

THE EFFECT OF ROLLING TEMPERATURE ON THE PROPERTIES OF  
BIODEGRADABLE MG-0.7CA ALLOY FOR IMPLANT APPLICATION

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To my beloved parents and my siblings

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

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## ABSTRAK

Aloi magnesium sebagai bahan implan biodegradasi telah diperkenalkan kepada ortopedik dan pakar pembedahan trauma sejak beberapa abad yang lepas. Magnesium tulen biasanya mempunyai saiz bijian yang besar. Fenomena ini boleh menyebabkan kekuatan pada bahan tersebut menjadi semakin lemah dalam sifat-sifat mekanik. Oleh sebab itu, dengan mengaloi magnesium tulen, ia boleh menjadikan bahan lebih kuat khas untuk sifat-sifat mekanik dan struktur. Dalam projek ini, kesan suhu mengelek ke atas sifat-sifat aloi Mg0.7Ca biodegradasi dikaji. Aloi telan dileburkan dan dituang dengan menggunakan teknik tuangan die graviti dan digelek dengan lima suhu yang berlainan 165°C, 225°C, 285°C, 345°C dan 405°C dengan pengurangan sebanyak 1mm, 2mm dan 3mm. Struktur mikro pada sampel dianalisis oleh optik dan imbasan mikroskop elektron bersama-sama dengan pembelauan sinar-X. Untuk menentukan sifat-sifat mekanik, aloi tuangan dan aloi gelek yang telah melalui ujian tegasan dan ujian kekerasan. Hasilnya, kadar purata tegasan tertinggi aloi gelek dicapai pada suhu 345°C dan nilainya adalah 158MPa dan aloi tuangan nilainya adalah 101MPa. Walaubagaimanapun, dengan peningkatan suhu dan pengurangan memberikan nilai kekerasan yang rendah. Untuk menentukan kelakuan kakisan aloi, ujian elektrokimia telah dijalankan ke atas sampel dalam simulasi bendalir badan. Hasil menunjukkan bahawa, semakin meningkat suhu mengelek ditambah dengan pengurangan, semakin meningkatlah kadar hakisan pada aloi. Walaupun proses mengelek memperbaiki kekuatan bahan, tetapi ia tidak memenuhi syarat kakisan aloi dalam implan biodegradasi.

## ABSTRACT

Magnesium alloys as biodegradable implants have been introduced to the orthopedic and trauma surgery since past several centuries. Pure magnesium tended a large grain size and as a result of these phenomena will be weakening in mechanical properties. Consequently, by alloying the pure magnesium makes the material stronger in state of mechanical properties and its structure. In this present project, the effects of rolling temperature on properties of biodegradable Mg0.7Ca alloy were investigated. Alloy was cast by gravity die casting and rolled with five different temperatures 165°C, 225°C, 285°C, 345°C and 405°C with reduction of 1mm, 2mm and 3mm. Microstructure of the sample was analyzed by optical and scanning electron microscope together with X-ray diffraction. To determine the mechanical properties, as-cast and as-rolled were through a tensile test and hardness test. As a result, the highest average maximum tensile stress was achieved at 345°C that is 158 MPa and as-cast was 101MPa. However, by increasing the temperature and reduction gives a low hardness value. To determine the corrosion behavior of the alloy, electrochemical test was conducted on the sample in simulated body fluid. The result shows, by increasing the temperature of the rolling process supported by reduction, were increasing the corrosion rate of some degree of the alloy. Although rolling process improved the strength, it does not satisfy the corrosion behavior of the alloy in implant application.

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

In 19<sup>th</sup> century the revolution of bone implants are very well known throughout the world and steel as a core material, produce a fastener element and implant into human body for centuries. From time to time, the scientists realize that steel is heavy and give a drawback to the human body. For example, steel made from nickel-plating steel and vanadium steel later exchanged the carbon steel materials as steel corrodes certainly in human body. Most of the biodegradable materials nowadays are made from polymeric and ceramic material. But, most of the material is unsatisfactory in terms of mechanical strength and biocompatibility. Furthermore, some of the material are not sustainable in a body fluid and corrode when implant in long period of time. The application of magnesium alloy is widely used in most industries such as automotive, biomedical, aerospace and microelectronic. This project emphasis on the microstructure of Mg0.7Ca with several electrochemical and mechanical tests. The experiments include

investigation of the properties of the material before and after rolling process. The temperature of rolling was be regulated and microstructure was monitor. Project involved two phases, the first phase concentrate on the composition of 0.7% wt of calcium that was added to pure Mg and cast using gravity diecasting process. The results of this project establish the mechanical properties, microstructure analysis and corrosion rate from the gravity diecasting process. The second phase was concentrate on rolling process with as-cast was preheat to the regulated temperature and on going the rolling process with difference reduction of thickness. Any significant result of the rolling process was inspected through a same test as the as-cast.

## **1.2 Background and Rational**

Magnesium alloys as biodegradable implants have been introduced to the orthopedic and trauma surgery since the past several centuries. Pure magnesium tended to have large grain size compare to magnesium alloy. As a result these phenomena will be weakening in mechanical properties. Consequently, by alloying the pure magnesium makes the material stronger in terms of mechanical properties and it structure. Previous study shows, adding with 0.7% calcium in pure magnesium will increase strength, refinement the grain size and optimize alloying that compatible with the requirement of biomaterial of implant application [9]. Moreover by increasing calcium contain, corrosion rate were decreasing potentially. However, because of the hexagonal close packed (HCP) structure of magnesium, ductility was poor in room temperature due to the limited slips planes. By increasing the temperature, it is simultaneously increase the slip in the slip system and indirectly strengthen the material properties.

The existing biodegradable materials for implant application such as stainless steel, cobalt-chromium alloys, titanium and titanium alloys and nickel-titanium alloys

have many drawbacks. For example stainless steel is slightly heavier than titanium although it is a corrosion resistance material. Meanwhile, for titanium and its alloys is more expensive compared to magnesium alloys. In general, biomaterials interact directly with human body and there are several failures that attack human body during interaction. Toxicity and corrosion are examples that can harm human body, as a result can cause a bone cancer and neighboring tissues. It was observed that small percentage of Ca additions (0.2 and 0.5% Ca) distinctly improve the corrosion resistance, but further Ca addition (1% Ca) increases the corrosion rate [13].

Because of the light weight behavior with density  $1.74\text{g/cm}^3$ , magnesium is considered a non-ferrous metal that can be most attractive material to some researchers compared with aluminium that is slightly heavier with density  $2.7\text{g/cm}^3$ . Magnesium alloy is not a new material for biomedical industry. In addition, for implant medical application, the use of metallic material in orthopedic is one of the options to replace bone fracture since magnesium alloy is similar with natural bone. The structure of the magnesium alloy is not only depending on the material behavior, but also related to the material processing. In rolling process, the rolling temperature and rolling speed is one of significant factors to investigate either by rolling the material below the recrystallization temperature will give a fine grain structure or vice versa.

### **1.3 Problem statement**

Since magnesium is a biodegradable material and rolling is a secondary process to produce plate in an implant application, rolling of Mg-0.7Ca (%wt) alloy is still lacking below the recrystallization temperature (cold and warm working), since the cold working has many advantages compared with hot working, less study was reported on mechanical

properties of Mg-0.7Ca (%wt) after rolling process. Besides that, there was inconsistent study of the intermetallic phases that appear at the grain boundaries during the rolling process with a various temperature. In addition, study of intermetallic phases that influence to failure is still less in the industry of biomaterial.

#### **1.4 Objectives**

- i. To determine the effect of rolling temperature on a microstructure, mechanical properties and biocompatibility of Mg-0.7Ca (%wt).

#### **1.5 Scope of Study**

The study was conducted within the scopes as follow:

- i. The magnesium alloy investigated was Mg-0.7Ca (%wt).
- ii. The casting of Mg-0.7Ca was conduct with gravity diecasting technique.
- iii. The works rolling temperature was 165°C, 225°C, 285°C, 345°C and 405°C and regulated speed at 2 mm/min

- iv. The responds of experiment was:-
- a) Microstructure of as-cast and as-roll
  - b) Corrosion rate (mm/yr) for different temperature
  - c) Mechanical properties of Mg-0.7Ca (hardness and tensile)

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