

**PROCESSING AND EVALUATION OF AN INVESTMENT CAST
MAGNESIUM-BASE ALLOYS**

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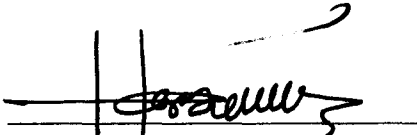
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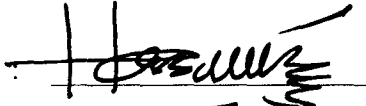
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Buat Ma dan anak-anak yang tabah.

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ABSTRACT

The research reported in this thesis was conducted in two phases. The first phase considered the suppression of mould-metal reaction. The approaches were to: cast the alloy with four pouring and mould preheat temperature combinations i.e. 680°C and 780°C pouring temperatures, and 300°C and 600°C mould preheat temperatures in five different mould refractory materials. CaF₂ was then used as an inhibitor with the most suitable of the five refractory materials and the moulds were cast with the best pouring/mould preheat temperature combination. The results showed that the best casting was produced by pouring the alloy at the temperature combination of 680°C and 300°C pouring and mould preheat temperature respectively in a zirconia refractory mould without an inhibitor. Incorporating CaF₂ inhibitor in the mould at levels of 20 wt% and 5 wt% of the zirconia poured at 680°C and 300°C, pouring and mould preheat temperatures respectively, produced a casting with a relatively bright metallic appearance. Further experimentation on the addition of 20 wt% of CaF₂ in a zirconia mould poured in the temperature range between 680°C and 780°C and with a 300°C mould preheat temperature revealed that the mould-metal reaction occurred above the pouring temperature of 740°C. The second phase concentrated on establishing the procedure for grain refinement and considered mould inoculation and vibration methods to grain refine the alloy. The effect of two melt conditions, i.e. zirconium-treated and zirconium-free melts, was investigated for four moulds, which had mould face coats containing 0%, 0.5%, 5% and 10% zirconium. The melts were poured at a temperature of 740°C in moulds preheated to 300°C. The investigation using vibration was conducted for two melt conditions, namely zirconium-treated and zirconium-free melts. Four mould vibration conditions were considered: no vibration, and vibration at frequencies of 5, 9 and 13 Hz. The results showed that none of the in-mould additions exhibited any effect on grain refinement for either of the melts. The investigation on the effect of vibration showed that the grain size decreased as the frequency was increased for both the zirconium-treated and zirconium-free melts. Mechanical properties were determined for samples produced using three casting conditions: zirconium-treated melt vibrated at a frequency of 13 Hz; zirconium-treated melt without vibration and a zirconium-free melt without vibration. The results showed that the highest yield strength and hardness were 69.31 MPa and 63.86 Hv respectively. These were obtained from the tensile test sample produced from the melt treated with zirconium and vibrated at the frequency of 13 Hz. Correspondingly, the grain size was found to be the smallest with a grain size of 56.9 µm when compared to the other castings.

ABSTRAK

Penyelidikan yang dilapurkan di dalam tesis ini telah dijalankan dalam dua fasa. Fasa pertama menekankan kepada menghentikan tindakbalas acuan-logam. Pendekatan yang diambil ialah dengan: menuang aloi tersebut ke dalam lima jenis bahan acuan pada empat kombinasi suhu penuangan/acuan, iaitu suhu penuangan 680°C dan 780°C dan suhu acuan 300°C dan 600°C. Penuangan juga dijalankan pada kombinasi suhu penuangan/acuan dan bahan acuan yang terbaik dengan campuran CaF₂. Keputusan menunjukkan bahawa tuangan yang terbaik dihasilkan dari bahan acuan zirconia dan dituang pada kombinasi suhu 680°C dan 300°C masing-masing suhu penuangan dan acuan. Mencampurkan CaF₂ pada kadar 20% dan 5% berat zirconia dan menuangnya pada suhu kombinasi 680°C dan 300°C masing-masing suhu penuangan dan acuan menghasilkan tuangan yang berpermukaan berkilau. Ujikaji selanjutnya yang dijalankan pada acuan yang dicampurkan dengan 20% CaF₂ dan dituang pada suhu antara 680°C dan 780°C ke dalam acuan yang dipanaskan pada suhu 300°C menunjukkan tindakbalas acuan/logam berlaku di atas suhu 740°C. Fasa kedua penyelidikan menekankan kepada mengujudkan prosedur untuk menghasilkan butiran halus dengan mengambilkira kaedah pembenihan acuan dan getaran ke atas dua keadaan cairan logam iaitu zirkonium terawat dan zirkonium bebas. Bagi penggunaan kaedah pembenihan acuan, penyelidikan dilakukan ke atas empat acuan yang dicampurkan samada dengan 0%, 0.5%, 5% atau 10% zirkonium pada permukaan sebelah dalam acuan. Penyelidikan menggunakan kaedah getaran, empat keadaan berbeza digunakan, iaitu tanpa getaran, getaran pada 5, 9 dan 13 Hz. Bagi kedua-dua kaedah, cairan logam dituang pada suhu 740°C ke dalam acuan yang dipanaskan pada suhu 300°C. Keputusan ujikaji kaedah pembenihan acuan memperlihatkan tiada sebarang kesan diperolehi pada kedua-dua keadaan logam. Bagi ujikaji getaran, didapati bahawa semakin tinggi frekuensi getaran maka semakin halus bijirin tuangan. Keadaan ini berlaku bagi kedua-dua keadaan logam cair. Sifat mekanikal diperolehi dari sampel yang dihasilkan dari tiga keadaan penuangan: zirkonium terawat digetar pada frekuensi 13 Hz; zirkonium terawat tanpa getaran dan cairan bebas zirkonium tanpa getaran. Keputusan ujikaji menunjukkan bahawa kekuatan alah dan kekerasan tertinggi masing-masing bernilai 69.31 MPa dan 63.86 Hv diperolehi dari sampel yang dihasilkan dari logam yang dirawat dengan zirkonium dan digetarkan pada frekuensi 13 Hz. Bersamaan dengan keputusan tersebut, saiz butiran yang dihasilkan adalah yang paling halus dan bersaiz 56.9 µm dibandingkan dengan tuangan lain.

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NOMENCLATURE

Chemical Symbols

AZ91A	- Magnesium-Aluminium 9%-Zinc
$\text{Al}_2\text{O}_3\text{SiO}_2$	- Mullite
Al_4C_3	- Aluminium carbide
AlN	- Aluminium nitride
B_2O_3	- Boron trioxide
BF_3	- Boron Trifluoride
$(\text{C}_2\text{F}_4)_n$	- Teflon/Polytetrafluoroethylene
CaF_2	- Calcium fluoride
CO_2	- Carbon dioxide
Fe_2SiO_4	- Fayalite
Fe_3O_3	- Ferrum(II) oxide
FeCl_3	- Ferrum (III) Chloride
FeO	- Ferric oxide
H_3BO_3	- Boric acid
H_3PO_4	- Phosphoric acid
HCl	- Hydrochloric Acid
HF	- Hydrofluoric acid
HNO_3	- Nitric acid
KBF_4	- Potassium borofluoride
Mg_2Si	- Magnesium silicide
MgCl_2	- Magnesium chloride
MgO	- Magnesium oxide
MnCl_2	- Manganese chloride
Na_2ZrO_3	- Sodium zirconates
$\text{Na}_2\text{ZrSi}_4\text{O}_{11}$	- Sodium zirconium silicates

NH_4BF_4	- Ammonium boron tetrafluoride
NH_4HF_2	- Ammonium hydrogen difluoride
NH_4SO_4	- Ammonium Sulphate
OH^-	- hydroxyl
SF_6	- Sulphur hexafluoride
$\text{Si}(\text{OC}_2\text{H}_5)_4$	- tetraethylorthosilicate
SiO_2	- Silica
SO_2	- Sulphur dioxide
ZrO_2	- Zirconia
ZrSiO_4	- Zircon
ZRE1	- Magnesium-Zinc 3%-Rare Earth 3%-Zirconium 0.6%
Zr	- Zirconium

Greek Symbols

σ_{YS}	- Tensile yield stress
η	- Density

Abbreviations

AFS	- American Foundrymen Society
ASM	- American Society of Materials
ASTM	- American Society for Testing Materials
AVCC	- Audio-Video-Computer-Communication
CAD	- Computer Aided Design
CAM	- Computer Aided Manufacturing
CNC	- Computer Numerical Control
CS	- Replicast Ceramic Shell
FDM	- Fused Deposition Modelling
GWP	- Global Warming Potential
IMA	- International Magnesium Association
LOM	- Laminated Object Manufacturing
MEL	- Magnesium Elektron

SEM	- Scanning Electron Microscope
SLA	- Stereolithography
SLS	- Selective Laser Sintering
UTS	- Ultimate Tensile Strength
UV	- Ultra Violet

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CHAPTER I

INTRODUCTION

The low density of magnesium alloys, at approximately 1.8-1.85 g/cm³, makes them particularly suitable for applications in which weight reduction is important. These include transportation, specifically automotive and aeronautical, and electronics products, especially those that are portable. In addition to low density, magnesium alloys also offer ease of machining, excellent castability, good weldability, superior damping characteristics and excellent electro-magnetic interference shielding

In the automotive industry, gradual increase in weight in the range of 25% to 30% [Porro and Beatrice, 1996] has been caused by the adaptation of the product to ever-stricter safety standards and to increasing customer demands for vehicle comfort, performance and reliability. The attainment for low-fuel consumption and emission reduction has been difficult. It has been estimated that a 10% reduction in weight yields an approximate 5.5% improvement in fuel economy. The use of magnesium was seen to provide an alternative route to compensate the increase in weight and fulfil the demands imposed on the automotive industry.

The aeronautic industry used magnesium alloys for reasons of weight reduction. However, in the past few decades it has changed to aluminium alloys. The drop in magnesium demand in the 1970's and 80's and the change to aluminium was attributed to deficiencies in magnesium alloys that were discovered at that time. The most quoted deficiency was poor corrosion resistance. The situation was worsened

by the lack of high strength alloys with good fatigue and creep properties. The wide availability of aluminium alloys at comparatively lower prices was also one of the decisive factors why aluminium was preferred for structural material. With the factors related to the size of aircraft, emission regulations and the requirements of economy in the construction and operation of aircraft, there are today signs of reversion to the use of components manufactured from magnesium [Zeumer, 1998].

Advances in melting technology and the development of new alloys to cater for more stringent applications and corrosion resistance has reduced concerns surrounding the use of magnesium. The development of fluxless melting, using a small amount of a mixture of SF₆ and CO₂ gas, by Hanawalt [1975] [Hanawalt and Okada, 1972] contributed to a major breakthrough in the melting of magnesium. Defects due to melting flux inclusions, which act as the nucleating agent for corrosion, can be eliminated. The preconceptions about corrosion behaviour which were responsible for the sharp decline in magnesium consumption in the 80's, can now be largely overcome by the development of high purity alloys, the use of special protective coatings and design. The high price of magnesium is a function of the volume of material used, as use increases the price should decrease and magnesium will become competitive with aluminium and engineering plastics. In contrast to other materials, the price of magnesium is very stable due to the large, mature material supply base and well developed secondary markets [Ruden and Albright, 1994].

At the cutting edge of info-communications technology, equipment is being downsized and made portable. Properties such as low density, damping capacity, electrical conductivity and thermal conductivity have made magnesium alloys ideal for attaining such design objectives.

Currently, most magnesium castings are produced by die casting although sand casting is still used. More than 90% of the shipments of magnesium alloy are consumed in the manufacture of high pressure die castings [Edgar, 2000] as pressure die casting caters for the manufacture of precision parts in large volumes. Sand casting, which is traditionally lower in cost but produces poor surface quality and dimensional accuracy, mainly caters for low volume production. It has also been

used for prototyping die cast components [Vecchiarelli, 1992] along with plaster moulding [Fantetti and Thorvaldsen, 1991]. With the current demand for the economic production of parts, there is a niche for the production of low volume, high precision components. This research is aimed at providing a process for the production of low volume, high precision parts using the investment casting process.

Traditionally, the investment casting process has used silica-base materials, in particular alumino-silicate ($\text{Al}_2\text{O}_3\text{-SiO}_2$), for mould production. It is unfortunate that, due to the high reactivity characteristics of magnesium, reactions at the mould-metal interface are likely to happen. These reactions cause serious problems for the casting of magnesium in investment casting moulds and must be eliminated or minimised to a tolerable degree if the full potential of the process is to be exploited. When casting magnesium in investment moulds, or other refractory aggregate moulds, inhibitors are used to suppress the metal mould-reaction. In investment casting, a KBF_4 inhibitor incorporated in the mould combined with mould flushing using sulphur dioxide gas, or, alternatively, mould flushing with a SF_6/CO_2 gas mixture have been used. For sand casting, sulphur and boric acid have been added in small quantities as inhibitors mixed with the moulding sand. The mechanism of suppression of mould-metal reactions in sand casting may be by/in the form of: the formation of a protective film on the surface of the molten magnesium alloy as it enters and takes the form of the mould; the formation of a protective atmosphere in the mould cavity; or by the formation of a protective atmosphere around the grains forming the mould [AFS, 1965]. This may also be the basis of inhibition for use in investment casting.

The processing of magnesium by the investment casting process often requires that the mould be heated to a high temperature with the objective of assisting the filling process, especially when thin sections are involved. However, this causes slow cooling which generates a relatively coarse grain despite the presence of zirconium as the grain-refining agent. Therefore, there is also a need to find alternative means to enhance the grain refinement with zirconium or at least restrict grain coarsening.

1.1 Problem Statement

There is a need to establish a successful inhibiting procedure or suitable investment mould material to overcome the mould - metal reaction that occurs during the pouring of magnesium alloy into alumino-silicate investment casting moulds. There is also a need to maximise the mechanical properties of investment cast magnesium alloys through the enhancement of the grain refinement process and control of the production process variables. Process optimisation, through enhancement of the grain refining process and control of mould and melt temperature, should maximise the mechanical properties of the alloy.

1.2 Objectives of the Research

The principal objectives of the research are as follows:

- i. To develop a mould/inhibiting procedure that effectively suppresses metal-mould reactions ;
- ii. To maximise the mechanical properties of the alloy by process optimisation considering:
 - a. Grain refinement enhancement;
 - b. Metal and mould temperature control;
- iii. To recommend a practical mould production procedure and metal treatment/processing route for casting of magnesium alloy.

1.3 Importance of the Research

The continuing pressure to reduce component weight in the automotive, aerospace and electronic products industries has generated renewed interest in magnesium alloys. However, processing of magnesium alloys by casting is currently limited to die or sand casting. The former caters for high volume, high precision part

manufacture and the latter for low volume, low precision production with reasonably acceptable finish. Therefore, there is a specific need to develop a low volume production, high precision process to cater for a specific demand.

1.4 Scope of the Research

The research was conducted within the following limits:

1. The magnesium base alloy investigated was zirconium-containing ZRE1 alloy.
2. The casting process was the ceramic shell, lost wax investment casting process.

1.5 Thesis Outline

The thesis is presented in 8 chapters. Chapter I highlights the general aspects of magnesium alloys, the demand areas and their associated problems, particularly in melting and casting. Chapter II describes the various aspects of magnesium alloys with emphasis given to the melting technology of magnesium, mould-metal reactions associated with casting in sand aggregate moulds and grain refinement methods. The various approaches used for suppression of mould-metal reactions for other alloys, especially in titanium casting, are also highlighted. The methodology for microstructural analysis and testing procedures for magnesium alloys are briefly presented.

The investment casting process used in the research is presented in Chapter III. The chapter highlights the capabilities and the drawbacks of the process, classification of the process, the materials normally used and the technology involved in the investment casting process.

The experimental programme used to achieve the objectives mentioned in Chapter I is presented in Chapter IV. The chapter describes the experiments conducted for the two phases of programme; suppression of mould-metal reactions and grain refining of magnesium alloy with vibration and in-mould methods. It also highlights the general procedures used in mould production, melting and casting, microstructural analysis, hardness testing and tensile testing.

Chapter V describes the results of the experiments conducted in Chapter IV. It consists of 8 sections and is presented in the form of descriptions, tables, photographs, micrographs and graphs.

Chapter VI presents the discussion of the results presented in Chapter V. It also consists of 8 sections. Chapter VII concludes the findings based on the results and discussion described in Chapter V and VI respectively. Finally, Chapter VIII outlines the suggestions for future work.

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