentice Hall.
- Cai, S., Lei, T., Li, N., & Feng, F. (2012). Effects of Zn on microstructure, mechanical properties and corrosion behavior of Mg-Zn alloys. *Materials Science and Engineering C*, 32(8), 2570–2577. <https://doi.org/10.1016/j.msec.2012.07.042>
- Cao, F., Shi, Z., Hofstetter, J., Uggowitzer, P. J., Song, G., Liu, M., & Atrens, A. (2013). Corrosion of ultra-high-purity Mg in 3.5% NaCl solution saturated with Mg(OH)₂. *Corrosion Science*, 75, 78–99.
- Cao, F., Shi, Z., Song, G. L., Liu, M., Dargusch, M. S., & Atrens, A. (2015). Influence of hot rolling on the corrosion behavior of several Mg–X alloys. *Corrosion Science*, 90, 176–191. <https://doi.org/10.1016/j.corsci.2014.10.012>

- Cataldi, P., Athanassiou, A., & Bayer, I. S. (2018). Graphene nanoplatelets-based advanced materials and recent progress in sustainable applications. In *Applied Sciences (Switzerland)* (Vol. 8, Issue 9). MDPI AG. <https://doi.org/10.3390/app8091438>
- Chen, B., Wu, H., Yi, R., Wang, W., Xu, H., Zhang, S., Peng, H., Ma, J., Jiang, H., Zan, R., Qiao, S., Sun, Y., Hou, P., Han, P., Ni, J., & Zhang, X. (2020). In vitro crevice corrosion of biodegradable magnesium in different solutions. *Journal of Materials Science and Technology*, 52, 83–88. <https://doi.org/10.1016/j.jmst.2020.04.014>
- Chen, J., Tan, L., Yu, X., Etim, I. P., Ibrahim, M., & Yang, K. (2018). Mechanical properties of magnesium alloys for medical application: A review. *Journal of the Mechanical Behavior of Biomedical Materials*, 87, 68–79.
- Chen, J., Wang, J., Han, E., & Ke, W. (2008). Effects of dust and salt particles on the formation and spreading of micro-droplets on AZ91 magnesium alloy. *Corrosion Science*, 50(5), 1449–1459. <https://doi.org/10.1016/j.corsci.2008.01.027>
- Chen, L. Y., Konishi, H., Fehrenbacher, A., Ma, C., Xu, J. Q., Choi, H., Xu, H. F., Pfefferkorn, F. E., & Li, X. C. (2012). Novel nanoprocessing route for bulk graphene nanoplatelets reinforced metal matrix nanocomposites. *Scripta Materialia*, 67(1), 29–32. <https://doi.org/10.1016/j.scriptamat.2012.03.013>
- Chen, L., Zhao, Y., Hou, H., Zhang, T., Liang, J., Li, M., & Li, J. (2019). Development of AZ91D magnesium alloy-graphene nanoplatelets composites using thixomolding process. *Journal of Alloys and Compounds*, 778, 359–374.
- Chen, T., Yuan, Y., Wu, J., Liu, T., Chen, X., Tang, A., & Pan, F. (2019). Alloy Design Strategies of the Native Anti-corrosion Magnesium Alloy. *Minerals, Metals and Materials Series*, 169–173. https://doi.org/10.1007/978-3-030-05789-3_25

- Chen, Y., Xu, Z., Smith, C., & Sankar, J. (2014). Recent advances on the development of magnesium alloys for biodegradable implants. *Acta Biomaterialia*, 10(11), 4561–4573. <https://doi.org/10.1016/j.actbio.2014.07.005>
- Cheng, J., Li, Y., Huang, X., Wang, Q., Mei, A., & Shen, P. K. (2015). Highly stable electrocatalysts supported on nitrogen-self-doped three-dimensional graphene-like networks with hierarchical porous structures. *Journal of Materials Chemistry A*, 3(4), 1492–1497. <https://doi.org/10.1039/C4TA05552G>
- Čížek, L., Greger, M., Pawlica, L., Dobrzański, L. A., & Tański, T. (2004). Study of selected properties of magnesium alloy AZ91 after heat treatment and forming. *Journal of Materials Processing Technology*, 157–158(SPEC. ISS.), 466–471.
- Czerwinski, F. (2014). Controlling the ignition and flammability of magnesium for aerospace applications. In *Corrosion Science* (Vol. 86, pp. 1–16). Elsevier Ltd. <https://doi.org/10.1016/j.corsci.2014.04.047>
- Danaie, M., Asmussen, R. M., Jakupi, P., Shoesmith, D. W., & Botton, G. A. (2013). The role of aluminum distribution on the local corrosion resistance of the microstructure in a sand-cast AM50 alloy. *Corrosion Science*, 77, 151–163.
- Dhand, V., Rhee, K. Y., Kim, H. J., & Jung, D. H. (2017). *A Comprehensive Review of Graphene Nanocomposites: Research Status and Trends 2013*, 158-158.
- Ding, Y., Wen, C., Hodgson, P., & Li, Y. (2014). Effects of alloying elements on the corrosion behavior and biocompatibility of biodegradable magnesium alloys: a review. *J. Mater. Chem. B*, 2(14), 1912–1933. <https://doi.org/10.1039/C3TB21746A>

- Dobrzański, L. A., & Tański, T. (2009). Influence of Aluminium Content on Behaviour of Magnesium Cast Alloys in Bentonite Sand Mould. *Solid State Phenomena*, 147–149, 764–769. <https://doi.org/10.4028/www.scientific.net/SSP.147-149.764>
- Dong, J., Lin, T., Shao, H., Wang, H., Wang, X., Song, K., & Li, Q. (2022). Advances in degradation behavior of biomedical magnesium alloys: A review. *Journal of Alloys and Compounds*, 908, 164600.
- Dorozhkin, S. V. (2014). Calcium orthophosphate coatings on magnesium and its biodegradable alloys. *Acta Biomaterialia*, 10(7), 2919–2934.
- Du, X., Du, W., Wang, Z., Liu, K., & Li, S. (2019). Defects in graphene nanoplatelets and their interface behavior to reinforce magnesium alloys. *Applied Surface Science*, 484(November 2018), 414–423. <https://doi.org/10.1016/j.apsusc.2019.04.111>
- Dziubińska, A., Gontarz, A., Dziubiński, M., & Barszcz, M. (2016). The Forming of Magnesium Alloy Forgings for Aircraft and Automotive Applications. *Advances in Science and Technology Research Journal*, 10(31), 158–168.
- Easton, M., Beer, A., Barnett, M., Davies, C., Dunlop, G., Durandet, Y., Blacket, S., Hilditch, T., & Beggs, P. (2008). Magnesium alloy applications in automotive structures. *Jom*, 60(11), 57–62. <https://doi.org/10.1007/s11837-008-0150-8>
- Feliu, S., Maffiotte, C., Galván, J. C., & Barranco, V. (2011). Atmospheric corrosion of magnesium alloys AZ31 and AZ61 under continuous condensation conditions. *Corrosion Science*, 53(5), 1865–1872.
- Feng, A., & Han, Y. (2010). The microstructure, mechanical and corrosion properties of calcium polyphosphate reinforced ZK60A magnesium alloy composites. *Journal of Alloys and Compounds*, 504(2), 585–593.

- Gaines, L., Cuenca, R., Stodolsky, F., & Wu, S. (1996). Potential Automotive Uses of Wrought Magnesium Alloys. *Automotive Technology Development*, 1, 7.
- Gandel, D. S., Birbilis, N., Easton, M. A., & Gibson, M. A. (2010). Influence of manganese, Zirconium and Iron on the corrosion of Magnesium. *Corrosion & Prevention*, 8, 1–11.
- Ghali, E., Dietzel, W., & Kainer, K. U. (2013). General and localized corrosion of magnesium alloys: A critical review. *Journal of Materials Engineering and Performance*, 22(10), 2875–2891. <https://doi.org/10.1007/s11665-013-0730-9>
- A Ghavanini, F., & Theander, H. (2015). Graphene feasibility study and foresight study for transport infrastructures.
- Ghosh, S., Calizo, I., Teweldebrhan, D., Pokatilov, E. P., Nika, D. L., Balandin, A. A., Bao, W., Miao, F., & Lau, C. N. (2008). Extremely high thermal conductivity of graphene: Prospects for thermal management applications in nanoelectronic circuits. *Applied Physics Letters*, 92(15), 1–4. <https://doi.org/10.1063/1.2907977>
- Goh, C. S., Wei, J., Lee, L. C., & Gupta, M. (2006). Development of novel carbon nanotube reinforced magnesium nanocomposites using the powder metallurgy technique. *Nanotechnology*, 17(1), 7–12. <https://doi.org/10.1088/0957-4484/17/1/002>
- Gray, J. E., & Luan, B. (2002). Protective coatings on magnesium and its alloys — a critical review. *Journal of Alloys and Compounds*, 336, 88–113.
- Gu, C., Lian, J., He, J., Jiang, Z., & Jiang, Q. (2006). High corrosion-resistance nanocrystalline Ni coating on AZ91D magnesium alloy. *Surface and Coatings Technology*, 200(18–19), 5413–5418. <https://doi.org/10.1016/j.surfcoat.2005.07.001>

- Guo, M. X., Du, J. Q., Zheng, C. H., Zhang, J. S., & Zhuang, L. Z. (2019). Influence of Zn contents on precipitation and corrosion of Al-Mg-Si-Cu-Zn alloys for automotive applications. *Journal of Alloys and Compounds*, 778, 256–270.
- Gupta, M. (2017). Utilizing Magnesium based Materials to Reduce Green House Gas Emissions in Aerospace Sector. *Aeronautics and Aerospace Open Access Journal*, 1(1). <https://doi.org/10.15406/aaaj.2017.01.00005>
- Gupta, M., & Ling, S. N. M. (2011). *Magnesium, magnesium alloys, and magnesium composites*. John Wiley & Sons.
- Gwyne, B., & Lyon, P. (2007, October 29). *Magnesium Alloys in Aerospace Applications, Past Concerns, Current Solutions*. Magnesium Elektron. https://www.fire.tc.faa.gov/2007conference/files/Materials_Fire_Safety/WedAM/GwynneMagnesium/GwynneMagnesiumPres.pdf
- Hafeez, N., Liu, S., Lu, E., Wang, L., Liu, R., Lu, W., & Zhang, L.-C. (2019). Mechanical behavior and phase transformation of β -type Ti-35Nb-2Ta-3Zr alloy fabricated by 3D-Printing. *Journal of Alloys and Compounds*, 790, 117–126. <https://doi.org/10.1016/j.jallcom.2019.03.138>
- Hassan, S. F., & Gupta, M. (2002). Development of ductile magnesium composite materials using titanium as reinforcement. *Journal of Alloys and Compounds*, 345(1–2), 246–251. [https://doi.org/10.1016/S0925-8388\(02\)00413-9](https://doi.org/10.1016/S0925-8388(02)00413-9)
- Hughes, C. (2011, January 4). *The promise and challenges of ultra high bypass ratio engine technology and integration*. National Aeronautics and Space Administration. <https://ntrs.nasa.gov/api/citations/20110011737/downloads/20110011737.pdf>

- James, T. H. (1943). Rate of Solution of Magnesium in Acids. *Journal of the American Chemical Society*, 65(1), 39–41. <https://doi.org/10.1021/ja01241a012>
- Jayasathyakawin, S., Ravichandran, M., Baskar, N., Chairman, C. A., & Balasundaram, R. (2020). Magnesium matrix composite for biomedical applications through powder metallurgy – Review. *Materials Today: Proceedings*, 27, 736–741.
- Jiang, J., Geng, X., & Zhang, X. (2023). Stress corrosion cracking of magnesium alloys: A review. *Journal of Magnesium and Alloys*.
- Jiang, Q. C., Li, X. L., & Wang, H. Y. (2003). Fabrication of TiC particulate reinforced magnesium matrix composites. *Scripta Materialia*, 48(6), 713–717. [https://doi.org/10.1016/S1359-6462\(02\)00551-1](https://doi.org/10.1016/S1359-6462(02)00551-1)
- Jones, R. M. (2018). *Mechanics of composite materials*. CRC press.
- Jönsson, M., & Persson, D. (2010). The influence of the microstructure on the atmospheric corrosion behaviour of magnesium alloys AZ91D and AM50. *Corrosion Science*, 52(3), 1077–1085. <https://doi.org/10.1016/j.corsci.2009.11.036>
- Joost, W. J., & Krajewski, P. E. (2017). Towards magnesium alloys for high-volume automotive applications. *Scripta Materialia*, 128, 107–112.
- Kainer, -Ing. K. U., & John Wiley & Sons. (2000). *Magnesium alloys and their applications*. Wiley-VCH Verlag GmbH.
- Kekare, P. R., & Kulkarni, A. (2021). In vitro study of Biodegradation in Mg-alloys by Isothermal Calorimetry.

- Kirkland, N. T., Kolbeinsson, I., Woodfield, T., Dias, G. J., & Staiger, M. P. (2011). Synthesis and properties of topologically ordered porous magnesium. *Materials Science and Engineering: B*, 176(20), 1666–1672.
- Kondoh, K., Oginuma, H., Tuzuki, R., & Aizawa, T. (2003). Magnesium Matrix Composite with Solid-state Synthesized Mg₂Si Dispersoids. *Materials Transactions*, 44(4), 611–618. <https://doi.org/10.2320/matertrans.44.611>
- Kulekci, M. K. (2008). Magnesium and its alloys applications in automotive industry. *The International Journal of Advanced Manufacturing Technology*, 39(9–10), 851–865. <https://doi.org/10.1007/s00170-007-1279-2>
- Kumar, K., Gill, R. S., & Batra, U. (2018). Challenges and opportunities for biodegradable magnesium alloy implants. In *Materials Technology* (Vol. 33, Issue 2, pp. 153–172). Taylor and Francis Ltd. <https://doi.org/10.1080/10667857.2017.1377973>
- Li, G., Zhang, J., Wu, R., Feng, Y., Liu, S., Wang, X., Jiao, Y., Yang, Q., & Meng, J. (2018). Development of high mechanical properties and moderate thermal conductivity cast Mg alloy with multiple RE via heat treatment. *Journal of Materials Science and Technology*, 34(7), 1076–1084. <https://doi.org/10.1016/j.jmst.2017.12.011>
- Li, Y. Gang, Wei, Y. Hui, Hou, L. Feng, & Han, P. Ju. (2013). Atmospheric corrosion of AM60 Mg alloys in an industrial city environment. *Corrosion Science*, 69, 67–76.
- Liao, J., Hotta, M., Motoda, S. I., & Shinohara, T. (2013). Atmospheric corrosion of two field-exposed AZ31B magnesium alloys with different grain size. *Corrosion Science*, 71, 53–61. <https://doi.org/10.1016/j.corsci.2013.02.003>
- Liao, J., Hotta, M., & Yamamoto, N. (2012). Corrosion behavior of fine-grained AZ31B magnesium alloy. *Corrosion Science*, 61, 208–214.

- Lindström, R., Johansson, L. G., Thompson, G. E., Skeldon, P., & Svensson, J. E. (2004). Corrosion of magnesium in humid air. *Corrosion Science*, 46(5), 1141–1158. <https://doi.org/10.1016/j.corsci.2003.09.010>
- Liu, C., Shen, J., Yeung, K. W. K., & Tjong, S. C. (2017). Development and Antibacterial Performance of Novel Polylactic Acid-Graphene Oxide-Silver Nanoparticle Hybrid Nanocomposite Mats Prepared by Electrospinning. *ACS Biomaterials Science and Engineering*, 3(3), 471–486. <https://doi.org/10.1021/acsbiomaterials.6b00766>
- Liu, H., Yan, Y., Wu, X., Fang, H., Chu, X., Huang, J., Zhang, J., Song, J., & Yu, K. (2021). Effects of Al and Sn on microstructure, corrosion behavior and electrochemical performance of Mg–Al-based anodes for magnesium-air batteries. *Journal of Alloys and Compounds*, 859. <https://doi.org/10.1016/j.jallcom.2020.157755>
- Liu, X., Wu, K., Gao, L., Wang, L., & Shi, X. (2022). Biomaterial strategies for the application of reproductive tissue engineering. *Bioactive Materials*, 14, 86–96. <https://doi.org/10.1016/j.bioactmat.2021.11.023>
- Lotfpoor, M., Emamy, M., Dehghanian, C., & Tavighi, K. (2017). Influence of Cu Addition on the Structure, Mechanical and Corrosion Properties of Cast Mg-2%Zn Alloy. *Journal of Materials Engineering and Performance*, 26(5), 2136–2150. <https://doi.org/10.1007/s11665-017-2672-0>
- Luo, A., Renaud, J., Nakatsugawa, I., & Plourde, J. (1995). Magnesium castings for automotive applications. *Jom*, 47(7), 28–31. <https://doi.org/10.1007/BF03221226>
- Makar, G. L., & Kruger, J. (1993). Corrosion of magnesium. *International Materials Reviews*, 38(3), 138–153. <https://doi.org/10.1179/imr.1993.38.3.138>

- Manohar, G., Pandey, K. M., & Maity, S. R. (2021). Characterization of Boron Carbide (B₄C) particle reinforced aluminium metal matrix composites fabricated by powder metallurgy techniques – A review. *Materials Today: Proceedings*, 45, 6882–6888. <https://doi.org/10.1016/j.matpr.2020.12.1087>
- Matthews, F. L., & Rawlings, R. D. (1999). *Composite materials: engineering and science*. Woodhead Publishing.
- Merino, M. C., Pardo, A., Arrabal, R., Merino, S., Casajús, P., & Mohedano, M. (2010). Influence of chloride ion concentration and temperature on the corrosion of Mg–Al alloys in salt fog. *Corrosion Science*, 52(5), 1696–1704.
- Mingo, B., Arrabal, R., Mohedano, M., Llamazares, Y., Matykina, E., Yerokhin, A., & Pardo, A. (2018). Influence of sealing post-treatments on the corrosion resistance of PEO coated AZ91 magnesium alloy. *Applied Surface Science*, 433, 653–667. <https://doi.org/10.1016/j.apsusc.2017.10.083>
- Mining, M., Higashi, K., & Prefecture, O. (1996). Tensile strength, ductility and fracture of magnesium-silicon alloys. *Journal of Materials Science*, 31, 1529–1535.
- Mordike, B. L., & Ebert, T. (2001). Magnesium Properties - applications - potential. *Materials Science and Engineering A*, 302(1), 37–45. [https://doi.org/10.1016/S0921-5093\(00\)01351-4](https://doi.org/10.1016/S0921-5093(00)01351-4)
- Niu, X., Shen, H., & Fu, J. (2018). Microstructure and mechanical properties of selective laser melted Mg-9 wt%Al powder mixture. *Materials Letters*, 221, 4–7. <https://doi.org/10.1016/j.matlet.2018.03.068>
- Nogita, K., Ockert, S., Pierce, J., Greaves, M. C., Gourlay, C. M., & Dahle, A. K. (2009). Engineering the Mg-Mg₂Ni eutectic transformation to produce improved hydrogen

- storage alloys. *International Journal of Hydrogen Energy*, 34(18), 7686–7691.
<https://doi.org/10.1016/j.ijhydene.2009.07.036>
- Nordlien, J. H., Ono, S., Masuko, N., & Nisancioglu, K. (1997). A TEM investigation of naturally formed oxide films on pure magnesium. *Corrosion Science*, 39(8), 1397–1414. [https://doi.org/10.1016/S0010-938X\(97\)00037-1](https://doi.org/10.1016/S0010-938X(97)00037-1)
- Orlov, D., Ralston, K. D., Birbilis, N., & Estrin, Y. (2011). Enhanced corrosion resistance of Mg alloy ZK60 after processing by integrated extrusion and equal channel angular pressing. *Acta Materialia*, 59(15), 6176–6186.
- Pettersella, G., & Westengen, H. (1996). Microstructure of a pressure die cast magnesium 4wt.% alloy modified with rare earth additions. *Materials Science and Engineering: A*, 207, 115–120. [https://doi.org/10.1016/0921-5093\(95\)10035-0](https://doi.org/10.1016/0921-5093(95)10035-0)
- Polmear, I. J. (1994). Magnesium alloys and applications. *Materials Science and Technology*, 10(1), 1–16. <https://doi.org/10.1179/026708394790163401>
- Pourbaix, M. (1974). Atlas of electrochemical equilibria in aqueous solution. *NACE*.
- Prasad, S. V. S., Prasad, S. B., Verma, K., Mishra, R. K., Kumar, V., & Singh, S. (2022). The role and significance of Magnesium in modern day research-A review. In *Journal of Magnesium and Alloys* (Vol. 10, Issue 1, pp. 1–61). National Engg. Research Center for Magnesium Alloys. <https://doi.org/10.1016/j.jma.2021.05.012>
- Pryor, M. J., & Astley, D. J. (2013). Bimetallic Corrosion. *Corrosion: Third Edition*, 1, 1:213-1:243. <https://doi.org/10.1016/B978-0-08-052351-4.50015-7>
- Pu, Z., Song, G. L., Yang, S., Outeiro, J. C., Dillon, O. W., Puleo, D. A., & Jawahir, I. S. (2012). Grain refined and basal textured surface produced by burnishing for improved corrosion performance of AZ31B Mg alloy. *Corrosion Science*, 57, 192–201.

- Pu, Z., Yang, S., Song, G. L., Dillon, O. W., Puleo, D. A., & Jawahir, I. S. (2011). Ultrafine-grained surface layer on Mg–Al–Zn alloy produced by cryogenic burnishing for enhanced corrosion resistance. *Scripta Materialia*, 65(6), 520–523.
- Qiao, Z., Shi, Z., Hort, N., Zainal Abidin, N. I., & Atrens, A. (2012). Corrosion behaviour of a nominally high purity Mg ingot produced by permanent mould direct chill casting. *Corrosion Science*, 61, 185–207. <https://doi.org/10.1016/j.corsci.2012.04.030>
- Ralston, K. D., & Birbilis, N. (2010). Effect of grain size on corrosion: a review. *Corrosion*, 66(7), 075005–075005.
- Rashad, M., Pan, F., Asif, M., & Chen, X. (2017). Corrosion behavior of magnesium-graphene composites in sodium chloride solutions. *Journal of Magnesium and Alloys*, 5(3), 271–276. <https://doi.org/10.1016/j.jma.2017.06.003>
- Rashad, M., Pan, F., Asif, M., & Tang, A. (2014). Powder metallurgy of Mg-1%Al-1%Sn alloy reinforced with low content of graphene nanoplatelets (GNPs). *Journal of Industrial and Engineering Chemistry*, 20(6), 4250–4255.
- Rashad, M., Pan, F., Hu, H., Asif, M., Hussain, S., & She, J. (2015). Enhanced tensile properties of magnesium composites reinforced with graphene nanoplatelets. *Materials Science and Engineering A*, 630, 36–44. <https://doi.org/10.1016/j.msea.2015.02.002>
- Rashad, M., Pan, F., Lin, D., & Asif, M. (2016). High temperature mechanical behavior of AZ61 magnesium alloy reinforced with graphene nanoplatelets. *Materials and Design*, 89, 1242–1250. <https://doi.org/10.1016/j.matdes.2015.10.101>
- Rashad, M., Pan, F., Tang, A., Asif, M., Hussain, S., Gou, J., & Mao, J. (2015). Improved strength and ductility of magnesium with addition of aluminum and graphene

- nanoplatelets (Al+GNPs) using semi powder metallurgy method. *Journal of Industrial and Engineering Chemistry*, 23, 243–250. <https://doi.org/10.1016/j.jiec.2014.08.024>
- Rashad, M., Pan, F., Tang, A., Asif, M., She, J., Gou, J., Mao, J., & Hu, H. (2015). Development of magnesium-graphene nanoplatelets composite. *Journal of Composite Materials*, 49(3), 285–293. <https://doi.org/10.1177/0021998313518360>
- Rodzi, S. N. H. M., & Hussain, Z. (2018). The effect of milling time on properties of magnesium-based composite fabricated via powder metallurgy. *International Journal of Current Research in Science, Engineering & Technology*, 1, 510-515.
- Romanowski, C. (2019). Magnesium Alloy Sheet for Transportation Applications. *Minerals, Metals and Materials Series*, 3–12. https://doi.org/10.1007/978-3-030-05789-3_1
- Saberi, A., Bakhsheshi Rad, H. R., Karamian, E., Kasiri-Asgarani, M., & Ghomi, H. (2020). Magnesium-graphene nano-platelet composites: Corrosion behavior, mechanical and biological properties. *Journal of Alloys and Compounds*, 821, 153379.
- Safari, N., Toroghinejad, M. R., & Kharaziha, M. (2019). Influence of copper on the structural, mechanical, and biological characteristics of Mg–1Al–Cu alloy. *Materials Chemistry and Physics*, 237(4), 121838.
- Saris, N.E. L., Mervaala, E., Karppanen, H., Khawaja, J. A., & Lewenstam, A. (2000). Magnesium: An update on physiological, clinical and analytical aspects. *Clinica Chimica Acta*, 294(1), 1–26.
- Say, Y., Guler, O., & Dikici, B. (2020). Carbon nanotube (CNT) reinforced magnesium matrix composites: The effect of CNT ratio on their mechanical properties and corrosion resistance. *Materials Science and Engineering A*, 798.

- Seetharaman, S., Jayalakshmi, S., Arvind Singh, R., & Gupta, M. (2022). The Potential of Magnesium-Based Materials for Engineering and Biomedical Applications. *Journal of the Indian Institute of Science*, 102(1), 421–437. <https://doi.org/10.1007/s41745-021-00274-w>
- Sekar, P., Sanna, N., & Desai, V. (2020). Enhancement of resistance to galvanic corrosion of ZE41 Mg alloy by equal channel angular pressing. *Materials and Corrosion*, 71(4), 571–584. <https://doi.org/10.1002/maco.201911337>
- Seth, P. P., Singh, N., Singh, M., Prakash, O., & Kumar, D. (2020). Formation of fine Mg₂Si phase in Mg–Si alloy via solid-state sintering using high energy ball milling. *Journal of Alloys and Compounds*, 821, 153205. <https://doi.org/10.1016/j.jallcom.2019.153205>
- Sezer, N., Evis, Z., Kayhan, S. M., Tahmasebifar, A., & Koç, M. (2018). Review of magnesium-based biomaterials and their applications. *Journal of Magnesium and Alloys*, 6(1), 23–43. <https://doi.org/10.1016/j.jma.2018.02.003>
- Shi, Z., Liu, M., & Atrens, A. (2010). Measurement of the corrosion rate of magnesium alloys using Tafel extrapolation. *Corrosion Science*, 52(2), 579–588. <https://doi.org/10.1016/j.corsci.2009.10.016>
- Shuai, C., Guo, W., Wu, P., Yang, W., Hu, S., Xia, Y., & Feng, P. (2018). A graphene oxide-Ag co-dispersing nanosystem: Dual synergistic effects on antibacterial activities and mechanical properties of polymer scaffolds. *Chemical Engineering Journal*, 347, 322–333. <https://doi.org/10.1016/j.cej.2018.04.092>
- Slooff, F. A., Zhou, J., Duszczyk, J., & Katgerman, L. (2007). Constitutive analysis of wrought magnesium alloy Mg-Al₄-Zn₁. *Scripta Materialia*, 57(8), 759–762. <https://doi.org/10.1016/j.scriptamat.2007.06.023>

- Song, G., & Atrens, A. (2000). Corrosion Mechanisms of Magnesium Alloys. *Advanced Engineering Materials*, 1, 11–33.
- Song, G., & Atrens, A. (2003). Understanding Magnesium Corrosion—A Framework for Improved Alloy Performance. *Advanced Engineering Materials*, 5(12), 837–858. <https://doi.org/10.1002/adem.200310405>
- Song, G., Johannesson, B., Hapugoda, S., & StJohn, D. (2004). Galvanic corrosion of magnesium alloy AZ91D in contact with an aluminium alloy, steel and zinc. *Corrosion Science*, 46(4), 955–977. [https://doi.org/10.1016/S0010-938X\(03\)00190-2](https://doi.org/10.1016/S0010-938X(03)00190-2)
- Song, G. L., Dudney, N. J., Li, J., Sacci, R. L., & Thomson, J. K. (2014). The possibility of forming a sacrificial anode coating for Mg. In *Corrosion Science* (Vol. 87, pp. 11–14). Elsevier Ltd. <https://doi.org/10.1016/j.corsci.2014.07.007>
- Stachel, A., & KG, R. A. S. G. C. (2010). Light Weight Alloys in Seat Design. *The Sixth Triennial International Aircraft Fire and Cabin Safety Research Conference*.
- Stankovich, S., Dikin, D. A., Dommett, G. H. B., Kohlhaas, K. M., Zimney, E. J., Stach, E. A., Piner, R. D., Nguyen, S. B. T., & Ruoff, R. S. (2006). Graphene-based composite materials. *Nature*, 442(7100), 282–286. <https://doi.org/10.1038/nature04969>
- Sun, X., Li, C., Dai, X., Zhao, L., Li, B., Wang, H., Liang, C., Li, H., & Fan, J. (2020). Microstructures and properties of graphene-nanoplatelet-reinforced magnesium-matrix composites fabricated by an in situ reaction process. *Journal of Alloys and Compounds*, 835. <https://doi.org/10.1016/j.jallcom.2020.155125>
- Sun, Y., Wu, H., Wang, W., Zan, R., Peng, H., Zhang, S., & Zhang, X. (2019). Translational status of biomedical Mg devices in China. *Bioactive Materials*, 4, 358–365. <https://doi.org/10.1016/j.bioactmat.2019.11.001>

- Tacikowski, M., Banaszek, M., & Smolik, J. (2014). Corrosion-resistant composite titanium nitride layers produced on the AZ91D magnesium alloy by a hybrid method. *Vacuum*, 99, 298–302. <https://doi.org/10.1016/j.vacuum.2013.06.018>
- Tamura, Y., Kida, Y., Tamehiro, H., Kono, N., Soda, H., & McLean, A. (2008). The effect of manganese on the precipitation of Mg₁₇Al₁₂ phase in magnesium alloy AZ 91. *Journal of Materials Science*, 43(4), 1249–1258. <https://doi.org/10.1007/s10853-007-2276-4>
- Tjong, S. C. (2013). Recent progress in the development and properties of novel metal matrix nanocomposites reinforced with carbon nanotubes and graphene nanosheets. *Materials Science and Engineering R: Reports*, 74(10), 281–350.
- Turan, M. E., Sun, Y., Akgul, Y., Turen, Y., & Ahlatci, H. (2017). The effect of GNPs on wear and corrosion behaviors of pure magnesium. *Journal of Alloys and Compounds*, 724, 14–23. <https://doi.org/10.1016/j.jallcom.2017.07.022>
- Unigovski, Y., Eliezer, A., Abramov, E., Snir, Y., & Gutman, E. M. (2003). Corrosion fatigue of extruded magnesium alloys. *Materials Science and Engineering A*, 360(1–2), 132–139. [https://doi.org/10.1016/S0921-5093\(03\)00409-X](https://doi.org/10.1016/S0921-5093(03)00409-X)
- Vilarigues, M., Fernandes, J. C. S., Alves, L. C., & da Silva, R. C. (2008). Electrochemical behaviour of chromium-implanted magnesium in hydroxide, chloride and sulphate solutions. *Surface and Coatings Technology*, 202(17), 4086–4093.
- Wang, Y., Zhang, F., Wang, Y., Duan, Y., Wang, K., Zhang, W., & Hu, J. (2019). Effect of Zn content on the microstructure and mechanical properties of Mg-Gd-Y-Zr alloys. *Materials Science and Engineering A*, 745, 149–158.

- Wendt, A., Weiss, K., Ben-Dov, A., Bamberger, M., & Bronfin, B. (2016). Magnesium castings in aeronautics applications—Special requirements. *Essential readings in magnesium technology*, 65-69.
- Witte, F. (2010). The history of biodegradable magnesium implants: a review. *Acta biomaterialia*, 6(5), 1680-1692. <https://doi.org/10.1016/j.actbio.2010.02.028>
- Wu, G., Fan, Y., Gao, H., Zhai, C., & Zhu, Y. P. (2005). The effect of Ca and rare earth elements on the microstructure, mechanical properties and corrosion behavior of AZ91D. *Materials Science and Engineering: A*, 408(1–2), 255–263. <https://doi.org/10.1016/J.MSEA.2005.08.011>
- Wu, H., Zhang, C., Lou, T., Chen, B., Yi, R., Wang, W., Zhang, R., Zuo, M., Xu, H., Han, P., Zhang, S., Ni, J., & Zhang, X. (2019). Crevice corrosion – A newly observed mechanism of degradation in biomedical magnesium. *Acta Biomaterialia*, 98, 152–159. <https://doi.org/10.1016/j.actbio.2019.06.013>
- Wu, K. C., Yeh, J. W., & Chang, S. Y. (2015). Refined microstructure and improved mechanical properties of high-ratio extruded AZ91-xSn magnesium alloy. *Materials Chemistry and Physics*, 162, 757–763.
- Wu, L., Wu, R., Hou, L., Zhang, J., & Zhang, M. (2018). Microstructure, mechanical properties and wear performance of AZ31 matrix composites reinforced by graphene nanoplatelets (GNPs). *Journal of Alloys and Compounds*, 750, 530–536. <https://doi.org/10.1016/j.jallcom.2018.04.035>
- Xiao, K., Dong, C., Wei, D., Wu, J., & Li, X. (2016). Galvanic corrosion of magnesium alloy and aluminum alloy by kelvin probe. *Journal Wuhan University of Technology, Materials Science Edition*, 31(1), 204–210.

- Xiao, P., Gao, Y., Yang, C., Li, Y., Huang, X., Liu, Q., Zhao, S., Xu, F., & Gupta, M. (2020). Strengthening and toughening mechanisms of Mg matrix composites reinforced with specific spatial arrangement of in-situ TiB₂ nanoparticles. *Composites Part B: Engineering*, 198(April), 108174. <https://doi.org/10.1016/j.compositesb.2020.108174>
- Yamada, Y., Shimojima, K., Sakaguchi, Y., Mabuchi, M., Nakamura, M., & Asahina, T. (1999). Processing of an Open-Cell AZ91 Magnesium Alloy with a Low Density of 0.05 g/cm³. 18, 1477–1480.
- Yamashita, A., Horita, Z., & Langdon, T. G. (2001). Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation. *Materials Science and Engineering: A*, 300(1–2), 142–147. [https://doi.org/10.1016/S0921-5093\(00\)01660-9](https://doi.org/10.1016/S0921-5093(00)01660-9)
- Yu, Z., Tang, A., He, J., Gao, Z., She, J., Liu, J., & Pan, F. (2018). Effect of high content of manganese on microstructure, texture and mechanical properties of magnesium alloy. *Materials Characterization*, 136, 310–317.
- Yung, K. C., Wu, W. M., Pierpoint, M. P., & Kusmartsev, F. v. (2013). Introduction to graphene electronics - a new era of digital transistors and devices. *Contemporary Physics*, 54(5), 233–251. <https://doi.org/10.1080/00107514.2013.833701>
- Zeng, R. C., Zhang, J., Huang, W. J., Dietzel, W., Kainer, K. U., Blawert, C., & Wei, K. (2006). Review of studies on corrosion of magnesium alloys. *Transactions of Nonferrous Metals Society of China*, 16, s763–s771. [https://doi.org/10.1016/S1003-6326\(06\)60297-5](https://doi.org/10.1016/S1003-6326(06)60297-5)

- Zhang, C., Wu, L., Liu, H., Huang, G., Jiang, B., Atrens, A., & Pan, F. (2020). Microstructure and corrosion behavior of Mg-Sc binary alloys in 3.5 wt.% NaCl solution. *Corrosion Science*, 174(3), 108831.
- Zhao, M. C., Liu, M., Song, G., & Atrens, A. (2008a). Influence of the β -phase morphology on the corrosion of the Mg alloy AZ91. *Corrosion Science*, 50(7), 1939–1953. <https://doi.org/10.1016/j.corsci.2008.04.010>
- Zhao, M. C., Liu, M., Song, G. L., & Atrens, A. (2008b). Influence of pH and chloride ion concentration on the corrosion of Mg alloy ZE41. *Corrosion Science*, 50(11), 3168–3178. <https://doi.org/10.1016/j.corsci.2008.08.023>
- Zhao, T., Hu, Y., He, B., Zhang, C., Zheng, T., & Pan, F. (2019). Effect of manganese on microstructure and properties of Mg-2Gd magnesium alloy. *Materials Science and Engineering A*, 765(August), 138292. <https://doi.org/10.1016/j.msea.2019.138292>
- Zheng, Q., & Kim, J.-K. (2015). *Graphene for Transparent Conductors*. <https://doi.org/10.1007/978-1-4939-2769-2>
- Zhou, M. Y., Ren, L. B., Fan, L. L., Zhang, Y. W. X., Lu, T. H., Quan, G. F., & Gupta, M. (2020). Progress in research on hybrid metal matrix composites. In *Journal of Alloys and Compounds* (Vol. 838). Elsevier Ltd.

APPENDICES

Appendix A: Journal Publications

1. **Abang Beniamin, A. M. H.**, Noor Mohamed, N. H., & Yusof, M. (2023). The Effect of Graphene Nanoplatelets Content on the Hardness of Mg6%Zn0.2%Mn Composites. *Pertanika Journal of Science and Technology*, 3, 1493–1502.

Appendix B: Experimental Equipment, Materials, and Setup



Mg powder, Mn powder, Zn powder and GNPs powder for sample preparation



GNPs powder being stirred in ethanol solution using magnetic stirrer



Hydraulic compressing machine



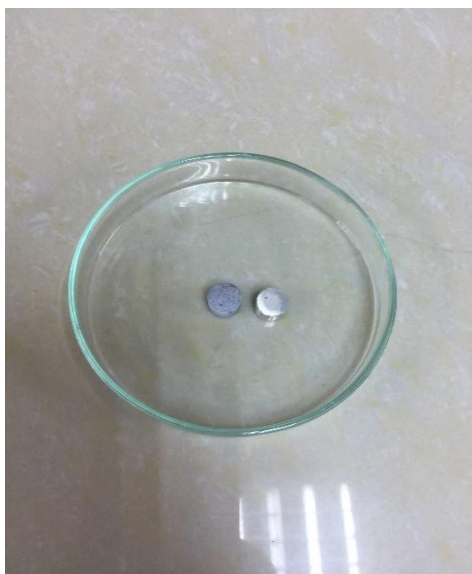
Sample compactor for powder metallurgy: consist of 1) Body 2) Die 3) Stopper 4) Compactor



Tube furnace with argon gas environment



Fabricated samples after sintered in the tube furnace



Unpolished HP Mg sample and polished HP Mg sample



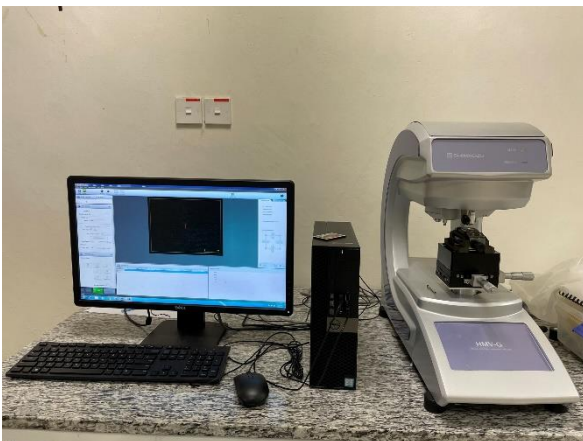
Samples are prepared to be mounted using epoxy resin



3D optical microscope



Scanning electron microscope (SEM)
with EDX



Vickers microhardness testing machine



Grinding and polishing machine



NaCl powder and $\text{Mg}(\text{OH})_2$ powder
used in immersion test



Experimental setup for immersion test



Dessicator used for store samples
after immersion test



Cr_2O_3 and AgNO_3 used for cleaning
samples to remove corrosion products