

Contents

List of Figures	xiv
List of Tables	xx
Abbreviations	xxii
Symbols	xxiii
Preface	xxiv
1. Introduction	1
1.1 Composites	1
1.2 Classification of composites	2
1.3 Processing routes for metal-matrix composites	5
1.3.1 Solid-state processing	6
1.3.2 Liquid-phase synthesis	7
1.3.3 In situ synthesis	8
1.3.4 Two-phase process	8
1.4 Corrosion in materials	9
1.4.1 Types of corrosion	10
1.4.1.1 Chemical/Dry corrosion	10

1.4.1.2 Electrochemical / wet corrosion	10
1.5 Applications of metal matrix composites	11
2. Literature review	14
2.1 Introduction	14
2.2 Magnesium	14
2.3 Magnesium based alloys and composite	15
2.4 Chemical properties of magnesium and its alloys	16
2.5 Characteristics of bioactive glasses and their applications	18
2.6 Biological applications of magnesium based composites	19
2.7 Processing methods of magnesium-based composites	28
2.8 Summary	29
3. Objective of the work	31
4. Materials and method	34
4.1 Introduction	34
4.2 Preparation of magnesium based alloy and composites	34
4.2.1 Preparation of magnesium alloy	35
4.2.2 Preparation of HAp	35

4.2.3 Preparation of S45P7 BAG	36
4.2.4 Preparation of 1393 BAG	37
4.3 Processing of raw materials to prepare different composites	37
4.3.1 Raw Materials	37
4.3.2 Weighing and mixing	42
4.3.3 Compaction	44
4.3.4 Sintering	45
4.4 Characterization techniques	46
4.4.1 Physical/Microstructure characterization	46
4.4.1.1 X-ray Diffraction (XRD)	47
4.4.1.2 Scanning electron microscopy (SEM)	49
4.4.1.3 Actual density, theoretical density and percentage relative density	51
4.4.2 Mechanical characterization	52
4.4.2.1 Hardness	52
4.4.2.2 Young's modulus	55
4.4.2.3 Flexural strength	57
4.4.2.4 Compressive strength	58

4.4.3 Electrochemical characterization	58
4.4.3.1 Preparation of Simulated Body Fluid	59
4.4.3.2 Corrosion tests (weight loss method)	60
4.4.3.3 Corrosion tests (potentiodynamic method)	62
4.4.3.4 Atomic absorption spectroscopy (Ion release estimation)	64
4.4.3.5 Fourier-transform infrared spectroscopy (FTIR)	66
4.4.4 Biological testing	67
4.4.4.1 Cell lines and cell culture	67
4.4.4.2 Cell viability assay	67
4.4.4.3 Cell growth inhibition assay	68
4.4.4.4 Cytotoxicity assay	68
4.4.4.5 Hemolysis assay	69
5. Synthesis and characterization of Mg₂₀Zn₂Mn based composite reinforced with HAp and S45P7 BAG	70
5.1 Introduction	70
5.2 Preparation of Composites	71
5.3 Results and Discussion	72
5.3.1 X-ray Diffraction (XRD)	72

5.3.2 Microstructure evolution	77
5.3.3 Fourier Transform Infrared Spectroscopy	81
5.3.4 Mechanical characteristics	84
5.4 Summary of the chapter	86
6. Synthesis and characterization of Mg₃Al₂Zn_{0.6}Ca._xBAG composite	88
6.1 Introduction	88
6.2 Results and Discussion	89
6.2.1 X-ray Diffraction (XRD)	89
6.2.2 Microstructure evolution	91
6.2.3 Physical and mechanical characteristics	99
6.2.3.1 Total Porosity and Densification	99
6.2.3.2 Mechanical characteristics	100
6.2.4 Electrochemical behavior	101
6.2.5 Ion release estimation	108
6.3 Summary of the chapter	110
7. Biological properties of Mg₃Al₂Zn_{0.6}Ca._xBAG composite	112
7.1 Introduction	112
7.2 Results and Discussion	113

7.2.1 Cell viability, growth inhibition & cytotoxicity assay	113
7.2.2 Hemolysis assay	117
7.3 Comparative analysis of mechanical and corrosion properties of bone tissues and materials for orthopedic implants	118
7.4 Summary of the chapter	119
8. Conclusions	120
Bibliography	123
List of publications	153

List of Figures

1.1 Classification of composites based on the reinforcement phase	3
1.2 Classification of composites based on matrix phase	3
1.3 Different routes for synthesis of metal matrix composites	6
4.1 Flow diagram for synthesis of different composites by powder metallurgy route	34
4.2 Digital image of magnesium powder	38
4.3 Digital image of zinc powder	38
4.4 Digital image of aluminum powder	39
4.5 Digital image of manganese powder	39
4.6 Digital image of calcium powder	40
4.7 Digital image of HAp powder	40
4.8 Digital image of S45P7 BAG	41
4.9 Digital image of 1393 BAG powder	41
4.10 Planetary ball mill	43
4.11 Die and punch assembly for preparation of pellets	44
4.12 Hydraulic press	45
4.13 Different characterizations techniques	46
4.14 Schematic of diffraction of X-rays by a crystal	47

4.15 X-ray diffraction measurement setup	48
4.16 Scanning electron microscopy setup	50
4.17 Vickers's hardness technique used for hardness measurement	53
4.18 Vickers's hardness equipment	54
4.19 Young's modulus NDT testing equipment	56
4.20 UTM machine for mechanical testing	57
4.21 Vacuum oven	61
4.22 Schematic Layout of experiment setup used for corrosion test by potentiodynamic method	62
4.23 Schematic of the three neck cell used for electrodes mounting	63
4.24 Tafel extrapolation technique for finding corrosion parameters	64
4.25 Atomic absorption spectroscopy setup	65
4.26 FTIR measurement set up	66
5.1 Flow diagram of preparation of composite specimens	70
5.2 Reference peak for zinc crystal	72
5.3 Reference peak for magnesium crystal	72
5.4 XRD peak of unfired base alloy (Mg ₂₀ Zn ₂ Mn)	73
5.5 XRD patterns of unfired alloy-bioactive glass composite in various proportions	74

5.6 XRD patterns for unfired alloy-HAp composite in various proportions	74
5.7 XRD patterns of unfired Alloy-HAp-bioactive glass composites	75
5.8 XRD patterns for fired Alloy-HAp composite	75
5.9 XRD patterns for fired Alloy- bioactive glass composite	76
5.10 XRD patterns for fired Alloy-HAp-bioactive glass composite	76
5.11 SEM image of sample containing HAp	77
5.12 (a) SEM images of composite with 10% HAp showing evolution of gases	78
5.12 (b) Shows HAp flakes on the surface of the sample HAp	78
5.13 SEM image of sample containing S45P7 BAG	79
5.14 (a) SEM images of sample with 5 % HAp and 5 % BAG spread over whole crystals	80
5.14 (b) SEM images of sample with 10 %HAp and 10% BAG	80
5.15 FTIR spectrum of HAp	81
5.16 FTIR spectrum of S45P7 bioactive glass when kept in SBF solution for 7 days	81
5.17 FTIR spectrum of S45P7 bioactive glass when kept in SBF solution for 14 days	82
5.18 FTIR spectrum of composite samples with reinforcement of (a) 10 wt% HAp, (b) 10 wt% bio-glass, (c) 5 wt% each of HAp and bio-glass and (d) 10 wt% HAp + 10 wt% bio-glass after 14 days of immersion in SBF	83

6.1 XRD pattern of Mg ₃ Al ₂ Zn _{0.6} Ca & Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG composites after sintering	89
6.2 XRD pattern of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after sintering	90
6.3 SEM images of samples after sintering (before immersion test) (a) Mg ₃ Al ₂ Zn _{0.6} Ca, (b) Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG, (c) Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG	91
6.4 SEM & EDS images of composites Mg ₃ Al ₂ Zn _{0.6} Ca a) 7 days b) 14 days c)EDS analysis and Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG d) 7 days e) 14 days f) EDS analysis	92
6.5 SEM with EDX images of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF for 1 days at magnification (a) 2000X, (b)5000X, (c)10000X, (d)20000X	93
6.6 SEM with EDX images of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF for 3 days at magnification (a) 1000X, (b)2000X, (c)5000X, (d)10000X	94
6.7 SEM and EDX images of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF for 5 days at magnification (a) 2000X, (b) 5000X, (c) 10000X, (d) 20000X	95
6.8 SEM and EDX images of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF for 7 days at magnification (a) 500X, (b) 2000X, (c)5000X, (d)10000X	96
6.9 SEM and EDX images of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF for 14 days at magnification (a) 2000X, (b)5000X, (c)10000X, (d)30000X	97
6.10 XRD pattern of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after 1,3,5,7 and 14 days of immersion in SBF	98

6.11 Potentiodynamic polarization curves of Mg ₃ Al ₂ Zn _{0.6} Ca alloy after immersion in SBF solution	102
6.12 Potentiodynamic polarization curves of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG composite after immersion in SBF solution	102
6.13 Potentiodynamic polarization curves of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite after immersion in SBF solution	103
6.14 Variation of corrosion rate of Mg ₃ Al ₂ Zn _{0.6} Ca & Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG after immersion for different durations in SBF solution	105
6.15 Variation of corrosion rate of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG after immersion for different durations in SBF solution	105
6.16 Release of Magnesium ions with immersion time for Mg ₃ Al ₂ Zn _{0.6} Ca & Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG composite	109
6.17 Release of Magnesium ions with immersion time for Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG composite	109
7.1 Effects of 0% 1393BAG, 10% 1393BAG, 15% 1393BAG and 20% 1393BAG against K562 cells with respect to proliferative potential (A), viability (B) and cytotoxicity (C) as judged by MTT, XTT and LDH release assay respectively	114
7.2 Effects of 0% 1393BAG, 10% 1393BAG, 15% 1393BAG and 20% 1393BAG against DL cells with respect to proliferative potential (A), viability (B) and cytotoxicity (C) as judged by MTT assay	115

7.3 Analysis of biocompatibility in lymphocyte and monocytes in the presence of 0% 1393BAG, 10% 1393BAG, 15% 1393BAG and 20% 1393BAG with respect to viability (A & B) and cytotoxicity (C & D) as judged by XTT and LDH release assay	116
7.4(A) Concentration dependent haemolysis of RBC in the presence of 0% 1393BAG, 10% 1393BAG, 15% 1393BAG and 20% 1393BAG. Bright field images of RBC treated with (B) 0BAG, (C) 10BAG, (D) 15BAG, and (E) 20BAG	117

List of Tables

2.1 Physical properties of magnesium	15
4.1 Composition of base alloys	35
4.2 Characteristics of materials used	42
4.3: Average particle size of base alloy and reinforcement	44
4.4 Ion concentrations of human blood plasma and SBF	59
4.5 Reagents used to prepare simulated body fluid	60
5.1 Alloy-reinforcement proportions	71
5.2 Phases detected in fired and unfired samples	77
5.3 Mechanical properties of different composites	85
6.1 Nomenclature of the composites prepared	88
6.2 Total porosity and densification of different composites	99
6.3 Mechanical properties of different composites	100
6.4 Corrosion potential, current density and corrosion rate of Mg ₃ Al ₂ Zn _{0.6} Ca in potentiodynamic test	103
6.5 Corrosion potential, current density and corrosion rate of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₀ BAG in potentiodynamic test	104
6.6 Corrosion potential, current density and corrosion rate of Mg ₃ Al ₂ Zn _{0.6} Ca ₁₅ BAG in potentiodynamic test	104

Abbreviations

AAS	A tomic A bsorption S pectroscopy
ASTM	A merican S ociety for T esting and M aterials
BAG	B io A ctive G lass
BSE	B ack S cattered E lectron
EDS/EDX	E nergy D ispersive X -ray
FTIR	F ourier T ransform I nfrared S pectroscopy
HAp	H ydroxy A patite
JCPDS	J oint C ommittee on P owder D iffraction
MMCs	M etal M atrix C omposites
OCP	O pen C ircuit P otential
SBF	S imulated B ody F luid
SEM	S canning E lectron M icroscopy
TCP	T ricalcium P hosphate
XRD	X R ay D iffraction

Symbols

$^{\circ}\text{C}$	Degree Centigrade
α	Alpha
β	Beta
θ	Theta
λ	Lambda
Hv	Vickers Hardness
I_{corr}	Corrosion current
E_{corr}	Corrosion potential
C_r	Corrosion rate
%	Percentage
©	Copyright
μm	Micrometer
nm	Nanometer
cm	Centimeter
Kg	Kilogram
G	Gram
min	Minute
Sec	Second
\AA	Angstrom
ρ	Density