

# COMPARISON OF MICROSTRUCTURES AND PROPERTIES OF AE42 MAGNESIUM ALLOY AND ITS COMPOSITES

This thesis is submitted in the partial fulfilment of the requirement

For the degree of Bachelor of Technology

In

**Metallurgical and Materials Engineering**

By

**NITISH KUMAR**

**(111MM0353)**

**And**

**RISHABH AGARWAL**

**(111MM0387)**



**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

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Under the Guidance of

**Prof. Ashok Kumar Mondal**



**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

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**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

## **CERTIFICATE**

This is to certify that the thesis entitled ‘Comparison of the Microstructures and Properties of AE42 Magnesium alloy and its Composites’ submitted by NITISH KUMAR (111MM0353) and RISHABH AGARWAL (111MM0387) in partial fulfilment of the requirements for the award of Bachelor of Technology degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela, is an original work carried out by them under my supervision and guidance.

The matter embodied in the thesis has not been submitted to any other University/institute for the award of any degree or diploma.

Date: 6<sup>th</sup> May, 2015

**Prof. Ashok Kumar Mondal**

**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

## **ACKNOWLEDGEMENT**

We would like to express our sincere gratitude to Prof. S. C. Mishra, Head of the Department, Metallurgical and Materials Engineering, NIT Rourkela for giving us an opportunity to work on this project and provide the valuable resources of the department.

We would like to express our deep sense of gratitude and indebtedness to our guide, Prof. Ashok Kumar Mondal, Department of Metallurgical and Materials Engineering, NIT Rourkela, for his valuable guidance, constant encouragement and kind help throughout the project work and the execution of the dissertation work.

We would also like to convey our thankfulness to all the staff members of MME Department, NITR who in some way or the other has provided us valuable guidance, suggestion and extended their help for this project.

Date: 6<sup>th</sup> May, 2015

NITISH KUMAR (111MM0353)

RISHABH AGARWAL (111MM0387)

Department of Metallurgical and Materials Engineering

National Institute of Technology, Rourkela

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## **ABSTRACT**

Magnesium as an energy proficient material, has the potential to replace steel, aluminium and some plastic-based materials. There is a great interest in using magnesium (Mg) alloys in the automotive industry due to greater environmental concern. Fuel resources are limited so it should be conserved and the harmful emissions in the environment should be reduced. Magnesium with a density of  $1.74 \text{ gm/cm}^3$  is a light metal and is suitable for automotive purpose.

In this investigation the microstructure and properties of AE42 magnesium alloy and its composites reinforced with saffil short (essentially  $\delta\text{-Al}_2\text{O}_3$ ) fibres and SiC particles has been studied. Both optical and SEM characterization study is carried out. Hardness values reveals that the composites are more promising than the alloy. Wear study is carried out on Ball on Plate Wear Tester at a normal load of 5N and 10N at rotational speed of 25 rpm. Though wear rate increases with the normal load, composites show more resistance to indentation than the AE42 alloy. Large curly chips are observed in case of magnesium alloy. Immersion test reveals that composites are more prone to corrosion due to galvanic cell creation within itself because of the presence of fibres and SiC particles.

*Keywords:* Magnesium alloy, Composite, Hardness, SEM, Optical, Wear, Corrosion

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## CHAPTER 1: INTRODUCTION

### 1.1 INTRODUCTION

Magnesium (Mg) (density:  $1.738 \text{ gm/cm}^3$  and 35% lesser in weight than Al) is the lightest structural metal and therefore, has an added advantage for the applications in the automobile and aviation industries. In addition, it has high specific strength, good castability, good damping capacity, good machinability, and good weldability but under the controlled atmosphere. It also shows improved corrosion resistance with high purity magnesium [1]. Due to these factors extensive use of magnesium-based alloys can be seen. The uses of Mg alloys are limited in the interior parts of automobile (like steering wheels, front control panels, clutch pedals, brake pedals etc.) where temperature is not an issue.

The conventional Mg alloys (AZ and AM series) are having excellent castability, less cost and reasonable strength at ambient temperature. Magnesium alloys have turned into the appealing possibility for structural applications because of their high specific strength, which prompts the weight decrease bringing about an extensive financial advantage.

The real development region of magnesium alloys is in the vehicles powertrain applications, which requires high temperature applications. For powertrain segments, a number of creep-resistance magnesium alloys has been developed [2]. On the other hand, the use of Mg alloys is limited up to  $200^\circ\text{C}$ , above which metal matrix composites (MMCs) must be developed. As particle reinforced magnesium-MMCs do not increase creep properties much and also they might actually deteriorate the creep properties, short fibre reinforced MMCs are wanted for such applications. In any case, they are expensive and exhibit anisotropic properties, which can be overcome by developing hybrid composites. The partial substitution of expensive short fibres by cheap particles diminishes the expense as well as anisotropy. In the present investigation, the microstructure and wear properties of the AE42 magnesium alloy and its

composite reinforced with saffil (essentially  $\delta$ -Al<sub>2</sub>O<sub>3</sub>) short fibres and SiC particles has been investigated.

## **1.2 OBJECTIVE AND SCOPE**

The objective of this project is to evaluate the microstructure and to compare the properties of magnesium alloy and its composites. Magnesium being an energy efficient metal, has the potential to replace many conventional materials currently used in the automotive industry. Because of its high strength to weight ratio, this metal is a favourable choice for many industries. Hence the scope of work will include:

- Microstructural evaluation of the magnesium alloy as well as its composites by both Optical and SEM micrography.
- Hardness measurement of the specimens with the help of Vickers hardness tester.
- Wear study of the given specimens with the help of Ball on Plate Wear machine at different normal loads, say 5N, 10N.
- Immersion corrosion test to get an idea of the effect of aggressive environment on the specimens preferably NaCl solution of given concentration.

## CHAPTER 2: LITERATURE SURVEY

### 2.1 BACKGROUND

Magnesium is the eighth most abundant metal in the earth's crust. If we consider only 3.8 km, then it is the third most abundant metallic material. Ocean water also contains a lot of magnesium ions. Hence magnesium can be extracted either from the hydrosphere or it can be extracted from lithosphere. When compared to aluminium, it can be extracted from earth crust only. There are a number of routes available for the extraction of magnesium.

Magnesium is a very light metallic material. Its density is  $1.74 \text{ gm/cm}^3$ , which is about two-thirds of that of aluminium and one-fourth of that of steel. Light weight combined with high specific mechanical properties makes this material very much suitable for weight-critical applications. Table 2.1 compares the densities of most used structural materials [3].

Materials	Density(kg/cm <sup>3</sup> )
Steel	7.2
Titanium	4.51
Aluminium	2.71
Magnesium	1.74
Plastic	1.0-1.7

Table 2.1: Density Comparison of Various Structural Materials

Magnesium accounts for 2.7% of the earth crust though this metal is not found in the elemental form. Common compounds of magnesium are magnesite ( $\text{MgCO}_3$ ), dolomite ( $\text{MgCO}_3$ ), and carnallite ( $\text{KCl.MgCl}_2.6\text{H}_2\text{O}$ ). Magnesium is the most promising light weight metal but this metal is currently not utilized to its fullest because of certain processing difficulties.

## **2.2 PROPERTIES OF PURE MAGNESIUM METAL**

### **2.2.1 Crystal Structure and Atomic Properties**

Symbol	Mg
Classification	Alkaline earth metal
Atomic number	12.0
Atomic weight	24.3
Atomic volume	14.0 cm <sup>3</sup> /mole
Atomic radius	0.160 nm
Ionic radius	0.072 nm
Crystal structure	Hexagonal-Closed-Packed (HCP)

### **2.2.2 Physical Properties**

Density	1.738 gm/cm <sup>3</sup>
Melting point	650 °C
Boiling point	1090 °C

### **2.2.3 Mechanical Properties**

Yield strength	80 MPa
Ultimate tensile strength	190 MPa

## **2.3 APPLICATIONS OF MAGNESIUM METAL**

### **2.3.1 Automotive Applications**

Magnesium metal and its alloys because of its light weight have become the most favourable candidate for the applications in the automotive industry. With the increasing demand of energy, the prices of crude oils are skyrocketing. Industries are constantly searching for materials which can take the place of conventional materials. Magnesium and its alloys have earned great reputation because of its light weight as a result having high specific strength.

Hence this novel magnesium metal is being developed for its applications especially in the automotive industry. By the use of this metal, weight of the automobile can be decreased thus increasing the fuel efficiency. A huge amount of energy can be saved in this way.

### **2.3.2 Aerospace Applications**

Weight reduction in the aerospace industry is very important owing to the need of fuel efficiency and emission reduction. In the recent years magnesium based materials are extensively used in the military aircraft as well as civil aircrafts. To reduce the lift-off weight these alloys are also used in spacecraft and missiles.

### **2.3.3 Medical Applications**

Magnesium alloys can be used as orthopaedic biomaterials. It is also used for implanting the load bearing applications in the medicine industry. When compared to bone, its compressive yield strength and elastic modulus values are more comparable to magnesium than any other material. Human body contains about 1 mole of magnesium and half of it is present in the bone tissues. It takes part in many metabolic activities of human body and is nontoxic to human body. So magnesium has good biocompatibility. Also it is biodegradable in the human body fluid by corrosion.

### **2.3.4 Sports Applications**

Ability of magnesium and its alloys to form into intricate shapes made this alloy a favourable choice for the sports industry. Archery bows, tennis rackets, golf clubs etc. uses magnesium based handles. Frames of bicycle made from magnesium alloys are capable of shock absorbing and vibrations. This allows the rider to have a comfortable ride.

### **2.3.5 Electronic Applications**

This metal is used in the automotive industry to make electronic appliances more handy and portable. Hence the materials which are used to make the appliances should be durable. This metal is as light as plastic. But this metal show a great advancement in strength, heat transfer, and has a good ability to shield electromagnetic interferences. It is also used in the heat sinking devices and in the arms of hard drive reader because of its good heat transfer property.

## **2.4 MAGNESIUM-BASED MMCs AND PROCESSING ROUTES**

The applications of magnesium based alloys is limited. Magnesium alloys can be used up to 200 °C but above this temperature application requires the development of magnesium-based metal matrix composites (MMCs). This requires the reinforcement of magnesium with ceramic materials. The matrix in this case is the magnesium and reinforcement is the ceramic materials. The reinforcement can be ceramic particles, ceramic fibres or both. When two types of reinforcements are used, then it is called hybrid reinforcement. The coefficient of thermal expansion of matrix and reinforcement is different. As a result the interface has to withstand the resulting stresses. Hence interface need to be strong. This requires the good wettability of reinforcement by the matrix. In a number of variety of ways the reinforcements can be used to vary the properties. This can be done either by using two reinforcements at a time, using large

fraction of one of the reinforcement or just using a single reinforcement. The amount of reinforcement and the alignment also plays the role. This is especially applicable for fibres.

#### **2.4.1 Reinforcements for Magnesium MMCs**

Ceramic particles and carbon fibres are extensively used for the reinforcements of magnesium. Generally metallic particles and fibres are not used because of their poor corrosion properties. When more than one reinforcement is used in the making of MMCs, it is called hybrid composite.

##### **2.4.1.1 Particle Reinforcement**

Non-abrasive ceramic particles are usually chosen as the reinforcing materials. Typical examples of particle reinforcements are nitrides (BN, AlN, TiN, and ZrN), carbides (B<sub>4</sub>C, ZrC, SiC, TiC, W<sub>2</sub>C, and WC), borides (TiB<sub>2</sub>, ZrB<sub>2</sub>, WB) and oxides (ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>). The interaction between the matrix and the reinforcement is also taken in to account. The shape and size of the reinforcements also affect the properties of the composite. Generally the sharp edge reinforcements are avoided as these acts as stress raisers and may initiate the crack.

##### **2.4.1.2 Fibre Reinforcement**

When compared to particles, there are fewer fibre reinforcements available. Single fibres are called monofilaments. Multifilaments are having huge number of fibres in the range of 5-25 μm [4]. Carbon fibres are used for magnesium based composites in R&D projects. This is mainly because of their high tensile strength, high modulus, low density, and low coefficient of thermal expansion, good electrical conductivity and because of high availability. There are two different precursors available for the processing of the carbon fibres. These are Polyacrylonitrile based (PAN) fibres and pitch based fibres. Alumina based continuous long fibres based on oxides are mostly used. These alumina based fibres offer good processing abilities because of good wetting ability by the magnesium matrix.

### 2.4.1.3 Whiskers Reinforcement

Whiskers are single crystals having needle like shape. They are having an aspect ratio of around 10. Diameter is in the range of 1  $\mu\text{m}$ . Whiskers are having low defect density. But whiskers are very thin and small which had led to the discussions of health risks. If they are inhaled, it leads to potential carcinogenetic. Figure 3.1 shows the types of reinforcements commonly used in the making of metal matrix composites (MMCs).

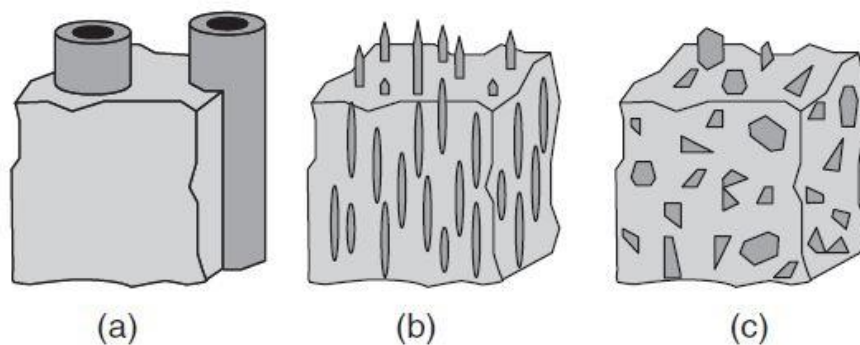


Figure 2.1: Different types of reinforcements commonly used. (a) Continuous long fibres, (b) Discontinuous short fibres or whiskers, (c) Particle reinforcements

### 2.4.2 Routes for processing Magnesium Composites

A number of different processing routes are available which are used to produce magnesium-based composites materials. The processing routes can be divided into liquid phase or ingot metallurgical route and solid phase or powder metallurgical route. The first step in the fabrication process is the blending of the magnesium alloy powders.

#### 2.4.2.1 Powder Metallurgical Process

This process can produce components having a strength about 80-120% greater than conventional casting methods. The cast magnesium ingot is ground to powder by machining because magnesium is a soft metal and it can be rasp like wood. High strain deformations can



be caused by the mechanical machining of the magnesium alloy resulting in nano-crystalline structures. These nanocrystalline materials are used in the making of finished components having strength greater than that of the castings. The strength is greater than the castings because the cast structure is having columnar and coarse grained structure. Hence granular magnesium powders are utilized for the making of high strength components.

#### 2.4.2.2 Ingot Metallurgical Route

Liquid phase process or ingot metallurgical route results in a very good wetting between the magnesium matrix and the reinforcements. These processes are very much inexpensive processing technologies. Few most common processes are discussed.

#### 2.4.2.3 Stir Casting

Stir casting is the conventional, cheapest and easiest processing route to produce short reinforced composite materials. In this process, whiskers, short fibres, or particles can be used. These reinforcements are simply introduced into the matrix when it is in the molten state. Stirring is done to get a good wetting between the matrix and the reinforcement. Figure 2.2 shows the schematic sketch of stir casting.

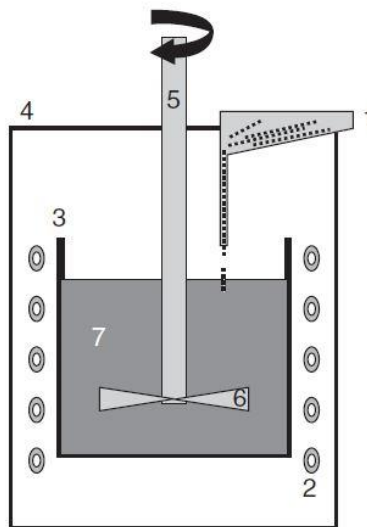


Figure 2.2: Stir Casting Technique

As per the sketch, (1) Reinforcements are added to the molten matrix. (2) The heated crucible. (3) Crucible embedded in the vacuum chamber. (4) The vacuum chamber. (5) The stirrer. (6) The rotating vortex. (7) The molten matrix.

#### 2.4.2.4 Compocasting

The main difference between the stir casting and Compocasting is that the former is performed above the liquidus temperature and the latter is performed below the liquidus and solidus temperature. The matrix is in the form of slurry which is the semi-solid material. A fraction of the solid material is surrounded by the liquid. When no reinforcements are introduced this is called rheocasting. But addition of the reinforcements is called Compocasting. Since a fraction of the material is in the solid state, an additional shear force is required compared to stir casting. The advantages over stir casting are: (1) Semi-solid state has lower tendency for burning as temperature is lower. (2) The viscosity is higher hence tendency for settling is reduced. (3) The probability for degradation is lower because of the lower temperature.

#### 2.4.2.5 Melt-Infiltration Casting

Also called squeeze casting and it is widely used for the fabrication of the fibre-reinforced composites. The fraction of fibres used is usually in between 10-60% and has volume fraction of 15-25%. The fibres are distributed in a planar isotropic manner.

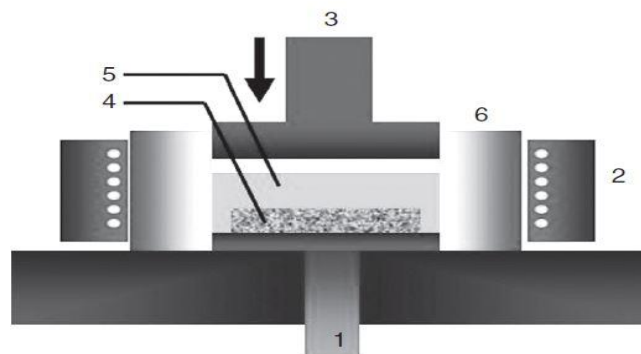


Figure 2.3: Schematic Squeeze casting

As per the sketch, (1) the solidified part which is taken out by the ejector. (2) Heating coils. (3) Preformed hydraulic ram. (4) Heated preforms. (5) Superheat melt. (6) Preheated mould

#### **2.4.2.5 Spray Casting**

In this process a stream of molten metal is atomized by the pressure of an inert gas, such as nitrogen or argon. The reinforcements which are generally the ceramic particles are added simultaneously. Generally this process is used for the manufacturing of semi-finished products which can be later forged or extruded. To collect the partially solidified materials, substrate is located under stream. A final consolidated step is needed to reach its full density.

#### **4.2.4.6 Gas pressure assisted infiltration casting**

It is very much similar to the squeeze-casting technique. It requires prefabricated preforms for the manufacturing of composite materials. Here gas pressure is used instead of ram. The total infiltration time is much greater compared to squeeze casting as a result of which a strong interface is formed between the matrix and the reinforcement. This also depends on the time of contact with the melt.

## **2.5 LITERATURE REVIEW**

Friedrich et al. [5] made a research on new age of magnesium alloy in automotive industry and found out the actual and potential uses of magnesium in different automotive components. The essential prerequisites for adopting an integrated approach for development of Mg alloy in this sector involved greater use of in-house recycling to have cost reduction, adaptation of existing casting and forming techniques, development of new Mg alloy with improved property profile and incorporation of this in multi-material design concept. Many alloys were then developed to find their application in different industries. Some of the commonly used Mg alloys included AZ91, AE42, and MRI155 and so on.

Aune et al. [6] studied the microstructure and creep behaviour of AE42 Mg die-casting alloy. They found out that above 150°C  $Al_{11}RE_3$  diminishes in volume fraction and that of  $Al_2RE$  increases. Also, there is concurrent formation of  $Mg_{17}Al_{12}$ . There was a sharp decrease in creep resistance at 175°C either due to reduced presence of lamellar  $Al_{11}RE_3$  or due to appearance of  $Mg_{17}Al_{12}$ .

The effect of varying Al and RE content on mechanical properties of die cast magnesium alloy was studied by Powell et al. [7]. They provided mechanical property data on AE-type magnesium alloys. The aluminium and rare earth addition to magnesium tends to improve creep resistance and stress relaxation performance attributes of the base metal.

Changes in tensile strength, elongation etc. was also observed. Huang et al. [8] studied the evolution of microstructure and hardness of AE42 alloy after heat treatment. They found out that in few investigations carried out on the solid solution and ageing processes of AE42 alloy, showed that High Pressure Die Casting (HPDC) is one of the main production route for this alloy. But alloy produced after this process has limited heat treatment opportunities because of the blistering of cast parts which is a result of internal porosity. Also it was observed that there was no apparent change in microstructure after heat treatment.

## CHAPTER 3: EXPERIMENTAL DETAILS

### 3.1 MATERIALS USED

#### 3.1.1 AE42 Magnesium alloy

In this investigation magnesium alloy and its composite reinforced with saffil short fibres and SiC particles were used. The magnesium alloy required for the study was prepared by squeeze casting technique. The furnace which is a bottom pour stir type was allowed to reach up to 850 °C. 752 gm of Mg, 32 gm of Al, 16 gm of RE were added into the furnace. The melt was stirred for 10 minutes. The molten mixture was then poured in to the preheated die. Simultaneously hydraulic pressure was used to apply pressure. The cast was allowed to cool. Figure 3.1 is a bottom pouring type furnace which was used for casting.

The as-cast magnesium alloy was then cut into the required shapes. One of the small cube shaped alloy was used for microstructural characterization and for various experimental procedure. The chemical composition details of this alloy is given in Table 3.1.

Element	Al	Zn	Mn	Si	Ce	La	Nd	Pr	Th	Be	Mg
Wt%	3.9	0.01	0.30	0.01	1.2	0.6	0.4	0.1	0.6	0.001	93.1

Table 3.1: Chemical composition of AE42 alloy



Figure 3.1: Bottom pouring type furnace used for melting

### 3.1.2 Composites of Magnesium alloy

The composition details and important properties of saffil fibres and SiC particles are given in Table 3.2. The AE42 magnesium alloy based composites have been used in the present investigation (Mg-4.0 wt. % Al-2.0 wt. % Rare Earth (RE)-0.2 wt. % Mn). One of the magnesium alloy is reinforced with saffil short fibres which is having a diameter of 3-8  $\mu\text{m}$  and length of 200  $\mu\text{m}$  up to 20% by volume. The round shaped SiC particles having a diameter of 40  $\mu\text{m}$  up to 10 vol. % and 10 vol. % of saffil short fibres are used in the making of second sample. The volume percentage of the reinforcements in both the composites is kept same, up to 20vol. %.

Reinforcement	Saffil fibres	SiC particles
Chemical Composition	$\delta\text{-Al}_2\text{O}_3$	SiC
Melting temperature ( $^{\circ}\text{C}$ )	2000	2700
Application temperature( $^{\circ}\text{C}$ )	1600	1650
Young's Modulus(GPa)	300	200-300
Tensile Strength(MPa)	2000	-
Density( $\text{gm}/\text{cm}^3$ )	3.3	3.2
Mohr's Hardness	7.0	9.7

Table 3.2: Properties of Saffil fibres and SiC particles.

Squeeze casting process was used for the making of the both the magnesium alloy and its composite. A preheated preform which was containing the saffil short fibres in a planar random distribution was transferred in to a die which was maintained at 200  $^{\circ}\text{C}$ .

### 3.2 SPECIMEN PREPARATION FOR METALLOGRAPHIC OBSERVATION

Metallography is the study of the microstructure of the materials. Metallographic study is the first step in the characterization of the materials. This study need the careful preparation of the

sample. Hence our main aim is to produce a flat, mirror like finish of the sample with scratch free surface. Magnesium alloys being soft metals are have to be carefully polished because they are very prone to wear and abrasion during polishing.

Following steps are involved in the preparation of the sample-

1. Cutting a desired specimen from the ingot which is cast.
2. Initial Grinding
3. Paper Polishing
4. Cloth Polishing
5. Etching

**3.2.1 Cutting a Specimen from the Ingot:** A square shaped sample was cut from the ingot which was casted by squeeze casting technique. Being a soft metal hacksaw blade was used to cut the sample to its desired length.

**3.2.2 Initial Grinding:** To ground the sample initially, belt grinder was used. This step was necessary to make the sample flat. Intermediate rotation of the sample was done to remove the scratches and hacksaw lining if any. The magnesium alloy was having a greater abrasion compared to its composited during the belt grinding. Thus it gives an initial impression that the magnesium composites are having higher strength than the AE42 magnesium alloy.

**3.2.3 Paper Polishing:** After making the surfaces flat by grinding operation the next step was the paper polishing. Different grades of paper was ranging from 1/0 to 4/0. 1/0 paper was used for rough paper polishing followed by 2/0, 3/0 and finally 4/0 paper was used for fine polishing. After 4/0 paper, it was made sure that the scratches were only in one direction. This was done by rotating the sample by 90° in each change of the paper. The sample was washed with soap solution which was made from distilled water and was dried.

**3.2.4 Cloth Polishing:** The cloth used for cloth polishing was velvet cloth. Only magnesium metal was polished with this cloth so that it does not get contaminated by other metals. The



abrasive used for cloth polishing was a diamond paste. The speed of the rotating wheel was kept at low rpm so that the sample does not get burnt owing to heat generation during polishing. Also diamond spray was used as a lubricating agent. A mirror finish surface was needed to see the microstructure, so the polishing was done accordingly. Figure 3.2 shows a rotating disc covered with cloth.



Figure 3.2: Rotating Disc for cloth polishing

**3.2.5 Etching:** The etching is done with a purpose of selectively colouring the micro-constituents so that they can be resolved under microscope. The etchant used in this investigation was a picral solution having a chemical solution of 20 ml distilled water, 10 ml acetic acid, 5 ml picric acid, and 100 ml ethanol.

### 3.3 SCANNING ELECTRON MICROSCOPY

**SEM** micrographs of the AE42 magnesium alloy and the composite before and after the wear tests were taken. The specimens before the wear tests were prepared and etched with a solution of 100 ml ethanol, 10ml acetic acid, 6 ml picric acid in 20 ml distilled water. Before that the sample was polished accordingly. The SEM analysis of the wear paths was done to investigate the changes which took place and the AE42 magnesium alloy was compared with the composites. Figure 3.3 is Scanning Electron Microscope.



Figure 3.3: Scanning Electron Microscope

### **3.4 VICKER'S MICRO-HARDNESS TESTING**

The standard definition of the hardness is the resistance of the material to indentation, wear, abrasion and deformation. But hardness has many meanings depending upon the experience of the person involved.

The Vickers hardness test utilises a square-base diamond pyramid as an indenter. The hardness is reported in terms of DPH (Diamond Pyramid Hardness) or VPH (Vickers Pyramid Hardness). It is defined as load applied divided by the surface area of the indentation. This area is calculated by the measurement of the diagonals of the square impression. Figure 3.4 shows the indenter and the measurement of the impression area.

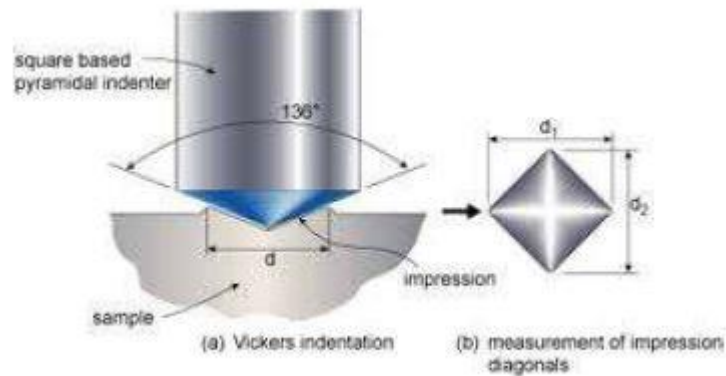


Figure 3.4: (a) Vickers Indenter (b) Measurement of diagonals

The load applied was 500gmf for a period of 10 seconds in all the three samples. The mathematical formulation of the Vickers hardness formula is-

$$DPH = \frac{2 P \sin\left(\frac{\theta}{2}\right)}{L^2} = 1.854P/L^2$$

Where P= applied load which is in Kg

L= average length of the diagonals, mm

$\theta$ = angle between opposite faces of diamond = 136°

### 3.5 WEAR STUDY

Wear is a very complex process and it involves most of the failures. It involves a very complex chemical, mechanical, and physical processes. Failure in general sense means that the specimen has reached such critical point that after that it can't be used.

Wear in a generalized way can be defined as loss of weight or mass during a process in which a surface is in contact. Wear mechanism can be divide into three types-

- 1) Diffusion Wear
- 2) Abrasion Wear
- 3) Diffusion Wear

In this study abrasion wear is carried out which involves the removal of the material on the surface by the use of a very hard constituents i.e. indenter. A diamond indenter was used in this process. The indenter used was Ball on Plate Wear Tester. Figure 3.5 shows the machine used in the wear study. Wear loss (in terms of vertical penetration of the indenter) as a function of sliding distance (cm) at an applied load of 5N and 10N at 25 rpm speed (linear speed of 0.00362 and 0.00524 m/s) on a 2 mm and 4mm diameter track was carried out. The sliding time used was 10 minutes.

The SEM images of the worn-out surfaces and wear debris of both the magnesium alloy and the composites was examined after the wear tests.



Figure 3.5: Ball on Plate Indenter Wear Tester

### 3.6 IMMERSION CORROSION TEST

Immersion corrosion test was carried out in a five weight percent NaCl salt solution. Distilled water and AR grade NaCl powder was used to prepare the salt solution. The samples were cleaned with acetone and distilled water. The samples were dried with the help of a blower. The initial weight of the specimens were taken. Let the initial weight be  $W_0$ . The specimens were dipped in the salt solution for a duration of 24 hours. Magnesium being a very reactive metal, dissolves very quickly. It gives an initial impression that, it is very much corrosive in the salt solution. After the immersion test, the specimens were taken out and the corroded surfaces were cleaned and the sample was dried. The specimens were weighed to get the final weight ( $W_1$ ). Hence the difference in weight between  $W_1$  and  $W_0$  gives the weight loss. The specimens were hanged in a 250 ml beaker with the help of thread. The whole test was carried out at room temperature i.e. 25 °C. Figure 3.6 shows the picture the beaker in which the specimens were hanged for 24 hours.



Figure 3.6: Specimens hanged in the beaker containing salt solution.

## CHAPTER 4: RESULTS AND DISCUSSION

### 4.1 MICROSTRUCTURAL CHARACTERIZATION

#### 4.1.1 Optical Microstructure

Figure 4.1(a), 4.1(b) and 4.3(c) shows the optical microstructure of the AE42 alloy and the composites.

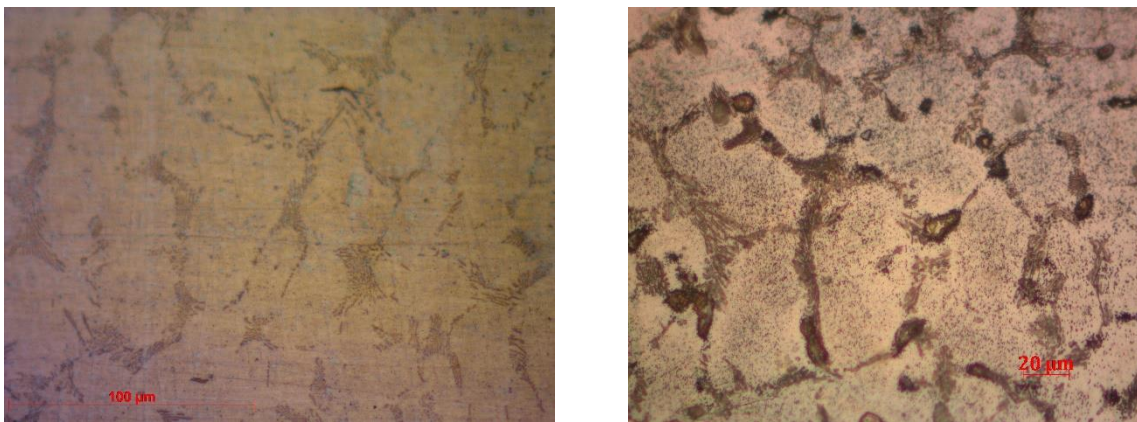


Figure 4.1(a): Optical micrograph of AE42 magnesium alloy.

Two distinct phases are clearly visible i.e.  $\alpha$ -magnesium which is the primary phase and Al<sub>4</sub>RE which is the intermetallic phase. Grains of magnesium which are almost polygonal in shape can be observed.

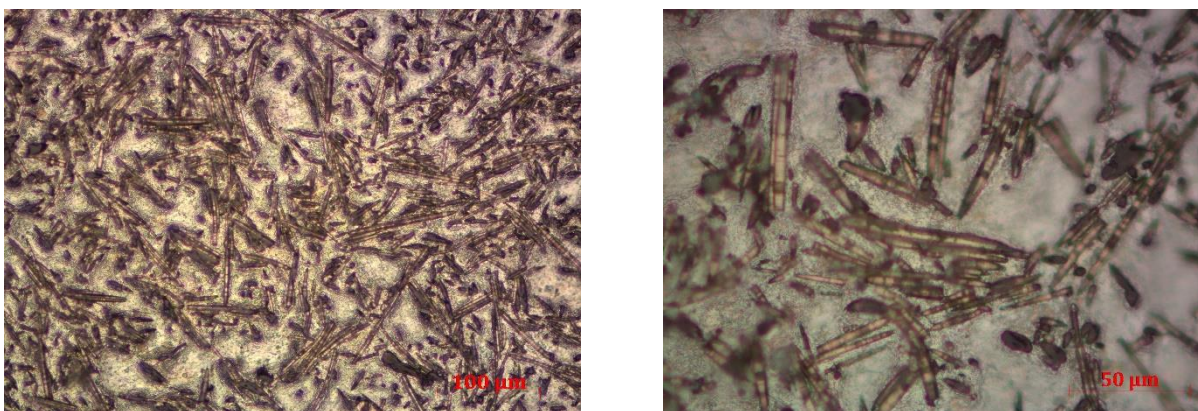


Figure 4.1(b): Optical micrograph of AE42+20% saffil composite.

The microstructure of the AE42+20% saffil reinforced composite shows the uniform distribution of the fibres in the magnesium alloy matrix. Micrograph reveals that no clusters of the fibres have been formed.

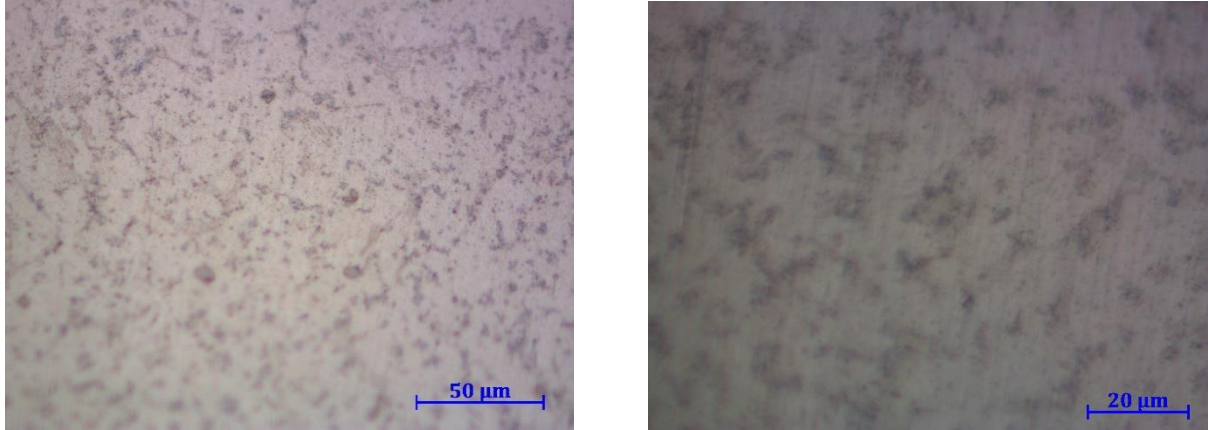


Figure 4.1(c): Optical micrograph of AE+10% SiC+10% Saffil fibres.

The microstructure of the AE42+10% SiC+10 % Saffil fibres reinforced composite shows a dense network of SiC particles and saffil short fibres which are uniformly distributed.

#### 4.1.2 SEM Micrograph

Figure 4.1(d), 4.1(e) and 4.1(f) shows the Scanning Electron Micrograph (SEM) of AE42 magnesium alloy and its composites

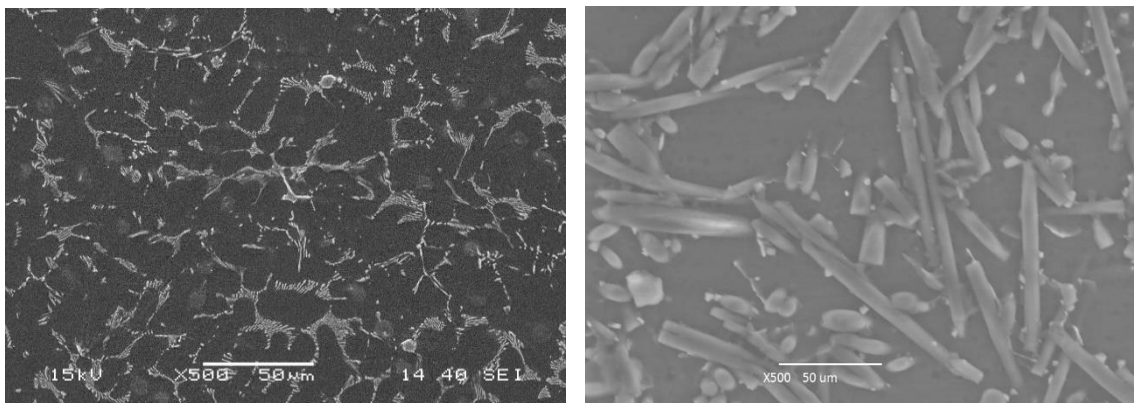


Figure 4.1(d): SEM of AE42 alloy

Figure 4.1(e): SEM of AE42+20% saffil

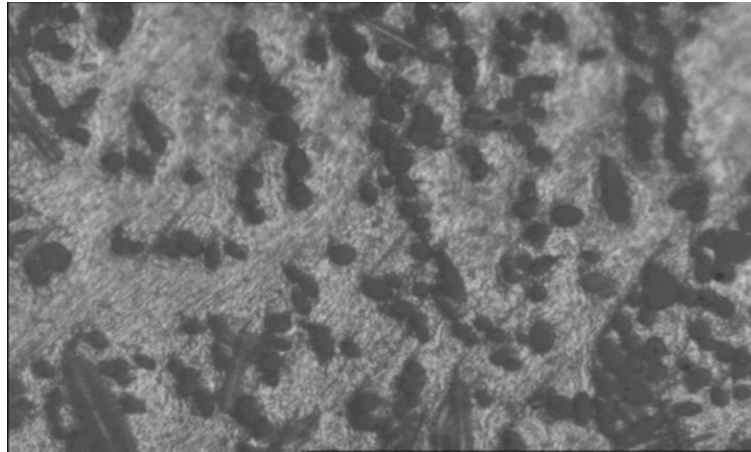


Figure 4.1(f): SEM micrograph of AE42+10% saffil fibres+10% SiC

Figure 4.1(d) depicts the SEM micrograph of as-cast AE42 magnesium alloy. Approximately polygonal grains of  $\alpha$ -Mg grains are visible. It can be estimated that the grain size is between 25-30  $\mu\text{m}$ . Intermetallic phase which is essentially  $\text{Al}_4\text{RE}$  is observed.

Figure 4.1(e) depicts the SEM micrograph of AE42 magnesium alloy and 20% saffil reinforced fibres composite. Here beside  $\alpha$ -Mg and  $\text{Al}_4\text{RE}$  phases, saffil fibres which is essentially  $\text{Al}_2\text{O}_3$  are observed.

Figure 4.1(f) depicts the SEM micrograph of AE42+10% SiC+ 10% saffil fibres reinforced composite. The microstructure consists dense compact of saffil short fibres and SiC particles along with magnesium matrix.

## 4.2 MICROHARDNESS

Table 4.1 shows the Microhardness values of AE42 alloy and the composites. The load applied in all the samples was 500 gmf and loading time was 10 seconds.

Figure 4.2(a) is a picture of the indentation mark on the specimen.



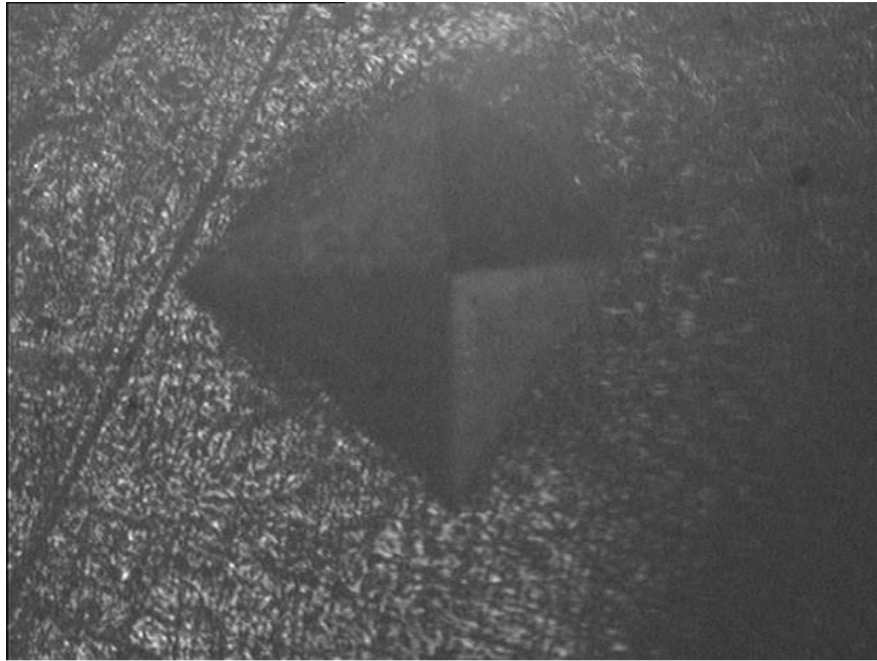


Figure 4.2(a): Indentation mark on the specimen

Specimen	1	2	3	4	Average (VPN)
AE42	73.5	73.9	74.9	80.8	75.78
AE42-20A	162.4	172.1	151.1	144.3	157.48
AE42-10A- 10S	271.3	271.9	254.9	234.5	258

Table 4.1: Hardness values of AE42 alloy and the composites

Figure 4.2(b) shows the variation of the hardness values of the AE42 magnesium alloy and the composites in the form of bar graph. From the table and the bar graph it is very much clear that the hardness value of 10% Sic+10% Saffil fibres reinforced composite is greater followed by

20% saffil fibres reinforced composite. The AE42 magnesium alloy is having the least hardness compared to its composites.

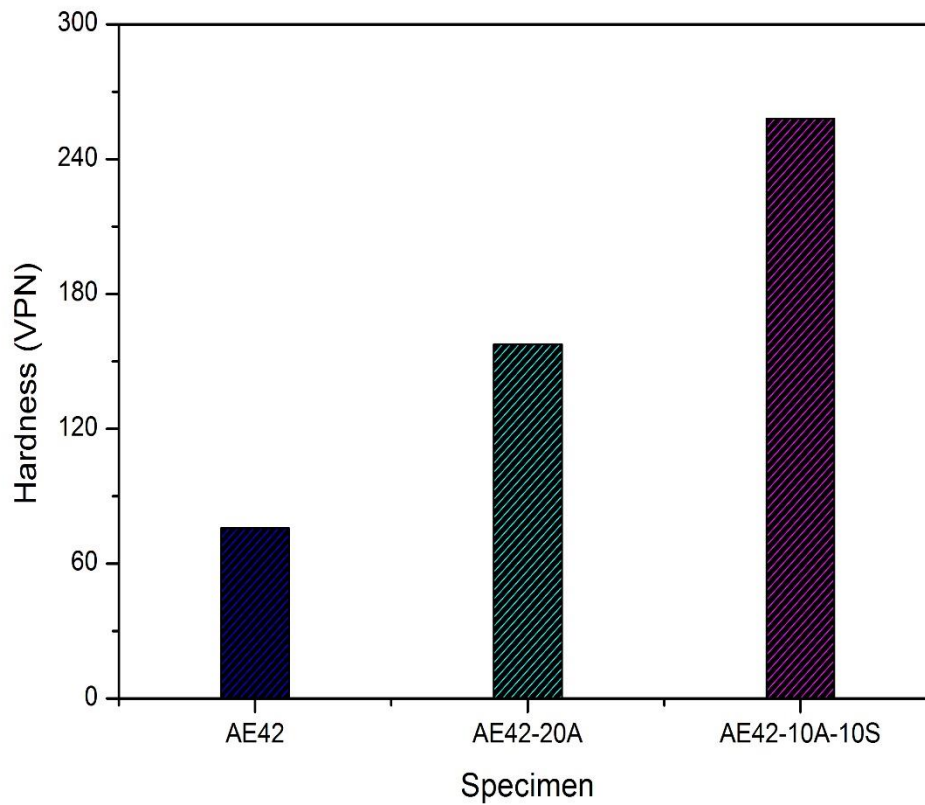


Figure 4.2(b): Variation of hardness values among the specimens.

Where, AE42 = AE42 Magnesium alloy

AE42-20A = AE42 Magnesium alloy+ 20% saffil fibres

AE42-10A-10S = AE42 Magnesium alloy+10% saffil fibres+10% SiC particles

### 4.3 WEAR STUDY

Wear study of AE42 magnesium alloy, 20% saffil fibres reinforced magnesium composite and 10% saffil fibers+10% SiC particles reinforces fibres were studied in dry sliding condition using a Ball on Plate Wear Tester at an applied load of 5 N and 10 N. The rotational speed was kept at 25 rpm, having a linear speed of 0.00262 m/s on a 2 mm diameter track and a linear speed of 0.00524 m/s on a 4 mm diameter track. Figure 4.3(a), 4.3(b), and 4.3(c) shows the variation in the wear loss. Variation is in terms of vertical penetration of the indenter or wear depth as a function of sliding distance (cm). Diamond indenter was used to prepare the track. In this investigation 5N load was used on 2mm diameter track radius and 10N load was used on 4mm diameter track radius at 25 rpm speed. The rotational speed of 25 rpm corresponds to 0.00262 m/s on the 2 mm diameter track and 0.00524 m/s on 4 mm diameter track.

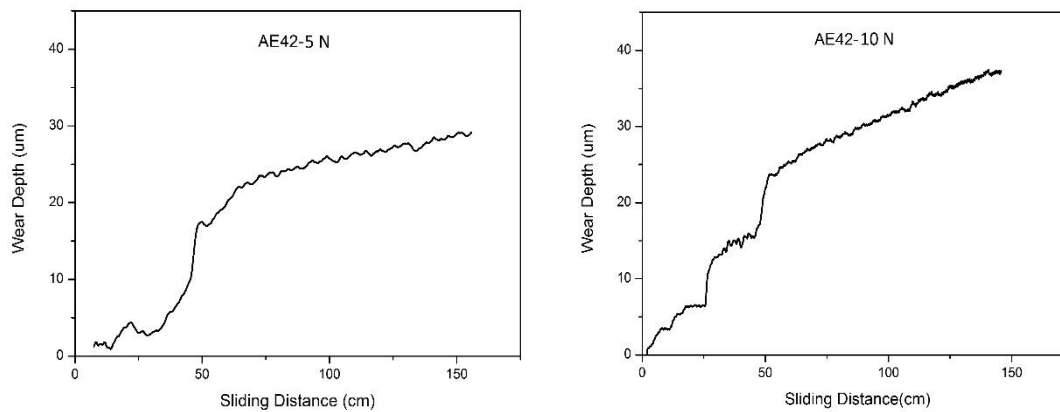


Figure 4.3(a)

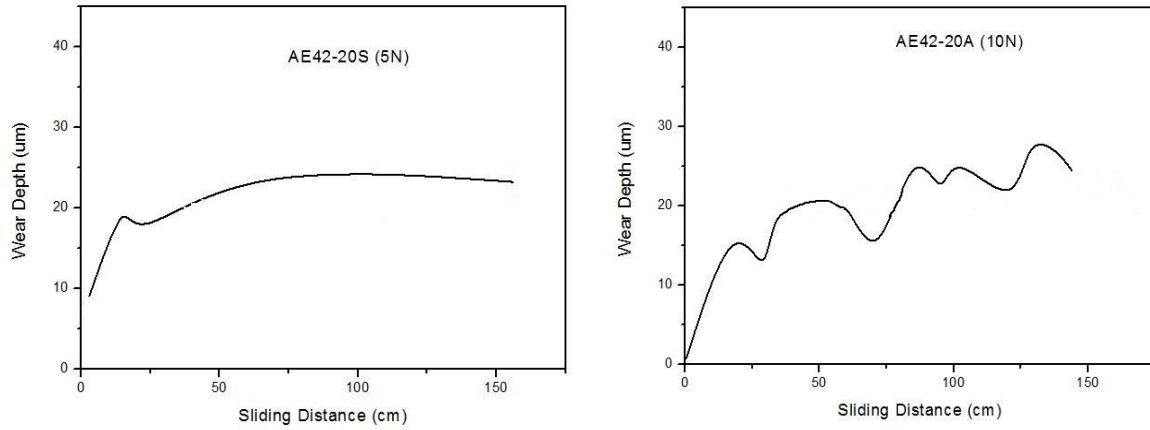


Figure 4.3(b)

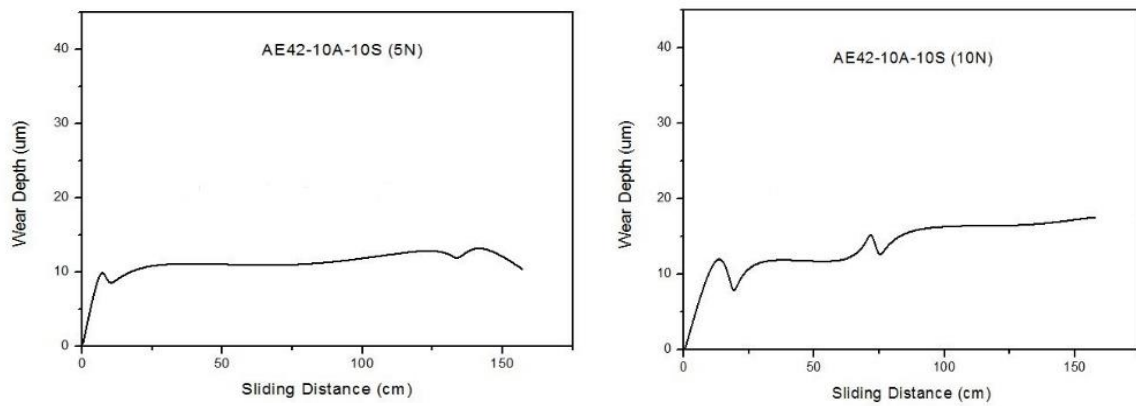


Figure 4.3(c)

Figure 4.3: (a) Variation of cumulative loss of wear as a function of sliding distance for AE42 Magnesium alloy.  
 (b) For 20% saffil fibres reinforced composite.  
 (c) For 10% saffil fibres+10% SiC particles reinforced composite.

Wear surfaces of the magnesium alloy and the composites were examined on a scanning electron microscope. The graphs gives an initial impression that the wear depth increases as the load increases for both the magnesium alloy and the composites. Figure 4.3(d) and 4.3(e) compares the wear loss for all the three specimens at a load of 5N and 10N respectively.

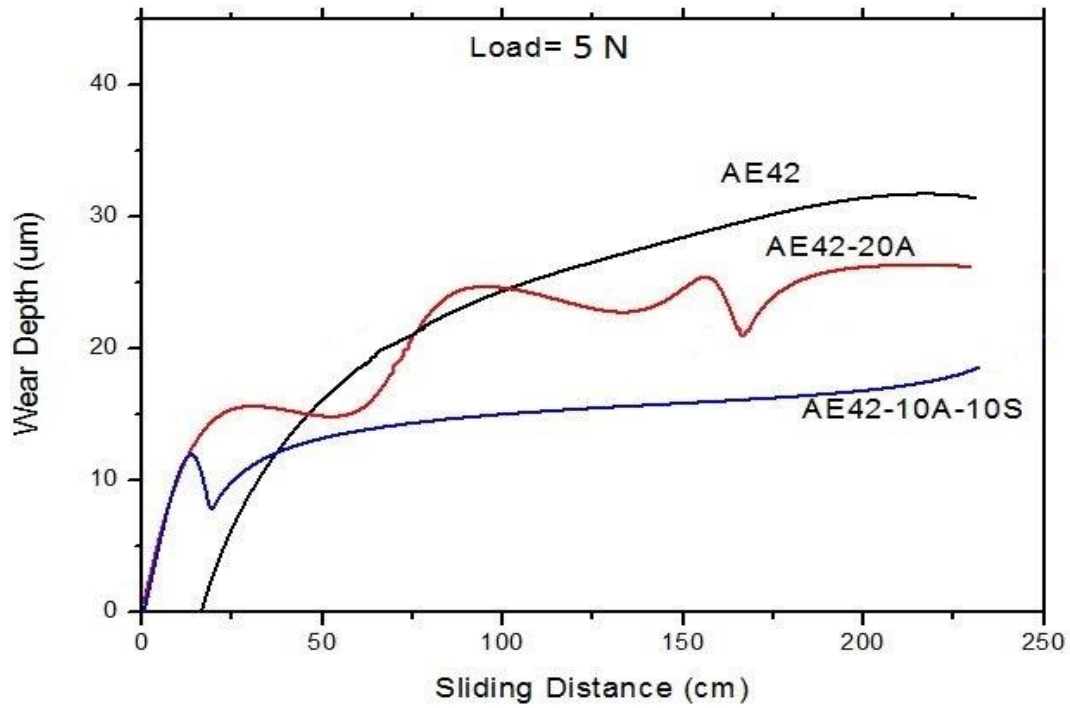


Figure 4.3(d): Comparison of wear loss of the specimens at a 5N load and 25 rpm rotational speed on a 2 mm track diameter.

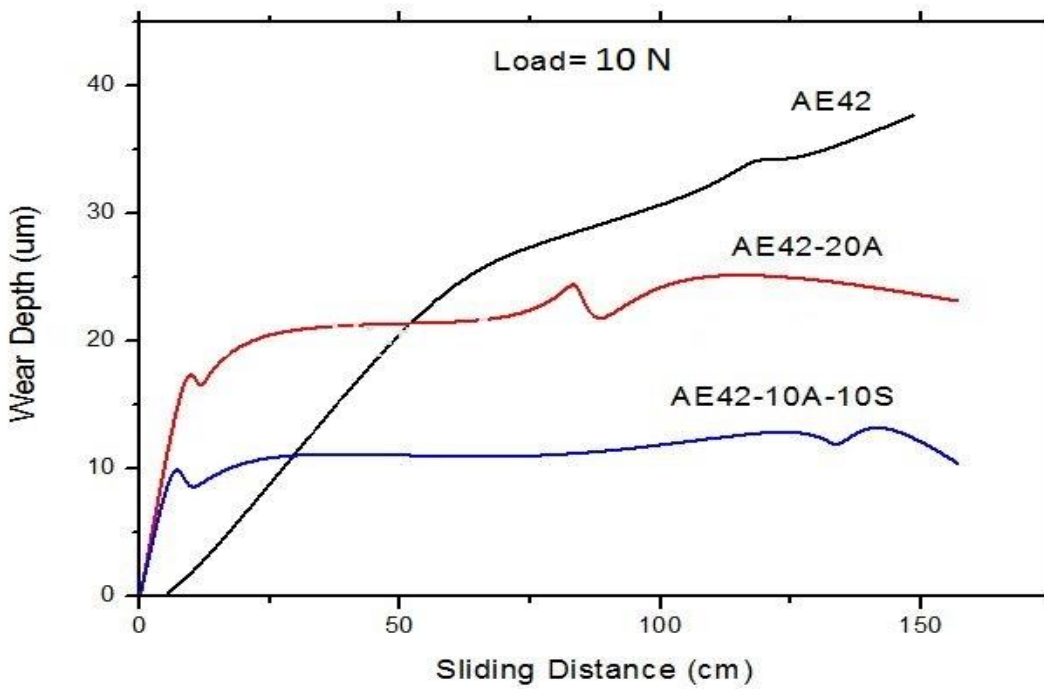


Figure 4.3(e): Comparison of wear loss of the specimens at 10 N load and 25 rpm rotational speed on a 4mm track diameter.

Comparison of the wear rate in terms of vertical penetration or wear depth for all the three specimens at 5N and 10N load is plotted in the figure 4.3(d) and 4.3(e). It is evident from the plot that the depth of penetration is highest in case of AE42 magnesium alloy followed by 20% saffil reinforced composite and least penetration is in the 10% saffil+ 10% SiC reinforced composites. Since the replacement of the expensive saffil fibres by cheap SiC particles increases the wear resistance, it is commercially beneficial. Increase in the vertical load in case of composites does not results in much increase in the wear depth. In can be concluded that composites are more resistance to wear than the alloy.

### ANALYSIS OF THE SEM MICROGRAPHS

Figure 4.3(f), 4.3(g), and 4.3(h) show the micrographs of the worn wear surfaces of the AE42 magnesium alloy, 20% saffil reinforced composite and 10% saffil+10% SiC reinforced composite respectively.

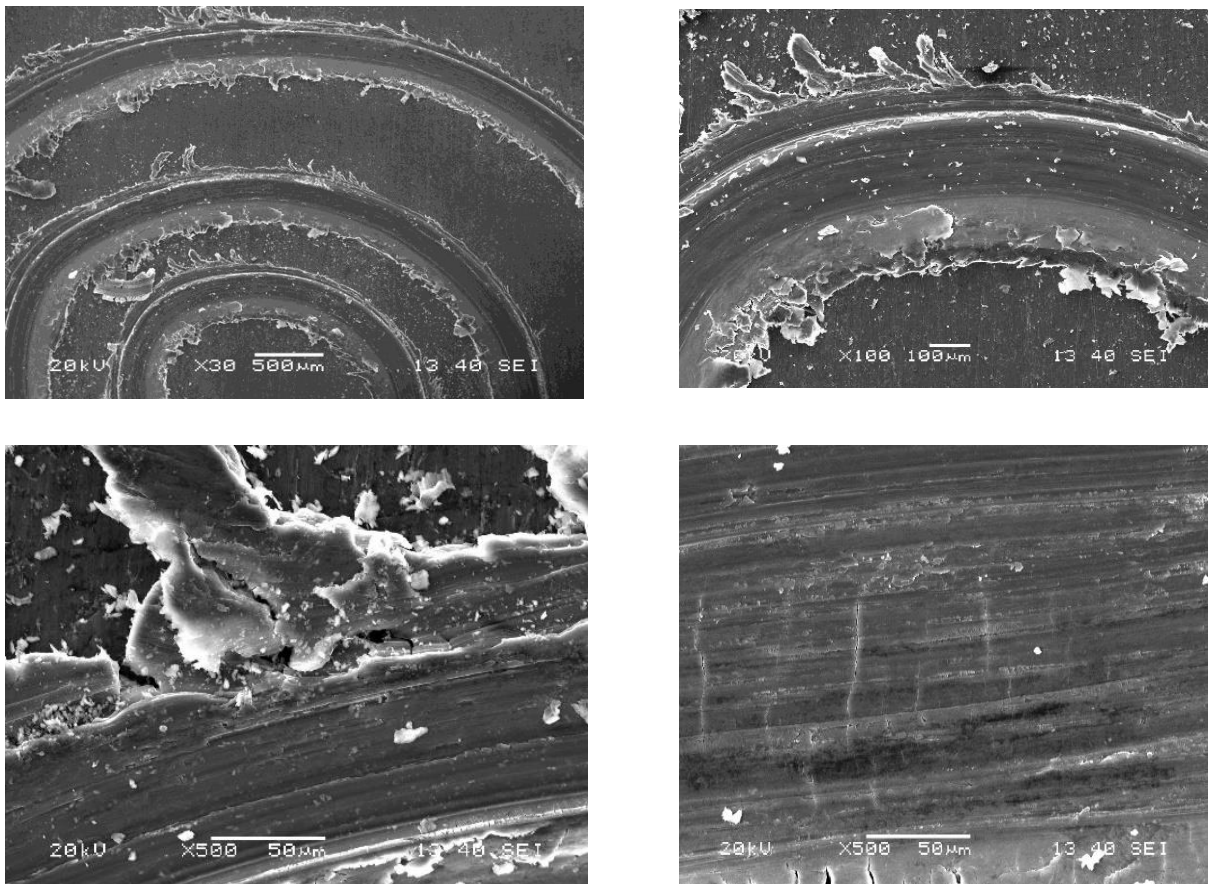


Figure 4.3(f)

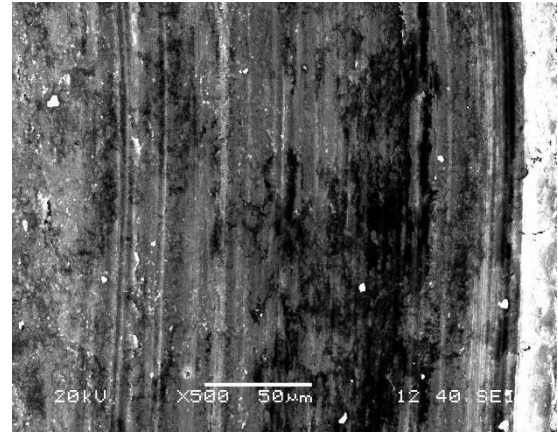
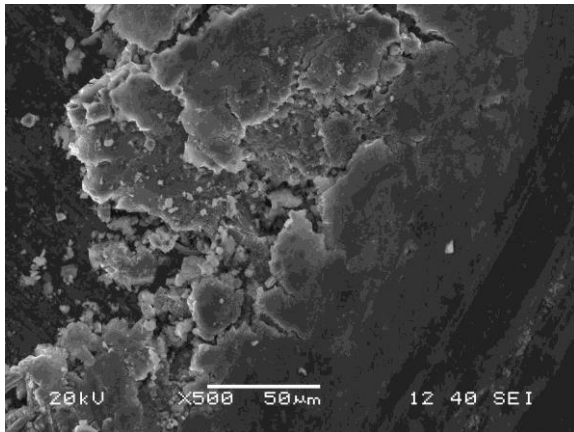
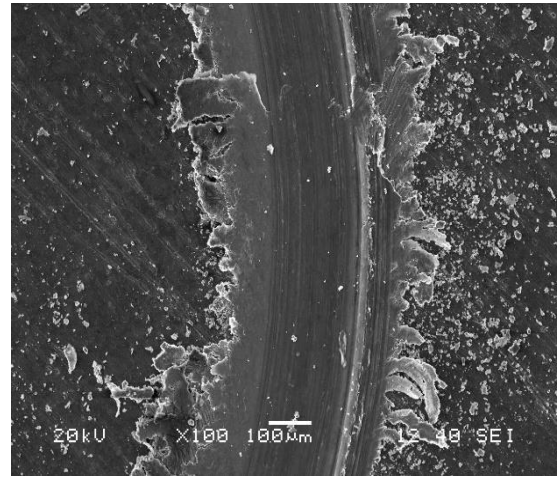
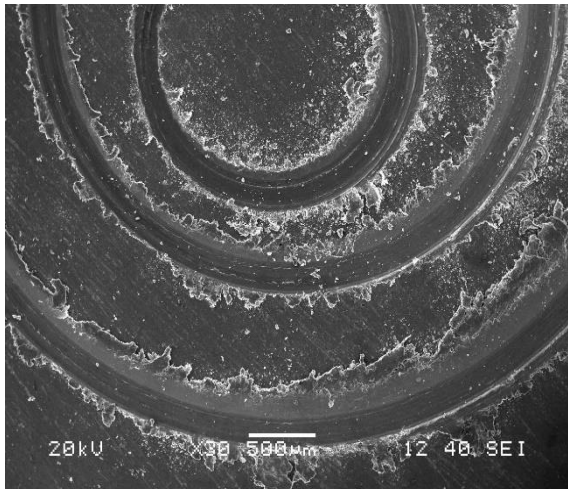


Figure 4.3(g)

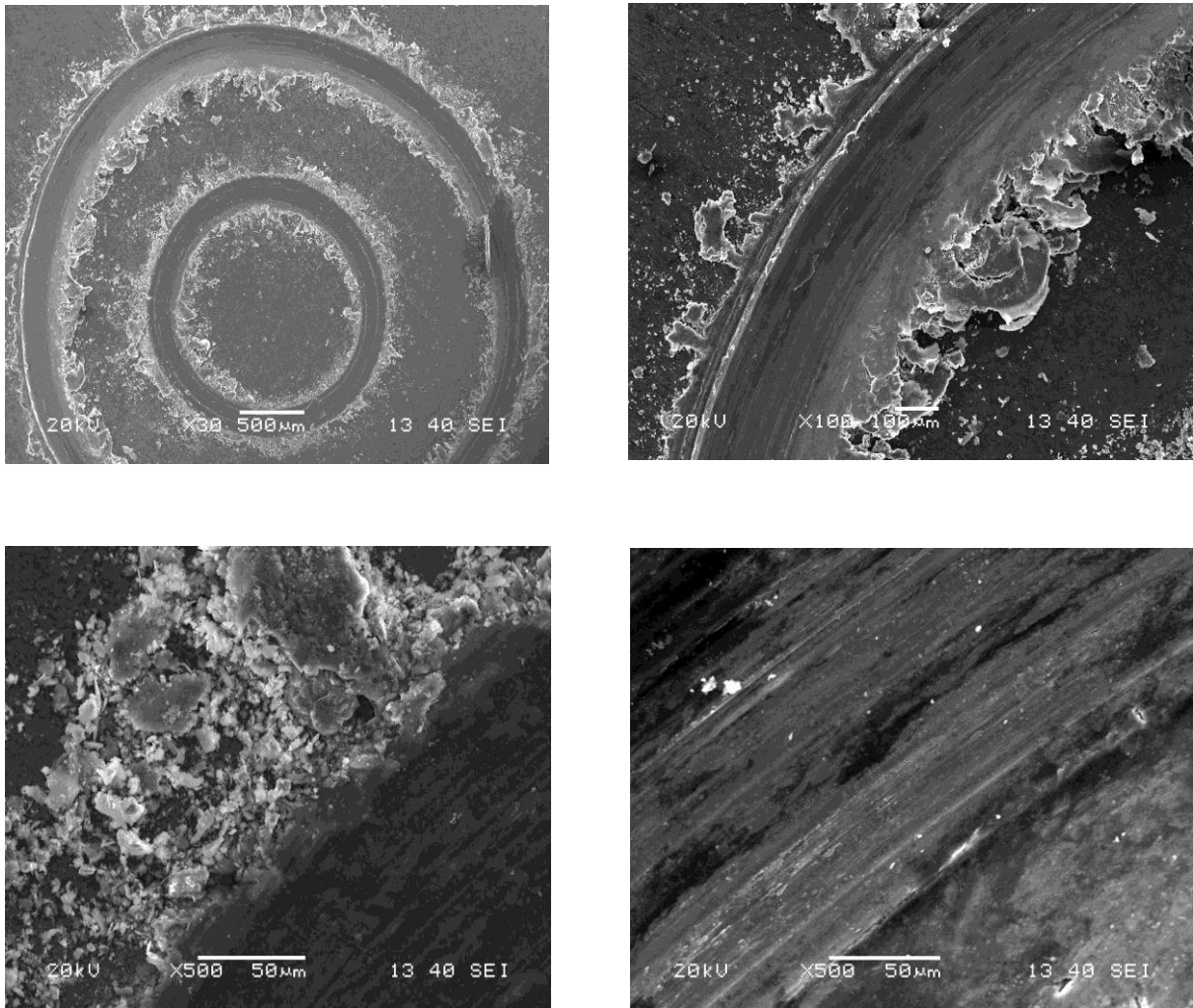


Figure 4.3(h)

Figure 4.3 (f): SEM micrograph of Wear tracks of AE42 magnesium alloy.

4.3 (g): SEM micrograph of Wear tracks of 20% saffil reinforced composite.

4.3 (h): SEM micrograph of Wear tracks of 10% saffil +10% SiC reinforced composite

A number of grooves are seen in the track of the alloy as well as composite. But the spacing between the grooves is more in magnesium alloy compared to the composites, least in the SiC reinforced composite. Also the track width and depth is more in the magnesium alloy compared to the composites, and least in the SiC reinforced composite. Large craters can be observed in the magnesium alloy from where the materials has been dislodged. The wear craters observed in the composites are few and smaller. The wear track of SiC reinforced composite id free of



debris indicating that the material loss has taken place in the form of chips. The materials are not attached to the track as compared to the alloy in which large craters are seen as worn materials are attached to the track periphery.

#### 4.4 IMMERSION CORROSION TEST

Immersion corrosion test was carried out in a five percent NaCl solution. The specimens were immersed in the salt solution for a duration of 24 hours. Table 4.2 shows the weight loss of the specimens due to corrosion which occurred in the salt solution.

Specimen	Initial weight(gm)	Final weight(gm)	Weight loss (%)
AE42	0.86	0.7764	9.72
AE42-20A	0.86	0.597	30.58
AE42-10A-10S	0.65	0.4248	34.64

Table 4.2: Percentage weight loss of the specimens

From the table it is very much evident that composite having SiC is very much vulnerable to the corrosive environment. A surface film essentially of magnesium oxide is formed when the alloy is exposed to atmosphere. In the presence of moisture this magnesium oxide changes to magnesium hydroxide. This protects the magnesium alloy from the extensive corrosion. The above weight loss results reveals that as the volume percentage of reinforcements increases in the magnesium alloy, their corrosion tendency also increases. Higher corrosion tendency is exhibited by both the composites compared to the magnesium alloy. It can also be seen that for the same volume percentage of reinforcement, composite containing SiC particles showed more corrosion. The poor resistance to corrosion of the composites can be attribute to the lack of surface film on the surface of the sample. Also there is a formation of micro-galvanic cell between the magnesium matrix and the reinforcements. The SiC particles acted as a site for the

reduction of the oxygen. Figure 4.4 shows the variation in the weight loss in terms of percentage of the initial weight.

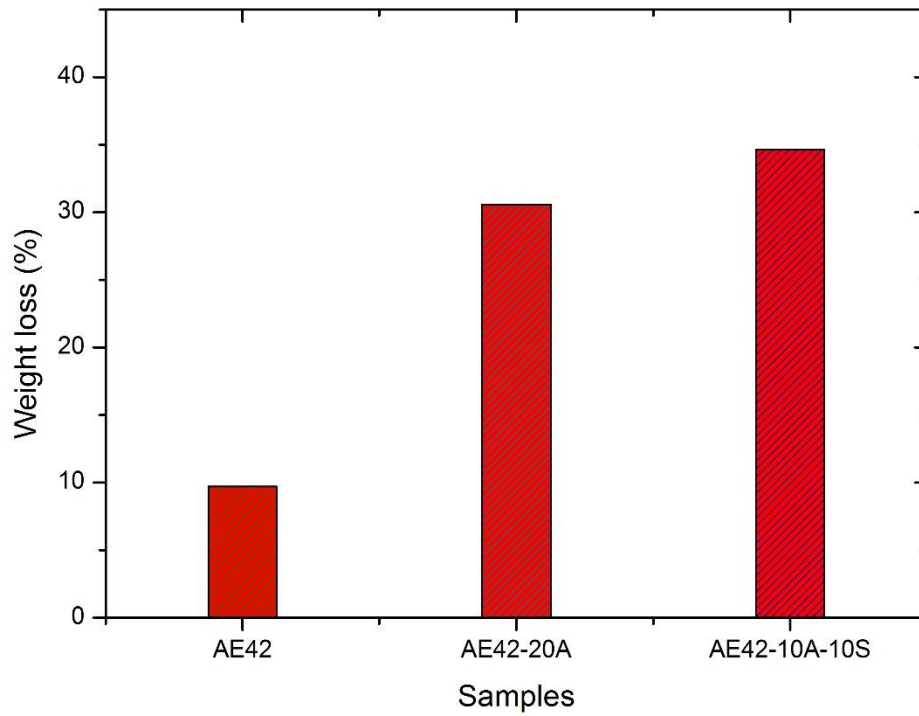


Figure 4.4: Weight loss of the specimens due to corrosion in the 5% NaCl solution.

Here, AE42 is the as-cast magnesium alloy

AE42-20A is the magnesium alloy containing 20 vol. % of saffil ( $\delta$ -Al<sub>2</sub>O<sub>3</sub>) fibres

AE42-10A-10S is the magnesium alloy containing 10 vol. % of saffil fibres and 10 vol. % of SiC particles

## CHAPTER 5: CONCLUSIONS

1. The microstructure of the magnesium alloy essentially consists of  $\alpha$ -mg matrix and Al<sub>4</sub>RE intermetallic compounds. Composites along with the magnesium matrix reveals saffil fibres and SiC particles.
2. SEM micrographs of the AE42 magnesium alloy shows polygonal grains of the magnesium matrix and dense compact of saffil fibres and SiC particles in the composites.
3. Hardness value of SiC reinforced composite is highest followed by 20% saffil fibres reinforced composites. Hardness value of magnesium alloy is least.
4. Wear resistance of SiC reinforced composite is best among the three specimens. Wear depth is highest for the magnesium alloy and it is having a wide wear track. Also large wear craters are there in magnesium alloy compared to the composites.
5. Composites are more prone to corrosion. This is because of the absence of protective oxide layer of magnesium oxide which is present on the surface of the magnesium alloy but absent in the case of composites.

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