# MAGNESIUM BASED MATERIAL SYSTEM FOR ORTHOPAEDIC APPLICATION

### **A DISSERTATION**

Submitted in partial fulfilment of the requirements for the award of the degree

of

### MASTER OF TECHNOLOGY

in

# METALLURGICAL AND MATERIALS ENGINEERING

(With specialization in Materials Engineering)

By

SATISH JAISWAL



#### DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY ROORKEE

ROORKEE – 247667 (INDIA)

MAY 2016

#### **CANDIDATE'S DECLARATION**

I hereby declare that the proposed work presented in this dissertation entitled "Magnesium based material system for Orthopaedic Application" is in partial fulfilment of the requirements for the award of the degree of Master of Technology in Materials Engineering, submitted in the Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee is an authentic record of my own work carried out during the period of July 2015 to May 2016 under the supervision of Dr. Debrupa Lahiri, Assistant Professor, Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, India.

The matter presented in this dissertation has not been submitted by me for the award of any other degree.

Dated:

Place:

(Satish Jaiswal)

#### **CERTIFICATE**

This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief

Dr. Debrupa Lahiri Assistant Professor Metallurgical and Materials Engg Indian Institute of Technology Roorkee Roorkee-247667 (India) First and foremost, all praise is to God.

I would like to express my sincere and deep gratitude to my guide, Dr. Debrupa Lahiri, Assistant professor at Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, for their encouragement, suggestions, and continuous support during my M.Tech. Study at IIT Roorkee. I would like to thank my professor for giving the opportunity to work on my Master thesis. I am so fortunate to work under your supervision for my project and immensely grateful to all the effort taken by my professor throughout my project.

I am extremely thankful to Dr. Anjan Sil, Professor and Head of the department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, for his help to carry out this dissertation.

I am also thankful to Dr. Partha Roy, professor, Department of Biotechnology and Dr. Murali, Post doctoral fellow, Centre for Nanotechnology, Indian Institute of Technology Roorkee, for providing the cell culture facility with free access to his lab.

I also wish to express my appreciation to Mr. Manoj kumar R, Mr. Vijayesh kumar, Mr. Soumyaranjan Nayak, Ms. Pallavi Gupta and Ms. Ankita Bisht for reviewing my work and serving on my committee.

I cannot forget to thank all my group members especially Mr. Mukul Srivastava and Mr. Nikhil Mohandas for their help and support during my work. I will always remember the laughter and good times we had together.

Special thanks is given to my family for their love, continuous support, understanding and constant encouragement.

LIST OF FIGURESi	
LIST OF TABLESiii	
ABSTRACTiv	
CHAPTER 1: INTRODUCTION1	
CHAPTER 2: LITERATURE SURVEY5	
2.1 Introduction to biomaterials	5
2.1.1 Biomaterials: An overview5	
2.1.2 The journey of Biomaterials	5
2.2 Characteristic of metallic Biomaterials	7
2.2.1 Mechanical Properties	7
2.2.2 High Corrosion resistance	)
2.2.3 Excellent Biocompatibility9	)
2.2.4 Osseo-integration	)
2.3 Current Implant Materials10	)
CHAPTER 3: PLAN OF WORK14	
CHAPTER 4: EXPERIMENTAL DETAILS15	
4.1 Materials and Methods15	
4.1.1 Synthesis of Hydroxyapatite15	
4.1.2 Composite fabrication and sample processing17	
4.2 Physical characteristic of powders and composite	I
4.3 Characterization of corrosion behaviour	
4.3.1 Potentiodynamic polarization study19	
4.3.2 Immersion Testing	)
4.4 Mechanical characterization20	)
4.5 Biocompatibility and cell culture test20	)
CHAPTER 5: RESULT AND DISCUSSION	

## **CONTENTS**

5.1 Morphology of Hydroxyapatite	
5.2 Characterization of powders	
5.2.1 XRD Analysis	23
5.2.2 SEM and EDS Analysis	25
5.3 Characterization of the sintered compacts	27
5.3.1 Physical Analysis	27
5.3.2 XRD Analysis	
5.3.3 SEM and EDS Analysis	29
5.4 Mechanical Characterization	
5.4.1 Vicker's Hardness	
5.4.2 Compression Analysis	
5.5 Corrosion Analysis	34
5.5.1 Potentiodynamic Polarization Test	
5.5.2 Immersion Test	
5.6 Biocompatibility	41
CHAPTER 6 CONCUSIONS	44
CHAPTER 7 FUTURE SCOPE	45
CHAPTER 8 PRESENTATION	46
CHAPTER 9 REFERENCES	47

## **LIST OF FIGURES**

FIGURE NO.	TITLE	PAGE
1	The required characteristics of biomaterials for biomedical application	7
2	Flow chart for plan of work	14
3	Flow chart for synthesis of Hydroxyapatite	16
4	Processing route for fabrication of composite	17
5	SEM micrograph of HA powder	22
6	XRD Pattern of synthesized Hydroxyapatite powder	23
7	XRD Pattern of powder (a) Mg and, (b) Zn	24
8	SEM micrograph of powder (a) Mg (b) Zn and (c) Mixed and EDS of 5% HA powder (d) selected area micrograph, (e) Elemental analysis	26
9	Digital image of the sintered pellets	28
10	XRD pattern of sintered compacts	29
11	FESEM micrograph of fractured surface of all the sintered compacts (a-d)	30

12	Elemental mapping of Mg-3Zn-5HA pellet	31
13	Average hardness value for all the compacts	32
14	Stress-strain curve for compression testing	33
15	Representative curve for Potentiodynamic polarization test	35
16	Representative curve for mass gain after Immersion testing	37
17	Representative curve for mass loss after removal of the corrosion product	37
18	Concentration of Mg <sup>2+</sup> obtained from the SBF retrieved from the Immersion testing by Atomic Absorption Spectroscopy (AAS)	38
19	FESEM micrograph of all the immersed samples for different duration	39
20	The digital images of all the immersed samples for different duration	40
21	Fluorescence images of hFOB cells stained with Acridine orange (AO) after 1day of seeding on control (a), 5%HA (b) and 10%HA (c)control	42
22	Graph between cell viability and number of days shown for hFOB cell viability for day 1,3 and 5 of cell seeding on all the samples.	43

### **LIST OF TABLES**

TABLE NO.	TITLE	PAGE NO.
1	Three generation of biomaterials	6
2	Types of biomaterials in biomedical Application	7
3	Typical Mechanical Properties of Biomaterials	8
4	Cobalt based alloy	11
5	Classification of Stainless steel	11
6	Relative density values for all the composites	27
7	The result of compression testing	33
8	$I_{\text{corr}}$ and $E_{\text{corr}}$ value for representative Potentiodynamic polarization curve	35

#### **ABSTRACT**

The development of biodegradable implants has grown into one of the important areas in medical science. It has fascinated researchers because of the importance in supporting tissue revival and healing, which generally occurs by degradation of the biodegradable implant material and taking up of the place by in-growing new tissue. Its degradability becomes more important for the accessories used to support fractured and damaged bones. Biodegradable accessories help in avoiding the requirement of revision surgery to remove those accessories after healing is complete. The biodegradable orthopaedic materials available in the current market are mainly made of polymers or ceramics. These orthopaedic accessories have an unsatisfactory mechanical strength when used for load-bearing parts. Magnesium and its alloys can be suitable candidate for this purpose due to their outstanding strength to weight ratio, biodegradability, non-toxicity and mechanical properties, similar to natural bone. The major drawback of magnesium is bioactivity and low corrosion resistance in the body, which also influences its mechanical and physical characteristics in service condition. In the present study, an effort has been taken to improve the corrosion resistance, bioactivity and mechanical strength of biodegradable magnesium alloys by synthesizing a Mg alloy matrix composite reinforced with different mass fraction of thermally treated hydroxyapatite  $[Ca_{10}(PO_4)_6(OH)_2]$ . The composite is fabricated through powder consolidation route. All the powders were mixed thoroughly with the probe sonicator, cold-compacted and sintered at 550°C. Morphologies, structure and density of the powders and sintered samples were characterized by X-ray diffraction, scanning electron microscopy and helium pycnometer. The in vitro bio-corrosion (biodegradability) and mechanical behaviours of samples are investigated by electrochemical and compression tests. The experimental results showed that addition of HA could slow down the corrosion rate and improve the mechanical properties of biodegradable magnesium alloy. These studies give an insight on the effect of HA reinforcement on corrosion resistant and mechanical behaviour of composite. Keywords: Magnesium, Hydroxyapatite, Composite,