

THE INFLUENCES OF HOLMIUM ON THE SOLIDIFICATION,
MICROSTRUCTURE AND MECHANICAL PROPERTIES OF
ELEKTRON 21 MAGNESIUM ALLOYS

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DEDICATION

Teristimewa buat emak tersayang, Romlah Abu Bakar & baba, Shahizan Sudin..

Untuk adik-beradik tercinta..

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Shahril Akmar..*

Terima kasih atas sokongan dari semua sudut..

Ini untuk kalian..



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ABSTRACT

The solidification parameter of Elektron 21 magnesium alloys with various Ho concentrations were investigated using computer-aided cooling curve thermal analysis system, and the solidification microstructure and phase constitution of the investigated alloys were characterized by SEM, EDX, and XRD. The mechanical properties were also investigated comparatively. The Ho was selected due to the ability to develop new intermetallic phases together with Mg and have a positive potential to reflect the properties of Mg alloys. As the outcome from this study, the cooling parameter of Elektron 21 with lower addition Ho content, the nucleation and growth temperature of α -Mg in Elektron 21 magnesium alloy decreased, where the Ho changed the solution degree of Zn, which resulted in refinement of microstructure. The results of solidification parameters showed the addition of 0.083 and 0.16 wt. % Ho cause a decrease of the solidification temperature alloys, which lead to the grain size about 64.0 %, which being a most effective addition. The microstructure of the result indicated that the intermetallic phases in the Elektron 21 cast alloy consisted mainly of α -Mg matrix, $Mg_{12}Nd$, $Mg_{41}Nd_5$, Mg_3Gd and Mg_3Nd phase. The lower addition of Ho consists of Mg-Zn-Ho phase appears in the cast alloys. However, after adding 0.5 to 3.0 wt. % Ho, the Mg-Zn-Ho phase was suppressed and the phase has earlier been mixed Mg_2Ho and Mg_3Ho phase. Moreover, the addition of 0.083 wt. % of Ho exhibited an excellent improvement of ultimate tensile strength, yield strength and hardness of 139.7 MPa, 105.62 MPa and 103.52 Hv, respectively. The solidification, microstructure and mechanical properties of Elektron 21 magnesium alloys influenced by the Ho concentration in the magnesium alloys; Elektron 21 with 0.083 wt.% Ho exhibited the refinement of microstructure and displayed the best properties compared to other alloys.



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ABSTRAK

Parameter pemejalan aloi magnesium Elektron 21 dengan pelbagai kepekatan Ho telah disiasat dengan menggunakan sistem analisis haba lengkung penyejukan bantuan komputer, dan penyusunan mikrostruktur dan fasa perlembagaan aloi yang disiasat telah dicirikan oleh SEM, EDX, dan XRD. Sifat-sifat mekanik juga disiasat secara relatif. Ho dipilih disebabkan oleh keupayaannya untuk membentuk fasa baru bersama Mg dan mempunyai potensi yang positif terhadap sifat aloi Mg. Berdasarkan hasil dari kajian ini, ia didapati dari parameter penyejukan Elektron 21 dengan 0.083, 0.16 dan 0.32, nukleasi dan suhu pertumbuhan α -Mg dalam aloi Magnesium Elektron 21 menurun dengan penambahan yang lebih rendah, di mana Ho telah mengubah tahap pelarutan Zn dan mikrostruktur tuangan aloi adalah sangat ditapis dengan penambahan Ho yang lebih rendah. Hasil parameter menunjukkan penambahan 0.083 dan 0.16 % Ho menyebabkan penurunan aloi suhu pepejal, yang membawa kepada saiz bijian kira-kira 64.0 %, yang merupakan penambahan yang paling berkesan. Struktur mikro hasil menunjukkan bahawa antarafasa dalam Elektron 21 terdiri terutamanya daripada matriks α -Mg, $Mg_{12}Nd$, $Mg_{41}Nd_5$, Mg_3Gd dan Mg_3Nd . Penambahan yang lebih rendah Ho terdiri daripada fasa Mg-Zn-Ho yang muncul dalam aloi tuangan. Walau bagaimanapun, selepas menambah 0.5 hingga 3.0 % berat Ho, fasa Mg-Zn-Ho ditindas dan fasa sebelum ini telah bercampur fasa Mg_2Ho dan Mg_3Ho . Selain itu, penambahan 0.083 % Ho mempamerkan peningkatan tegasan mutakhir, pemanjangan dan kekerasan 139.7 MPa, 105.62 MPa dan 103.52 Hv. Pemisahan, mikrostruktur dan sifat-sifat mekanik elektron 21 aloi magnesium dipengaruhi oleh kepekatan Ho dalam aloi magnesium; Elektron 21 dengan 0.083 % Ho mempamerkan penghalusan mikrostruktur dan mempamerkan sifat terbaik berbanding dengan aloi lain



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LIST OF ABBREVIATIONS

ΔT_s	-	Solidification temperature range
Δt_s	-	Solidification time range
Ar	-	Argon
ASTM	-	American Society Testing Method
EDS	-	Electron diffraction spectroscopy
FKMP	-	Fakulti Kejuruteraan Mekanikal dan Pembuatan
Gd	-	Gadolinium
Ho	-	Holmium
HRE	-	Heavy rare earth
Hv	-	Hardness value
IQ	-	Icosahedral
Mg	-	Magnesium
Nd	-	Neodymium
RE	-	Rare earth
SAED	-	Selected area electron diffraction
SF6	-	Sulfur hexafluoride
TEM	-	Transmission electron microscopy
T_N	-	Nucleation temperature
t_N	-	Nucleation time
T_s	-	Solidification temperature
t_s	-	Solidification time
Zn	-	Zinc
Zr	-	Zirconium
α	-	alpha
β	-	beta
UTHM	-	Universiti Tun Hussien Onn Malaysia

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A List of publication



CHAPTER 1

INTRODUCTION

1.1 Background of study

Magnesium is the lightest structural metal, with a density 1.74 g/cm^3 , which found successfully use in a variety of applications (Brown, 2006; Luo, 2013a). In 1808, Sir Humphrey Davy discovered the magnesium oxide of a newly recognized metal (Neelameggham, 2013). The primary production of magnesium has greatly increased in the past decade, as shown schematically in Figure 1.1 (Wulandari *et al.*, 2010). In late 90's, China leads and being the main producer of primary magnesium production compared to other country (Wulandari *et al.*, 2010). It is used as an alloy material, where it can be formed by any of the bulk working process including castings, forgings, extruding, rolled sheet and plate (Brown, 2006).

As reported by United State Geological Survey, the leading use, which is 35 percentages for primary metal was aluminium-base alloys that used in packaging, transportation and other applications. Primary metal consumption accounted for 15 percentages of structural uses of magnesium (castings and wrought products). Consumption of primary metal on desulfurization of iron, steel and other uses sharing the same percentage which is 10 % (Bray, 2015).

Figure 1.2 shows the magnesium consumption in the last ten years and its forecast until 2015 (Luo, 2013a). The third most-commonly used structural metal is magnesium and followed by steel and aluminum (Luo, 2013b). As the lightest metallic materials, with its density about one-fourth of steel and two-thirds of aluminium, magnesium are considered to be promising candidate which offer significant oppurtunities for lightweight application in automotive, aerospace, power

tools, and 3C (computer, communication and consumer products) industries (Luo, 2013b; Wang *et al.*, 2016).

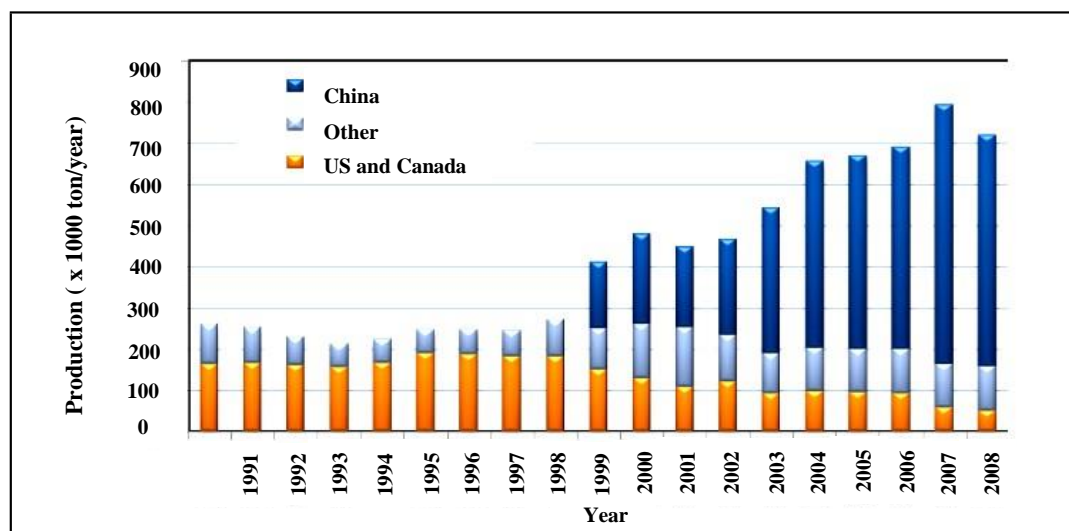


Figure 1.1: Primary magnesium production from International Magnesium Association (2010) (Wulandari *et al.*, 2010).

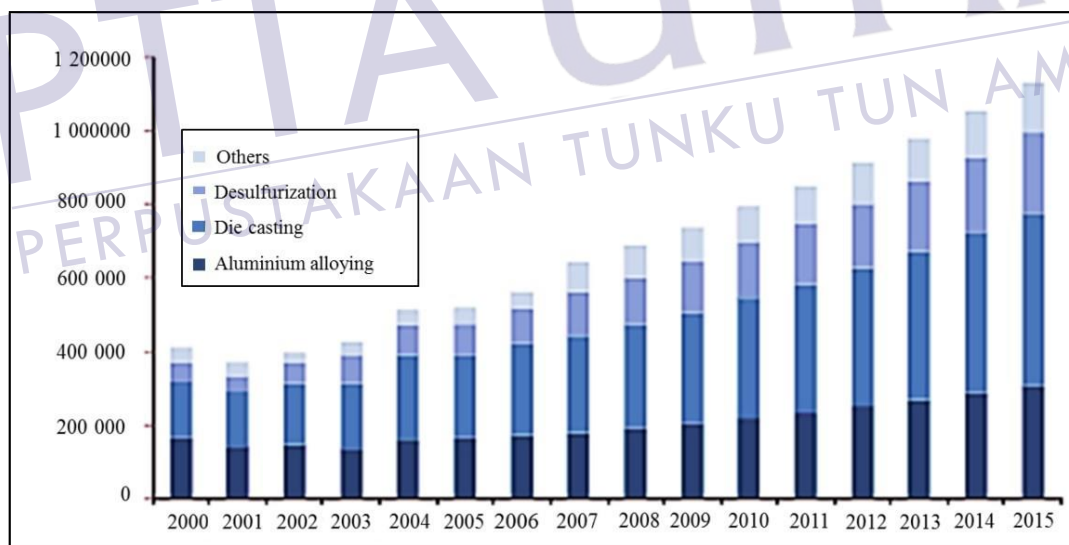


Figure 1.2: Magnesium consumption by end uses in 2000–2015 (Luo, 2013a)

Magnesium alloys are divided into two groups which is cast magnesium alloys and wrought magnesium alloys. More than 90% of structural components of magnesium alloys are produced by casting. Currently, casting is still the main industry forming method for magnesium alloys (Li *et al.*, 2011b; Sahoo & Thomas, 2011). Cast aluminium alloys have been extensively studied and served as structural components

in automobiles, aircraft, and computers due to their high specific strength and good castability (Zhang & Zhao, 2012).

Numerous studies on different addition into magnesium cast alloys have been discovered. A lot of researches have been developed for the approach to improving the strength of magnesium alloys by alloying elements. The addition of rare earth (RE) element into magnesium alloys were known for the excellent performance at both the elevated temperature and the room temperature (*Sun et al.*, 2015; *Zhang et al.*, 2012; *Zhang et al.*, 2015). Recently, by optimizing the thermo-mechanical process and the addition of trace amounts of rare earth elements are believed to improve the quality of properties, especially strength and ductility by microstructure optimization and various strengthening mechanisms, including grain refinement and grain boundary optimization (*Tekumalla et al.*, 2014). However, to our knowledge, the effect of holmium addition on the solidification, microstructure and mechanical properties of magnesium alloy has not been investigated. Therefore, expending holmium for future application is another reason for this study. Previous researcher had found (by reducing the amount of Holmium will optimize the microstructure due to well combination between Ho and the other element) the lower addition of Holmium will optimize the microstructure where Ho combined well with the other element where presence Ho-containing intermetallic phase and thus improved the corrosion resistance of Mg-9Al alloys (*Zhou et al.*, 2006). A moderate addition of holmium also significantly enhanced the strength and ductility as the microstructure refined and the fracture modes changed from partial brittle to ductile fracture (*Charles et al.*, 2016). It is known that the size of a grain of the alloy without RE is larger compare to the alloy with RE addition; therefore affect the mechanical properties of the alloys (*Liu et al.*, 2010; *Meng et al.*, 2015). Also, according to the existing literature, highlights that the precipitation of dense and fine rare earth containing dispersed phases within the matrix grains, and along the grain boundaries of these alloys generally strengthen the base material (*Bettles et al.*, 2009; *Zhu et al.*, 2010).

Recently, new alloys known as Elektron 21 alloys (contains of 2.5 weight percent neodymium, 1.5 weight percent gadolinium, 0.5 weight percent zinc, 0.5 weight percent zirconium and magnesium as remainder) seeking an attention among the researchers to introduce the new design of magnesium alloy. However, the work studies by Kielbus (2010), found the further aging treatment decreased the hardness

value due to the formation of $Mg_{41}Nd_5$ phase on the grain α -Mg grain boundaries. The majority work done with Mg-2.8Nd-1.5Gd-0.5Zn-0.5Zr or known as Elektron 21 magnesium alloys is focusing on the improvement of corrosion resistance and creep (Pikos *et al.*, 2014), constituent phase (Kielbus *et al.*, 2010) and corrosion (Kielbus, 2007)(Kannan *et al.*, 2008). However, there are no findings on the effect of rare earth metal addition on the Elektron 21 magnesium alloys. Therefore, this study is focusing on the benefits obtained by monitoring the solidification parameters of metallurgical phase transformation together with the microstructural behaviour and mechanical properties with different addition of Ho.

1.2 Problem statement

Magnesium alloys have received the broad attentions in the industry due to their competitive strength to density ratio, but poor ductility and strength has limit wide range of its applications as engineering materials. Magnesium alloys containing rare earth elements (RE) have great application potentials in the fields of automobiles and aerospace due to their high specific strength and better heat-resistant property compared with those without RE addition. Holmium (Ho) is known as one of promising element in the heavy rare earth group and used as an alloying element especially magnesium. It has a high solid solubility in the magnesium and there is a potential of Ho to form a larger solution which contribute to the enhancement of the properties of alloys. Recently, a lot of studies focusing on the processing and improvement of the properties on magnesium alloys, however, only fewer investigations concerned on the effects of the RE on the solidification parameters together with the corresponding microstructure and properties had been conducted. As a result, the application of magnesium cast alloys containing rare earth element is still hindered by lack of knowledge about their solidification characteristic together with the corresponding microstructure and properties.

1.3 Objectives

The objectives of this research are:

- i. To study the effect of Ho on solidification parameters of metallurgical phases.

- ii. To investigate the role of Ho on microstructure and formation of intermetallic phases.
- iii. To collerate the effect on mechanical properties and behaviour with the finding on microstructure and thermal analysis.

1.4 Scopes of the study

In order to realize the above objectives the following scopes was formulated.

- a) Mg-Nd-Gd-Zn-Zr (Elektron 21) magnesium alloy was used in this present investigation as a base alloy was provided from the Magnesium Elektron (MEL).
- b) The composition of Nd (2.6-3.1 wt. %), Gd (1.0-1.7 wt. %), Zn (0.2-0.5 wt. %), Zr (saturated) and Mg as the remainder were used in this study as base alloy.
- c) The addition of Holmium in the Elektron 21 magnesium alloys is 0, 0.083, 0.16, 0.32, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 wt. %.
- d) The casting experimental was conducted in the Foundry Lab UTHM.
- e) The solidification of the sample was obtained based on recording analysis of the temperature versus time data using Computer-aided cooling curve analysis (CA-CCA).
- f) Microstructural and phase analysis studies of magnesium alloy using optical microscopy, scanning electron microscope and X-ray diffraction.
 - i) Optical microscope (OM) to determine the quantitative information from the grain size and volume fraction of the sample
 - ii) Scanning Electron Microscope (SEM) with Energy Dispersive Spectrometer (EDS) is a qualitative and quantitative analytical technique to determine chemical composition.
 - iii) Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). X-ray diffraction is used to identify and characterize the compounds based on the diffraction pattern.

- g) Vicker's Hardness Test and Ultimate Tensile Strength Test (UTS) are used to obtain the mechanical properties of the samples and provide the quantitative measurement.

1.5 Significant of study

Generally, a study on the magnesium casting process is relatively new and through this study, it will give us the opportunity to improve the knowledge of the mechanism of the casting process. The higher demands of magnesium alloys allow us to improve the way of casting method to produce the alloys with an excellent property. A quality of casting was identified in this study by monitoring the solidification, together with the corresponding microstructure and mechanical properties of the alloys. Therefore through this study, it is beneficial approach especially for foundry engineers, once the solidification, microstructure and mechanical properties is understood, it is capable to improve the existing alloys and also it may applied in the field of various applications for real.

1.6 Outline of thesis

This thesis was divided into five chapters. Chapter 1 will cover background of the study in this research. It will brief the introduction of magnesium and the effect of rare earth in this solidification method corresponding to the microstructure and mechanical properties. This chapter also includes problem statement and related to the objectives of the research. There are some limitations when running this experimental which were further explained in the scope of study. The important and purpose of the study were briefly explained in significant of the research. After an introduction of the research background the previous literatures were reviewed in Chapter 1, Chapter 2 begins with a concise introduction which supported by detail overview of previous theoretical studies in effects of rare earth elements on the solidification, microstructure and mechanical properties of magnesium alloys. Studies include the magnesium alloys theories, the effect of rare earth on microstructure and mechanical properties of magnesium alloys. The solidification behavior in magnesium alloys is also summarized as well in Chapter 2 from previous

studies. Chapter 3 reported the experimental procedures and technologies in details. In Chapter 4, the results of experimental are presented, including the solidification parameters of Holmium as an addition of Mg-2.8Nd-1.5Gd-0.5Zn-0.5Zr (Elektron 21) magnesium alloys, the X-ray diffraction analysis of alloys as confirmation of the phase occurred, scanning electron microscope to analyzed the chemical composition of cast alloys, optical microscope to measure the grain size and volume fraction of cast alloy. Also, Chapter 4 were covered the tensile test and hardness test results from the experiment. In Chapter 5, the conclusion and recommendation were made based on the presented result from the previous chapter.



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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Recently, magnesium (Mg) has become a focus and succeed in attracting the automotive industry which contribute to fuel efficiency and environmental conservation and also for aircraft construction (Meng *et al.*, 2015). The manufacturer is always looking forward with the improvement on magnesium itself, especially in saving the weight without losing the material properties (Hao *et al.* 2012). Compared to the other structural metal, magnesium has the lowest densities density (1.8 g/cm^3) which is over four times lighter than steel (7.85 g/cm^3) and 35 percent lighter than aluminium (2.7 g/cm^3) (Hamdy *et al.*, 2012). Magnesium offers an excellent casting ability, high specific strength and stiffness, good machining ability, good weldability and also having integrated recycling possible, compared to aluminum and steel (Davis, 1997; Fergus *et al.*, 2015).

Magnesium is an alkaline earth metal and it is the 8th most abundant element which constituting 2% of the total mass of the earth's crust and dissolved in seawater (Kainer, 2006; Luo, 2013a). In the 19th century, the first metal was produced using an electrolysis of a mixture of magnesia and mercury oxide (Luo, 2013a). Nowadays, the study of magnesium has been developed and broadly used in the engineering application, especially in an automotive and aerospace. However, magnesium rarely used in a pure form itself in the engineering application without being alloyed with other metals. Recently, the addition of rare earth element is widely used to obtain the strong and lightweight where are needed for structural uses (Avedesian & Baker, 1999). The introduction of rare earth addition into the magnesium alloys displayed an



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excellent result which led to the grain refinement and also the mechanical properties (Zhu *et al.*, 2013).

2.2 Alloys designation

Magnesium alloys usually referred to their American Society for Testing and Materials (ASTM) designations which, together with their temper designations, are given in a four-part code (Koshal, 2014). Determination of the International Code for magnesium alloys is still not established. The methods used by ASTM B275 (standard practice for collection of certain non-ferrous metals and alloys, cast and wrought) have a tendency to prevail. In this system, the main alloying element refers to both first letter (refer to Table 2.1). In the nominal weight percent composition of a number of these elements will be rounded to the nearest whole number. Generally it refers to increasing the quality and suffix letters A, B, C are given chronologically. In this study, EV31A or known also as Elektron 21 is an alloy containing approximately 3 percent rare earth and 1 percent gadolinium

Table 2.1: ASTM codes for magnesium's alloying elements (Avedesian & Baker, 1999; Koshal, 2014; Mathaudhu *et al.*, 2016)

Letter alloying element	Alloying element	Letter alloying element	Alloying element
A	Aluminium	N	Nickel
B	Bismuth	P	Lead
C	Copper	Q	Silver
D	Cadmium	R	Chromium
E	Rare Earth	S	Silicon
F	Iron	T	Tin
H	Thorium	V	Gadolinium
J	Strontium	W	Yttrium
K	Zirconium	X	Any other
L	Lithium	Y	Antimony
M	Manganese	Z	Zinc

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