

TRIBOLOGICAL INVESTIGATION OF Mg-Sr-Gd ALLOY FOR ORTHOPEDIC APPLICATION

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

by

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SATHYABAMA

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

**Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of Rahul Srivatsan M R (37150149) and Rushwant Gnanasekar (37150158) who carried out the project entitled **“TRIBOLOGICAL INVESTIGATION OF Mg-Sr-Gd ALLOY FOR ORTHOPEDIC APPLICATION”** under my supervision from December 2020 to April 2021.

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ABSTRACT

Magnesium (Mg) and its alloys have been widely researched for orthopedic applications recently. Mg alloys have stupendous advantages over the commercially available stainless steel, Co-Cr-Ni alloy and titanium implants. Till date, extensive mechanical, in-vitro and in-vivo studies have been done to improve the biomedical performance of Mg alloys through alloying, processing conditions, surface modification etc. A limitation of these current metallic biomaterials is the possible release of toxic metallic ions through corrosion or wear processes that lead to inflammatory cascades which reduce biocompatibility and cause tissue loss.

The intention behind this study is to prepare a Magnesium alloy with improved wear properties. Four different samples of Mg alloy containing Strontium and Gadolinium were prepared. The alloy was developed on the principle of microalloying strategy. Wear analysis were carried out using Pin on a disc wear apparatus. The variation in wear properties of Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-4Gd, Mg-2Sr-8Gd alloys upon adding Gadolinium (Gd) to Mg-2Sr base alloy under the same condition has been analysed.

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LIST OF SYMBOLS AND ABBREVIATIONS

%	Percentage
$\mu\text{g}/(\text{Nm.})$	Microgram per Newton metre
$\mu\text{g}/\text{m}$	Microgram per metre
$\mu\text{g}/\text{N}$	Microgram per Newton
μm	Micron
Ag	Silver
Al	Aluminum
Ba	Barium
Be	Beryllium
Cd	Cadmium
Ce	Cerium
cm	Centimeter
Co	Cobalt
Cr	Chromium
Cu	Copper
d	Average grain diameter
$\text{g}/(\text{Nm.})$	Gram per Newton metre
g/cm^3	Gram per Centimeter cube

g/m	Gram per metre
g/N	Gram per Newton
Gd	Gadolinium
g	Gram
GPa	Giga-Pascal
H ₂	Hydrogen
HAVSRC	Human Aortic Vascular Smooth Muscle cells
HCP	Hexagonal Closed Package
In	Inches
kNm/kg	Kilo newton metre per kilogram
k	Strengthening coefficient
La	Lanthanum
Mg	Magnesium
mg	Milligram
ml/cm ² /day	Milliliters per centimeter squared per day
mm ³ /(Nm.)	Millimeter cube per Newton metre
mm ³ /m	Millimeter cube per metre
mm ³ /N	Millimeter cube per Newton
m	Metre
mm	Millimeter

Mn	Manganese
MPa	Megapascal
Ni	Nickel
N	Newton
Pa	Pascal
Pb	Lead
pH	Potential of Hydrogen
PO ₄	Phosphate
SF ₆	Sulphur Hexafluoride
Sr	Strontium
THR	Total hip replacement
Th	Thorium
Ti	Titanium
TJR	Total joint replacement
V	Vanadium
Zn	Zinc
σ_0	Material constant
σ	Yield strength

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CHAPTER 1

INTRODUCTION

1.1 ORTHOPEDICS

Orthopedic surgery, also known as orthopedics, is a form of surgery that deals with musculoskeletal problems. Orthopedic surgeons treat musculoskeletal trauma, spine diseases, athletic accidents, degenerative diseases, cancers, tumors, and congenital abnormalities with both surgical and nonsurgical methods.

The musculoskeletal system is treated by orthopedic surgeons. This entails:

- Injury or condition will be diagnosed.
- Medication, exercise, casting, surgery, or other options are performed to better the condition.
- physiotherapy or regular exercising can be prescribed to help restore movement, strength, and function.
- To avoid injuries or delay the progression of disease, prevention with awareness and treatment plans is important.

Although orthopedic surgeons treat all parts of the musculoskeletal system, many specialize in particular areas, such as the foot, wrist, knee, spine, shoulder, or hip. They can even opt to specialize in areas such as pediatrics, trauma, or physiology in sports. Some orthopedic surgeons have many areas of expertise.

1.1.1 Surgical treatment

Orthopedic surgeons conduct a vast list of procedures. The following are examples of common procedures:

Arthroscopy: A technique that uses specialized cameras and equipment to view, diagnose, and treat problems within a joint.

Fusion: A "welding" technique where bones are joined together with internal devices and bone grafts (ex: metal rods) to form a bone that is independent and solid.

Internal fixation: A technique that uses metal plates, pins, or screws to hold broken bone fragments in place until they heal.

Joint replacement (partial, complete, and revision): It includes removing an arthritic or weakened joint and using an alternate such as prosthesis.

Osteotomy: It is the cutting and repositioning of a bone to correct a deformity.

Soft tissue repair: The mending of soft tissue, such as torn tendons or ligaments.

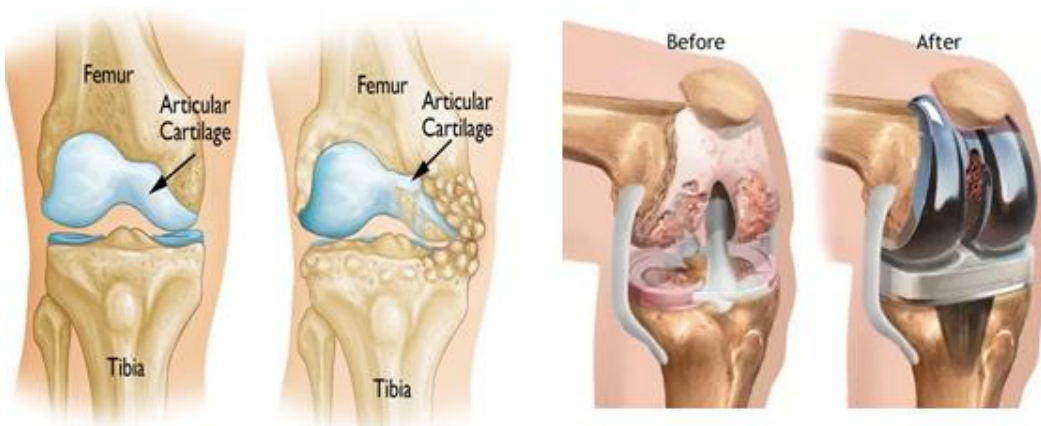


Fig 1.1: Osteotomy and Joint replacement using implant

1.1.2 Orthopedic Implants

The word "orthopaedic implants" refers to implants that are used to repair a missing joint or a broken bone. Orthopaedic implants account for a substantial portion of all medical implants made. Chromium, cobalt, molybdenum, copper, titanium, and zirconium alloys make up the bulk of orthopaedic implants. Metal allergy, metal implant failure, and joint replacement complications are all listed as drawbacks of metallic orthopaedic implants. Device malfunction, discomfort, metallic tanning on the underlying tissue, muscle necrosis, periprosthetic fibrosis, and prosthesis loosening are all severe complications of orthopaedic implants.

Metal allergy and system failure have been recorded in orthopaedic implants used for Total Hip Arthroplasty (THA) and Total knee Arthroplasty (TKA). Failure of metallic orthopaedic implants causes discomfort, loss of stability, disturbed rotation, and pain, which can lead to infection. Hypersensitivity of metal reactions to orthopaedic implants were studied by Wendy et al. Bone screws with self-tapping threads, which are used as elements that help in fastening of prosthetics, are another form of orthopaedic implant. The most common materials used to make bone screws are steel and titanium screws. Recent development in the area of improved bone screw performance has led to the investigation of magnesium (Mg) and screws of Mg coating.

Miniaturization decreases pressure on the screw implant's surface, resulting in a bone screw with a low risk of fracture. Self-tapping, self-lubrication and surface functionalization thread profiles, as well as the use of bioactive materials, were used to increase the speed of the grown of bones and healing of it.

The tapping efficiency of the abrasives is improved, which reduces friction during the screwing-in phase. The implant's mass and size are reduced, which reduces patient recovery time and allows resorbable implants to be used in places like small limbs and the skull.

1.1.2.1 Permanent Implants

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

1.1.2.2 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 this temporary implant there are two types of materials are used.

- I. Non-Degradable Material
- II. Bio Degradable Material

1.1.2.3 Non degradable implants

Non-Degradable materials has 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 for the cost reduction of the second surgery both patients and doctors are showing their interest on the bio degradable implants.

1.1.2.4 Biodegradable implants

Ceramic implants and biodegradable composites are gaining popularity. Because of their ability to provide a required mechanical function for tissue reconstruction after the specified function in the body. There are various barriers, such as the mechanical properties of alloys of Mg degrading faster than anticipated while working on the tissue re-modelling process. Surface coatings and modifications are proposed as an option to achieve the extended mechanical integrity of these materials.

Age, physical health, infection risk, and fracture's type are all important factors in deciding which ceramic implants and bioresorbable composites are required. Biodegradable/bioresorbable polymers are currently being used successfully in pharmaceutical and biomedical applications.

However, these polymers' degradation rates are uncontrollable; they lack mechanical strength and cause inflammation in an acidic environment. Few metallic polymer composites, such as Mg/PCL's moduli of compression have been demonstrated that are comparable to human cancellous bone, as well as amazing osteoblastic differentiation and cytocompatibility properties.

Biodegradable/bioresorbable composites also need a lot of basic research and development for use in bioimplants, according to the existing state of the art. To avoid metal-related toxicity inside the body and to prevent corrosion in the body, metals must be carefully chosen.

Many other considerations must be taken into account, such as the effect of the pH transition on neighboring tissue and metabolic activity in the supplementary phases. Magnesium-based implants have a bright future ahead of them in the current scenario.

1.1.2.5 Future of orthopedic implants

The constant research and development of implants has led the orthopedic industry to improve every year. The new methods of building implants consist of creating new alloys and suitable technologies. The future of such implants seems to have not only therapeutic benefits but also the ability to diagnose. Orthopedic implants have found their application in calculating physical parameters such as force, strain and temperature from within the body and these details are used to treat the body in a quicker manner. The implant data that has been diagnosed contributes to the improvements in implant design, surgical procedure, and postoperative treatment and recovery strategies. Despite decades of testing, smart implants are yet to become a regular part of clinical practice, with a few exceptions. This is primarily due to the fact that integrating existing sensor technology necessitates substantial implant adjustment. In future, the sensors

shall be smaller and inexpensive as compared with today's technology. This brings the orthopedic industry to a level where only little to almost no modification needs to be done for the implants to be efficient.

1.1.2.6 Infections due to orthopedic implants

Because of the failure of implants and the need for revisional procedures under a particular time period in patients who may require complicated, expensive and frequent surgeries, orthopedic implants, particularly replacements of the joints of hip and knee, have a less of a lifetime. As a result, extending the life of implants by many decades will relieve substantial patient pain and save money on health care. Orthopedists usually use calcium phosphate mixtures made up of hydroxyapatite and tricalcium phosphates. Hydroxyapatite has been used as a coating agent on orthopedic implants to enhance the bioactivity of metallic surfaces, which blends the strength of metals with the bioactivity of ceramics, due to their chemical and structural resemblance to the inorganic component of bone. Implant-related surgery is becoming more common, according to reports. Despite advances in surgical procedures, infection can complicate up to 5% of implant-related surgeries. Infection has also resulted to stymie bone tissue healing. Surgical debridement and systemic antibiotic administration are the most common treatments for implant-related infections. Antibiotics administered systemically would not be able to treat a deep implant infection. Many of the contaminated implants would have to be removed. The most popular treatments for implant-related infections are surgical debridement and systemic antibiotic administration. Antibiotics given intravenously will be unsuccessful in treating a deep implant infection. It will be compulsory to take out all of the infected implants.

1.2 MAGNESIUM IMPLANTS IN ORTHOPEDICS

1.2.1 Introduction

In recent years, the advancement of biomaterials for fracture fixation and joint replacement has had a significant impact on orthopedic surgery. Biomaterials make a major contribution to the advancement of human health and well-being. Strains, sprains, dislocations, and fractures are common injuries that cause pain and disability in humans. Fractures are essentially a split in the bone caused by forces that are greater than the osseous tissue's strength. Apart from injuries, age, gender, bone strength, and already existing history of medical conditions all influence the risk of fracture. The majority of fractures are known as traumatic fractures because they occur due to excessive external forces.

Metallic materials, ceramic materials, and polymeric materials are the three primary types of biological implant materials. Metallic materials, such as stainless steel, cobalt-chromium alloys, and titanium alloys, have been widely used in orthopedic applications due to their mechanical strength. Metallic materials will still play a major role as biomaterials in repairing or change of diseased or damaged bone tissue. Metals, as opposed to ceramics or polymeric materials, are better suited for load-bearing applications because of their abilities such as high resistance to fracture and mechanical strength.

The potential outage of toxic metallic ions and/or particles due to wear processes or corrosion that trigger inflammatory cascades that decrease the biocompatibility and causes loss of tissue is a disadvantage of these existing metallic biomaterials. Furthermore, current metallic biomaterials' elastic moduli are not well matched to those of naturally forming bone tissue, resulting in stressed outcomes that can result in decreased stimuli for new bone growth and remodeling, decreasing implant stability. In the case of pins, pipes, and screws used to stabilize extreme fractures, current metallic biomaterials are essentially neutral in vivo, remaining as fixtures for a lifetime that must be removed by a second surgical procedure after the tissue has healed sufficiently. Recurrent surgery increases the costs of the health-care system as well as the patient's morbidity. The physical and mechanical properties of various implants are tabulated in Table 1.1.

Table1.1: Properties of different implant metals and metallic alloys in comparison to the human bone

Properties	Natural bone	Magnesium	Ti alloy	Co-Cr alloy	Stainless steel	Synthetic hydroxyapatite
Density(g/cm ³)	1.8-2.1	1.74–2.0	4.4–4.5	8.3–9.2	7.9–8.1	3.1
Elastic modulus (GPa)	3–20	41–45	110–117	230	189–205	73–117
Compressive yield strength (MPa)	130–180	65–100	758–1117	450–1000	170–310	600
Fracture toughness	3–6	15–40	55–115	N/A	50–200	0.7

1.2.2 Metallic Implants evolution

Metallic materials have seen a substantial increase in use in orthopedic surgery for the manufacture of orthopedic devices such as permanent implants (hip replacement, total joint replacement), temporary implants (bone plates, pins etc.), and permanent implants (prosthesis, etc.) screws, among other items. Mg's efficacy as a biodegradable implant has been around for over a century. In Hank's solution, the surface reaction of commercially available AZ91 and AZ31 alloys is investigated. intended to be used in clinical settings. The quickness with which Mg corrosion is related to the hydrogen (H₂) release. In a few experiments from the mid-nineteenth century, it was discovered that gas inhibits was suggested as a possible solution. The quickness with which in a few studies from the mid-nineteenth century, corrosion of magnesium was linked to hydrogen (H₂) gas release. Mg was suggested as a possible solution. The research, in contrast, was ignited in with a stronger understanding of corrosion kinetics in the early 2000s, the strategies developed to control Mg degradation allowed fractures to heal without the requirement for another surgery to take out the implant. The development of biodegradable Mg-based materials is advancing in order to design orthopedic implants. Magnesium (Mg) and the alloys of magnesium must be biocompatible to be considered as biomaterials. The increasing toxicity of commercial Mg alloying elements presents a potential threat, making their use in

biomaterials more difficult. In this case, extreme caution should be taken in selecting biocompatible elements, optimizing composition design for new biodegradable Mg alloys with desired bio-mechanical properties, and using practical processing techniques to ensure defect-free products.

1.2.3 Metallic Implants – Pros, Cons and Application in orthopedics

Table 1.2: Metallic materials – Pros, Cons and Application

Material	Pros	Cons	Application
316L Stainless steel	Easily available Great properties for fabrication. Biocompatible Toughness	Increased modulus Poor resistance to corrosion. Poor resistance to wear. reaction in the tissues surrounding the implant. Stress shielding effect	Wires, Bone plates, screws in bone and pins.
Co-Cr alloys	Corrosion resistance fatigue and wear strength is high. Biocompatibility on longer run.	Expensive Machining needs expertise. Effect in shielding of stress. Modulus is high. Biologically toxic because of the release of Cr, Ni and Co ions.	Temporary implants wires and bone plates. Total hip replacements (THR) Hard-on-hard or Stem bearing system.
Ti alloys	High corrosion resistance, Modulus is less, High strength as compared to stainless steels, Light in weight	Poor resistance to wear. Poor ductile ability. Expensive.	Fracture Screws, Fasteners, nails, rods, wires and screws, Femoral hip stems, Total replacement of joints. (TJR) arthroplasty-knees and hips.
Mg alloys	Biocompatible, Biodegradable Bioresorbable, Similar Young's modulus and density of bone, less stress shielding effect, Light weight.	Hydrogen evolution during degradation	Bone screws, Bone plates, bone pins etc.

1.2.4 Key properties of Mg based implants

Table 1.3: Key properties of Mg based implants

Property	Characteristics	Details
Density	Low	Mg density (1.738 g/cm^3) is approximate to that of cortical bone ($1.75\text{-}2.1 \text{ g/cm}^3$)
Specific strength	High	strength-to-weight ratio of nearly 130 kNm/kg .
High damping capacity	High	Mg has the absorbing ability of energy of any metal can be used for bearing of load.
Machinability and dimensional accuracy	High	Mg is a structural metal that is easy to machine, and stable final dimensions are achieved easily. Consequently, the production of complex shapes is easy, which is important for the often-intricate shapes that are used medical field.
Stress shielding	Less	Stress-shielding-related problems can be greatly reduced for many orthopedic implants as Mg density is very close to that of bone.
Biocompatibility	Good	Mg is biocompatible and it increases the rate of formation of bone.
Degradation	Good	Corrosion of Mg in the body would gradually result in degrading completely and it would be beneficial for patients having temporary time period to a bodily implant.

1.2.5 Limitations of Mg based implants

- Elastic modulus

Low modulus of elasticity of Mg may be advantageous with respect to resistance to stress and it is important to ensure that any bodily implant is made to resist its load without disturbing its form.

- Degradation

Mg implants usually degrade completely, but at a less rate to the speed of the re-modelling of bone.

- Hydrogen evolution

The released H₂ gas gets collected at the soft tissues in the corners at severe degradation rate. The hydrogen evolution rates for various Mg alloys containing Al, Mn and Zn are documented as 0.01 ml/cm²/day.

1.3 MECHANICAL CAPACITY AND DEGRADATION PERFORMANCE IMPROVEMENT METHODS

Magnesium has a low strength when cast and a high rate of degradation. Properties can be strengthened by using the right metals to alloy and executing environment. The elements used to alloy in self-resorbable Mg alloys must be chosen not only for their mechanical abilities, also for helping their biocompatibility and degradation.

Magnesium gains strength due to the grain refining and strong solution reinforcing mechanisms of alloying elements. Solid-solution stabilization, refinement of grain, and precipitation hardening are some of the ways alloying elements help to improve mechanical properties. The addition of alloying elements with a higher solubility limit are used to reinforce the matrix.

Strengthening of grain size is summarized by the Hall-Petch relation:

$$\sigma_{yp} = \sigma_o + \frac{k}{\sqrt{d}}$$

where σ is told as yield strength, σ_o is said to be the material constant, k is the coefficient of strengthening and d is the average diameter of the grain.

The HCP structure of magnesium ensures solid solutions when alloying elements are added. Grain processing is a tried-and-true way to improve the mechanical properties and corrosion resistance of magnesium-based alloys.

1.4 DESIGN OF MAGNESIUM AND ALLOYS OF MAGNESIUM FOR ORTHOPEDIC APPLICATION

The biocompatibility and mechanical properties of biodegradable Mg alloys must be considered when choosing alloying components. By enhancing Mg's strength by solid solution strengthening and grain refining, alloying elements can be added.

The development of biocompatible Mg alloys based on toxicity, strengthening ability and degradation rate.

The alloy corrosion products should be free toxicity and quickly absorbed, dissolved, and excreted by the surrounding tissues. Toxic, allergic, and nutrient elements are the three forms of elements found in the human body.

- 1) Toxic elements: Ba, Be, Th, Cd, Pb
- 2) Allergic elements: Cu, Ce, V, Pr, Ni, Co, La, Al, Cr
- 3) Nutrient elements: Sr, Sn, Zn, Mn, Ca

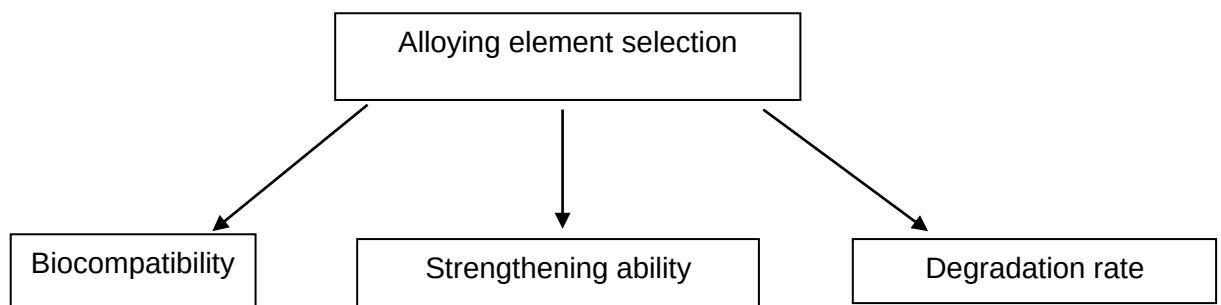


Fig 1.2: Selecting alloying element

1.5 IMPORTANCE OF ALLOYING ELEMENTS

Magnesium is one amongst the seven macro minerals essential for good health. A healthy adult body needs 325–425 mg of magnesium per day. In the average adult body of 70kgs, there are approximately 19 mg of Mg, with approximately 65 percent of that contained in Teeth and bone and the other sources distributed among the blood, body fluids, organs, and other tissues.

The modulus of elasticity of magnesium (4145 GPa) is higher as compared to iron (211.4 GPa) or zinc (90 GPa) and is similar as of natural bone (320 GPa). Since the implant bears the majority of the load, a mismatch in elastic modulus may trigger bone stress shielding. This Incompatibility of the bone can lead to serious clinical problems, such as loosening of the implant, damage in the healing process, thickening of the skeleton and chronic inflammation.

Furthermore, when used as bone fixtures, magnesium implants have been shown to promote the development of new bone. The high corrosion rates and subsequent loss of mechanical integrity of Mg-based alloys at pH levels varying in range 7.3 and 7.8 and high chloride environments continue to limit their widespread use.

Moreover, the rapid development of hydrogen gas bubbles, which typically occurs during the first week after surgery, can be a side effect of Mg-based implants. Despite the fact that magnesium is favored for implant applications, faster corrosion and hydrogen evolution remain a major concern.

Magnesium and its alloys, which have, high specific stiffness, specific strength, a similar modulus to human bones, and unique biodegrading ability, are attracting increasing interest for biodegradable materials applications. Mg alloys that have been alloyed and have had their surfaces treated help to slow down the rate of deterioration until the broken bones have healed completely.

It is an essential micro-mineral that, along with Ca and Zn, is responsible for about many biochemical reactions in the human body. Calcium, buoyed by magnesium, has been shown in several studies to improve bone mineral density. Ca, Zn, Mn,

and Sr are the important and most keen alloying nutritional elements (Sr). The effect of various alloying elements on bone implants is tabulated in Table 1.4.

Table 1.4: Mechanical properties of Alloying elements

Alloying element	Mechanical properties
Ca	Grain refinement, Improves corrosion resistance
Zn	Reduce hydrogen gas evolution during bio-corrosion, Improves yield stress
Mn	Improve corrosion resistance
Sr	Increase bone mass and reduce the occurrence of fractures. Refinement of grains
Sn	Increases compressive strength and corrosion resistance
Ag	Antibacterial effect, Improves tensile strength, Less corrosion resistance

1.5.1 Strengthening ability

The role of alloying elements in improving mechanical properties is considered as one of the major disadvantages. Ni, Zn, Ca, Al and Cu can all improve strength and ductility. Some elements, such as Bi, Sn, Sb, and Pb, increase magnesium's strength while decreasing its ductility. Ni, Cu, Fe, and Co, which are considered impurities, should be avoided if at all possible.

Impurities in Mg-based alloys must be carefully regulated, otherwise the implant can degrade and cause toxic effects inside the human body. As Mg alloys are reacted with alloying materials, the presence of impurities weakens the mechanical integrity of the alloy by forming secondary phases.

1.5.2 Degradation rate

Magnesium alloys must have enough strength not only when they are implanted, but also when they weaken over time and in contact with body fluids, in order to be used as a weight-bearing implant. It's also important that the implants stay in place

at least until the bone heals. As a result, numerous studies to assess mass loss and strength evolution during the implantation or immersion phase have been conducted. Understanding galvanic and pitting corrosion, two of the important common forms of corrosion that Mg alloys exhibit, is important for efficiently tuning the composition and microstructure.

Since magnesium reacts anodically when it comes into contact with other metals, galvanic corrosion occurs. When the concentration of an alloying element exceeds the corresponding maximum solid solubility in magnesium, galvanic couples occur. The galvanic effect magnitude is determined by a variety of variables, including the magnesium matrix's crystal orientation, the type of secondary phases and impurity particles, the solution in which the alloy is immersed and the grain size.

Corrosion behavior is often influenced by the concentration and distribution of secondary phases. Corrosion efficiency is usually enhanced by a fine and consistent distribution of secondary phases. By minimizing internal galvanic corrosion, alloying elements and other intermetallic phases with electrochemical potential similar to magnesium (2.37 V) will increase corrosion resistance by reducing the internal galvanic corrosion. Magnesium, as the element with the lowest solubility limit, acts as a cathodic site, causing extremely harmful corrosion.

1.6 FABRICATION

1.6.1 Hot and cold working

Magnesium alloys harden quickly with some kind of cold work, so they can't be cold molded extensively without annealing. Sharp bending, spinning, and drawing must be done at temperatures between 500- and 600-degrees Fahrenheit (260 and 316 degrees Celsius), while gentle bending around broad radii can be done cold. The effects of slow formation are superior to fast shaping. Since the press allows for more time for metal movement, it is favored over hammer forging. The temperature range for plastic forging is 500 to 800 degrees Fahrenheit (260 to 427 degrees Celsius). Owing to a lack of usable deformation mechanisms, metal worked outside of this range is easily broken.

1.6.2 Casting

In casting, magnesium alloys, especially precipitation-hardened alloys, are used. Casting techniques include sand, permanent mould, and die casting, but plaster-of-Paris casting has yet to be based. Since magnesium reacts with moisture in the sand, forming magnesium oxide and liberating hydrogen, sand casting in green-sand moulds necessitates a special technique.

The oxide causes blackened areas on the casting's surface known as burns, and the released hydrogen can cause porosity. To avoid the reaction, inhibitors like Sulphur, boric acid, ethylene glycol, or ammonium fluoride are mixed into the damp sand.

All gravity-fed moulds need a large column of molten metal to generate enough pressure to push gas bubbles out of the casting and allow the metal to take the mold's detail. In certain cases, the casting wall thickness should be at least 5/32 in permanent mould castings are made of the same alloys as sand castings and have similar physical properties. Aluminum moulds can also be modified to produce magnesium-alloy castings since the solidification shrinkage of magnesium is similar to that of aluminum (although it may be necessary to change the gating). For mass production of small pieces, pressure cold-chamber castings are used. Action of the fluid metal with the cold die causes rapid solidification, resulting in a casting with a thick structure and excellent physical properties. The finish and dimensional accuracy are excellent, and only extreme precision necessitates machining. These castings are usually not heat treated.

1.6.3 Welding, Soldering and riveting

Many common magnesium alloys can be welded and cut with gas or resistance welding equipment, but not with an oxygen torch. Magnesium alloys are not welded to other metals because brittle intermetallic compounds can form when they are welded together, and the combination of metals can promote corrosion. Magnesium alloys can only be soldered to repair surface defects in components. The solders are much more corrosive than aluminum solders, and the components

should never be subjected to stress. Aluminum or aluminum-magnesium alloy rivets are commonly used in riveted joints in common Mg alloy structures.

Since they must be powered when hot, magnesium rivets are rarely used. Since punching tends to give a rough edge to the hole and trigger stress concentrations, the rivet holes must be drilled, especially in heavy sheet and extruded sections.

1.7 ROLE OF STRONTIUM (Sr)

The element strontium is essential in the human body because it promotes the growth of osteoblasts and prevents bone resorption. It 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. It is also reported that addition of Sr in Mg alloy increased the both mechanical and corrosion properties. Sr can enhance corrosion and mechanical properties by refining grain structure, decreasing shrinkage porosity, and increasing relative density of castings. Some trace elements chemically similar to Ca, such as Sr, have pharmacological effects on bone when present at levels higher than those necessary for normal cell physiology. Sr is found in bone and connective tissue in amounts ranging from 320 to 400 mg in the human body. Strontium has been discovered to have anabolic properties in bone, suggesting that it may have significant beneficial effects on bone balance in both normal and osteopenic animals, in addition to its antiresorptive properties.

As a result, strontium has been suggested as a possible cure for osteoporosis. Strontium may substitute calcium to some degree in various biochemical processes in the body, including replacing a small proportion of the calcium in hydroxyapatite crystals of calcified tissues such as bones and teeth. Since it is chemically similar to calcium. These tissues are enhanced by the presence of Sr in these crystals. Strontium also helps to pull calcium into the bones. Sr has a solubility of 0.11 weight percent in Mg. It aids grain refinement and improves Mg's corrosion resistance.

1.8 ROLE OF GADOLINIUM (Gd)

Table 1.5: Density of alloying elements

Property	Sr	Gd	Zn	Ca	RE
Density(g/cm ³)	2.64	7.90	7.13	1.55	4.47

From Table 1.5, we can infer that when it comes to bone implants density plays an important role. Gadolinium (Gd) with a density of 7.9g/cm³ makes it the denser in comparison with other common alloying elements such as Strontium, Zinc, Calcium and rare earth elements. Thus, it can be considered as one of the best substitutes to make the bones denser.

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, many cast magnesium alloys containing Gd and additional REE have been studied. Gd has a suitable biocompatibility and high solubility in Mg. Recent studies states that Mg-Gd alloys are developed as a possible material for bone implant applications. the composition of Gd was optimised when taking into account its mechanical properties and corrosion resistance.

Also, studies have shown that addition of Gadolinium decreases the wear rate and improves the wear resistance of the alloys. Although Gadolinium (Gd) alone is toxic to human beings, but when reinforced as alloying elements makes it safer to use in the human body.

1.9 PRESENT STUDY

This study focuses on developing an alloy material based on micro alloying strategy with improved tribological properties for bone implants used in orthopedics. Magnesium is selected as the parent metal. Mg alloys have attracted high interest in recent years due to their tremendous biocompatibility, biodegradation, density and elastic modulus which are similar to human tissues. These properties make them promising for biodegradable medical implants, such as cardiovascular Gadolinium (implantation, and bone tissues. A few novel biodegradable Mg alloys which include Mg-Sr, Mg-Zn, Mg-Ca, and Mg-RE (rare earth), have been developed recently.

Since it has many key benefits which includes low density, high specific strength, high damping capacity, high machinability & dimensional accuracy, low stress shielding, good biocompatibility and degradation. Strontium and Gadolinium are selected as the alloying elements. Strontium promotes the growth of osteoblasts and prevents bone resorption whereas Gadolinium enhances the density of the implants.

Four Mg based alloys were used in this study: Mg-2Sr, Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-8Gd. The alloys were prepared using permanent mold casting. The composition of Gd was varied in each alloy sample. The samples were then placed on a Pin on a disc wear apparatus. Wear properties like wear rate, resistance and co efficient of friction of each sample were noted and analyzed. SEM was used to examine the wear tracks, worn surfaces, and wear debris of the alloys.

The wear behavior of commercial Mg alloys has been widely studied for industrial application. However, there have been less reports on the wear resistance of novel biomedical Mg alloys. As a result, studying the wear behavior of biodegradable alloys is important. Studies have shown biodegradable Mg-Sr demonstrate good biocompatibility and prevents osteoporosis. From the table above, Gd is much denser and suggests it can be used instead of Zn, Ca, RE as reinforcement. In this study, we are going to investigate the variations in wear property of Mg-Sr alloy upon the addition of Gadolinium (Gd).

CHAPTER 2

LITERATURE SURVEY

Many researchers have made immense contributions in the field of material science. There are several alloys which were discovered and put into use in several medical applications. Research was done on how the addition of Sr and Gd impact the wear properties of Magnesium based alloys. Studies on the biocompatibility, degradation, biological response of Magnesium based alloys were also investigated.

2.1 Mg-Sr CASE STUDY

(Hongyan Tang et al.,2014) studied the production of Mg-Sr-RE (RE = La, Nd, Gd) alloys for stent applications based on a microalloying strategy that aims to regulate biosafety while also improving degradation. The results reveal that the built Mg-Sr-RE alloys are close to being a single-phase solid solution, with total alloying additions limited to 0.5 wt.%. Mg-Sr-RE alloy has 0.5–2 times the ductility of binary Mg-Sr while having 45–66% lower corrosion rates. Mg-Sr-RE alloys reduce HAVSMC proliferation, stimulate HUVEC formation, and endothelialization - related gene expression in both indirect and direct cell assays, suggesting that they can help in the healing of stent-assisted vascular injuries. The Mg-Sr-RE microalloying alloys have a fine-grained microstructure and a high ductility between 20–40%. As compared to Mg-Sr power, corrosion rates are reduced by 47–68%. Surprisingly, during a 10-day culture cycle, RE microalloying improved endothelial cell binding, spreading, and proliferation on the surface of Mg-Sr-RE alloys while inhibiting smooth muscle cell proliferation.

(Junjie Han et al.,2014) studied ways to assess and correlate the degradation of Mg–Sr alloys for grafting with their clinical requirements. Microstructure and efficiency of Mg–Sr alloys were investigated, as well as in vitro degradation and biological properties such as in vitro cytocompatibility and in vivo implantation. The results revealed that due to the galvanic corrosion and intergranular distribution of the second phase and micro-, the as-cast Mg–Sr alloy degraded at a faster rate

than the as-extruded alloy. However, the coating safety, which was found to be cytocompatibility and suitable for bone repair after in vivo implantation, could tailor the initial degradation.

(FupoHea et al.,2014) studied how Magnesium and Strontium promote osteogenesis while suppressing osteoclastic activity. They are nontoxic at high concentrations, making magnesium-strontium phosphate bioceramics ideal for treating osteoporotic bone defects. The researcher wanted to see how strontium affects cell mechanical strength and biological production. The magnesium-strontium phosphate bioceramics, with the exception of $\text{Mg-2.9Sr-0.1(PO}_4)_2$ and $\text{Mg-2.25-Sr0.75-(PO}_4)_2$, had substantial compressive strength, according to the findings. Overall performance of $\text{Mg}_{2.75}\text{Sr}_{0.25}(\text{PO}_4)_2$ bioceramics has reflected by considerably high compressive strength, in vitro osteogenesis and osteoclastic activities. As a result, the bioceramic $\text{Mg-2.75Sr-0.25-(PO}_4)_2$ is the perfect substitute biomaterial for osteoporotic bone regeneration.

(Junjie Han et al.,2015) developed magnesium strontium alloys (Mg-Sr) for the bioresorbable bone substitute application. Strontium (Sr) is a bioactive factor that can help to promote bone formation and density. Sr has been shown to increase osteoprotegerin expression and decrease receptor activator of nuclear factor kappa B ligand expression in osteoblasts, as well as promote osteoblastic cell replication by phosphorylating the calcium sensing receptor and signal-regulated kinase 1/2. Intermetallic phases strengthened precipitation and galvanic corrosion as a result of the addition of Sr as an alloying element. Mg-Sr alloys were tested for mechanical strength, degradation rate, and cytocompatibility, and found to have major advantages over commercial bone substitutes.

(Junjie Han et al.,2015) SEM and EDS were used to analyze the surface and cross-section morphology of the Mg-Sr alloy. The phase composition was investigated using XRD. The chemical composition of the coatings was determined using X-ray photoelectron spectroscopy with a spot size of 500 nm diameter. The energy resolution for the high resolution and survey scans was 0.1 and 1 eV, respectively. As-cast Mg-Sr microstructure had the usual metallographic microstructural characteristic of α -Mg grains, with intermetallic

phases precipitating along grain boundaries. The microstructure of the grains became homogeneous after extrusion, and the intermetallic phases were split and detected within the grains. Back-scattered electron images of Mg–Sr alloys demonstrated the presence of Sr in grain boundaries and white particles dispersed in the microstructure. The chemical compositions of the matrix and the second phase at grain boundaries were calculated using EDS analysis, with the amount of Sr contained more in the second phase at grain boundaries. The degradation of Mg–Sr alloys differed depending on their state and was also influenced by coating safety.

(Mei Li et al.,2016) Endogenous and exogenous bone-graft materials are frequently needed. To help, fill voids, and boost biologic repairing of skeletal defects caused by traumatic or non-traumatic injury. Autologous bone remains the "gold standard" for promoting bone repair and regeneration, However, its supply may be restricted, and the method for harvesting the material is fraught with dangers. Several studies have shown that Mg-Sr alloys have potential benefits such as adequate mechanical performance, good bio compatibility, and osteo-conductive properties, making them ideal for bone replacement alternatives.

(W. Wang et al.,2019) studied the Mg-Sr alloy samples upon conducting extrusion. After the extrusion procedure, broken intermetallic phases were detected in the microstructure of as-extruded Mg-1.5Sr without coating along the processing route. Furthermore, because of the complex nature of the process, non-uniform sized grains were detected. During the extrusion process, recrystallization occurs. The longitudinal image of a back-scattered electron (BSE) image. Long strip intermetallic phases were observed, while water-drop shaped second phases were observed in the transverse direction. The chemical composition of the second phase at grain boundaries was studied, with Sr being the most abundant element (25 wt.%).

(W. Wang et al.,2019) In trauma and orthopedic surgery, the repair of large bone defects remains a major challenge. The aim of Tissue engineering is to resolve this issue by integrating osteogenic signals, such as hybrid growth factors, into scaffolding biomaterials. However, when titanium alloys are fixed as orthopedic

implants, the effect of shielding stress and non-absorbable character are the key limitations. Since alloying and processing conditions may affect the microstructure and distribution of intermetallic phases, they can tailor Mg degradation and bioactivity. The efficacy of healing of bone of binary Mg-Sr alloy system in either casted or rolled condition for skeletal applications has been demonstrated in previous studies.

(W. Wang et al.,2019) Coated magnesium grafts demonstrated superior new bone growth, suggesting the possibility of using a biodegradable Mg-Sr alloy graft to treat segmental defects. Both of these factors contribute to a lack of bioactivity in bone formation. Meanwhile, indirect touch cell studies, X-ray radiographs, and histological photographs revealed that SrP coating had superior osteo-inductive properties, owing to greater release of nutrient elements such as Mg^{2+} , Ca^{2+} , and Sr^{2+} . Considering the effect of bioactive surface, proper rate of degradation, and beneficial ions released in the beginning stages of healing, SrP has proven to be an effective process of coating.

(Hu'seyin S, evik et al 2014) studied the effect of Strontium on the microstructure and mechanical properties of Mg-6Al-0.3Mn-0.3Ti-1Sn. The alloys were prepared using squeeze casting process under normal conditions. Results of Optical Microscopy (OM), Scanning electron microscopy (SEM), Energy Dispersive Spectrometer (EDS) and X-Ray diffraction (XRD) analysis were analyzed. The strontium addition altered the structure and grain size was refined, according to the findings. The alloys' yield strength and hardness increased steadily as the strontium content increased, while their elongation decreased. The tensile ability of the underlying alloy was also increasing may be taken up to 1% of the total weight.

As the Sr amount got increased, the grain size reduced. Many studies have shown that Sr has a major grain refining effect in Mg-Al alloy systems in the past. Sr has a poor solid solubility in Mg matrix, as is well known (wt. percent 0.11). Sr solute atoms are rejected onto the front end of solid/liquid interface during solidification, which inhibits grain growth and encourages nucleation in the melt. Furthermore, since Al_4Sr and Sr-Mg-Sn intermetallic particles have a higher formation temperature, they could serve as nucleation sites, resulting in finer grains with the addition of Sr.

The tensile abilities of base alloys with various strontium additions. The yield intensity improved significantly as the amount of Sr added increased, while the elongation decreased. The alloy containing 2 wt. percent Sr had the highest yield strength. The tensile strength of alloy 1 was also improved by adding Sr up to 1% by weight.

(X.N. Gu et al in 2012) Conducted tests on Mg-Sr alloys that ranges from 1 to 4% of strontium prepared by alloying of pure Mg (99.98%). And samples made (10*10*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 α -Mg with an HCP 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%. We calculate in terms of H₂ evolution and mass loss. In vitro results shown Mg-Sr can be used as temporary implant.

(Bin Jiang et al 2012) investigated the effect of Sr on microstructure and aging behavior of Mg-14Li alloys. The influence of Sr on the microstructure and ageing behavior of Mg-14 wt.% Li-xSr alloys was investigated. Both as-cast and as-extruded microstructures of Mg-14 wt.% Li-xSr alloys indicated that b(Li) and Mg-2Sr were the two primary phases. Surprisingly, the rise in Sr content coincides with the increase in Sr content.

The grain sizes of the as-extruded and as-cast Mg-14 wt. percent range from 0.14 wt. percent to 0.39 wt. percent. The influence of Sr on the microstructure and ageing behavior of Mg-14 wt.% Li-xSr alloys was investigated. The microstructures of Mg-14 wt. percent Li-xSr alloys as-extruded and as-cast revealed that the two primary phases were b(Li) and Mg-2Sr. Surprisingly, the increase in Sr content occurs at the same time as the increase in Sr content.

It was concluded that the grain size of both as-extruded and as-cast Mg–14Li alloys was found to decrease as the content of Sr increased. The primary phases in the alloys were classified as b(Li) and Mg₂Sr, with the fraction of volume of Mg₂Sr increasing as the content of Sr increases.

Because of the crystallography matching relationship, the Mg₂Sr became a fine granular compound after extrusion and can serve as a site for nucleation for dynamic recrystallization during extrusion. Moreover, after Li–0.19 wt.%Mg–14 wt.%Sr alloy is treated to age at standard room temperature and at 100°C for 384hrs, no sign of over-aging appears since the hardness at the micro level of this alloy keeps stable and both b(Li) and Mg₂Sr have little change during aging treatment.

2.2 Mg- Gd CASE STUDY

(Jie Zhang et al.,2014) sliding wear tests in dry conditions on t6-treated and as-cast Mg-3Gd-1Zn-0.4Zr (wt. percent, GZ31K) and Mg-6Gd-1Zn-0.4Zr (wt. percent, GZ61K) alloys using a ball-on-disk configuration. Under three separate applications of load 50N, 100N, 200N respectively, the alloys and wear rate and coefficient of friction were measured. SEM was used to investigate the morphologies of worn surfaces. As the load increases, the rate of wear for the as-cast Mg-Gd-Zn-Zr alloys increase. The wear rates of T6-treated Mg-Gd-Zn-Zr alloys, but in contrast, initially increase with the presence of needle-like precipitates in a large amount, but then it decreases due to apparent work hardening. Abrasion, plastic deformation, oxidation, adhesion, and delamination wear processes are all observed.

(H.R. Jafari Nodooshan et al.,2014) investigated the effects of various additions of Gd on the wear actions of Mg–xGd–3Y–0.5Zr alloys heat treated to T6. The tests for wear analysis were performed on an AISI 52100 style bearing steel ball counterface with a range of load of 3–15 N, a range of sliding speed of 0.035–0.193 m/s, a range in temperature of 25 to 200 °C, and a continuous sliding distance of 500 m using a wear apparatus with Ball-on-flat type apparatus. The

findings showed that as the sliding load increased, the wear rate of the tested alloys increased. The rate of wear of the Mg–6Gd–3Y–0.5Zr alloy was reduced by around 24% when the wear temperature was increased to 200 °C. Wear resistance of the alloys improved as wear speeds increased because of the birth of stable oxide layers on the worn surfaces. Under the same conditions, the alloy that has 12 wt. percent Gd demonstrated higher wear resistance than the lower Gd containing alloy contents.

(Yong Liu et al.,2014) Extrusion was used to make Gd in a Mg alloy with the composition Mg-1.5Zn-0.15Gd (at percent). Commercially pure Mg (99.9%), Zn (99.9%), and Gd are used as raw materials (99.9 percent). The materials were melted at 1023 degrees Fahrenheit and poured into a permanent mould at 993 degrees Fahrenheit. At 573 K, the Mg-1.5Zn-0.15Gd ingot was extruded into a rod (= 30 mm) with an 11:1 reduction ratio. A computerised ball-on-disk wear testing system (HIT-1) was used to perform wear tests of dry sliding on the surface parallel to the extrusion direction at room temperature (293.4 K, relative humidity 40%). The experiments were carried out with various loads (10, 7, and 5 N) and range of sliding speeds from 0.05 to 0.7 m/s for a total of 300 seconds.

(Yong Liu et al.,2014) The microstructure of Mg-1.5Zn-0.15Gd alloys, as seen in SEM photographs, is typical of wrought magnesium alloys, with the Mg matrix and coarse secondary phases. There tend to be a lot more fine-grained precipitates with a uniform distribution. After the exam, the fraction of volume of secondary phase rises from 0.5 percent of the as-extruded sample to 4.3 percent. Meanwhile, after 24h, the alloy's hardness increased marginally from 52 Hv to 60 Hv. The FC of the extruded alloy reduces from 0.39 in 0.05 m/s to 0.35 in 0.5 m/s under a 5 N load. The difference in friction coefficient between the Mg-1.5Zn-0.15Gd alloy before and after CT, however, is less than 5%.

(Yong Liu et al.,2014) With rising sliding speed, the rate of wear of Mg-Gd alloy decreases. At sliding speeds ranging from 0.05m/s to 0.7m/s, two distinct wear mechanisms were discovered. Abrasive wear occurs at a high rate during 0.05 m/s in low-speed sliding.

(Yong Liu et al.,2014) After cryogenic treatment, the wear resistance ability of Mg-Zn-Gd alloy was increased. the hard-phase support effect is weakened due to the formation of the layer of oxidation on the wear surfaces during the wear process, such an improvement is small at high speeds of sliding with oxidative wear. According to the temperature increment when the sliding process occurs, a wear map was proposed to reflect the key wear mechanisms of Mg-Zn-Gd alloy. In a function of load and sliding speed, the wear process could be easily explained as abrasive wear and oxidative wear.

(YulingXu et al 2016) investigated the effect of Gd on yield strength and hardness of Mg alloys. By combining Zr and hot extrusion, different grain sizes of alloys were formed. After extrusion and solid solution treatment, the alloys were tested for compression/tensile yield strength and hardness. tensile/compressive and hardness data were used to measure Hall Petch constants.

The findings showed that Mg–Gd alloys having different grain sizes and similar Gd content had nearly identical hardness, indicating that the size of grains had little influence on hardness. With increasing Gd content, there occurred a linear increase in hardness (dHv/dc 25 kg mm²/at percent Gd).

Before extrusion, the Mg–2Gd alloy produced the largest grain size, which was higher than pure Mg. With increasing Gd content, there is a clear reduction in grain size. The average grain size of Mg–Gd–Zr alloys was about 350 m with low Gd content, and it decreased to 130 and 100 m for Mg–10Gd–Zr and Mg–15Gd–Zr alloys, respectively.

2.3 SUMMARY OF LITERATURE SURVEY

Several literatures have been studied thoroughly and many results were analyzed. These results helped us in choosing the alloying elements for bone implants. The current research has many facts to understand and based on that the conclusions are incorporated. The following are the conclusions drawn from the literature survey:

- The alloys containing different Gd contents showed similar wear mechanisms in the same conditions.
- In contrast, the powerful mechanism of wear at the higher loads was determined to be a mixture of abrasion and delamination wear.
- Abrasion controls the wear mechanism under the 50 N range, while adhesion is the main mechanism of wear under 100 N, and deformation of plastic has a huge influence on the morphology of surface under 200 N.
- In addition, oxidation can be observed under every condition of wear occurrence, and the detection of delamination is under 200 N.
- Magnesium and strontium promote osteogenesis by suppressing osteoclastic activity
- The strontium addition altered the structure and grain size was refined, according to the findings.
- Mg-Sr alloys have potential benefits such as adequate bio compatibility, good mechanical performance, and osteo-conductive properties, making them ideal for bone replacement alternatives.
- The alloys' yield strength and hardness increased steadily as the strontium content increased, while their elongation decreased.
- The tensile strength of the underlying alloy was also increased.
- The yield intensity improved significantly as the Sr amount that was added increased, while the elongation decreased.
- The alloy containing 2 wt. percent Sr had the highest yield strength.
- The tensile strength of alloy 1 was also improved by adding Sr up to 1% by weight.
- They are nontoxic at high concentrations, making magnesium-strontium phosphate bioceramics ideal for treating osteoporotic bone defects.
- Mg-Sr coated implants possessed appropriate degradation rate, in addition to, dual biological function of bioactive surface and ionic dissolution, indicated its promising application in segmental defects regeneration.
- Intermetallic phases were observed to be broken along the processing path in the microstructure analysis of the extruded Mg-1.5Sr with no coating after the extrusion method.

- Furthermore, because of the complex nature of the process, grain sizes with non-uniformity were present.
- Before extrusion, the Mg alloy had a larger grain size even than pure Mg.
- After addition of Gd, the increase in hardness was linear and decrease in the grain size was observed.
- It was also found that the formation of the layer of oxidation on the wear surfaces is weakened the hard-phase support effect during the wear process at high sliding speeds.
- At sliding speeds ranging from 0.053 to 0.17 m/s, two distinct wear mechanisms were discovered. Abrasive wear occurs at a high rate during low-speed sliding at 0.053 m/s.
- As the sliding speed reaches 0.5 m/s, the wearing mechanism of the alloy oxidative wear with a low coefficient of friction and rate of wear.
- Mg–Gd alloys with almost the same content of Gd and different grain sizes had nearly identical hardness, indicating that grain size had lesser influence on hardness.
- After the addition of Gd, the volume fraction of secondary phase rises from 0.5 percent of the as-extruded sample to 4.3 percent.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

To investigate the wear property of the Magnesium alloy when they are alloyed with Strontium and Gadolinium for treating bone defects effectively. To analyse the changes in the grain size and the microstructure of the alloys.

3.2 SCOPE

- To prepare Magnesium alloys with Strontium and Gadolinium which can be used for bone implants
- To check the biocompatibility of the Magnesium alloy used for orthopaedic applications after adding Strontium and Gadolinium.
- Taking the Mg-2Sr alloy as base alloy and adding the gadolinium to it with weight ratio of Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-8Gd.
- To check the difference in the wear properties of the samples and analyse whether the addition of Gd impacts the wear property of the Mg-2Sr base alloy.

CHAPTER 4

MATERIALS AND EXPERIMENTAL METHOD

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 Kalpadi, 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)
- IV. Hot Mould

4.3 WORKFLOW PROCESS:

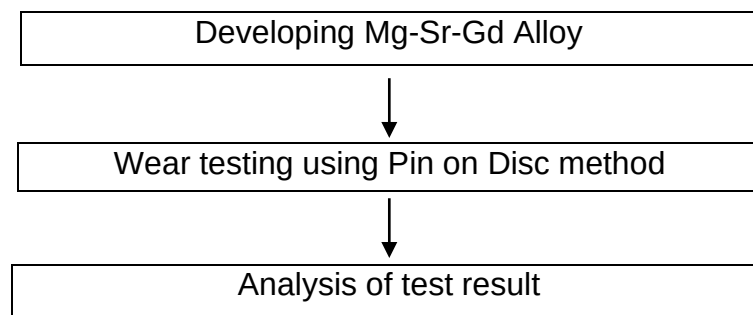


Fig 4.1: Workflow

Four Mg-based materials were used in this study: Mg-2Sr, Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-8Gd. Casting of the materials were done at Matrics Laboratory, Kanyakumari. For the casting, pure materials were used: Mg (99.98 %), Gd (99.98 %), Sr (99.98 %).

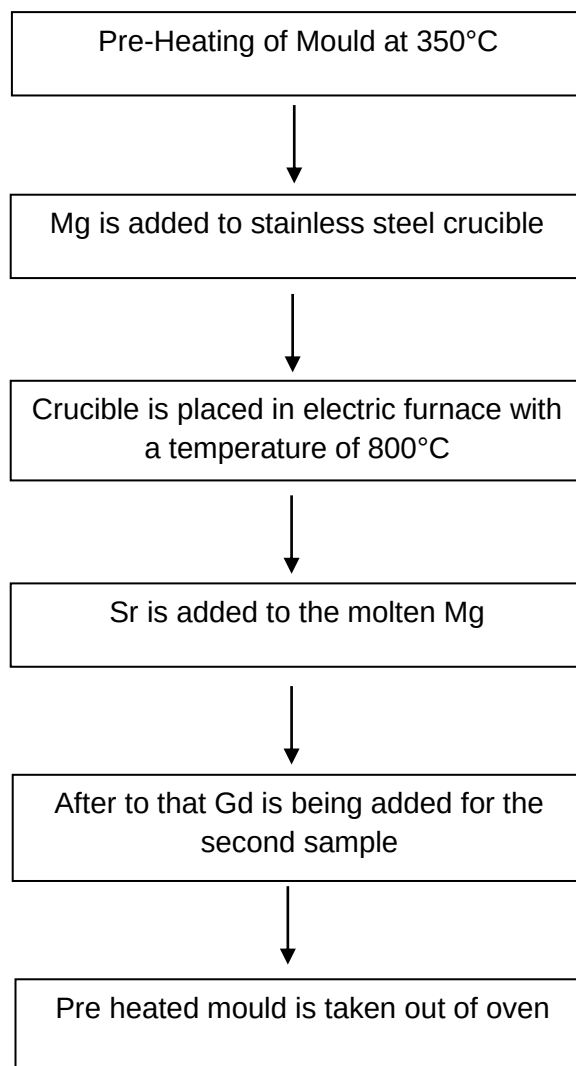
The Mg-2Sr was produced by permanent mold gravity casting. the melt was held for 45 minutes, after melting the pure Mg, at 750°C and Mg-Sr (%wt.) base alloy was added with continuous stirring for 10 minutes and was reheated to 750°C. The melt was poured into a preheated (400°C) permanent die-cast steel mold using a mold release agent.

The Mg-2Sr-2Gd (wt.%), Mg-2Sr-4Gd, Mg-2Sr-8Gd alloys were produced by a permanent mold gravity casting process, a stainless-steel crucible was preheated to 500°C~550°C in a 2 KW electrical resistance furnace under argon (Ar) protection. A magnesium ingot with a purity of 99.98% was placed into the stainless-steel crucible for heating and melting. Ar gas (2% wt.) and SF₆(98%) flew through the crucible to prevent oxidation during heating and melting. The heating temperature was then increased to 720°C and the dried industrial pure gadolinium (Gd) and Mg-Sr (2%wt. Sr) base alloy were added to the metallic mixture.

In order to ensure that the alloy elements adequately reacted with each other, the magnesium alloy mixture was then stirred. The mixture was then uniformly distributed. After the magnesium alloy was maintained in the melting state for 30 minutes, the alloy was de-slagged and cast into a die-cast mold to form ingots.

4.4 CASTING PROCESS

- i. Di steel mould is pre heated at 350°C in an oven.
- ii. Mg metal placed in a stainless-steel crucible is charged and melted, with the furnace temperature set at 800 °C
- iii. Skimming is done using SF₆ and Ar gas to remove any material or particles that are floating on the Mg surface.
- iv. After melting, Mg-Sr base alloy and Gd is being added.
- v. The pre heated mould is taken out from the oven.
- vi. The melted Mg alloy is poured into the pre heated mould and casted using gravity casting method.



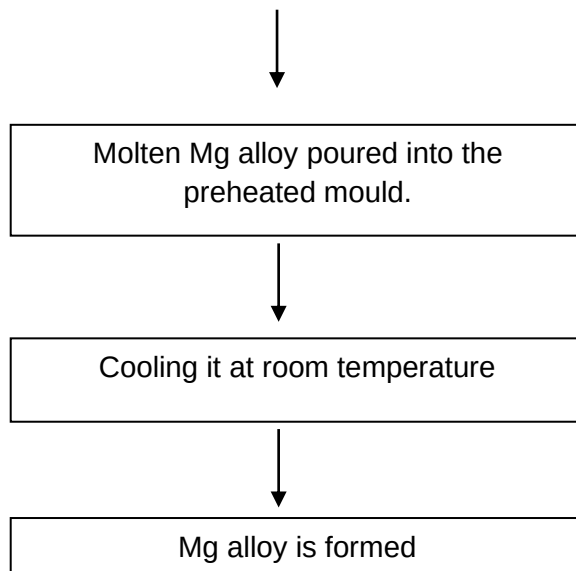


Figure 4.2: Alloy preparation



Fig 4.3: Melting of Mg



Fig 4.4: Skimming using Ar, SF₆ gas



Fig 4.5: Pouring Mg alloy into the mould



Fig 4.6: Gravity Casted Mg-2Sr alloy

4.5 TESTING WEAR PROPERTIES - PIN ON DISC METHOD

In this project, we have tested the tribological property using the Pin on disc test. The mechanism is quite simple considering the work it offers.

Wear test was carried out to analyse the wear performance and to investigate the wear mechanism.

From a material standpoint, the test is used to assess a material's wear property in order to determine if it is suitable for a particular wear application.

Wear tests are carried out in surface engineering to assess the potential of using a particular surface engineering technology to minimise wear for a specific application, as well as to investigate the impact of treatment conditions (processing parameters) on wear efficiency, in order to achieve optimal surface treatment conditions.

4.5.1 Environments of wear testing

Various types of tribometers are used to characterise materials for friction and wear (typically wear rates and wear resistance), with the pin on disc the most popular. The method's popularity stems from its relative simplicity and abundance of tribological contacts that can be well represented by a simple pin on disc motion: from dry bolt screw contacts to rail wheels to rail contact and lubricated biological implant contact. Several motion modes, such as unidirectional, fretting modes, and, more recently, some other complex motion patterns, are usually tested in this test. Typically, the experiments can be conducted in accordance with any testing environment.

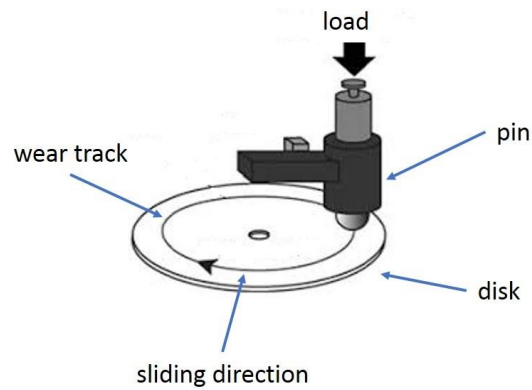


Figure 4.7: Pin on a disc principle

(Source: <https://www.tribonet.org/wiki/pin-on-disk-test/>)

The pin on disc tribometer consists of the following parts:

- Load
- Pin
- disc

Wear testers, tribotesters, and tribometers are all terms for wear testing equipment. Wear, friction, and lubrication are all specified in the prefix "tribo-." Hundreds, if not thousands, of different wear testing setups and procedures are used in laboratories all over the world and recorded in technical literature. A wear tester will still require two components loaded against and moving relative to each other, regardless of how different one arrangement is from another. A motor or an electro-magnetic system can power the movement. The substance or portion under investigation is generally referred to as a specimen, while the other is referred to as a counterface. The working principles of three of the most commonly used laboratory wear testers are shown below as examples.



Figure 4.8: Pin on disc wear testing machine

The pin on disc test wear testing machine is represented in Fig 4.3. Under the given load, the stationary pin is pressed against the spinning disc. The pin may be any shape, but the most common shapes are spherical (ball or lens) or cylindrical, which are easier to align (flat pins are typically subject to certain misalignment which can lead to non-uniform loading and difficulties for theoretical analysis). The friction force, wear, and temperature are all continuously monitored during the test.



Figure 4.9: Pin used in the wear testing machine

The examination can be carried out both dry and lubricated. The pin is usually submerged in the lubricant bath in a lubricated situation. After a certain number of cycles, the test is stopped, and additional data processing steps typically include surface topography analysis to determine wear volume and surface roughness evolution, SEM/EDX analysis for chemical composition/particles analysis, tribofilms, and microstructural analysis. If the test was conducted in a lubricated setting, the oil could be examined for wear particle size and additive degradation.

There are some important factors and test conditions to be taken care of while performing the wear test. Setting up a rubbing experiment couldn't be easier – all you have to do is rub two surfaces against each other. However, it appears that a variety of factors will affect test outcomes, and the test design should be as similar as possible to the actual tribological contact under investigation. When designing a pin on disc wear test protocol, keep the following considerations in mind:

- **Shape of the bolt:** In most cases, the pin may be any type. A ball pin or a lens is used in the ball on disc test. Pins that are flat or chip-shaped may also be used.

- **Alignment of pins:** If the pin has a flat end, it must be balanced, or else non-uniform loading will occur, resulting in non-uniform wear and probably overloaded conditions. Flat specimens should not have sharp edges; otherwise, chipping can occur.
- **Material for pins:** It's crucial to pick the right pin stuff. It's a must to note that the tribological activity of a given pair of materials varies depending on which material is used for the pin and which material is used for the disc. This is especially true when 'soft' and 'hard' materials come into touch. This distinction arises from the fact that the softer material is subjected to the most wear. If the ball on disc case is taken into account, a softer ball can wear faster and form a flat-on-flat contact with a pressure profile that differs greatly from the original one. If the disc is harder, a groove may form on the back, resulting in ploughing wear.
- **Pin location:** A pin can be inserted into the top of the disc. The pin, on the other hand, is often pressed against the disc from the rim. It's usually done to keep the wear particles from coming back into touch after they've developed or to capture them for further study. The wear particles will fall down due to gravity.

4.5.2 Levels of wear testing

Testing in a laboratory: Small samples are tested in the lab under simulated laboratory conditions to see if the surface engineering technology ensures further investigation in order to see under what treatment conditions the greatest wear resistance improvement can be achieved.

Test simulation of components: The optimised surface treatment state will then be used to treat a few individual joint sections, such as the femoral head or cup, which will then be evaluated in a hip-joint simulator under conditions as similar to those for a real human joint as possible, such as lubricated with body liquid, temperature around 38°C, and human walking or running.

Field test: Only after the surface-treated parts have passed the simulation tests and display a significant improvement in wear resistance while retaining other

properties (such as corrosion and biocompatibility) may in-service tests be performed. If there are no immediate side effects after implantation, the surface-treated joint parts can be inserted in a human body and examined (monitored) for a long time.

4.5.3 Wear measurement

After a wear inspection, wear analysis is used to assess the number of materials removed (or worn away) (and in reality, after a part in service for a period of time). Depending on the purpose of the test, the degree of wear, the geometry and size of the test specimens, and often the availability of a measuring facility, the material worn away may be expressed as weight (mass) loss, volume loss, or linear dimension change.

Wear measurement techniques include using a precision balance to calculate mass loss, profiling surfaces, and measuring the wear depth or cross-sectional area of a wear track with a microscope to determine wear volume loss or linear dimensional change.

- **Mass loss**

When the worn surface is uneven and asymmetrical in shape, mass loss calculation with a precision balance is a simple approach for wear measurement. Before and after a wear test, the sample to be weighed is carefully washed, and the weight is measured. The weight loss caused by wear is represented by the difference in weight before and after the test. The gram (g) or milligram (mg) unit can be used.

- **Volume Loss**

According to the geometry of the wear track/scar, wear volume is usually determined from the depth, length, width and scar profile of wear track. The calculation is done with a surface profilometer, such as a stylus kind, or a microscope with scale. mm^3 or m^3 is the reporting unit for wear volume loss. Wear volume loss allows for a more accurate comparison of wear between

materials of various densities. When a wear track is unusual, however, calculating volume loss is difficult. If the materials are uniform and the density is known, the mass loss can be determined first, and then the volume loss can be estimated.

- **Linear dimension loss**

In many engineering situations, measuring wear by linear dimension change is very useful, particularly when a particular dimension, such as length, thickness, or diameter, is more important to the system's normal operation. A stylus style surface profilometer, a micrometre, or a microscope may be used. The linear dimension loss unit may be either m or mm.

- **Wear rate**

Under unit applied normal force and/or unit sliding distance, wear rates are measured results representing wear mass loss, volume loss, or linear dimension change. The wear rate can be represented in a number of ways. Table 1 gives several examples.

- **Wear resistance**

Wear resistance is a concept that is often used to describe a material's anti-wear properties. However, the scientific definition of wear resistance is ambiguous, and there is no standard unit for measuring it. Usually, the relative wear resistance is often calculated as the inverse of mass loss or volume loss. Relative wear resistance can also be measured by comparing the wear loss of a reference material to that of the investigated material under the same testing conditions. In any case, the definition of a numeric value of wear resistance should be clearly stated. Table 4.1 shows measurement methods used for calculating various wear losses.

Table 4.1: Wear losses measurements

	Measurement Methods	Units of Wear	Units of wear rate
Mass loss	• Direct measurement by a precision balance. • Calculated from volume loss for known density material.	μg g	$\mu\text{g}/\text{m}$, g/m , $\mu\text{g}/\text{N}$, g/N , $\mu\text{g}/(\text{Nm.})$, $\text{g}/(\text{Nm.})$.
Volume loss	• Calculated from depth, width, wear profile and/or other dimensions data of a wear track. Surface profile or microscopy techniques can be used for measurements. • Calculated from mass loss of known density material.	Mm^3	mm^3/m , mm^3/N , $\text{mm}^3/(\text{Nm.})$.
Linear dimension loss	• Direct measurement by surface profile, microscopy and other dimensions measurement techniques.	μm mm	$\mu\text{m}/\text{year}$ mm/year

4.5.4 Advantages of Pin on Disc method

A pin-on-disc system allows for unrestricted movement in one direction at a variety of linear velocities, depending only on the distance of the pin from the disc's middle. Since the "pin" can be moulded into a flat, a reciprocating mechanism allows for a wider wear track. Configurations closer to bearing loads are possible with a ball-on-spindle device. Another important benefit is that this process prevents heat or debris from being trapped.

4.5.5 Applications of Pin on Disc method

- The Pin on Disc Friction & Wear Test Rig is designed to determine the tribological properties of a wide range of materials under a number of standard load conditions.
- The pin-on-disc system operates at a variety of temperatures and in a variety of environments.
- This system functions smoothly in both dry and lubricated environments.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 WEAR ANALYSIS

Table 5.1: Wear results

Magnesium Alloy	Wear Rate 2kg 19.62N	Wear Rate 6Kg 58.8N	Wear Resistance 2Kg	Wear Resistance 6Kg	COF (2Kg)	COF (6Kg)
Mg – 2Sr	0.2241	0.14609	231.48	116.41	0.060	0.0705
Mg – 2Sr – 2Gd	0.21276	0.14541	239.80	116.95	0.0627	0.0663
Mg – 2Sr – 4Gd	0.2074	0.14456	246.30	117.65	0.0656	0.0615
Mg – 2Sr – 6Gd	0.18827	0.13044	271.03	130.38	0.0707	0.0659
Mg – 2Sr – 8Gd	0.17347	0.1216	294.17	140.15	0.0767	0.0581

The wear results tabulated in Table 5.1 confirms that there has been a subsequent decrease in the wear rate with both the loads. The wear rate decreases by the increase in the proportion of Gd. Naturally, the wear resistance is shown to increase as the proportion of Gd increases. The coefficient of friction (2kg) is also shown to increase. However, the coefficient of friction (6kg) fluctuates with the increasing proportion of Gd from Mg-2Sr up to Mg-2Sr-8Gd.

Table 5.2: Variation of Wear properties

Magnesium Alloy	Wear Rate % decrease 2kg 19.62N	Wear Rate % decrease 6Kg 58.8N	Wear Resistance % increase 2Kg	Wear Resistance % increase 6Kg	COF (2Kg) % variation	COF (6Kg) % variation
Mg – 2Sr	0	0	0	0	0	0
Mg – 2Sr – 2Gd	5.06024	0.46546	3.46955	0.46173	4.30622	5.95744
Mg – 2Sr – 4Gd	7.45203	1.04729	6.01705	1.05397	8.53658	12.76595
Mg – 2Sr – 6Gd	15.9883	10.71257	14.5924	10.71483	15.13437	6.52482
Mg – 2Sr – 8Gd	22.59259	16.76363	21.31081	16.93899	21.77314	11.83611

The variation of wear properties of Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-6Gd, Mg-2Sr-8Gd alloys in comparison with Mg-2Sr base alloy is tabulated in the above Table 5.2.

5.1.1 Wear rate

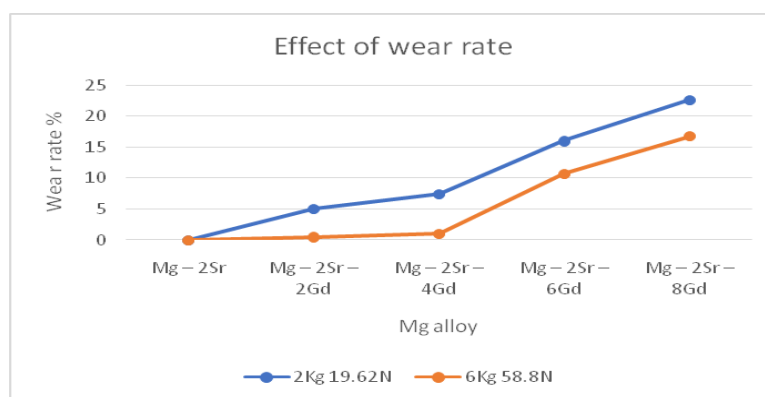


Fig 5.1: Wear rate variation

From Table 5.2, we can infer that there is a decrease in the wear rate upon adding Gd to Mg-2Sr base alloy. Wear rates is also less when load of 58.8N is applied compared to 19.62N.

From the above graph, there is a 22.6% decrease in the wear rate of Mg-2Sr-8Gd w.r.t Mg-2Sr base alloy under 19.62N load whereas there is only 16.7% decrease under 58.8N load.

Under 19.62N, 58.8N loads, Mg-2Sr-8Gd has the least wear rate of 0.17 and 0.12 respectively which confirms higher the wt.% composition of Gd in Mg-2Sr, lower the wear rate.

5.1.2 Wear resistance

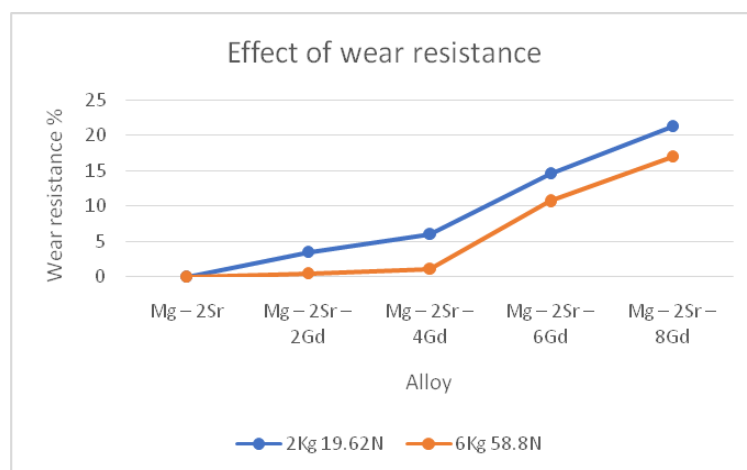


Fig 5.2: Wear resistance variation

There is a significant increase in wear resistance upon the addition of Gd to Mg-2Sr base alloy in both the loads.

From the above graph, under 19.62N load there is 22% increase in the wear resistance of Mg-2Sr-8Gd alloy w.r.t Mg-2Sr base alloy. However, under 58.8N load there is only 17% increase in wear resistance.

Under 19.62N, 58.8N loads, Mg-2Sr-8Gd has the highest wear resistance of 294 and 140 respectively which confirms higher the wt.% composition of Gd in Mg-2Sr base alloy, higher is the wear resistance.

5.1.3 Co-efficient of friction

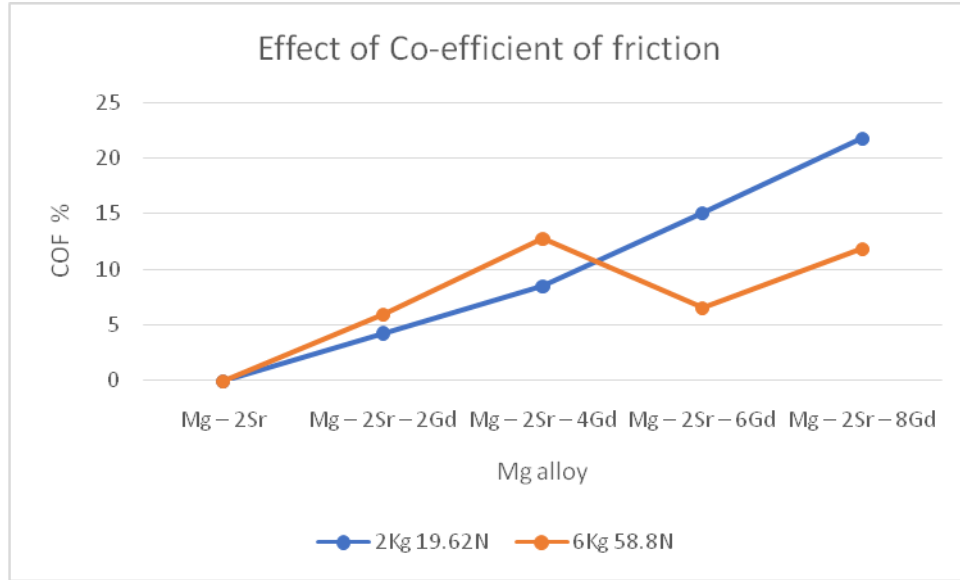


Fig 5.3: Co-efficient of friction variation

Under 19.62N load there is a steady increase in Co-efficient of friction. The above graph shows there is a significant increase upon adding Gd to Mg-2Sr base alloy. There is a maximum increase of 21% in coefficient of friction of Mg-2Sr-8Gd alloy w.r.t Mg-2Sr base alloy.

However, under 58.8N load the variation is not uniform. There is an increase of 6%, 12% in Mg-2Sr-2Gd and Mg-2Sr-4Gd alloy respectively. After that there is a sudden decrease of 6% in Mg-2Sr-6Gd and 11% increase in Mg-2Sr-8Gd alloy.

The significant decrease in the wear rate can be related to the refinement in grain size of the Mg alloy. The addition of strontium altered the structure and refined the grain size, according to the findings.

Studies have shown that the dominant wear mechanism at the higher loads was determined to be a mixture of abrasion and delamination wear. It was also observed that Abrasion dominates the wear mechanism at 19.62N while adhesion is the main wear mechanism under 58.8 N, and plastic deformation has great influence on the surface morphology. In addition, oxidation was observed under each wear condition, and delamination is detected.

In the microstructure, broken intermetallic phases were observed along the processing direction. Also, because of the complex nature of the process, non-uniform sized grains were present. After addition of Gd, the hardness increased linearly and decrease in the grain size was observed. The development of an oxidation layer on the wear surfaces also weakens the hard-phase support effect during the wear process at high sliding speeds, according to the findings.

Two distinct wear mechanisms were discovered at sliding speeds varying from 0.57 to 0.73 m/s. During low-speed sliding at 0.05 m/s, abrasive wear occurs at a high pace. The wear function transitions to oxidative wear with a low friction coefficient and rate of wear when the sliding speed exceeds 0.57 m/s.

According to some research, Mg–Gd alloys with similar Gd content but different grain sizes had nearly equal hardness, implying that grain size had little impact on hardness. The volume fraction of secondary phase increases from 0.5 percent of the as-extruded sample to 4.3 percent after Gd is added.

CHAPTER 6

SUMMARY AND CONCLUSION

Based on the research performed on Mg-2Sr, Mg-2Sr-2Gd, Mg-2Sr-4Gd, Mg-2Sr-8Gd It was found that addition of Gd to the Mg-2Sr base alloys helps in the improvement of the wear properties.

If we see the wear rate, when the wt.% of Gd is increased in Mg-2Sr base alloy, wear rate is significantly increased. Wear resistance on the other hand is significantly increased upon the addition of Gd to Mg-2Sr.

The results indicated that the Mg-2Sr-8Gd exhibited low wear rate compared with the other alloys. When the wt.% composition of Gd is increased, the wear rate decreases. The wear resistance of Mg-2Sr-8Gd alloy was the highest which confirms higher the wt.% of Gd in Mg-2Sr base alloy, the wear resistance of the alloy.

Magnesium and strontium promote osteogenesis by suppressing osteoclastic activity. The addition of strontium altered the structure and refined the grain size, according to the findings. Mg-Sr alloys have potential benefits such as adequate mechanical performance, excellent bio compatibility, and osteo-conductive properties, making them ideal for bone replacement alternatives. The addition of Gd significantly improved the wear properties by decreasing the wear rate and increasing the wear resistance of the Mg alloy.

Strontium proven to be biocompatible prevents Osteoporosis and also wear results shows us that subsequent addition of Gd enhanced the wear properties.

Thus, it can be concluded that Mg-Sr-Gd alloy can be the best substitutes for bone implants in Orthopedics.

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APPENDIX

Tribological investigation of Mg-Sr-Gd alloys for Orthopedic application

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Tribological Investigation of Magnesium Rare Earth Alloy for orthopaedic Application

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Abstract

Bio degradable magnesium alloys, has mechanical properties similar to human bone. Rare earth elements (REE) when alloyed with magnesium improve tribological and mechanical properties of Mg - REE alloy. Hence being used Mg REE alloys are new series orthopaedic implant materials used for bone replacement due to its bio compatibility, high strength, and very low degradation. The magnesium alloys can be strengthened by choosing the correct REE alloying element. More over Mg - REE Alloy gains higher strength due to grain refinement and strong solution reinforcing mechanism during gravity casting. This leads to a promising substitute for a complete permanent bone implant due to biodegradability nature. Several researchers have developed Mg alloys with Al, Ca, Zn and REE as reinforcing elements. In this research, tribological properties of Mg-2Sr, Mg-2Sr-2Gd, Mg-2Sr-4Gd, and Mg-2Sr-8Gd gravity casting in carried out and the wear rate, wear resistance and coefficient of friction is investigated.

Keywords: Magnesium (Mg), Strontium (Sr), Gadolinium (Gd), Tribology, Wear properties.

1. Introduction

Metallic products have seen a large rise in use in orthopaedic surgery for the production of orthopaedic devices, such as permanent implants (total joint replacement, hip replacement), temporary implants (pins, bone plates, etc.) and permanent implants (prosthesis, etc.) screws, and so on [1]. Metallic materials, ceramic materials, and polymeric materials are the three primary types of biological implant materials. Metallic materials, such as stainless steel, cobalt-chromium alloys, and titanium alloys, have been widely used in orthopaedic applications due to their mechanical strength.

Metallic materials will continue to play an important role as biomaterials in the repair or replacement of diseased or damaged bone tissue [2]. Metals, as opposed to ceramics or polymeric materials, are better suited for load-bearing applications due to their combination of high mechanical strength and fracture toughness. However, the potential release of toxic metallic ions and particles through corrosion or wear processes that trigger inflammatory cascades reduced biocompatibility and cause tissue loss is a disadvantage of these existing metallic biomaterials.

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