

BIOCOMPATIBLE AND METALLURGICAL PROPERTY INVESTIGATION OF BIODEGRADABLE MAGNESIUM ALLOYS

Submitted in partial fulfilment of the requirements for the award of
Bachelor of Engineering in Mechanical Engineering

by

RAVIPATI SASI VENKATA SREE DATTA (37150154)

SAGIRAJU SRI SAI KRISHNA SUMANTH VARMA (37150159)



**DEPARTMENT OF MECHANICAL ENGINEERING
SCHOOL OF MECHANICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

**Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE
JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119**

APRIL – 2021



SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**



Accredited with Grade “A” by NAAC | 12B Status by UGC | Approved by AICTE
Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai - 600 119

www.sathyabama.ac.in

DEPARTMENT OF MECHANICAL ENGINEERING

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of Ravipati Sasi Venkata Sreedatta (37150154) and Sagiraju Sri Sai Krishna Sumanth Varma (37150158) who carried out the project entitled **“BIOCOMPATIBLE AND METALLURGICAL PROPERTY INVESTIGATION OF BIODEGRADABLE MAGNESIUM ALLOYS”** under my supervision from December 2020 to April 2021.

Internal Guide

Mr. J R. DEEPAK M.E., (Ph.D.,)

Head of the Department

Dr. G. ARUNKUMAR, M.E., Ph.D.,

Submitted for Viva voce Examination held on 19/04/2021.

Internal Examiner

External Examiner

DECLARATION

We RAVIPATI SASI VENKATA SREE DATTA and SAGIRAJU SRI SAI KRISHNA SUMANTH VARMA hereby declare that the Project Report entitled “BIOCOMPATIBLE AND METALLURGICAL PROPERTY INVESTIGATION OF BIODEGARADABLE MAGNESIUM ALLOYS” done by us under the guidance of Mr. J. R. DEEPAK M.E., (Ph.D.) is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering degree in Mechanical engineering.

DATE: 19/04/2021

1.

2.

PLACE: CHENNAI

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of SATHYABAMA for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to Dr. S. PRAKASH, M.E., (PhD), Dean, School of Mechanical Engineering and Dr. G. ARUNKUMAR, M.E., (PhD), Head of the Department, Department of Mechanical engineering for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide Mr. J R. DEEPAK M.E., (Ph.D.), for his valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Mechanical engineering who were helpful in many ways for the completion of the project.

I wish to express my thanks to my parents and my friends who helped me in the duration of project in many ways and lead me to successfully complete the project.

ABSTRACT

Magnesium alloys are biodegradable and biocompatible which are actively using in medical applications. Magnesium (Mg) based biodegradable materials possess same mechanical properties as that of bone. Because of its biodegradability in the physiological world, this is a promising replacement for permanent implants. Pure Mg has low mechanical properties, so it is alloyed with other elements. To increase these properties different types of metals are used as alloy. Many Mg alloys are used like Al, Ca, Zn, Sr and also Rare earth metals. The present investigation is on Mg alloys casted with Strontium and rare earth element Gadolinium. With ranging the percentages of the Sr and Gd new Mg alloys are casted. Mg-2Sr and Mg-2Sr-2Gd as a new alloy material for the XRD investigation. Pure Mg has a grain size of $35\pm 10\mu\text{m}$ addition of Sr decrease the grain sizes of the Mg-Sr $23.9\pm 8.3\mu\text{m}$. The decrease in grain size was observed in the both Mg-2Sr and Mg-2Sr-2Gd through optical microscope. Addition of Gd into the Mg-2Sr master alloy there is a further decrease of grain size from $23.9\pm 8.3\mu\text{m}$ to $20.5\pm 7\mu\text{m}$ Addition of Gd in the alloy enhanced the microstructure which leads to improved metallurgical properties.

TABLE OF CONTENTS

CHAPTER No.	TITLE	PAGE No.
	ABSTRACT	iv
	LISTS OF SYMBOLS AND ABBREVIATIONS	viii
	LIST OF FIGURES	ix
	LIST OF TABLES	xi
1	INTRODUCTION	1
	1.1 MAGNESIUM	1
	1.1.1 APPLICATIONS OF MAGNESIUM	2
	1.1.2 TYPES OF Mg ALLOYS	3
	1.1.2.1 CAST ALLOYS	4
	1.1.2.2 WROUGHT ALLOYS	4
	1.2 STRONTIUM	5
	1.2.1 APPLICATIONS OF STRONTIUM	6
	1.3 GADOLINIUM	6
	1.3.1 APPLICATIONS OF GADOLINIUM	7
	1.4 IMPLANTS	7
	1.4.1 TYPES OF IMPLANTS	8
	1.4.2 PERMANENT IMPLANTS	8
	1.4.3 TEMPORARY IMPLANTS	9
	1.5 NON DEGRADABLE IMPLANTS	9
	1.6 BIODEGRADABLE IMPLANTS	9
	1.6.1 TYPES OF BIODEGRADABLE IMPLANTS	10
	1.7 BIO-DEGRADABLE POLYMERS	11
	1.8 Fe BASED BIODEGRADABLE IMPLANTS	11
	1.9 Zn BASED BIODEGRADABLE IMPLANTS	12
	1.10 Mg BASED BIODEGRADABLE IMPLANTS	12
	1.10.1 Mg-Al BASED IMPLANTS	13

CHAPTER No.	TITLE	PAGE No.
	1.10.2 Mg-Ca BASED IMPLANTS	13
	1.10.3 Mg-Zn BASED IMPLANTS	13
	1.10.4 Mg-Cu BASED IMPLANTS	14
	1.10.5 Mg-Zr BASED IMPLANTS	14
	1.10.6 Mg-REE BASED IMPLANTS	14
1.11	STUDIES ON Mg-Sr ALLOYS	15
1.12	STUDIES ON Mg-Gd ALLOYS	16
1.13	STUDIES ON Mg-Sr-Gd ALLOYS	16
1.14	STUDIES ON XRD (X-RAY DIFFRACTION)	17
	1.14.1 BASIC PRINCIPLES OF XRD	18
	1.14.2 WORKING PRINCIPLE OF XRD	19
	1.14.3 APPLICATIONS OF XRD	19
	1.14.4 USES OF XRD	20
	1.14.5 LIMITATIONS OF XRD	20
1.15	STUDIES ON OPTICAL MICROSCOPE	20
	1.15.1 PRINCIPLES OF OPTICAL MICROSCOPE	21
	1.15.2 MAIN PARTS OF MICROSCOPE	21
	1.15.3 EYEPIECE	21
	1.15.4 TURRET	22
	1.15.5 OBJECTIVE	22
	1.15.6 FOCUS WHEEL	22
	1.15.7 FRAME	22
	1.15.8 LIGHT SOURCE	23
	1.15.9 CONDENSER	23
	1.15.10 STAGE	23
	1.15.11 WORKING PRINCIPLE OF OPTICAL MICROSCOPE	23

CHAPTER No.	TITLE	PAGE No.
	1.15.12 APPLICATIONS OF OPTICAL MICROSCOPE	24
2	LITERATURE SURVEY	25
	2.1 Mg-Sr ALLOY	25
	2.2 Mg-Gd ALLOY	26
	2.3 Mg-Sr-Gd ALLOY	28
3	AIM AND SCOPE	28
	3.1 AIM	29
	3.2 SCOPE	29
4	EXPERIMENTAL OR MATERIALS AND METHODS	31
	4.1 MATERIALS	31
	4.2 COLLECTION OF MATERIALS	31
	4.3 CASTING PROCESS	31
	4.4 XRD TESTS	35
	4.5 OPTICAL MICROSCOPY	36
5	RESULT AND DISCUSSION	37
	5.1 XRD RESULTS OF Mg-2Sr	37
	5.2 XRD RESULTS OF Mg-2Sr-2Gd	38
	5.3 FWHM OF Mg-2Sr AND Mg-2Sr-2Gd	40
	5.4 D VALUE OF Mg-2Sr AND Mg-2Sr-2Gd	42
	5.5 OPTICAL MICROSTRUCTURE	43
6	SUMMARY AND CONCLUSIONS	45
	6.1 SUMMARY	45
	6.2 CONCLUSION	45
	REFERENCES	47
	APPENDIX	50

LIST OF SYMBOLS AND ABBREVIATIONS

μm	MICROMETER
Al	ALUMINIUM
Ca	CALCIUM
CGR	COMPOUND GROWTH RATE
Cu	COPPER
Dy	DYSPROSIUM
Fe	IRON
g/cm^3	GRAM PER CENTIMETERCUBE
Gd	GADOLINIUM
La	LANTHANIDE
Mg	MAGNESIUM
Nd	NEODYMIUM
REE	RARE EARTH ELEMENT
Sr	STRONTIUM
T_g	GLASS TRANSITION TEMPERATURE
Wt%	WEIGHT PERCENTAGE
XRD	X-RAY DIFFRACTION
Y	YITTRIUM
Zn	ZINC
Zr	ZIRCONIUM

LIST OF FIGURES

FIGURE No	TITLE	PAGE No.
1.1	Magnesium (Mg)	1
1.2	Applications of Magnesium in various sectors	3
1.3	Classification of Mg alloys	5
1.4	Strontium (Sr)	6
1.5	Gadolinium (Gd)	7
1.6	Orthopaedic Implant Application	8
1.7	Temporary Implant	9
1.8	Trends of using biomaterials	10
1.9	Biodegradable implant	10
1.10	Polymer implant	11
1.11	Flow chart of types of Implants	17
1.12	XRD machine	18
1.13	Working principle of XRD	19
1.14	Optical microscope	23
1.15	Working principle of optical Microscope	24
4.1	Process of Mg-REE Gravity Casting	32
4.2	Preheating of Mould	33
4.3	Placing of crucible with Mg in furnace	33
4.4	Skimming Process	34
4.5	Adding of Strontium	34
4.6	Pouring of molten metal into mould	34
4.7	Mg-2Sr alloy	35
4.8	Test Samples	36
4.9	Scheme of Research Work	36
5.1	Net intensity graph of Mg-2Sr	38
5.2	Net intensity graph of Mg-2Sr-2Gd	40
5.3	Highest Intensity Peak Data comparison	40
5.4	FWHM Value of Mg-2Sr and Mg-2Sr-2Gd	41

5.5	D Value comparison	42
5.6	Microstructures of (a) Mg-2Sr and (b) Mg-2Sr-2Gd	43

LIST OF TABLES

TABLE No	TITLE	PAGE No.
1.1	Production of Mg in different countries	2
1.2	Names of the Mg alloys under ASTM standards	4
1.3	Effects of Different Alloying Elements on Mg	15
5.1	XRD results of Mg-2Sr	38
5.2	XRD results of Mg-2Sr-2Gd	40
5.3	Maximum intensity values of Mg-2Sr	43
5.4	Maximum intensity values of Mg-2Sr-2Gd	44

CHAPTER 1

INTRODUCTION

1.1 MAGNESIUM

Magnesium is an alkali metal group element. It is the lightest structural metal on the market. Construction, vehicle, industrial, and medical sectors all use these compounds. Magnesium is the earth's eighth most abundant element (about 2.5 percent). The Figure 1.1 shows the magnesium element used for various applications.



Figure 1.1 Magnesium (Mg)

Magnesium is commercially produced by electrolysis of molten magnesium. Mg is found in nature as a mixture of three isotopes: Mg29 (79%), Mg26 (11%), and Mg25 (10%). Mg is a chemical element as well as a vital mineral in the human body. It aids in hundreds of vital bodily functions. Mg in the body aids in the formation of new bone cells. Furthermore, studies say that it may help to prevent bone loss. In contrast, Mg helps in the heart's blood pumping. Magnesium has the ability to relax the blood vessel walls. The production of magnesium in different countries is tabulated in table 1.1.

Table 1.1 Production of Mg in different countries

Rank	Country	Magnesium Production (in tonnes)
1	China	800
2	Russia	65
3	U. S	50
4	Israel	25
5	Kazakhstan	23
6	Ukraine	19
7	Brazil	15
8	Turkey	10
9	Korea	10
10	Iran	5

1.1.1 Applications of Magnesium

Humphry Davy used electrolysis to separate a small amount of pure metal from magnesium oxide. However, it was a French physicist, Antoine-Alexandre-Brutus Bussy, who developed a considerable amount of the metal in 1831 by treating magnesium chloride with potassium and then researching its properties.

Historically, the Germans used mg in aircraft during World War I. The term "elektron" was invented by Germans and is still in use today .Mg is used in automotive designs as it is the lightest material. In 1955, Mercedes used Mg for the sports car championship with the 300 SLR. In 1971, Porsche used mg alloy frames in its 917/053 models and won the Le Mans race.

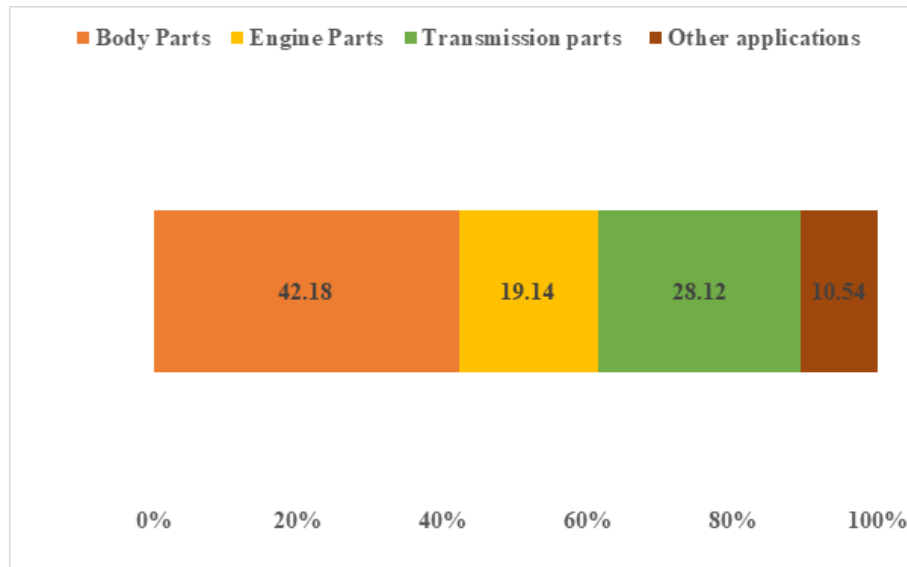


Figure 1.2 Applications of Magnesium in various sectors

Lambotte was the first to use pure Mg implants in 1906; however, due to widespread cavities due to gas, swelling and severe pain, the pure Mg plates had to be removed because the pure Mg implant deteriorated quickly in vivo and did not provide long-term mechanical support for fracture healing. Due to its low density and outstanding mechanical and electrical properties, magnesium is widely used in the manufacture of mobile phones, notebook and tablet computers, cameras, and other electronic components.

1.1.2 Types of Mg alloys

Magnesium alloys are two types cast alloys and wrought alloys. Mostly Mg cast alloys are used and research on wrought alloys are happening since 2003. Die-cast magnesium is used for camera bodies and lens part, cast Mg alloys are used for many components in modern automobiles and have been used in some high-performance vehicles. Casted Mg alloys are also used as implants in the medical industry as cardiovascular stents and orthopedic implants.

1.1.2.1 Cast alloys

Cast alloys are created by pouring molten metal into a mould and allowing it to solidify into the desired form. To increase the creep resistance mechanical properties, Mg is cast with other components. Magnesium cast alloys usually produce differing quantities of aluminium, manganese, and zinc as primary alloying elements, but never more than 10%. AZ63, AZ81, AZ91, ZK51, AM50, ZE41, AM60, HK31, QE22, QH21, WE54, HZ32, WE43, Elektron 21 are some of the types of casted Mg Alloys.

1.1.2.2 Wrought alloys

To achieve the desired form, wrought Mg alloys are produced using techniques such as forging, extrusion, and rolling operations. The primary alloying elements are aluminium, manganese, and zinc. Heat-treatable and non-heat-treatable magnesium wrought alloys are separated. AZ31, AZ61, AZ80, Elektron 675, ZK60, M1A, HK31, HM21, ZE41, ZC71, ZM21, AM40, AM50, AM60, K1A M1, ZK10, ZK20, ZK30, ZK40 these are some of the wrought Mg alloys.

Table 1.2 Mg alloys and its ASTM standards

Alloying Element	ASTM Standard
Mn	M
Al-Mn	AM
Al-Zn-Mn	AZ
Zr	K
Zn-Zr	ZK
Zn-Zr-REE	ZE
REE-Zr	EZ
Zn-Cu-Mn	ZC
Al-Si-Mn	AS

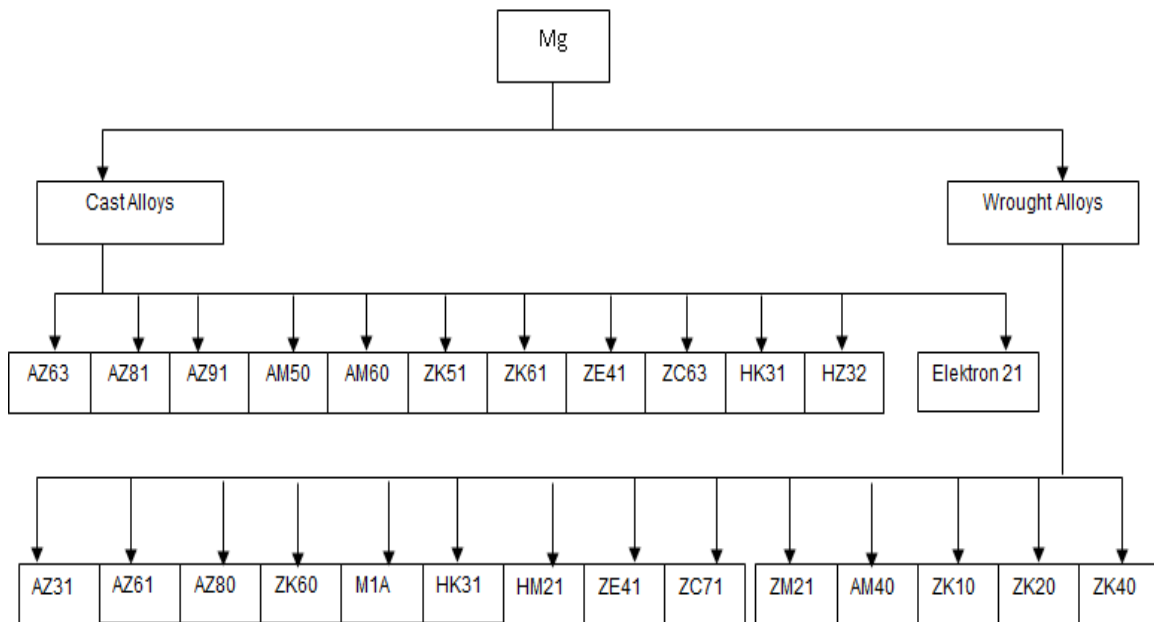


Figure 1.3 Classification of Mg alloys

1.2 STRONTIUM

Strontium is an alkaline earth metal which is extremely chemically reactive. It's a silvery-white yellowish shiny material smooth to touch. In a daily basis, the average human consumes about 2 milligrams of Sr. Sr ingested by adults appears to bind only to the surface of bones, but Sr may substitute calcium in the mineral of developing bones in infants, resulting in bone growth problems. Both strontium and strontianite are named after Strontian, a village in Scotland near which the mineral was discovered in 1790 by Adair Crawford and William Cruickshank.

It has a lighter consistency than calcium and a tougher consistency than barium. The melting point of strontium is 777° C, and the boiling point is 1377 degrees Celsius. Strontium only forms naturally in complexes of further elements, such as the minerals like strontianite and Celestine, owing to its intense reactivity to oxygen and water.



Figure 1.4 Strontium (Sr)

Strontium also bio accumulates in the body. Strontium increases bone density and decreases the possibility of vertebral, peripheral, and hip fractures.

1.2.1 Applications of Strontium

Sir Humphry Davy, an English chemist, was the first to isolate strontium in 1808 by electrolyzing a mixture of strontium chloride (SrCl_2) and mercuric oxide (HgO). Strontium is the 15th most plentiful metal on Earth and it is widely found in nature. Strontium is the 15th most plentiful metal on Earth, and it is found in abundance in nature.

The bright reds produced by strontium salts in fireworks and flares are well-known. It's also used in the manufacturing of ferrite magnets and the refinement of zinc. It can be used to produce electricity for space vehicles, remote weather stations and navigation buoys. Strontium chloride hexahydrate is used as an ingredient in tooth paste for sensitive teeth. As strontium is similar to the calcium it can penetrate into our body in the same way as calcium. Sr is a non-toxic element to the human body.

1.3 GADOLINIUM:

Gadolinium is a chemical element belongs to lanthanide group with the atomic number 64 and the symbol Gd. When the oxidation on gadolinium is extracted, it becomes a silvery-white metal. It only occurs in oxidised form in nature. Because of

their related chemical properties, it typically contains impurities of other rare-earths when isolated. Although gadolinium has no large-scale applications, it does have a wide range of specific applications. Green phosphors for colour TV tubes are also made from gadolinium compounds. Gadolinium is used as a biomaterial like vascular stents, orthopaedic implants by alloying with the other materials.



Figure 1.5 Gadolinium (Gd)

1.3.1 Applications of Gadolinium

The mineral gadolinium was named after Finnish chemist and geologist Johan Gadolin. It's unlikely for the metal to occur in nature because it's so reactive. Surprisingly, as previously mentioned, the mineral gadolinite only contains traces of this chemical.

Gd has very useful applications in alloy material Gd can increase the workability of alloys as well as their tolerance to high temperatures and oxidation. Gd is used for making of magnets electronic components and data storage disks. Gd is used is used in MRI scans as it reflects the image of the scan accurately. Gd is also non-toxic to the human body. China, the United States, Brazil, Sri Lanka, India, Australia are the major mining fields, with deposits estimated to reach one million tonnes.

1.4 IMPLANTS

Implants are the type of devices or tissues that are used to replace in the body when the actual part is getting damaged or lost by providing support to the organs. These

damages may be fractures, breakages or tissue damages etc. Most of the implants takes places in medical industry are orthopedically implants and dentistry implants.



Figure 1.6 Orthopedic Implant applications

1.4.1 TYPES OF IMPLANTS

There are many types of implants in medical industry. But the average usage of implants is in orthopaedic, dentistry, cardiovascular implants are mostly in the usage in every country. Dentistry implants are normal implants where the replacement of the tooth roots is made using metallic implants. These are removable and made to match our natural teeth. Whereas the cardiovascular implants are called as stents which are placed in artery walls which gives support to open the artery wall and improve the flow of blood to heart. This is one of the most important type of implants. And orthopaedic implants which are widely using in medical industry more than other two types of implants that discussed before. Orthopaedic implants are of two types.

- I. Permanent Implant
- II. Temporary Implants

1.4.2 Permanent Implants

Permanent orthopaedic implants are non-removable and placed in serious conditions. Some of them are Spinal Cord Implants, Knee joint replacement etc...

1.4.3 Temporary Implants

Temporary orthopaedic implants are removable and replaceable implants. These are used for bone breakages, fractures because these are regrowing in nature and they removed after the bone healing. In these temporary implants there are two types of materials are used.

- I. Non-Degradable Material
- II. Biodegradable Material

1.5 NON DEGRADABLE MATERIALS IMPLANTS

These implants are made of Non Degradable materials which have to be removed with another surgery after the healing process completed. The present medical industry uses mostly these non-degradable implants because of limited availability of the degradable implants. Most of the times these non-degradable implants are successful replacements. Due to the secondary surgery or to reduce the cost of the second surgery both patients and doctors are showing their interest on the biodegradable implants. How temporary implants are placed can be seen in the Figure 1.7.



Figure 1.7 Temporary implant

1.6 BIODEGRADABLE MATERIAL

A biodegradable material is a type of material where it undergoes degradation in the biological conditions without causing any affects to the surroundings. Biodegradable metals are designed to degrade in the body after performing their job inside the body.

The use of biodegradable material is that they don't need to be removed after the healing or no need of second surgery. These can also be used on children or young growing patients.

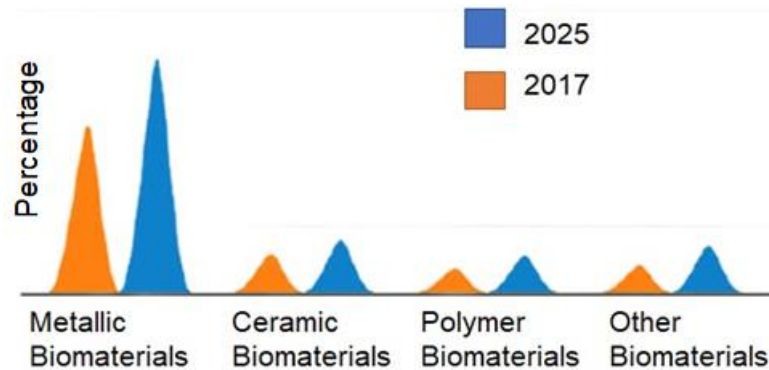


Figure 1.8 Trends of using biomaterials.

This Figure 1.8 shows the usage of biomaterials in 2017 and how compound growth rate (CGR) will increase by 2025. According to the surveys conducted the CGR will be 6.5% by the end of year 2025. This shows the interest towards the biomaterial implants. Figure 1.9 shows the biodegradable implant in orthopaedic surgery. The Figure 1.9 shows the placing of Biodegradable implant.



Figure 1.9 Biodegradable implant

1.6.1 Types of Biodegradable Materials

- I. Bio-degradable Polymers
- II. Iron (Fe) based biodegradable implants.

- III. Zinc (Zn) based biodegradable implants.
- IV. Magnesium (Mg) based biodegradable implants.

1.7 BIO-DEGRADABLE POLYMERS

Polymers are macromolecules made up of repeating monomers that are covalently bonded together. The chains in these compounds can be linear, branched, or cross-linked with other chains, and they can be amorphous or crystalline. Temperature affects polymer properties, so it's crucial to make biodegradable polymers with a glass transition temperature (T_g) higher than body temperature, as polymers become extremely flexible above their specified T_g . One of the most prevalent biomaterials used for bone repair and tissue engineering is biodegradable polymers. We can see the polymer implants in the Figure 1.10.

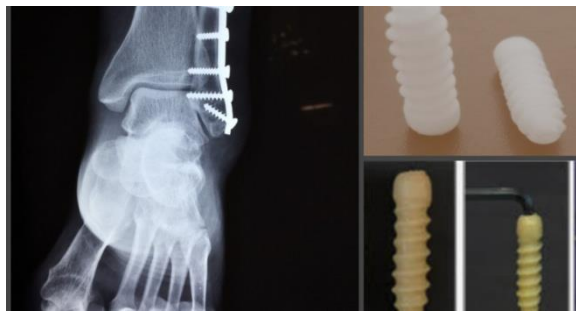


Figure 1.10 Polymer implant

Their biodegradability and regulated decomposition rates make them ideal for clinical use. Based on their nature, polymers may be classified as natural or synthetic. More crystalline materials have stronger intermolecular bonding and decay more slowly than amorphous polymers because crystallinity is a measure of organisation, relationships, and packaging in a substance. Natural polymers are primarily used to repair small bone fractures that do not impose high loads on the implant materials due to their intrinsic low strength.

1.8 Fe BASED BIODEGRADABLE IMPLANTS

Because of their slow decomposition, iron-based biodegradable implants are hard to come by. Iron-based implants degrade slowly, taking years to fully degrade. Another

issue with iron-based implants is their low corrosion resistance and mechanical properties. Iron cannot withstand the loads that are effective to the bone because of its low mechanical properties. Because of the low degradation levels, it remains in the body after the healing process is finished, causing problems for the patient by collecting in various tissues and being unable to bear the heavy loads on the site due to low mechanical properties.

1.9 Zn BASED BIODEGRADABLE IMPLANTS

In 2013, zinc-based biodegradable implants became famous. Zinc alloys, with reported yield strength of up to 300 MPa, are not as solid as titanium or stainless steel, despite the fact that their mechanical properties can be modified to meet specific specifications.

The degradation products of zinc alloys were found to be biocompatible. They are used very little in the industry because of their slow lifetime, which exceeds the anticipated healing times. Also, making standards of strength that are less than ideal. It's possible that the lack of inflammation can harm the body's tissues.

1.10 Mg BASED BIODEGRADABLE MATERIAL

Mg has received a great deal of interest as a biodegradable implant in recent decades. Mg and its alloys have been studied as bio-implant materials in a number of ways.

Mg and its alloys can provide better support and initial biochemical stability than biodegradable polymers. Mg is non-toxic and can even aid in the recovery of hard tissue after transplantation in the human body. Mg and some of its alloys are also attractive candidates for biodegradable implant applications.

The density of Mg-based metals (1.7–2.0 g/cm³) matches closely with the density of bone (1.8–2.1 g/cm³). Whereas the densities of other metals (titanium and stainless steel) are much higher or much lower. Mg exhibits elastic modulus that of similar to the human bone. Mg's biodegradability eliminates the need for revision surgery after implantation. This will prevent the conventional implant material from damaging the

bones during removal. Alloying is widely used to strengthen the properties of multiple elements. As we discussed in Mg and its alloys Mg is alloyed with some elements. The purpose of Mg alloying is to improve the mechanical, metallurgical, and corrosion resistance properties of the metal. Aluminium (Al), zirconium (Zr), strontium (Sr), zinc (Zn), calcium (Ca), and rare earth elements (REEs) such as yttrium (Y), gadolinium (Gd), lanthanum (La), and dysprosium (Dy) are the most widely utilized alloying elements. There are different affects with different elements used for alloying.

Mg-Al based implant

Al is a common alloying element in Mg, with concentrations ranging from 2 to 9 wt% in commercially available Mg-Al alloys. To improve both strength and corrosion resistance, Mg is alloyed with Al. The addition of Al in Mg strengthens the strength of Mg–Al alloys by promoting both solid solution and precipitation strengthening.

Although the mechanical strength and corrosion resistance of Mg–Al alloys are positive characteristics, there are some issues about Al's biocompatibility. The accumulation of Al in the body can cause neuronal injuries under certain circumstances, so a greater concentration of Al is undesirable.

1.10.1 Mg-Ca based implant

Calcium is one of the most essential minerals in bone structure. Ca is an attractive alloying element in Mg for biomedical applications because of this fact. The alloying of magnesium with calcium refines grain sizes, resulting in improved mechanical properties for magnesium. But increase content of Ca exceeding limit may create secondary phases in the alloy. Faster degradation of alloy leads to the problems of hydrogen evolution and mechanical strength should be addressed first.

1.10.2 Mg–Zn-based implants

Zn is one of the most abundant and nutritionally important components in the body. Solid-state and precipitation strengthening are both enhanced when Mg and Zn are alloyed. When compared to pure Mg, the alloying of Mg with Zn revealed an increase in corrosion resistance. The properties begin to deteriorate as the Zn content

increases. Zn is a preferred alloying element for Mg in biomedical applications due to its biocompatibility and excellent strengthening properties.

1.10.3 Mg–Cu-based implants

Cu has been used to improve bacterial resistance in a variety of alloys, including stainless steel and cobalt base alloys, but its alloying with Mg has received little attention. Cu is an antibacterial ingredient that plays a role in bone resorption and the immune system. Cu added above the tolerance limit has a negative impact on Mg corrosion resistance by causing localised corrosion. Despite the fact that the rate of degradation of Mg alloys increases as the concentration of Cu increases, it demonstrated excellent antibacterial properties without compromising biocompatibility.

1.10.4 Mg-Zr based implants

In Mg, zirconium (Zr) is the most widely used grain refiner. When Zr is added to Mg, it produces highly refined equiaxed grains. It also outperforms pure Mg in terms of corrosion resistance. Although Zr has a low solubility limit of 3.8 wt.% in Mg, it is suitable for alloying with Mg due to its excellent grain refinement property and good corrosion resistance. If we contribute more than 3.8 percent Zr, the particles remain unalloyed Zr.

1.10.5 Mg-REE based implants

In Mg alloys, REEs have been widely investigated as alloying components to increase creep resistance, corrosion resistance, and mechanical strength. When Mg-REE alloys were compared to Mg-Al, Sn, Ca RE elements Gd, and Nd alloys, the grain size of Mg-REE alloys was found to be improved. The addition of some REEs to Mg alloys will considerably increase corrosion resistance. RE element Gd showed excellent results on corrosion resistance when the content of Gd is kept fewer than 10%. Although Mg–REEs are not recommended for biocompatibility, a few studies have shown that they are biocompatible. Three Mg–RE alloys, Mg–La, Mg–Nd, and Mg–Gd, were tested for biocompatibility and found to have no adverse effects on

tissues. Mg alloys containing REEs had nearly the same biocompatibility as pure Mg, indicating that REEs are a good alloying material for degradable Mg alloys used in stents and orthopaedic implants. . Effects of the different alloying elements can be seen in Table 1.3.

Table 1.3 Effects of Different Alloying Elements on Mg

Element	Improved Mechanical Properties
Al	Yield strength, Tensile strength, Hardness, Elongation
Ca	Yield strength, Tensile strength, Bending strength, Hardness, Elongation, compressive strength
Zn	Yield strength, Tensile strength, compressive strength, Hardness, Elongation
Sr	Yield strength, Tensile strength
Zr	Yield strength, Tensile strength, compressive strength, Hardness, Elongation
Cu	Tensile strength, compressive strength, Hardness
REEs	Yield strength, Tensile strength, Hardness

1.11 STUDIES ON Mg-Sr ALLOY

Strontium is an alkali element belongs to the 2nd group of the periodic table. Sr is a trace element that available in human body bones and teeth. Sr accumulates in bones and improves the growth of the bone. Sr has already proved that it helps in reforming the bone by promoting the osteoclast. And also, it reported that addition of Sr in Mg alloy increased both mechanical and corrosion properties. Sr can refine the grain structure and decrease the shrinkage porosity and increase the relative density of castings which leads to improvement of corrosion and mechanical properties. Yuxiagwang and et all reported that Mg-2Sr(wt%) alloy shown the highest strength and lowest corrosion rate. Mg–Sr alloys have increased tensile and yield strengths up to 2 wt% Sr, but any further increase can result in a reduction in strength. Sr is not only biocompatible, but also bioactive and has a lot of potential for bone formation.

Because of these characteristics, Sr is a good material to alloy with Mg for implant applications. And also reported that it has acceptable biocompatibility and helps in bone formation without any adverse effects.

1.12 STUDIES ON Mg-Gd ALLOY

Gadolinium is the rarest earth metal, and it is one of the element from lanthanide series. Mg-Gd alloys are the best combination to increase the mechanical properties and corrosion resistance. Recently, a number of cast magnesium alloys containing gadolinium and additional REE have been studied. Gd has a suitable biocompatibility and high solubility in Mg [19]. And recent studies state that Mg-Gd alloys are developed as a possible material for bone implant applications. Considering both mechanical properties and corrosion resistance the composition of Gd was optimised. As an REE increase of Gd content from 0 to 2% shown increase in tensile strength. Because Mg-Gd inter-metallic phases are stable at high temperatures, Mg-Gd alloys have been shown to have good creep resistance. Mg alloyed with Gd showed refined grain size than any other REEs.

1.13 STUDIES ON Mg-Sr-Gd ALLOY

Studies on Mg-Sr-Gd are very less. As we seen using Magnesium and Gadolinium and Magnesium Strontium makes a lot of changes in the biomedical industries. As it enhances the microstructure and mechanical properties of the Magnesium. RE and Sr exhibit dose dependent cell behavior and low concentration of them is harmless to human body. Metallurgically, RE elements could weaken the texture and thus improve the ductility of magnesium alloys with the alloying concentration as low as 0.2 wt%. As Gd is an RE element it exhibits the same properties as discussed above. The classification of different types of implants can be seen in Figure 1.11.

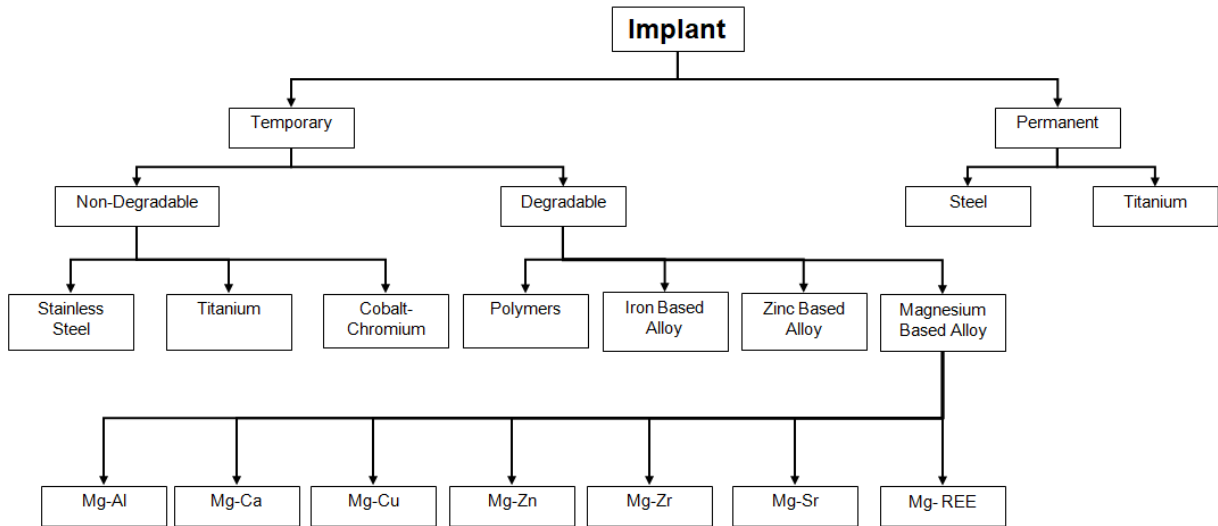


Figure 1.11 Classification of types of Implants

1.14 STUDIES ON XRD (X-RAY DIFFRACTION)

X-ray powder diffraction (XRD) is a swift analytical technique for determining unit cell dimensions and is generally used to classify the phases of crystalline materials. The substance to be analysed is finely ground and homogenised, and the bulk composition is measured on an average basis. The Figure 1.12 shows XRD (Bruker d2 phaser).



Figure 1.12 XRD Resting Machine

1.14.1 BASIC PRINCIPLES OF XRD

Max von Laue discovered in 1912 that crystalline materials serve as 3-D diffraction gratings for X-ray wavelengths same as plane spacing in a crystalline structure. X-ray diffraction is also often used in studying of crystal structures and atomic spacing.

X-ray diffraction is based on the constructive interference of monochromatic X-rays with a crystalline sample. X-rays are produced by a cathode ray tube, then filtered to produce monochromatic radiation, collimated to concentrate the signal, and directed at the sample. When Bragg's law ($n\lambda = 2d \sin \theta$) is satisfied, positive results from the association of incident rays through the sample. The wavelength of electromagnetic radiation is related to the diffraction angle as well as lattice spacing in a crystalline sample by this law. The diffracted X-rays that have been observed, collected, and counted are then analyzed. Sample is scanned through a range of 2θ angles will yield all possible lattice diffraction directions due to the arbitrary orientation of the powdered material. Converting diffraction peaks to d-spacing allows for mineral detection since each mineral has its own range of d-spacing. The most common process to do this is to compare d-spacing to standard reference patterns.

The generation of X-rays in an X-ray tube is the basis of all diffraction methods. The diffracted rays are obtained after these X-rays are guided at the sample. The angle in between the incident and diffracted rays is a crucial factor in all diffraction. Geochemical Instrumentation and XRD is a fast analytical technique for determining the phase of a crystalline substance.

1.14.2 WORKING principle OF XRD

The X-ray tube, sample holder, and ray-detector are the three fundamental components of an XRD. X-rays are produced in a cathode ray tube by heating a filament to produce electrons, then colliding the target material with electrons using voltage to accelerate electrons into the target. Characteristic X-ray spectra are formed when electrons have enough energy to dislodge the target material's inner shell. There are a number of spectra. The most famous are $K\alpha$ and $K\beta$. $K\alpha$ is made up of two parts: $K\alpha_1$ and $K\alpha_2$. $K\alpha_1$ has a much shorter wavelength and half the

amplitude of $K\alpha_2$. The target material's specific wavelengths define it (Cu, Fe, Mo, Cr). Filtering with foils or monochromatic crystals is needed to generate monochromatic rays for diffraction. Since $K\alpha_1$ and $K\alpha_2$ have similar wavelengths, a weighted average of the two can be used. $CuK\alpha$ radiation = $1.5418\text{\AA}$ is the most typical target material for single-crystal diffraction target material. When the sample and detector rotates, the power of the reflected rays is measured. A structural interference happens as the structure of the X-rays impinging on the sample follows the Bragg Equation, and peak amplitude occurs. This X-ray signal is detected and stored in a detector, which transforms it to a count rate, which is then transmitted to a system like a printer or a monitor. The sample rotates at an angle in the range of the collimated X-ray beam in an X-ray diffractometer, while the X-ray reader is positioned on an arm that rotates at an angle of 2θ to absorb the diffracted X-rays. A goniometer is a tool for maintaining and rotating the angle of a sample. For regular powder forms that are present in the X-ray scan, data is collected at 2θ ranging from 5° to 70° . The Figure 1.13 shows the working principle of XRD.

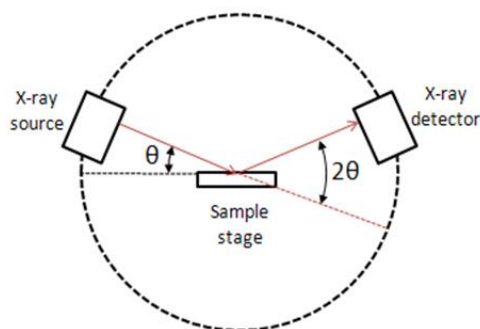


Figure 1.13 Working Principle of XRD

1.14.3 APPLICATIONS OF XRD

The most common method for identifying unknown crystalline materials is X-ray powder diffraction like minerals, inorganic compounds. In geology, environmental science, material science, engineering, and biology, the determination of unknown solids is important.

- Identify the fine-grained minerals that are difficult to assess optically, such as clays and mixed layer clays.
- Dimensions of unit cells are determined.
- Purity testing of a sample.

There are many different techniques in XRD that can also be used.

1.14.4 Uses of XRD

- A strong and faster less than 20 minutes technique for determining an unknown mineral.
- In the majority of instances, it provides a clear mineral definition. The basic sample testing is necessary.
- Units of XRD results are easily obtainable.
- This same data analysis is incredibly easy.

1.14.5 Limitations of XRD

- The main quality for recognizing an unknown is homogeneous and single phase.
- The detection limit for mixed materials is ~2% of sample.
- Indexing trends for non-isometric crystal systems is challenging for unit cell determinations.
- Peak overlay may occur, and it is worse for high-angle reflections.

1.15 STUDIES ON OPTICAL MICROSCOPE

1.15.1 Principle of optical microscope

An optical microscope, also defined also as light microscope, amplifies an image using a combination of light and lenses. Micro objects, such as cells, are studied with

optical microscopes. Because this type of microscope does not have the highest magnification, it could only see a minimal number of components when inspecting a cell. Organelles include lysosomes, the Golgi apparatus, ribosome's, and the cytosol, for example, weren't visible in cells. If an object has a wavelength smaller than light, the microscope will also not be capable of generating an image. Because cell components are usually opaque, they should be stained to be clear .With technological advances, it has changed over time to optimize resolution and clarity, allowing for a more adequate provision. The simple microscope, which uses a single lens, and the compound microscope, which requires different lenses, are the two types of optical microscopes.

1.15.2 Main parts of microscope

- Eyepiece.
- Turret.
- Objective.
- Focus wheel.
- Frame.
- Light source.
- Condenser.
- Stage.

1.15.3 Eyepiece

You look through the lens at the peak. These have double or maybe more lenses when focusing the picture. That magnification is typically 10times.

1.15.4 Turret

This contains two or three lenses and can easily be rotated to change magnification capacity. Beneficial to begin the lengthening at the lowest, then work your way upwards, when examining a slide for example.

1.15.5 Objective

Lens that reflect radiation One or more objective. Normally the lenses are in a cylindrical tunnel. The shortest lenses with both the lowest magnification, the longer the lens has the strongest magnification ability. The lenses are normally 4x, 10x and 40x focus.

1.15.6 Focus wheel

There are rollers that drive the stage vertically. Rollers for adjustment of ground and emphasis are also available. However, some microscopes facilitate focusing on the specimen.

1.15.7 Frame

The structure is composed of the arm and head, it acts as the microscope's bodywork. It requires the focus wheels and stage to be attached to the microscope.

1.15.8 Light Source

A medium of light used for a mirror. Manual light change through a wheel near the bases is required in most microscopes.

1.15.9 Condenser

The condenser lens' purpose is to concentrate the light on the sample. The condenser lens often has filters or a diaphragm in order to improve efficiency.

1.15.10 Stage

A platform below the target providing the platform to display the diaphragm. A opening in the middle of the stage allows light to pass in. The slide is usually held in place by arms on the stage. The Figure 1.14 shows the image of optical microscope.

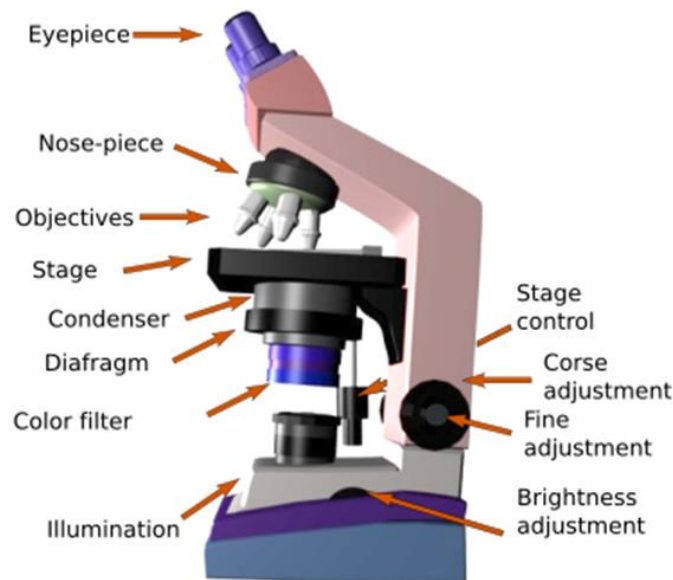


Figure 1.14 Optical Microscope

1.15.11 working of optical microscope

The most frequently used form of microscope is optical microscopes, also recognized as light microscopes, compound microscopes, which mean that they contain at least one handed lenses are the most realistic microscopes. Lenses are shaped glass or plastic pieces that bend light rays to magnify objects and make them all look larger than they are in two phases, this compound microscope magnifies an object. The mirror light is reflected in an efficient objective lens, which generates first magnification, from the specimen or the object to be seen. The lens image then is magnified again by the eyepiece, which works as a simple glass lens. Looking through the eyepiece lens exposes the magnified view. The Figure 1.15 shows the working principle of Optical Microscope.

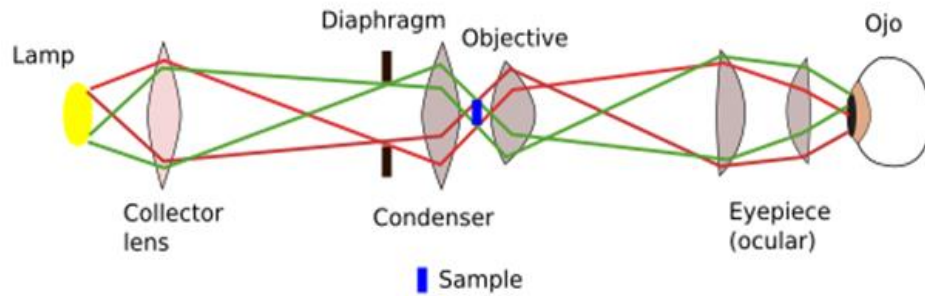


Figure 1.15 Working principle of Optical Microscope

1.15.12 Applications of optical microscope

The whole kind of microscopy is used in the metallurgical, industrial, biotechnology and microbiology fields. It enables one to differentiate between various cells and structures of the metals. These microscopes help in confirming the grain sizes and intermetallic structures of the metals and alloys.

CHAPTER 2

LITERATURE SURVEY

2.1 Mg-Sr Alloy

“(X.N. Gu et al., in 2012)”.. Conducted tests on Mg-Sr alloys ranging from 1 to 4% of strontium prepared by alloying of pure Mg(99.98%). And samples made ($10 \times 10 \times 1.5$ mm³) for microstructure characterization. An X-Ray diffractometer (Rigaku Dmax) used with X-Ray diffractometer used with cu ka radiation to find the crystal structure of the phases. According to the XRD results the various Mg-Sr alloy i.e., ranging 1 to 4% were composed of a-Mg with a hexagonal close packed crystal structure. Mg-Sr alloy exhibit finer grain sizes were like this for Mg-1Sr 32 ± 6.7 , Mg-2Sr 25.9 ± 8.3 , Mg-3Sr 23 ± 8.1 , Mg-4Sr 20.9 ± 8.8 .

“(M. Bornapour et al., in 2012)”.. conducted a research on Mg-Sr alloys varying the percentage of the Sr content. The degradation of Mg-sr alloy in the alloys which contains the content of Sr is less than 1%. It is calculated in terms of mass loss and H₂ evolution. In vitro results shown Mg-Sr has potential to be used as temporary iimplant.

“(H.S. Brar et al., in 2012)”.. Carried test on the mechanical and deterioration activity of three binary Mg–x wt. percent Sr (x = 0.5, 1.0, 1.5) alloys and three ternary Mg–x wt. percent Zn–0.5wt percent Sr (x = 2.0, 4.0, 6.0) alloys were investigated. When it came to degradation, Mg–0.5wt percent Sr had the lowest rate of degradation for binary alloys. The addition of Zn increased the corrosion risk at constant Sr content of 0.5wt percent, with the maximum rate for Mg–6.0wt percent Zn–0.5wt percent Sr. Mg–2.0wt percent Zn–0.5wt percent Sr and Mg–4.0wt percent Zn–0.5wt percent Sr were the alloys that better optimized both mechanical and corrosion behaviors. After 7 days in Hanks' solution, XRD patterns of the Mg–Sr and

Mg–Zn–Sr alloy samples confirm the presence of $\text{Mg}(\text{OH})_2$ on all of the specimens. The optical micrographs of the binary Mg–x wt. percent Sr ($x = 0.5, 1.0, 1.5$) alloys that have been solution-treated are noticeable. The typical grain sizes of Mg–0.5 wt. percent Sr and Mg–1.0 wt. percent Sr alloys are 379 μm and 390 μm , respectively. Mg–1.5 wt. Percent Sr had a grain size of 145 μm . The presence of the second process, $\text{Mg}_{17}\text{Sr}_2$, is confirmed by XRD analysis of these alloys.

“(Z. Li et al., in 2017)”.. carried out research on Mg-Zn-Zr-Sr (MZZS) To improve the mechanical properties and corrosion resistance of the biodegradable magnesium (Mg) alloy, it was smelted in vacuum under inert gas protection, and the bar content with dimensions of 8 mm diameter was then obtained via a hot-extrusion process. The XRD patterns depict the major phases found in MZS, MZZ, and MZZS alloys as-cast. In the MZS alloy, -Mg diffraction peaks were visible, as well as distinct diffraction peaks for $\text{Mg}_{17}\text{Sr}_2$ in the 2 theta degree. In the MZZS alloy with the same Sr material, the amplitude of $\text{Mg}_{17}\text{Sr}_2$ diffraction peaks at 38, 45, and 65 degrees increased dramatically. This means that, unlike $\text{Mg}_{17}\text{Sr}_2$ in the MZS alloy, $\text{Mg}_{17}\text{Sr}_2$ in the MZZS alloy increased substantially in crystallinity, and $\text{Mg}_{17}\text{Sr}_2$ size changed. $\text{Mg}_{17}\text{Sr}_2$'s diffraction peak position in MZZS and MZS alloys shifted to the right from its usual position, meaning that the lattice in $\text{Mg}_{17}\text{Sr}_2$ changed. The three alloys had very different microstructures; the MZS alloy had the largest average grain size, with a value of around 110.6 μm . Secondary phases in the MZS alloy were essentially uniform precipitates with dimensions of 2–3 μm .

2.2 Mg-Gd alloys

“(Dong Bian et al., in 2018)”.. conducted research on Mg-zn-Gd. Zn is used because Gd as a single alloyment may cause toxicity sometimes. To avoid superiority of Mg-Gd Zn is added . As Zn is also oone of the abundant and essential element in human body. Using resistance melting method billets were prepared 99.99 wt% Mg, 99.9 wt% zn and 99.95 wt% Gd. Mg-1.8Zn-0.8Gd, Mg-1.0Zn-2Gd were prepared

using the resistance melting. Microstructure showed finest grains with abundant twins inside. Much more particles are seen in Highest Gd content alloy that is Mg-1.0Zn-2Gd. And also the mechanical properties are also improved in the alloys. There is no much difference between the two alloys due to low added percentages.

“(Sankalp Agarwal et al., in 2016)”.. at Seoul university carried a research on Gd coated Mg alloy. AZ31 Mg alloy was cut into (15mm 25mm 2mm) and washed with acetone and distilled water. Gd was deposited using Electrophoretic deposition. Surface characterisation was done by using SEM on Gd coated AZ31 Mg. The results have shown that predominant impact and significant role in regulating biological behaviours. Low angle XRD analysis shown that presence of crystalline Mg. The formed Gd doesn't react with other coating elements. Therefore, Gd is an optimal material for transmit implant usage.

“(Miao Yang et al., in 2016)”.. worked on AM50 Mg alloy with different amounts of REE Gd. Gd is a prime candidate as a modifying rare element for Mg. The alloy was made by casting in an electric resistance furnace and a graphite crucible. Adding Gd in liquid molten AM50 Mg with different wt%. XRD results shown the phase changes presence of α -Mg, β -MgAl₂, AlGdMn. SEM results shown after addition of Gd in different (wt%) grain size decreased. Corrosion tests are carried out in NaCl sol. Time intervals for corrosion process is 24, 72, 168, 264, 336, 442 hrs separately. Increase in Gd observed the corrosion potential first decreased and then increased. Therefore, the RE element Gd contributes towards grain refinement, corrosion resistance.

“(N.Hort et al., in 2009)”.. Carried research on Mg-Gd alloys with varying the composition of Gd (2%, 5%, 10%, 15%). Following the weight percentages total 4 samples are prepared as Mg-2Sr, Mg-5Sr, Mg-10Sr, Mg-15Sr with the dimensions of 300mm×210×30mm. And the materials are investigated under three conditions as

cast condition, after solutionizing, and artificial ageing. Investigation on microstructure takes place by scanning electron microscope (SEM) and for the phase analysis X-ray diffraction (XRD) measurements are performed. Results from XRD states that there is no existence of pure Gd in the 1st three alloys Mg-2Gd, Mg-5Gd, Mg-10Gd only Mg peaks are formed. And also, it shown improvement in the mechanical properties. Addition of Gd improves the ultimate tensile strength (UTS), tensile yield strength(TYS) and elongation fraction(EL) .

2.3 Mg-Sr-Gd

“(Hongyan Tang et al., in 2019)”.. carried out a research on the Mg-Sr_RE element where they used La ,Nd , Gd as the RE elements to make the magnesium alloy. The Three alloys Mg-Sr-La, Mg-Sr-Nd and Mg-Sr-Gd casted. The Microstructure was observed using Optical microscope and X-Ray Diffractometer (XRD Bruker D8 focus) used to find the phases of alloys. The average grain size of the actual Mg-Sr was decreased with the adding of RE elements. The actual grain size of Mg-Sr is 39 ± 4.0 and adding of RE elements La, Nd, Gd changed to 29 ± 3.6 , 15 ± 2.8 , 12 ± 1.4 .XRD patterns states that there is no existence of second phases. It states that the present casted Mg-Sr-RE alloys are close to the single phase solid solution.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

- To prepare a biocompatible Mg alloy with Sr and Gd.
- To prepare magnesium alloys with strontium and gadolinium which can be used for biomedical applications.
- Preparation of Mg-2Sr by adding 2wt% of Sr in the Mg mould.
- Preparation of Mg-2Sr-2Gd by adding of 2wt% of Gd in Mg-2Sr master alloy.
- Taking the Mg-2Sr alloy as master alloy and adding the gadolinium to it with weight ratio of Mg-2Sr-2Gd.
- To check the difference between the Mg-2Sr and Mg-2Sr-2Gd microstructures and properties whether the enhancement takes place or not.
- Using XRD for detecting the phases and optical microscopy for the checking microstructure of the alloys.
- By checking the XRD patterns to conclude the mechanical properties of the samples.
- By checking the peaks concluding the structure of materials at the high intensity levels.
- To check the microstructure of Mg-Sr alloy after adding of Sr in Mg.
- To check the microstructure of Mg-2Sr-2Gd alloy after adding of Gd in Mg-2Sr master alloy.

3.2 SCOPE

- Making the use of alloy material for orthopaedic applications and biomedical applications.
- Enhancing the mechanical properties gives the alloy to perform better than the pure material.
- By decrease in grain size mechanical properties and metallurgical properties will improve by which it can be used as replacement for implant materials.

- By concluding the nature of the material if there is increase d values it shows decrease in the grain size. Decrease in the grain size improves grain boundaries.
- From the values of FWHM we can conclude the nature of the material whether amorphous or crystalline.
- Amorphous nature material consists improved mechanical properties. From this we can conclude about the properties partially.

CHAPTER 4

EXPERIMENTAL OR MATERIALS AND METHODS

4.1 MATERIALS

The following data represents the work materials used for the process. The main materials which are used for the experimental work are Magnesium(Mg), Strontium(Sr), Gadolinium(Gd).

4.2 COLLECTION OF MATERIALS

Raw materials which are used in this experiment were Magnesium, Strontium and Gadolinium. Which are available in the market as the material was processed and casted at Matrix technologies vellimalai, Chennai materials are procured by the industry and they are casted into a single alloy ranging the different percentages of the elements.

- I. Magnesium (Mg)
- II. Strontium (Sr)
- III. Gadolinium (Gd)

4.3 CASTING PROCESS

Mg is taken into the steel crucible and keep into an electric furnace at a temperature of 800° c. In the meantime, di steel mould is preheated with a temperature of 350°c and keep it ready. When the Mg is melted then Sr particles with the required weight is added into the mould. Skimming process is takes place with a gas SF₆ and Ar with a percentage of 98% Ar and 2% of SF₆. This molten metal is poured into the mould which is already preheated with temperature of 350°c. And set this to cool at room temperature. The schematic flow of process can be seen in Figure 4.1.

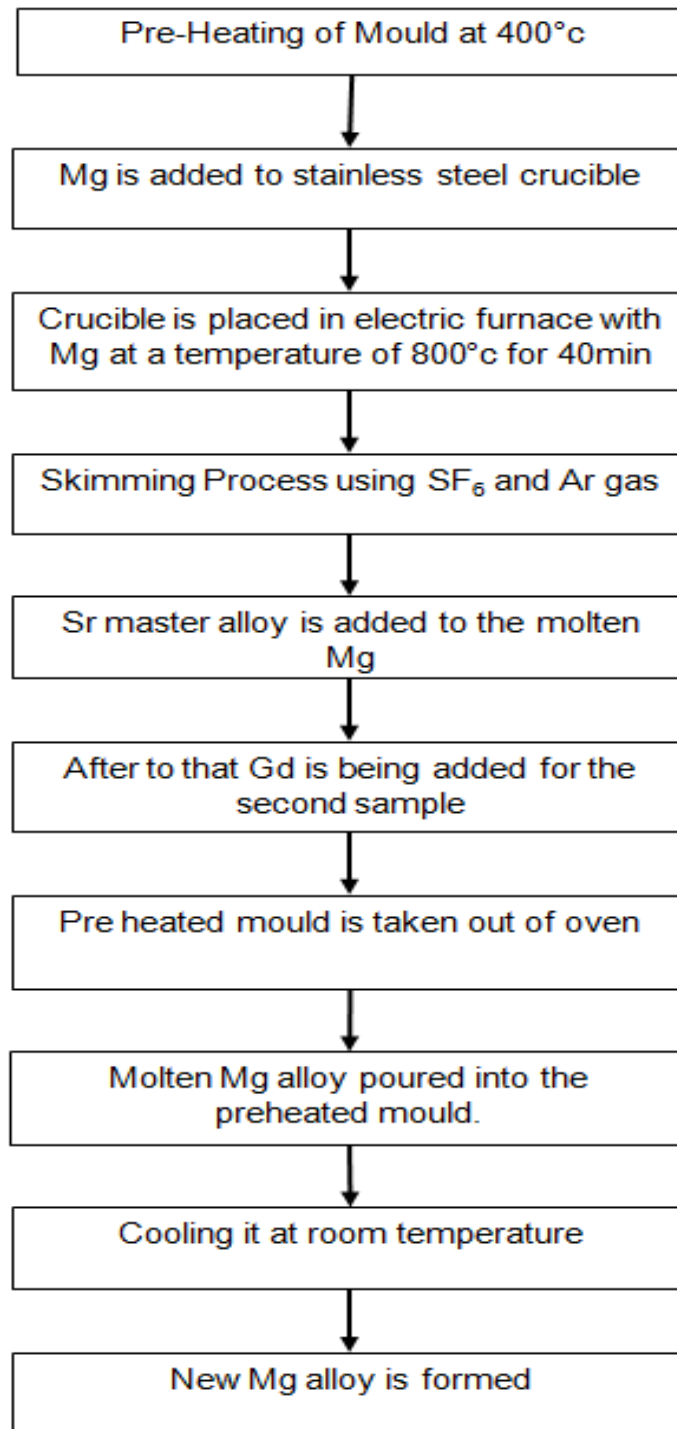


Figure 4.1 Process of Mg-REE gravity Casting



Figure 4.2 Preheating of Mould

In the above figure 4.2 we can see the preheating of mould in a oven before the molten alloy poured. This preheating helps in increase the surface quality of the castings. In the figure 4.3 it shows the process of keeping the Mg in a steel crucible and adding it into an electric furnace.



Figure 4.3 Placing Crucible with Mg in Furnace

In the figure 4.4 skimming process is shown. Skimming process is used to avoid the firing on top of the crucible inside the furnaces. For this process SF_6 and Ar gases are used.



Figure 4.4 Skimming Process

While the skimming process is taking place, addition of Sr in the molten Mg is taking place. This skimming process avoids firing while adding Sr. Addition of Sr is shown in figure 4.5.



Figure 4.5 Adding of Strontium

In figure 4.6 we can see the pouring of molten metal Mg and Sr into the hot mould which is preheated in an oven. This poured molten metal is cooled at room temperature.



Figure 4.6 Pouring of Molten metal into mould

The final output the cooled alloy can be seen in the figure 4.7. After this process completed again everything is cleaned and preparation of another sample is takes place in the same way with extra addition of Gd in it..



Figure 4.7 Mg-2Sr alloy

There will be a 10% wastage of the alloy due to the extra metal has to be removed from the material. And then the material is cut down and polished in to the required sizes for the tests conducts on it.

4.4 XRD TESTS

Prior to the tests the samples are prepared following the ASTM standards with the dimensions of (10×10×3mm). After the preparation of samples are polished and cleaned before testing. X-Ray Diffraction (XRD) is a laboratory-based technique commonly used for identification of crystalline materials and analysis of unit cell dimensions. Investigation of phase transitions are observed under the x-ray diffraction (Bruker d2 phaser). XRD tests taken on Mg-2Sr at room temperature the start angle is 10.002° and end angle is 80. 010°. The total time taken per step is 80 seconds. And the samples are undergone for testing. The pictures of the samples are given below figure 4.9. The same process is followed for another sample too.



Figure 4.8 Test Samples

Results of the XRD tests are discussed in chapter 5 results and discussions.

4.5 OPTICAL MICROSCOPY

An optical microscope, also defined also as light microscope, amplifies an image using a combination of light and lenses. Micro objects, such as cells, are studied with optical microscopes. The Mg-2Sr and Mg-2sr-2Gd are tested under the optical microscope for the microstructure. Images of the microstructures can be seen in the chapter *figure5.6*. The Figure 4.9 shows the research process flow.

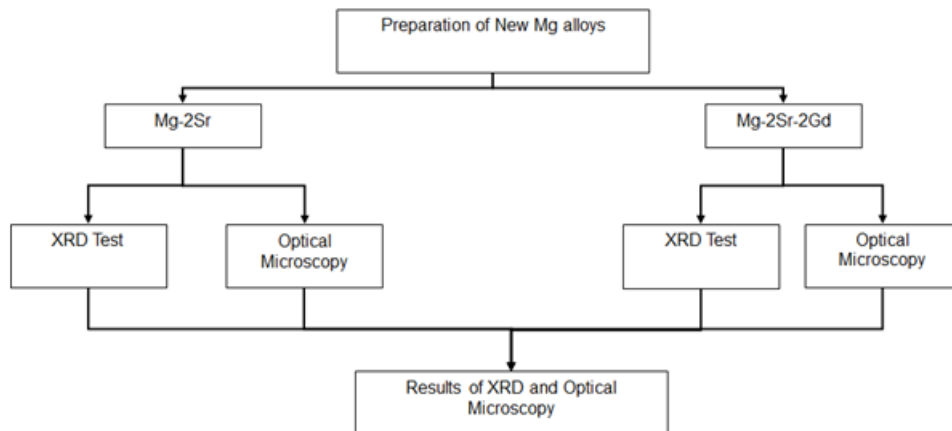


Figure 4.9 Scheme of Research Work

CHAPTER 5

RESULTS AND DISCUSSION

5.1 XRD RESULTS OF Mg-2Sr

With a wavelength of $1.54060\text{\AA}$ Mg-2Sr sample was investigated under XRD (Bruker d2 phaser) the start angle of the test is 10.002° and the end angle of the test was 80.010° conducted under the room temperature. Using the Cu as anode radiation takes place and the results were coated down in Table 5.1. The maximum intensity of the sample is observed at 36.593° as 9758.13 and the minimum intensity of the sample observed at 27.366° as 34.6963. And it shows the solid solution at the peak point and amorphous at the low intensity levels. The highest peak is formed at peak 8 with 36.593° , the total peaks formed for Mg-2Sr were 16 peaks which can be seen in Figure 5.1.

Table 5.1 XRD Results of Mg-2Sr

Peak Number	2-theta(deg)	d Value	Net Intensity	FWHM	Width
1	12.979°	6.81545	46.6169	0.242	0.121
2	24.097°	3.69024	115.062	0.244	0.122
3	27.366°	3.25635	34.6963	0.174	0.087
4	27.452°	3.2464	41.4957	0.174	0.087
5	29.366°	3.03902	124.308	0.217	0.108
6	32.17°	2.78024	2449.62	0.178	0.089
7	34.404°	2.60462	3819.74	0.205	0.102
8	36.593°	2.45368	9758.13	0.193	0.096
9	47.787°	1.9018	1300.58	0.21	0.105
10	57.314°	1.60625	956.044	0.204	0.102
11	63.036°	1.47351	1286.63	0.223	0.111
12	67.259°	1.39088	79.6213	0.189	0.094

Peak Number	2-theta(deg)	d Value	Net Intensity	FWHM	Width
13	68.578°	1.36731	896.971	0.222	0.111
14	69.937°	1.34403	541.614	0.212	0.106
15	72.447°	1.30352	167.924	0.207	0.103
16	77.752°	1.2273	97.217	0.211	0.106

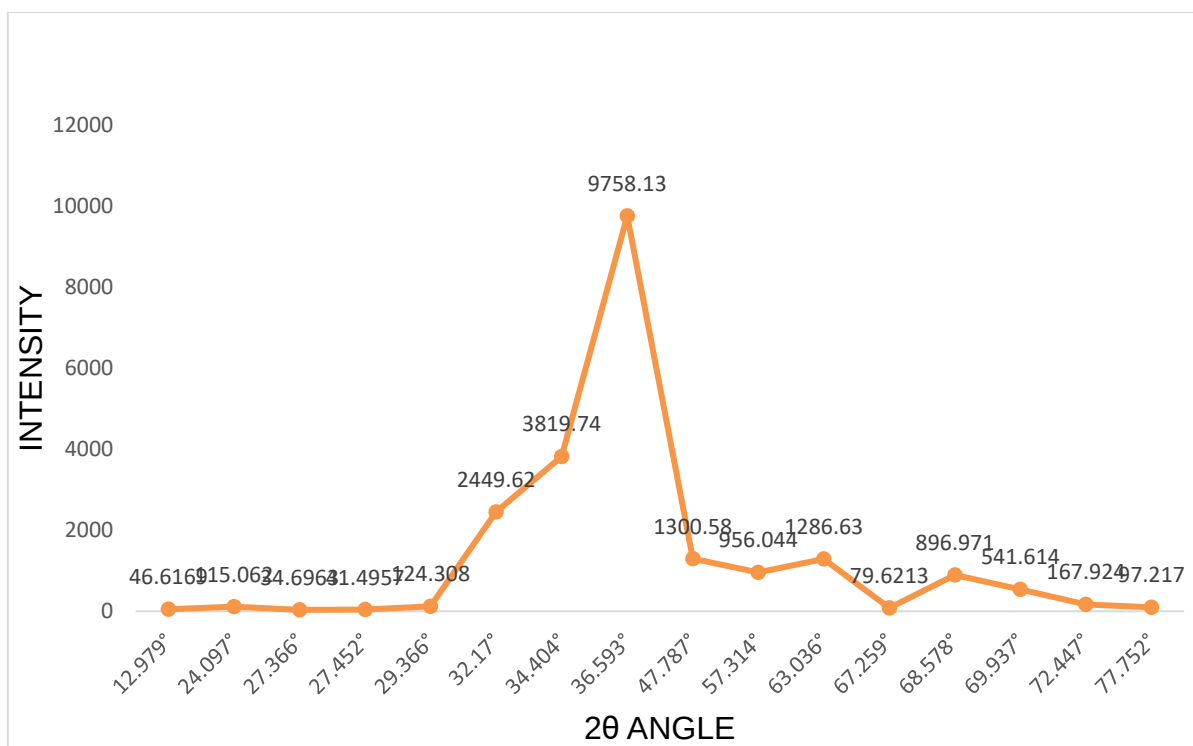


Figure 5.1 Net Intensity graph of Mg-2Sr

5.2 XRD RESULTS OF Mg-2Sr-2Gd

Mg-2Sr-2Gd sample was investigated under XRD(Bruker d2 phaser) the start angle of the test is 10.002° and the end angle of the test was 80.010° conducted under the room temperature. Using the Cu as anode radiation takes place and the results were coated down in Table 5.2. The maximum intensity of the sample is observed at

36.538° as 5432.28 and the minimum intensity of the sample observed at 77.687° as 34.0288. And it shows the solid solution at the peak point and amorphous at the low intensity levels. The highest peak is formed at peak 6 with 36.538°, the total peaks formed for Mg-2Sr-2Gd were 15 peaks which can be seen in Figure 5.2.

Table 5.2 XRD Results of Mg-2Sr-2Gd

Peak Number	2-theta(deg)	d Value	Net Intensity	FWHM	Width
1	24.048°	3.69761	82.4739	0.291	0.146
2	27.162°	3.28034	56.2531	0.15	0.075
3	29.255°	3.05031	91.4635	0.295	0.148
4	32.128°	2.78378	1429.93	0.262	0.131
5	34.317°	2.61102	1831.86	0.296	0.148
6	36.538°	2.45729	5432.28	0.294	0.147
7	47.752°	1.90311	588.471	0.323	0.161
8	57.216°	1.60876	651.04	0.305	0.153
9	62.939°	1.47554	559.742	0.319	0.159
10	67.115°	1.39352	40.2302	0.181	0.091
11	68.483°	1.36898	436.553	0.313	0.157
12	69.838°	1.34569	273.519	0.323	0.161
13	72.330°	1.30535	79.2619	0.228	0.114
14	77.585°	1.22952	46.5364	0.198	0.099
15	77.687°	1.22817	34.0288	0.198	0.099

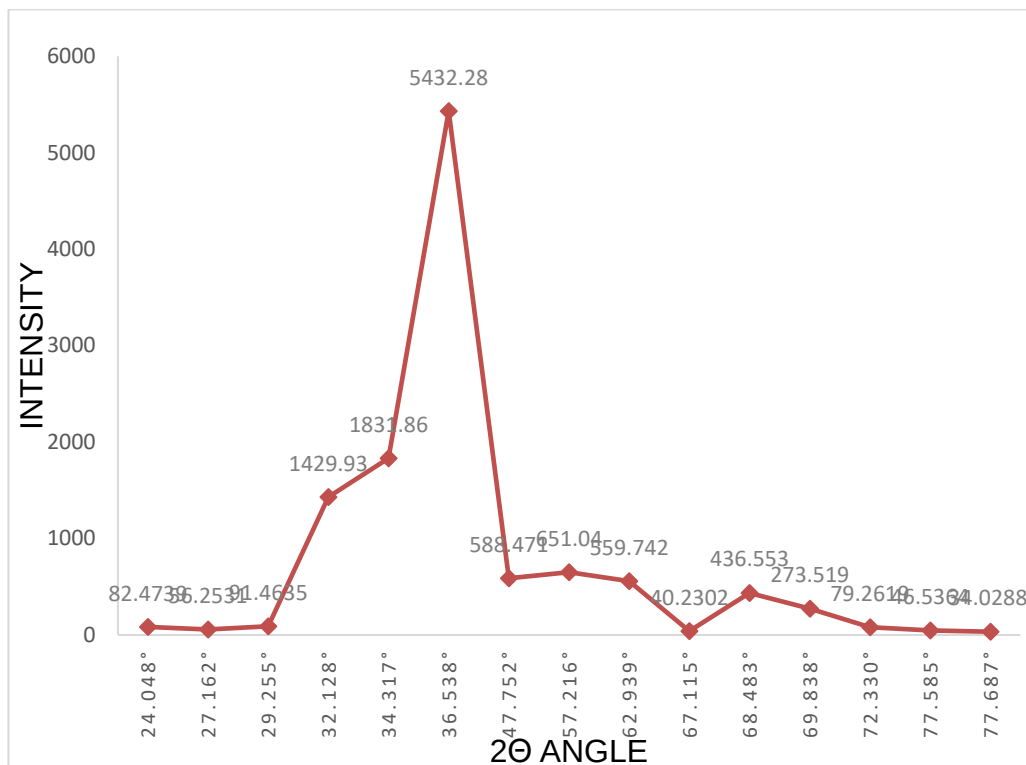


Figure 5.2 Net Intensity graph of Mg-2Sr-2Gd

The intensity graph of Mg-2Sr-2Gd was shown in the Figure 5.2. The total of 15 peaks was formed.

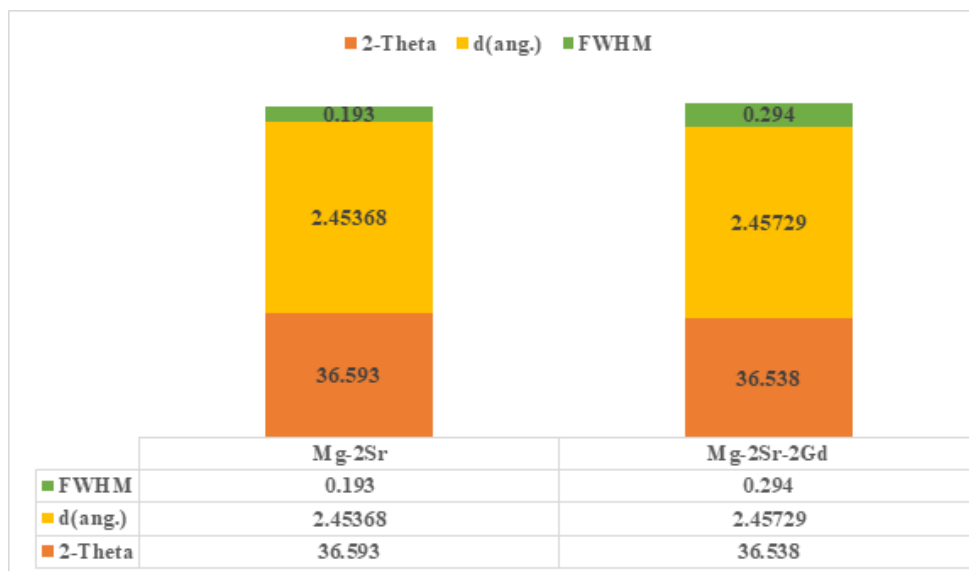


Figure 5.3 Highest Intensity Peak Data's of Mg-2Sr and Mg-2Sr-2Gd

In Figure 5.3 it shows the highest intensity peaks and the corresponding FWHM and D values for Mg-2Sr and Mg-2Sr-2Gd.

5.3 FWHM OF Mg-2Sr AND Mg-2Sr-2Gd

When the FWHM of Mg-2Sr and Mg-2Sr-2Gd compounds are compared as shown in the, the MG-2Sr compound showed relatively lower values, where the maximum FWHM was been observed in Mg-2Sr-2Gd. When the maximum values of each compound are compared, the FWHM value of Mg-2Sr resulted as 0.244, whereas for Mg-2Sr-2Gd the maximum value is 0.323. These are plotted on the graph can be seen in Figure 5.4.

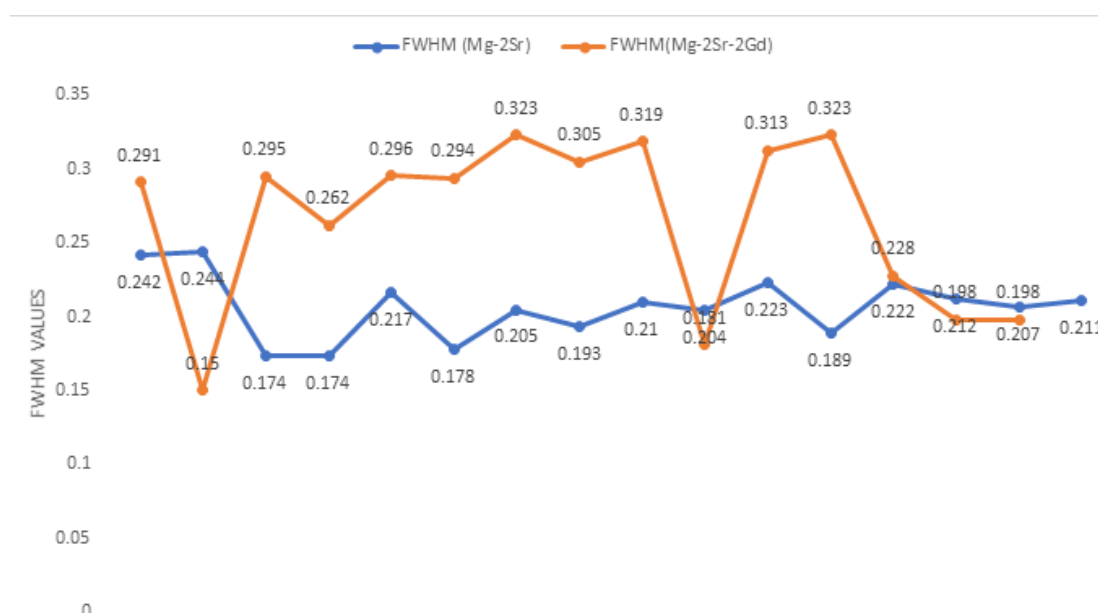


Figure 5.4 FWHM of Mg-2Sr and Mg-2Sr-2Gd

When the FWHM value is high the amorphous nature of the compound increases which leads to the betterment of mechanical properties in the compound. So among Mg-2Sr and Mg-2Sr-2Gd addition of Gd in the compounds resulted in the increase of FWHM value, which lead to the betterment of the mechanical properties. This can be either way stated as follows. The crystalline nature of the Mg2sr is high when compared to that of Mg-2Sr-2Gd.

Table 5.3 Maximum Intensity values of Mg-2Sr

Peak Number	2-theta(deg)	d Value	Net Intensity	FWHM	Width
6	32.17°	2.78024	2449.62	0.178	0.089
7	34.404°	2.60462	3819.74	0.205	0.102
8	36.593°	2.45368	9758.13	0.193	0.096
9	47.787°	1.9018	1300.58	0.21	0.105

We can observe the highest peak values of Mg-2Sr in the Table 5.3 and also the corresponding values of d values, FWHM can be seen in the table.

5.4 D VALUE OF Mg-2Sr AND Mg-2Sr-2Gd

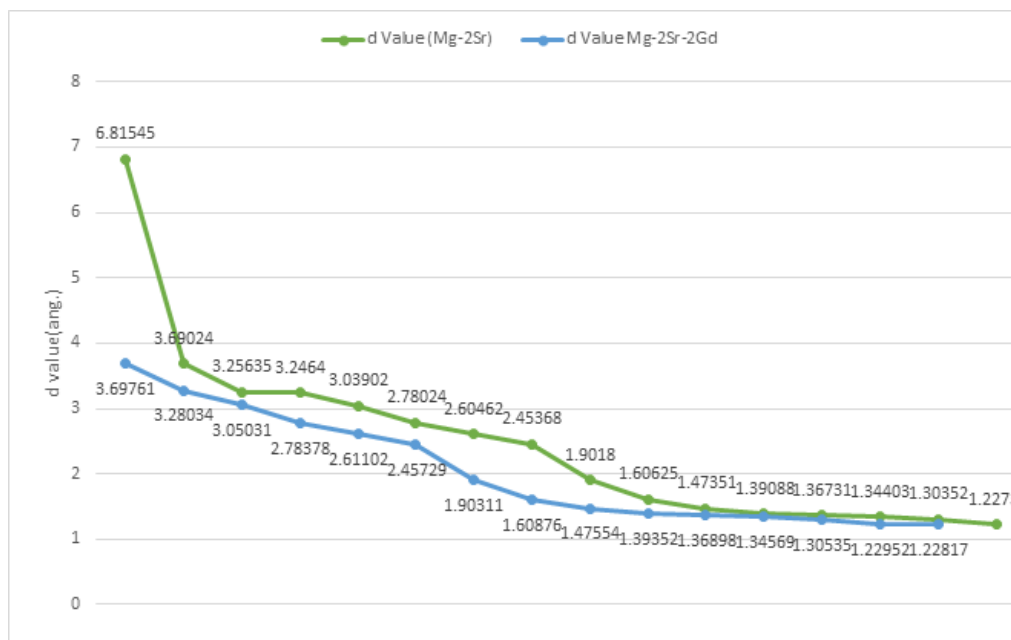


Figure 5.5 d value of Mg-2Sr and Mg-2Sr-2Gd

When the sample are cut and made into thickness of 3mm and XRD analysis is made, the following trends in D spacing values occur. From the d values of both Mg-2Sr and Mg-2Sr-2Gd we can find changes in the grain size. The d value of Mg-2Sr-2Gd is higher than the Mg-2Sr at maximum intensities. D value of Mg-2Sr at its highest intensity is 2.45368 for Mg-2Sr-2Gd is 2.45729 we can observe small increase in the d value. The Relationship between Grain size and d value is when Grain size decreases d value increases. Therefore, the grain size of the alloys decreased when Gd is added.

Table 5.4 Maximum Intensity values of Mg-2Sr-2Gd

Peak Number	2-theta(deg)	d Value	Net Intensity	FWHM	Width
5	34.317°	2.61102	1831.86	0.296	0.148
6	36.538°	2.45729	5432.28	0.294	0.147
7	47.752°	1.90311	588.471	0.323	0.161
8	57.216°	1.60876	651.04	0.305	0.153

We can observe the highest peak values of Mg-2Sr in the Table 5.4 and also the corresponding values of d values, FWHM can be seen in the table.

5.5 OPTICAL MICROSTRUCTURE

As the pure Mg has a grain size of $35 \pm 10 \mu\text{m}$ addition of Sr decrease the grain sizes of the Mg-Sr $23.9 \pm 8.3 \mu\text{m}$. Further addition of Gd into the Mg-2Sr master alloy there is a further decrease of grain size from $23.9 \pm 8.3 \mu\text{m}$ to $20.5 \pm 7 \mu\text{m}$. This shows that addition of Gd helps in decrease of grain size. Micro alloying with Gd results in 2 or 1.5 times increment in elongation However, we can see the difference in the grain structure of Mg by adding of Gd up to 10wt% further increase may decrease the mechanical properties of the Mg. The following Figure illustrates the microstructures.

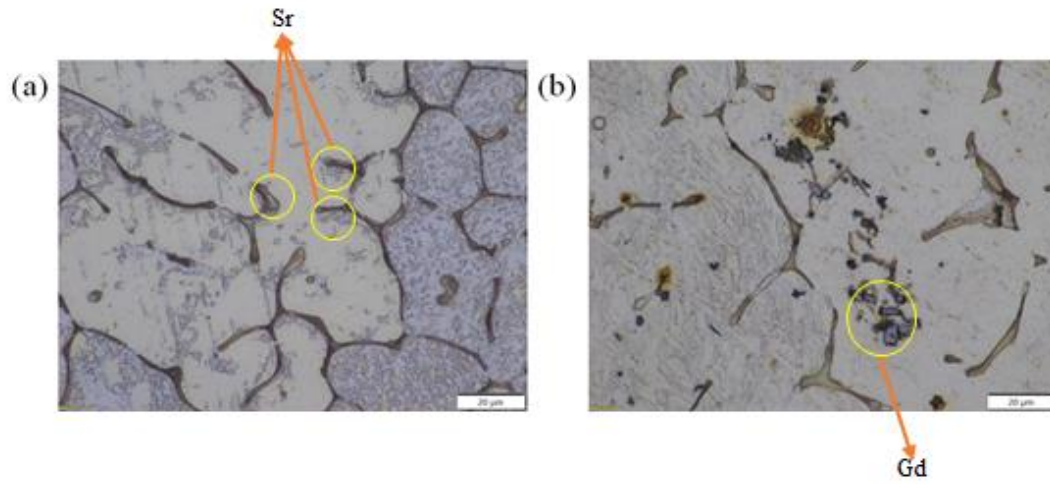


Figure 5.6 Microstructures of (a) Mg-2Sr and (b) Mg-2Sr-2Gd

CHAPTER 6

SUMMARY AND CONCLUSIONS

6.1 SUMMARY

Based on the research performed on Mg-2Sr and Mg-2Sr-2Gd it was found that addition of Sr to the Mg helps in the improvement of the mechanical properties and also it helps in refinement of the grain size.

If we see the second alloy Mg-2Sr-2Gd the grain size decreases further. That is more than the Mg-2Sr. And also, in XRD we can see that FWHM values are higher for Mg-2Sr-2Gd at higher intensities compared to Mg-2Sr. When the FWHM value is high the amorphous nature of the compound increases which leads to the betterment of mechanical properties in the compound.

6.2 CONCLUSION

- There is no phase change in the alloy as we see from the XRD results of both the samples.
- Addition of Sr in Mg contributes towards refinement of grain size and increase in mechanical properties.
- As we see the peak size indicates the crystalline form the alloy at the high intensity ranges.
- Pure Mg has an grain size of $35 \pm 10 \mu\text{m}$ addition of Sr decrease the grain sizes of the Mg-Sr $23.9 \pm 8.3 \mu\text{m}$.
- The decrease in grain size was observed in the both Mg-2Sr and Mg-2Sr-2Gd through optical microscope.
- Addition of Gd into the Mg-2Sr master alloy there is a further decrease of grain size from $23.9 \pm 8.3 \mu\text{m}$ to $20.5 \pm 7 \mu\text{m}$.
- Values FWHM shown difference between Mg-2Sr and Mg-2Sr-2Gd which concludes that amorphous nature increased with the adding of Gd.
- D value of the Mg-2Sr at maximum intensities is lower than the Mg-2Sr-2Gd values at maximum intensities.

- The Relationship between Grain size and d value is when Grain size decreases d value increases.
- Alloys possess good mechanical properties due to the amorphous nature caused by Gd.
- Decrease in grain size increase the grain boundaries. The more grain boundaries that exist, the higher the strength becomes.

REFERENCES

- [1] A. Kania, R. Nowosielski, A. Gawlas-Mucha, R. Babilas, Mechanical and Corrosion Properties of Mg-Based Alloys with Gd Addition, *Materials*. 12 (2019) 1775.
- [2] E. Baril, P. Labelle, M. Pekguleryuz, Elevated temperature Mg-Al-Sr: Creep resistance, mechanical properties, and microstructure, *JOM*. 55 (2003) 34–39.
- [3] H.S. Brar, J. Wong, M.V. Manuel, Investigation of the mechanical and degradation properties of Mg–Sr and Mg–Zn–Sr alloys for use as potential biodegradable implant materials, *Journal of the Mechanical Behavior of Biomedical Materials*. 7 (2012) 87–95.
- [4] H. Tang, F. Wang, D. Li, X. Gu, Y. Fan, Mechanical properties, degradation behaviors and biocompatibility of micro-alloyed Mg-Sr-RE alloys for stent applications, *Materials Letters*. 264 (2020) 127285.
- [5] H. Waizy, J.-M. Seitz, J. Reifenrath, A. Weizbauer, F.-W. Bach, A. Meyer-Lindenberg, B. Denkena, H. Windhagen, Biodegradable magnesium implants for orthopedic applications, *J Mater Sci*. 48 (2013) 39–50.
- [6] I.J. Polmear, Magnesium alloys and applications, *Materials Science and Technology*. 10 (1994) 1–16.
- [7] J.-H. Dong, L.-L. Tan, Y.-B. Ren, K. Yang, Effect of Microstructure on Corrosion Behavior of Mg–Sr Alloy in Hank’s Solution, *Acta Metall. Sin. (Engl. Lett.)*. 32 (2019) 305–320.
- [8] J. Song, J. She, D. Chen, F. Pan, Latest research advances on magnesium and magnesium alloys worldwide, *Journal of Magnesium and Alloys*. 8 (2020) 1–41..

- [9] K. Saranya, S. Bhuvaneswari, S. Chatterjee, N. Rajendran, Biocompatible gadolinium-coated magnesium alloy for biomedical applications, *J Mater Sci.* 55 (2020) 11582–11596.
- [10] M. Bornapour, N. Muja, D. Shum-Tim, M. Cerruti, M. Pekguleryuz, Biocompatibility and biodegradability of Mg–Sr alloys: The formation of Sr-substituted hydroxyapatite, *Acta Biomaterialia*. 9 (2013) 5319–5330.
- [11] M. Rahim, S. Ullah, P. Mueller, Advances and Challenges of Biodegradable Implant Materials with a Focus on Magnesium-Alloys and Bacterial Infections, *Metals*. 8 (2018) 532.
- [12] M. Yang, Z. Zhang, Y. Liu, X. Han, Corrosion and mechanical properties of AM50 magnesium alloy after modified by different amounts of rare earth element Gadolinium, *Open Physics*. 14 (2016) 444–451.
- [13] N. Hort, Y. Huang, D. Fechner, M. Störmer, C. Blawert, F. Witte, C. Vogt, H. Drücker, R. Willumeit, K.U. Kainer, Magnesium alloys as implant materials – Principles of property design for Mg–RE alloys☆, *Acta Biomaterialia*. 6 (2010) 1714–1725.
- [14] S. Agarwal, J. Curtin, B. Duffy, S. Jaiswal, Biodegradable magnesium alloys for orthopaedic applications: A review on corrosion, biocompatibility and surface modifications, *Materials Science and Engineering: C*. 68 (2016) 948–963.
- [15] U. Riaz, I. Shabib, W. Haider, The current trends of Mg alloys in biomedical applications—A review, *J. Biomed. Mater. Res.* 107 (2019) 1970–1996.
- [16] W. Wang, J. Han, X. Yang, M. Li, P. Wan, L. Tan, Y. Zhang, K. Yang, Novel biocompatible magnesium alloys design with nutrient alloying elements Si, Ca and Sr: Structure and properties characterization, *Materials Science and Engineering: B*. 214 (2016) 26–36.

- [17] X.N. Gu, X.H. Xie, N. Li, Y.F. Zheng, L. Qin, In vitro and in vivo studies on a Mg–Sr binary alloy system developed as a new kind of biodegradable metal, *Acta Biomaterialia*. 8 (2012) 2360–2374.
- [18] X. Xia, J.F. Nie, C.H.J. Davies, W.N. Tang, S.W. Xu, N. Birbilis, The Influence of Low Levels of Zinc, Calcium, Gadolinium, Strontium, and Zirconium on the Corrosion of Magnesium for Wrought Applications, *CORROSION*. 71 (2015) 1370–1386.
- [19] Y. Ding, C. Wen, P. Hodgson, Y. Li, Effects of alloying elements on the corrosion behavior and biocompatibility of biodegradable magnesium alloys: a review, *J. Mater. Chem. B*. 2 (2014) 1912–1933.
- [20] Y. Lu, Y. Huang, F. Feyerabend, R. Willumeit-Römer, K.U. Kainer, N. Hort, Microstructure and Mechanical Properties of Mg-Gd Alloys as Biodegradable Implant Materials, in: T.M. & Materials Society Metals (Ed.), TMS 2018 147th Annual Meeting & Exhibition Supplemental Proceedings, Springer International Publishing, Cham, 2018: pp. 253–262.
- [21] Yuxiang Wang, Di Tie, Renguo Guan, Ning Wang, Yingqiu Shang, Tong Cui and Junqiao Li, Microstructures, mechanical properties, and degradation behaviors of heat-treated Mg-Sr alloys as potential biodegradable implant materials
- [22] Z. Li, M. Chen, W. Li, H. Zheng, C. You, D. Liu, F. Jin, The synergistic effect of trace Sr and Zr on the microstructure and properties of a biodegradable Mg-Zn-Zr-Sr alloy, *Journal of Alloys and Compounds*. 702 (2017) 290–302.
- [23] Z. Sheikh, S. Najeeb, Z. Khurshid, V. Verma, H. Rashid, M. Glogauer, Biodegradable Materials for Bone Repair and Tissue Engineering Applications, *Materials*. 8 (2015) 5744–5794.

APPENDIX

For plag check x

ORIGINALITY REPORT

17 %

SIMILARITY INDEX

9 %

INTERNET SOURCES

11 %

PUBLICATIONS

7 %

STUDENT PAPERS

PRIMARY SOURCES

1

Usman Riaz, Ishraq Shabib, Waseem Haider.
"The current trends of Mg alloys in biomedical
applications—A review", Journal of Biomedical
Materials Research Part B: Applied
Biomaterials, 2018

Publication

3 %

2

Submitted to Higher Education Commission
Pakistan

Student Paper

1 %

3

en.wikipedia.org

Internet Source

1 %

4

Zhen Li, Minfang Chen, Wei Li, Haoran Zheng,
Chen You, Debao Liu, Feng Jin. "The
synergistic effect of trace Sr and Zr on the
microstructure and properties of a
biodegradable Mg-Zn-Zr-Sr alloy", Journal of
Alloys and Compounds, 2017

Publication

1 %

5

mdpi.com

Internet Source

1 %

Plagiarism Report from Turnitin

----- Forwarded message -----

From: fresm2021 - <fresm2021@cmrit.ac.in>

Date: Fri, Apr 9, 2021 at 10:51 AM

Subject: Acceptance and Registration Notification for Paper Id:MATPR-D-21-04061

To: <deepakcape@gmail.com>

Dear J R DEEPAK James Raj,

We are glad to inform you that your paper titled "XRD Investigation of Biodegradable Magnesium Rare Earth Alloy " (Paper Id: MATPR-D-21-04061) has been **PROVISIONALLY ACCEPTED WITH REVISIONS** for online presentation and publication in the " Materials today Proceedings " through FRESM21 to be held online organized by Department of Mechanical Engineering, CMR Institute of Technology, Bengaluru, India & Core-Facility Centre for Photochemistry and Nanomaterials Gyeongsang National University, South Korea during **April 23-24, 2021**.

Kindly incorporate reviewers comments via the Materials today Proceedings editorial manager website. **For your information, it may take a minimum of 14 days to receive the comments from the editor- Materials today.**

During revision please ensure that the similarity index in the revised paper should not exceed 15% (Excluding Reference)

Please note that the **final acceptance** will be received from Materials Today Proceedings after the successful submission of your **Revised paper & registration** process. Also, it is mandatory for any one of the authors to **present** their respective papers during the allotted session.

Now you are requested to proceed with the **REGISTRATION PROCESS**. Registration fee must be paid through **ONLINE** mode only.
The registration fee details

Mail of Acceptance from FRESM21

Materials Today: Proceedings XRD Investigation of Biodegradable Magnesium Rare Earth Alloy --Manuscript Draft--

Manuscript Number:	MATPR-D-21-04061
Article Type:	SI: FRESM'21
Keywords:	Magnesium (Mg), Strontium (Sr), Gadolinium (Gd), XRD, Mechanical properties.
Corresponding Author:	J R DEEPAK James Raj, BE - ME - (PhD) Sathyabama Institute of Science and Technology Chennai, INDIA
First Author:	J R DEEPAK James Raj, BE - ME - (PhD)
Order of Authors:	J R DEEPAK James Raj, BE - ME - (PhD) Arunkumar T Sasi Venkata Sree Datta Ravipati S S S K Sumanth Varma
Abstract:	Magnesium alloys are biodegradable and biocompatible which are actively using in medical application. Magnesium (Mg) based biodegradable materials possess same mechanical properties as that of bone. This shows Mg is promising substitute for implants due to biodegradability in the physiological environment (inside body). Pure Mg has low mechanical properties, so it is alloyed with other elements. To increase these properties different types of metals are used as alloy. Many Mg alloys are used like Al, Ca, Zn, Sr and also Rare earth metals. In this review we will discuss about the Mg-Gd and Mg-Sr alloys impact on Implants. Casting of Mg-2Sr and Mg-2Sr-2Gd as a new alloy material for the investigation of metallurgical properties.

Paper will be published on **Material Today: proceedings** after the presentation in FRESEM conference(April 23-24) with paper id **MATPR-D-21-04061**