THE PROPERTIES OF HOT EXTRUDED BINARY MGNESIUM CALCIUM ALLOY FOR USE AS BIODEGRADABLE MATERIAL WITHIN BONE

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To my beloved mother and father

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In the name of God, the most Gracious, the most Merciful

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ABSTRACT

The development of the suitable strategies to enhance the normally poor corrosion resistance and mechanical properties of magnesium alloys is an important issue that must be solved in order that magneiusm-based materials have wider biomedical applications. The work described in the thesis consists of two processing part: 1) Mg-xCa alloy preparation; in which five different mixture of commercially pure Mg (99.999%) and granules of Mg-40%Ca were die-cast and 2) extruding one of the as-cast Mg-xCa alloy from the casting process with optimum properties resulted from tests accomplished, at ram speeds of 26, 52 and 65rpm and temperatures 250°c, 350°c and 450°c. Optical microscopy observations of as-cast products and compositional analysis accomplished by EDS and X-RD showed that Mg-xCa (x= 0.7%, 1%, 1.9%, 2.8% and 3.6%) were composed of two phases, α(Mg) as the grain matrix and Mg2Ca on the grain boundaries. The hardness test results also showed that the Vickers hardness rates will increase by increasing the Ca content in the Mg-Ca alloys which was mainly due to the grain refinement and increment of Mg2Ca volume hindering the dislocations movements; According to the corrosion potential values, in-vitro corrosion rate and pH values, it was found out that binary Mg-Ca alloys with greater Ca contents corrodes faster than alloys containing less Ca; thus, increases the pH of the SBF solution and hydrogen evoluted from cathodic reaction more than others, that is predominantly due to grain refinement and increased Mg2Ca by adding more Ca. Finally by comparing the results acquired from tests and findings of Li et al. (2008) the Mg-1%Ca was selected as the the best compromise between mechanical properties, degradation behavior and biocompatibility of the implant. Since the higher ram speeds in extrusion develop finer grain structure (Y. Uematsu et al. 2006) it was expected that the corrosion rate decreased by grain refinement, but based on the results of electrochemical and immersion test conducted on as-extruded Mg-1%Ca products, the corrosion rate is lowest for the temperatures around 350 ºc and ram speeds of approximately 50 rpm. The hardness rates of the samples were enhanced at higher ram speeds and lower processing temperatures which is mainly because of grain refinement and possible phase precipitations.

ABSTRAK

Pembangunan strategi yang sesuai untuk meningkatkan ketahanan terhadap korosi biasanya miskin dan sifat mekanik dari gabungan magnesium merupakan isu penting yang harus dipecahkan supaya bahan magneiusm mempunyai aplikasi berasaskan bioperubatan yang lebih luas. kerja yang diperihalkan di dalam tesis ini terdiri daripada dua bahagian pemprosesan: 1) xCa gabungan Mg-persiapan, di mana lima campuran yang berbeza Mg komersil murni (99,999%) dan butiran Mg-40% Ca adalah mati-cast dan 2) satu ekstrusi dari gabungan Mg-xCa as-cast dari proses tuangan dengan sifat optimum yang diperolehi dari ujian selesai, pada kelajuan ram 26, 52 dan 65rpm dan suhu 250 ° c, 350 ° C dan 450 ° C. pemerhatian mikroskop optik produk sebagai-cor dan analisis komposisi semua oleh eds dan X-RD menunjukkan bahawa Mg-xCa (x = 0,7%, 1%, 1.9%, 2,8% dan 3,6%) yang terdiri daripada dua fasa, α (Mg) sebagai butiran matriks dan Mg2Ca pada batas butir. Keputusan ujian kekerasan juga menunjukkan bahawa tahap kekerasan Vickers akan meningkat dengan kenaikan kandungan Ca, Mg-Ca gabungan yang terutama disebabkan oleh pembaikan gandum dan kenaikan volume Mg2Ca menghalang gerakan dislokasi; Menurut nilai-nilai potensi korosi, di -vitro laju korosi dan nilai pH, dijumpai bahawa gabungan Mg-Ca binari dengan isi corrodes Ca lebih besar lebih cepat dari gabungan mengandungi kurang Ca, dengan demikian, meningkatkan pH larutan SBF dan hidrogen evoluted dari reaksi katodik lebih dari yang lain, bahawa adalah terutamanya kerana perbaikan gandum dan meningkatkan Mg2Ca dengan menambah lebih Ca. Akhirnya dengan membandingkan keputusan yang diperolehi dari ujian dan Penemuan Li et al. (2008) Mg-1% Ca dipilih sebagai kompromi terbaik antara sifat mekanik, degradasi perilaku dan biokompatibilitas implan. Kerana kelajuan ram yang lebih tinggi di ekstrusi mengembangkan struktur butir yang lebih halus (Uematsu Y. et al 2006). Diharapkan bahawa laju korosi menurun perbaikan butir, tetapi berdasarkan hasil elektrokimia dan perendaman dilakukan pada saat-diekstrusi Mg-1 % Ca produk, laju korosi terendah untuk suhu sekitar 350 º c dan kelajuan ram sekitar 50 rpm. tingkat kekerasan dari beberapa sampel yang dipertingkatkan pada kelajuan ram yang lebih tinggi dan suhu pemprosesan yang lebih rendah terutama karena perbaikan gandum dan hujan fasa mungkin.

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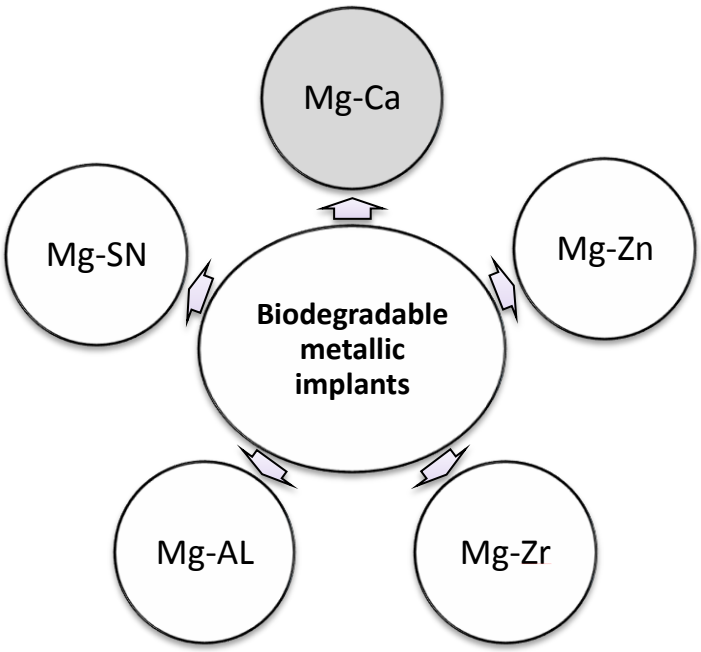
CHAPTER 1

INTRODUCTION

1.1 Background of the research

Modern medicine is hardly imaginable without biomaterials, that is biocompatible materials. It is a long time that replacing a part or a function of the body in a safe, reliable, economic, and physiologically acceptable manner is assisting the medical industry in proving more facilities for the patients suffering from different deseases. For this purpose different metallic and non-metallic biomaterials have been introduced; among which the metallic biomaterials are used as orthopedic prosthesis, mini plates and screws, and surgical tools. Titanium, stainless steel and cobalt which are classified as the permanent metallic biomaterials have the most applications in orthopedic implantation; but producing some problems, such as physical irritation, their inability to adopt wit growth, accumulation of metal in tissues and repeating the surgery, by these perpetual implants has motivated the researchers to work on the materials that can overcome the deficiencies. Consequently, magnesium based alloys with the ability to dissolve readily in aqueous solution (especially if contains chloride ion), were introduced as biodegradable implants. Most of the researches conducted on Mg-Al alloy system and Mg-RE alloy

system show better performance, compared to polymers, but neurotoxicity of Al and hepatotoxicity of rare elements motivate using calcium as an alloying element. But based on the fact that all the mechanical and chemical properties of materials are influenced by the chemical composition and processing conditions of the material, in order to improve the magnesium based alloys as biodegradable implants, the most compatible material selection and processing condition must be conducted to find the optimum composition and corresponding fabrication condition such as casting and forming variables.



1.2 Problem statement of the research

The degradability of magnesium and magnesium alloys in a physiological environment makes them desirable biodegradable biomaterials in many applications. However, their fast degradation rates in the human body and some problems such as neurotoxicity of Al and hepatotoxicity of rare elements impose a severe limitations

on using Mg-Al and Mg-RE; So Mg-Ca binary alloy which has compatibility to human body may be able to be used as biodegradable material.

At the same time, while rolling and forging can be used to enhance the properties of magnesium alloys and overcome the casting defects, inadequate surface finish, relatively not holding close tolerances and higher wastages plus higher costs for larger quantities constrain the application of these processes for use in implants production. Extrusion can be considered as a better alternative by offering extra benefits such as good flow of material, holding close tolerances, minimal waste, good surface finish specially at high speeds and maintaining mechanical properties.

1.3 Objectives of the project

Based on the problems defined in section 1.2 and the fact that all the mechanical and electrochemical properties and biocompatibility of materials are influenced by the microstructure of the material which is a function of chemical composition and processing conditions; the aim of this research is to study:

1) the effect of calcium content on the microstructure, mechanical and electrochemical properties and biocompatibility of magnesium calcium alloys;

2) the relationship between the extrusion parameters (extrusion rate and temperature) and the microstructure, mechanical and electrochemical properties and biocompatibility of magnesium calcium alloys.

1.4 Scope of the project

The scope of this project is as follows:

Part One: Preparation of Magnesium- Calcium alloys with Five different calcium content by Die casting method using argon gas as protective atmosphere.

Part Two: Impact extruding the alloy containing the optimum calcium content.

Part Three: Testing and Analysis

i. Corrosion test measurement by:
    a. Immersion test (ASTM G31 – 72)
    b. Electrochemical test (ASTM G5)
ii. Compositional analysis by:
    a. X-Ray diffractometery (XRD)
    b. Energy dispersive spectroscopy (EDAX)
iii. Micro structural analysis by:
    a. Secondary electron microscopy
    b. Optical microscopy
iv. Mechanical properties measurements
    a. Hardness test

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