

# **MICROSTRUCTURE AND PROPERTIES OF AZ91 MAGNESIUM ALLOY**

This thesis is submitted in the partial fulfillment of the requirement

For the degree of Bachelor of Technology

In

**Metallurgical and Materials Engineering**

By

**PRATIK AGARWAL**

**(111MM0014)**

**PALLAV SAHU**

**(111MM0380)**



**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

**2015**

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Under the Guidance of

**Prof. Ashok Kumar Mondal**



**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

**2015**



**Department of Metallurgical and Materials Engineering**

**National Institute of Technology, Rourkela**

## **CERTIFICATE**

This is to certify that the thesis entitled 'Microstructure and Properties of AZ91 magnesium alloy' submitted by PRATIK AGARWAL (111MM0014) and PALLAV SAHU (111MM0380) in partial fulfillment 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**

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## **ABSTRACT**

Magnesium is the lightest structural metal present on Earth's surface. With a density of 1.74g/cc, it gives a high strength to weight ratio. This has made it very useful for many applications like automobile, aeronautical etc. Magnesium has many limitations and because of which it cannot be used directly as a replacement of steel and aluminium.

Many alloys of magnesium have been developed to partially or completely overcome the limitations of magnesium without sacrificing other properties. One of the most commonly used commercial alloys is magnesium alloy AZ91, which gives better properties than magnesium.

In this project, microstructure and properties of AZ91 in two different casting conditions is compared. The microstructure is observed under the optical microscope. The hardness is measured by Vickers hardness tester. The wear resistance of the sample is compared. The SEM analysis of the wear tested sample is observed. The approximate corrosion rate is also calculated and compared.

*Keywords:* Magnesium alloy AZ91, Casting, Hardness, SEM, Optical, Wear, Corrosion

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# 1. INTRODUCTION

This project is based on the lightest structural metal present in abundance on the surface of earth, Magnesium. It is an element with atomic number 12 and mass 24. It belongs to the alkaline earth metals of the periodic table. It is the sixth most abundant element present on the surface of the earth and fourth most common element in the earth making almost 13% of the planets mass.

Magnesium has a density of 1.738gm/cc at 20°C which is almost one-fourth to that of steel and two-third of that of aluminum. This is the most important reason for the gaining importance of magnesium in structural applications. The parts which are formed with magnesium are light in weight which helps in increasing the fuel efficiency of a vehicle. Moreover the parts of the spacecrafts are also made of magnesium to decrease the weight of the shuttle. The alloys of this metal provide strength with reduction in weight without much increase in cost.

Some of the properties of magnesium are:

It has a HCP structure with  $c/a$  ratio = 1.624

(This value is closest to the ideal value of 1.633 than any other HCP metal.)

Ultimate tensile strength = 90 MPa

% elongation = 2-6%

Brinell Hardness = 30 MPa

In spite of very high effectiveness of magnesium, it cannot be directly used in many applications because:-

1. Magnesium is highly reactive, though once produced; it is coated in a thin layer of oxide, which partly inhibits this reactivity. The free metal burns with a characteristic brilliant-white light, making it a useful ingredient in flares.
2. Magnesium cannot withstand high temperature because of its low melting point of 650 degree C.
3. Magnesium has a high corrosion rate.
4. Magnesium also has a high creep rate.
5. Magnesium has high solidification shrinkage of about 4.2%. This makes it difficult to use magnesium in a work environment where there is a lot of temperature variations. Moreover care has to be taken while casting of magnesium alloy is done.

Due to these limitations, magnesium cannot be directly used for any application. Therefore, many alloys of magnesium have been developed by adding different metals like Aluminum,

Zinc, Zirconium, Manganese, Rare Earth elements, etc. These elements make different changes in the properties of magnesium alloy and hence make it usable in commercial scale.

One of the most commonly used magnesium alloy is AZ91. It has been used in many applications such as car seat frames, steering wheels, laptop frames, etc. and many others. The alloy's composition is 9% Al and 0.7% Zn in addition to these; some other elements are also present.

Alloying of a metal changes its properties like strength, hardness, corrosion resistance, etc. These changes can also be observed by applying different casting techniques. This project compares the microstructure and other properties of Magnesium alloy AZ91 under two different casting conditions:

- Gravity casted or simple sand casted
- Squeeze casted

The different methods applied for comparing the properties are as follows:

1. Casting for samples of both types is done.
2. The Microstructures are observed under Optical microscope under etched and un-etched condition.
3. The micro-hardness of the sample is tested by Vickers micro-hardness tester.
4. The behavior of material is observed for wear testing in increasing load condition.
5. The surface after wear testing is observed under Scanning Electron Microscope.
6. Corrosion testing of samples is done.

## 2. LITERATURE REVIEW:

The high strength to weight ratio of magnesium has always made researchers keen about the usability of it. But due to many limitations it cannot be used directly, so research work is under progress to overcome the drawbacks of magnesium by alloy addition.

1. Effect of addition of Aluminum on magnesium alloy: Aluminum imparts better casting properties, improves corrosion resistance and imparts excellent ambient temperature strength properties.
2. Effect of addition of Zinc on magnesium alloy: The presence of zinc provides solid solution strengthening to magnesium matrix. It is found that there is an increase in yield strength of magnesium alloys.
3. Effect of addition of manganese on magnesium alloy: Manganese is added in a small amount (0.3%) to increase the corrosion resistance of the alloy. Further addition does not have an increasing effect.
4. Effect of addition of calcium on magnesium alloy: Addition of 0.4% Ca reduces the grain size of the alloy. Increased addition progressively decreases the grain size. Further, strengthening of B-phase takes place and high temperature strength is improved without affecting the ductility. 0.8% Ca forms  $Al_2Ca$  precipitate which drastically decreases the ductility. So its addition is controlled.
5. Effect of Bismuth addition on magnesium alloy: It forms  $Mg_3Bi_2$  precipitates along the grain boundaries which is a thermally stable phase. This prevents the sliding of grains at elevated temperature.
6. The presence of antimony results in grain refinement of the compound. It also forms  $Mg_3Sb_2$  precipitates at the grain boundary which improves the room and elevated temperature strength, creep properties and fluidity.
7. Effect of Zirconium addition on magnesium alloy: It acts as an excellent grain refiner in magnesium alloys. But there is a limitation to its use. It cannot be used in presence of elements with which it forms stable compounds such as aluminum, manganese iron, etc.

The magnesium alloy AZ91 has main composition of 9% Aluminum, 1% Zinc and 0.3% Manganese. This alloy has high strength to weight ratio, high specific modulus and exhibits very good castability. But it loses its strength and other properties above 120°. This occurs due to the softening of the  $\beta$ -phase. Therefore, this material can only be used where the temperature does not exceed 120°. Many alloy additions are done in order to increase the high temperature usability such as Ca, Sb, Pb, etc. But care has to be taken that other properties are not sacrificed.

Many forms of Magnesium alloy AZ91 have been made, namely: AZ91A, AZ91B, AZ91C, AZ91D and AZ91E. Here, the last alphabet denotes the number of specific composition registered with the same major composition.

The alloy AZ91D is the major form of magnesium alloy which is used commercially because of its properties like:

- Good corrosion resistance.
- Low cost.
- Good strength and castability.

Some of the properties of magnesium alloy AZ91 are;

Tensile Strength – 250 MPa

% Elongation – 7%

Brinell Hardness – 70 HB

Alloys like AZ91A and AZ91B are used when the cost of material is further to be used and corrosion resistance is not a necessary criterion.

Magnesium alloy AZ91 shows good castability. Therefore, different casting methods can be applied for changing the properties of the alloy. The two different casting methods which show remarkable difference in properties are gravity casting and squeeze casting.

The difference in properties is due to the application of pressure for solidification and the usage of pre-heated die in squeeze casting process. The pre-heating of die is done to reduce the temperature gradient which is formed due to application of pressure. The pressure applied compacts the molten metal and leads to grain refinement of the material. The refined grain structure increases the strength, density and ductility of the material but it decreases the corrosion resistance due to the increase in surface area. As the thickness of the casted material increases, the effect of applied pressure decreases.

Due to the high rate of cooling, the chances of segregation while solidification of the sample decreases in case of squeeze casting. Whereas, in case of gravity casting, the inclusions and other products (if present) can get segregated and decrease the properties of the material. Different properties of gravity casted and squeeze casted samples can be measured by different processes. Some of these processes have been discussed below.

The difference in microstructures of the sample can be observed with the help of an optical microscope. The observations show the difference in grain size of the samples of gravity casted and squeeze casted magnesium alloys.

The hardness testing of the samples are done by Vickers hardness tester. This is one of the most common hardness measurement process and the results becomes the base for comparing with different materials. Due to the compaction process of the squeeze casted alloy, the hardness of this material is more than that of the conventional gravity casted samples. This can be easily observed with the help of Vickers hardness testing.

Wear is the surface erosion of a material due to application of frictional force on the surface of the material. Magnesium alloys shows very less resistance to wear. The wear resistance of both the samples can be compared with the help of a Ball on Plate Wear Tester. The wear depth vs Sliding distance plot can be made which shows the variation of wear with increase in load.

Scanning electron microscope is an apparatus which shows a magnified image of a material by focusing a beam of electrons on the surface of a specimen. It applies a Non Destructive testing method so no surface preparation is required. The surface analysis of the wear tested sample can be done. The microscope shows the variation of effect on the surrounding areas of the wear tracks with increasing load. The surface microstructure can also be easily observed with the help of a scanning electron microscope.

Corrosion testing of the materials is done by submerging the sample in NaCl solution. This is done to compare the effect of aggressive environment on the samples and their resistance to corrosion.

These comparisons shows the difference between the properties of the two types of casting techniques applied for the same composition of magnesium alloy AZ91.

## **3. EXPERIMENTAL PROCEDURES**

### **3.1. Casting:**

Casting is a process of fabrication of metals. In this process the metal or alloy is heated above its melting temperature to convert it to molten form. This molten metal is poured onto a mould. The mould consists of a hollow cavity which is of the shape of the required product. The molten metal is then allowed to cool and solidify.

Many casting procedures can be applied for magnesium alloys.

Two casting procedures have been studied:

1. Gravity casting;
2. Squeeze casting.

#### **3.1.1. Gravity casting**

Gravity casting is one of the basic metals casting process named after the material used for making the mould. This process has three basic requirements, a sand mould, a pattern and molten metal.

#### **Procedure:**

1. The sand has to be made loose and removal of inclusions is done.
2. The surface of sand is made plain with the help of a metal plate.
3. The pattern is kept on the surface.
4. Dry sand is sprayed over an area around the pattern.
5. The mould box is kept over the surface with the pattern at its center.
6. The positions of the riser and down runner are fixed with the help of conical wooden sticks.
7. Sand is poured onto the mould box to cover it completely and then pressed from top to ensure full packing with the help of a ram.
8. The riser and down runner are given proper shape and vents are made on different areas over the mould box with the help of a long stick.
9. The position of the mould box is marked with the help of wooden sticks.
10. The mould box is then lifted and the pattern is removed.
11. The runner is made and the mould is kept exactly back at the same place.
12. The metal is now poured through the down runner and then left for solidification.



Fig. 3.1. Mould for ingot casting.

### **3.1.2. Squeeze casting**

Squeeze casting is a type of casting process in which the solidification rate is increased by applying pressure on the molten metal. Moreover, the die used is preheated to an elevated temperature to prevent the formation of a high temperature gradient.

#### **Procedure:**

1. Die cavity, Which is located on the bed of a hydraulic press, was preheated and than apre-specified amount of molten metal was poured into it.
2. To close off the die cavity, the press was activated and the liquid metal was pressurized. This process was carried out very quickly because of the increase in solidification rate of the molten metal due to the application of pressure.
3. Until complete solidification, the pressure was held on the metal. This increases heat flow rate and eliminates macro/micro shrinkage porosity. The porosity formation due to dissolve gases in the molten metal was restricted, since nucleation of gas porosity is pressure-dependent,
4. At the end, the punch was withdrawn and the component was ejected.



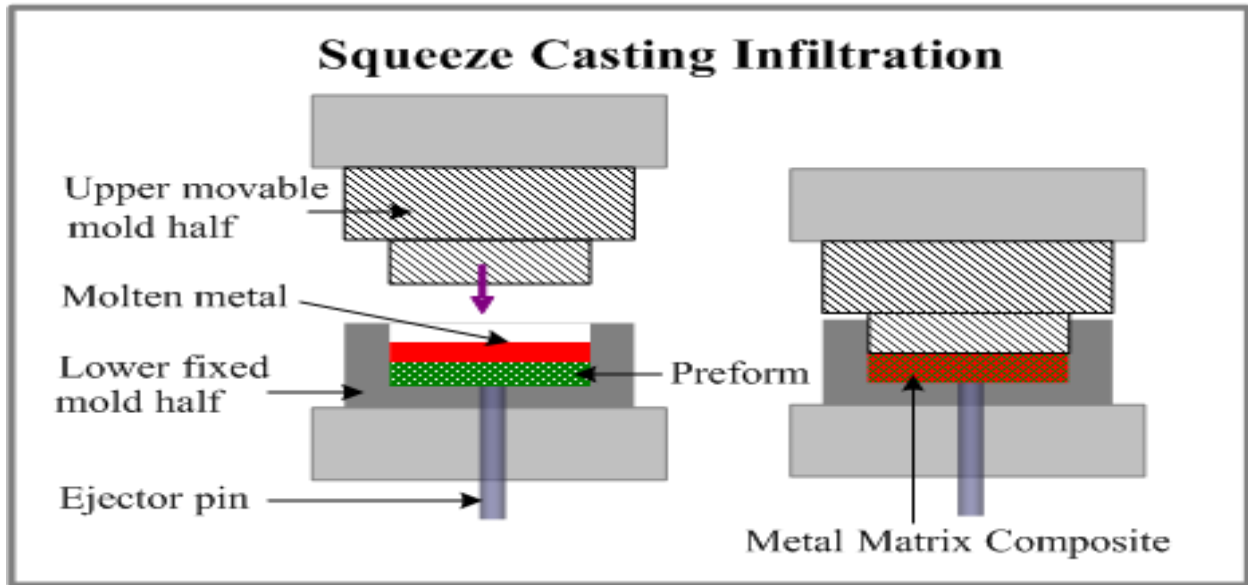


Fig. 3.2 Squeeze casting technique

The process of squeeze casting is preferred over traditional gravity casting because:

1. The pressure applied reduces the formation of blow holes, voids and other gas- associated defects.
2. The pressure applied prevents any solidification shrinkage to occur (very essential for magnesium because of its high solidification shrinkage).
3. It results in high material yield.

### 3.2. Cutting and Grinding of cast product.

The cast product formed cannot be used directly for further observations. It has to be cut to smaller sizes to make samples of the cast product. The cutting operation of the cast products is done using hack-saw.

#### The approximate size of the sample:

Length- 1cm.

Breath- 1cm

Height- 0.5cm

#### Number of samples:

Gravity casted- 2 samples.

Squeeze casted- 2 samples.

## Grinding

The samples prepared have rough edges and uneven surfaces. These samples have to be grinded on all sides to get a plane surface and remove any surface pits present. The grinding is done using belt grinder. Water can be used as coolant in this step because any reaction which occurs can be removed while polishing.

### 3.3. Cold Mounting

Cold mounting is process of mounting the sample without heating.

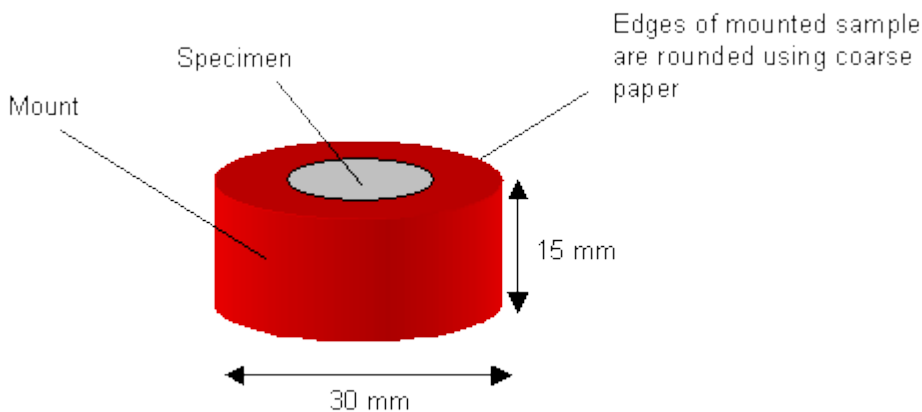


Fig.3.3 Cold Mounted sample

The materials used:

1. Epoxy resin
2. Hardener
3. Hollow Cylindrical plastic pipe of height 1.5 cm.
4. A glass plate

### Procedure:

- The grinded sample is kept on a glass plate with the surface to be polished kept upside down.
- The cylindrical pipe is kept such that the sample is positioned at the center.
- A few drops of the hardener is poured on and around the sample.
- The resin is then poured inside the pipe to fill it completely and few drops of hardener is again added from top.
- The whole apparatus is left for at least 8 hours without disturbance.
- After that the cold mounted sample can be used for further polishing treatments.
- This process is repeated for one sample of each of the cast products

## **3.4. Polishing**

The cold mounted sample is then polished by:

1. Sand paper/emery paper polishing.
2. Cloth polishing.

### **3.4.1. SAND PAPERS/EMERY PAPERS POLISHING**

Sand papers/emery papers of different categories are used for polishing such as:

- 0/0 paper – The paper is coated with very course grained sand particles. This paper is used to remove any surface imperfections generated due to cold mounting of the sample. The direction of movement of the sample does not have much importance at this stage.
- 1/0 paper – This paper is also coated with course grained sand particles, but these particles are finer than the 0/0 paper. This paper is used to polish the sample and align all the cracks in a single direction. Therefore from this stage onwards the direction of movement of the sample over the paper has the most important role. The sample is moved along a single direction from one end of the paper to the other end and then lifted to bring back to the start position. The sample is always kept with the same orientation. This step is repeated several times till the scratches visible on the surface of the sample are all aligned in the same direction and there is no surface defect observed.
- 2/0 paper – This paper is coated with comparatively finer sized particles of sand than that of the 1/0 paper. The sample is now rotated 90 degrees in clockwise or anticlockwise direction (which ever is convenient) from its initial direction and the polishing is continued in the same process as that in the 1/0 paper. This process is continued till all the scratches are aligned in the present direction of motion and no scratches are left in the perpendicular direction.
- 3/0 paper – This paper is coated with the even finer sized sand particles compared to that of the 2/0 paper. The sample is again rotated 90 degrees in clockwise or anticlockwise direction (which ever is convenient) from its current orientation. The polishing is again continued in the same process till all the scratches are aligned in the direction of motion with no scratches left in the perpendicular direction.

- 4/0 paper – This paper is coated with the finest sand particles of all the papers. Again the sample is rotated 90 degrees to its current orientation in clockwise or anticlockwise direction (which ever is convenient). The process is continued for a long time till all the scratches are aligned in the current direction of motion and no scratches are aligned in the perpendicular direction.

As the paper number increases, more finer polishing occurs. The size of the scratches decreases with increase in paper number. The number of scratch also increases with increase of paper number.

During paper polishing, care has to be taken that the force applied on the sample while polishing should not be very high because it will increase the depth of the scratch. Hence, more polishing will have to be done in the next step to change the orientation of the scratch.

If the orientation of all the scratches is not changed, then the scratches will not be completely removed from the sample. The pressure applied in the last polishing step with the 4/0 emery paper should be minimum. This is done so that the depth of scratches left on the surface will be very less and hence can be easily removed in the final cloth polishing process.

## 3.4.2. CLOTH POLISHING



Fig 3.4 Cloth used for cloth polishing of the samples

This is the final step of polishing and preparing the sample for observation. The sample polished from the 4/0 emery paper is then cloth polished to remove almost all the residual scratches left on the surface of the sample.

Cloth polishing is generally done by adding diamond paste to the cloth and frequently spraying HIFIN fluid onto the surface of the cloth while polishing.

The sample is placed onto the surface of the cloth polisher such that the orientation of the scratches is parallel to the direction of the imaginary radius drawn from the center to the sample.

Magnesium is highly susceptible to react with water so water cannot be used as a coolant while polishing because a layer of corroded material will be formed on the surface which cannot be removed. So, kerosene is used as coolant during polishing of magnesium sample. Kerosene has very less reactivity with magnesium and it evaporates very easily due to increased temperature.

The surface of the magnesium has a tendency to get burnt with rise in temperature in presence of oxygen. So, during cloth polishing, care has to be taken that the pressure applied is not too high to burn the surface of the sample. Proper usage of coolant is required.

### 3.5. Optical Microscope Observations

The optical microscope, often referred to as the "light microscope", is a type of microscope which uses visible light and a system of lenses to magnify images of small samples.



Fig 3.5 Optical Microscope

#### Principle of working of Optical microscope

An optical microscope works on the principle of conversion and diversion of light on passing through a lens. It creates a magnified image of an object specimen with an objective lens and magnifies the image further more with an eyepiece to allow the user to observe it by the naked eye. The optical microscope's magnification depends upon the combined magnification of the lens and the objective.

Table 3.1 Magnification of Optical Microscope

Sl. No.	Magnification of Eyepiece lens	Magnification of the Objective lens	Total magnification
1	10X	5X	50X
2	10X	10X	100X
3	10X	20X	200X
4	10X	50X	500X
5	10X	100X	1000X

### 3.6. VICKERS HARDNESS TESTING

Vickers hardness testing is a process of hardness measurement of a specimen. It measures the resistance of a specimen to indentation on application of constant load for small period of time.

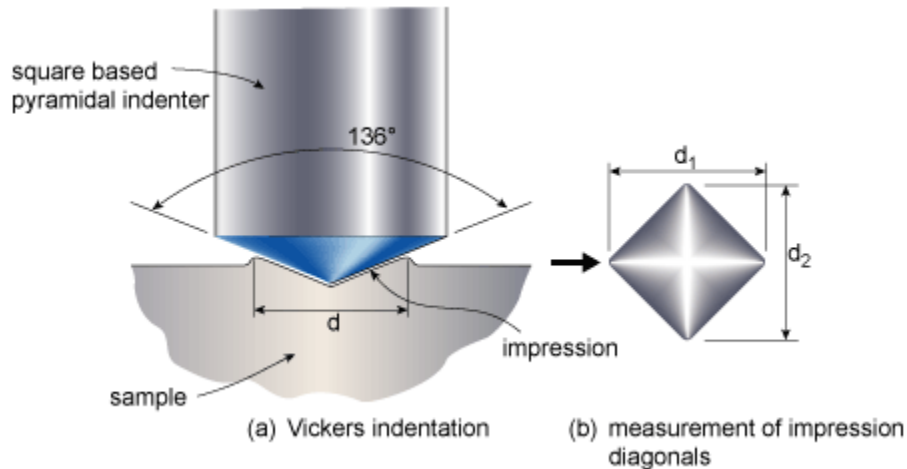


Fig. 3.6 (a)Vickers Indentation (b)Impression on the sample

The indenter used for Vickers hardness test is a square based pyramidal diamond indenter.

#### Procedure:

- The polished scratch free surface of the specimen is kept on the Vickers hardness apparatus.
- The optical microscope is focused on the area where indentation is to be made.
- The load to be applied and the dwell time are specified.
- Loading is commenced.
- With the help of the optical microscope and the measurement scales, the lengths of the diagonals are measured and the hardness value is calculated.

The hardness value is calculated using the following formula:

$$\text{Hardness value} = \frac{2 \cdot \sin(\theta/2) \cdot F}{d_1 \cdot d_2}$$

Where, F is the load applied

d1 is the length of one diagonal

d2 is the length of the other diagonal.

$\theta$  is the base angle of the indenter. The value of  $\theta$  is  $136^\circ$  of Vickers hardness testing and the length for both the diagonals are considered equal.

## 3.7. WEAR TESTING



Fig 3.7 Ball on Plate Wear Tester

### Procedure:

- The samples are grinded and polished to remove the surface imperfections (if any) present on the sample.  
Note: During wear testing it has to be taken care that the sample parallel and flat surfaces on both sides else a lot of variation will be seen in the observation. The height of the sample is a very important consideration and it cannot be more than 0.4 cm. Therefore, grinding of the sample may be required to get the optimum height.
- The sample is now fixed on the wear apparatus with the help of screws. Care has to be taken that the sample is firmly fixed so that it does not move during the testing operation.
- The ball indenter is fixed to get the required track diameter.
- The desired load, time and the rotation speed are fixed and the initial value of wear is made 0 micron.
- The software on the desktop is turned on to take the readings of wear.
- The name of the specimen and other specifications are entered in the software.
- The machine and the software are started consecutively.
- The machine and the software are stopped after the specified time.
- The same process is repeated for the second reading but the value of load and track diameters are changed.



### 3.8. Scanning Electron Microscope Observation



Fig. 3.8 Scanning Electron Microscope

A Scanning electron Microscope (SEM) is a device which is used to produce images of a sample by scanning the sample with focused electron beams. The electron beam interacts with atoms in the sample and produces various signals which are detected by the microscope. These signals contain information about the topography of samples and its composition. In the microscope, electron beams are scanned and beam's position is combined with the detected signal to form an image. SEM has a very high resolution of greater than 1 nm. Samples can be observed in high vacuum or in a low vacuum and it can also be observed in wet conditions (in environmental SEM) and at a wide range of temperatures.

SEM is a process of Non-Destructive Testing of a specimen so the wear tested sample is directly observed under Scanning electron microscope. There was requirement of no sample preparation operation. Care has to be taken that surface of the sample is not disturbed so the sample is covered with aluminum foil after wear testing is done before SEM analysis is done. The specimen was in high vacuum condition while observation.

## 3.9. Immersion Test/Corrosion Test

This test is done to find the corrosion rate of a material.

Materials used:

- Squeeze casted and gravity casted ingot samples
- NaCl powder - 35gms
- Distilled water – 1 Liter
- Chromium trioxide – 150 ml

Apparatus used:

- A 1 Liter beaker.
- Two 500ml conical flasks
- Two 500ml measuring beakers
- Wooden sticks
- Thread
- Weighing machine
- Tweezers

### Procedure:

1. The sample is grinded on all surfaces to remove any surface imperfections like pits and cracks.
2. The sample is polished to reduce the size and number of scratches on the surface of specimen.
3. The initial weight of the specimen is observed.
4. A 1 Liter beaker is filled with distilled water and 35gms NaCl powder is mixed to make a 3.5% NaCl solution.
5. The prepared solution is transferred to the two conical flasks.
6. The samples of each casting type are tied with a thread to the wooden stick such that when the samples are hung from the neck of the flask, they remain completely immersed in the solution without touching the bottom surface.
7. The flask is covered with aluminum foil and left for 24 hours.
8. Then the samples are taken out, cleaned with distilled water and their weights are measured after drying.
9. These samples are now dipped in Chromium trioxide solution for 10 minutes to remove all the corrosion product.
10. The sample is again cleaned with distilled water and their weights are measured after drying.



Fig. 3.9.1 Initialcorrosion testing sample and solution



Fig. 3.9.2 Sample and solution after 24 hours of corrosion testing



# 4.RESULTS AND DISCUSSION:

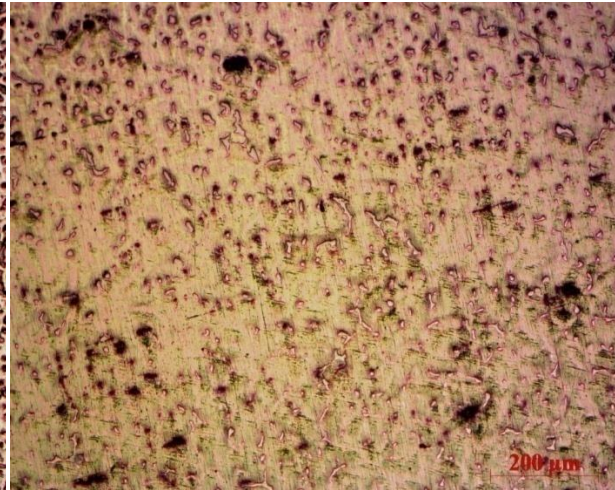
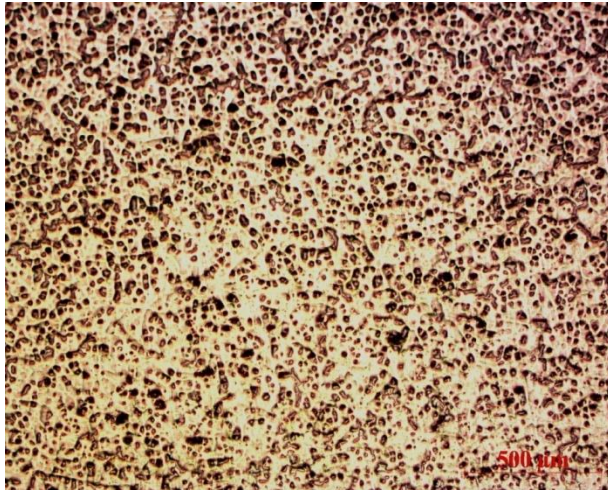
## 4.1. Optical Microscope observations:

The microstructure of:

1. Gravity casted ingot sample (un-etched):

a. Magnification -50X

b. Magnification – 100X



c. Magnification – 200X

d. Magnification – 500X

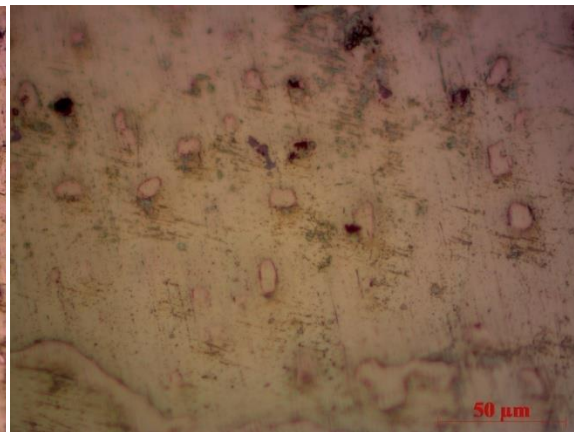
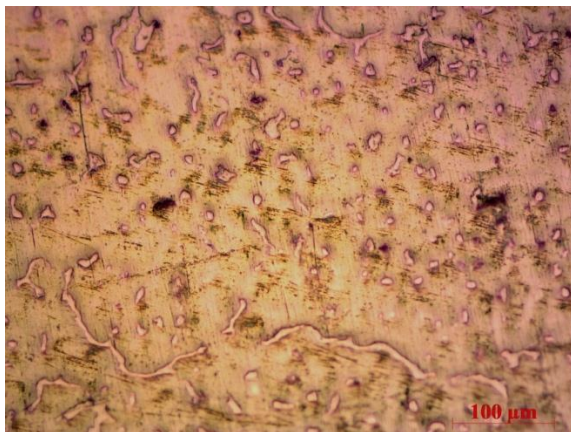


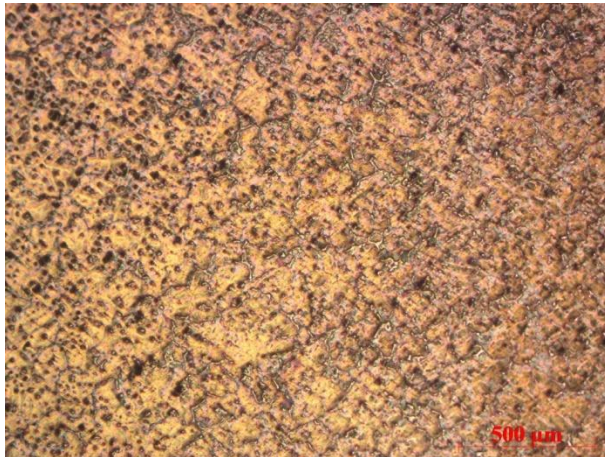
Fig.4.1.1 Gravity casted ingot sample (un-etched):



2. Microstructure of gravity casted (etched):

a. Magnification – 50X

b. Magnification – 100X



c. Magnification – 200X

d. Magnification – 500X

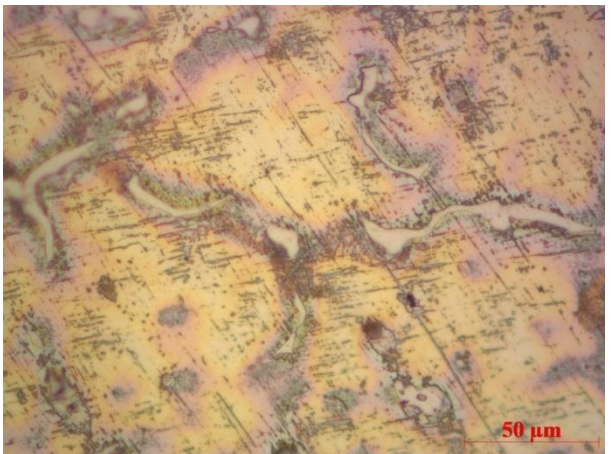
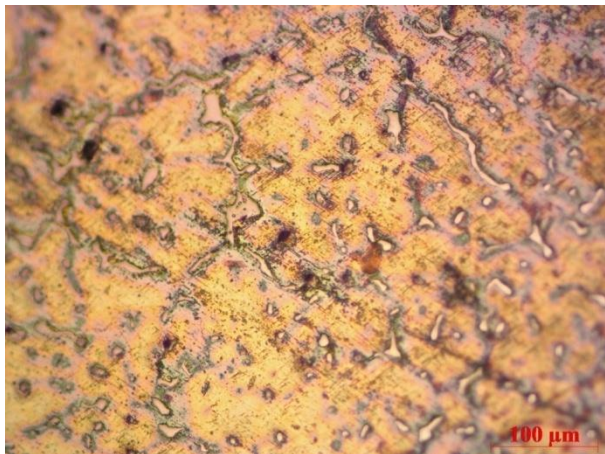
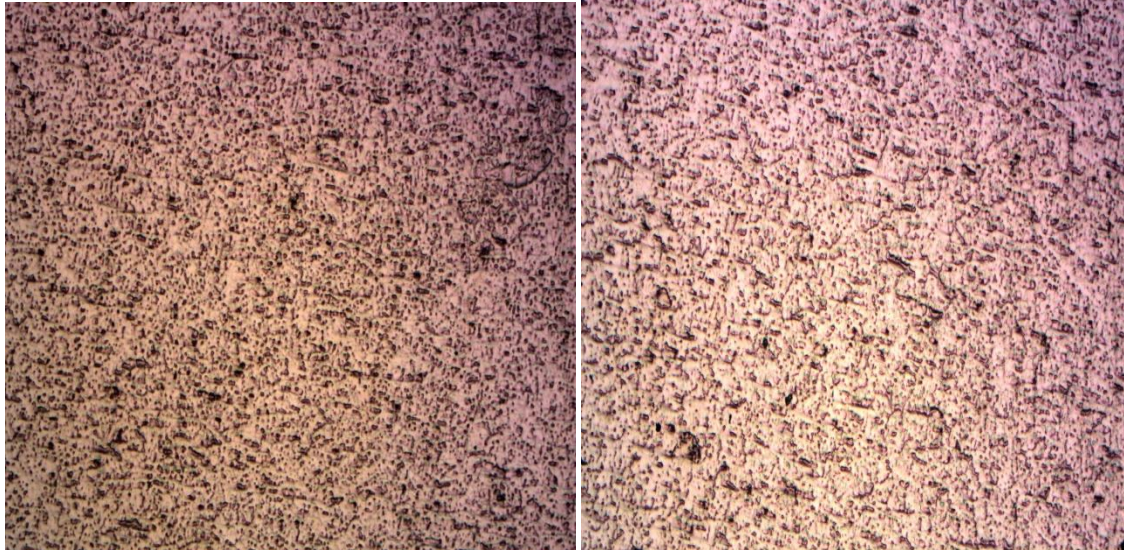


Fig 4.1.2 Microstructure of gravity casted (etched):



3. Microstructure of Squeeze cast ingot (un-etched):  
a. Magnification – 50X



a. Magnification – 100X

c. Magnification – 200X

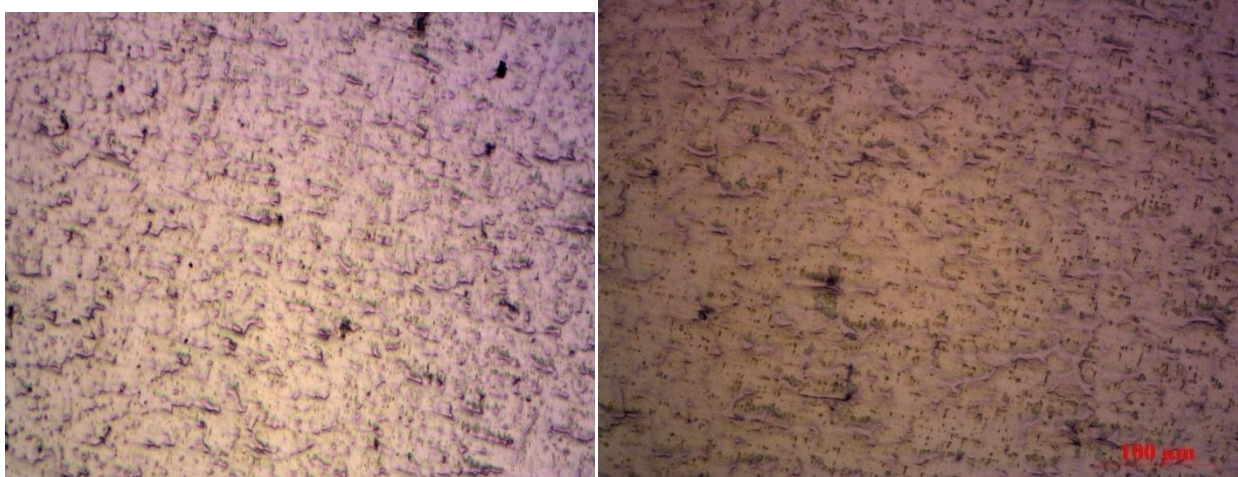


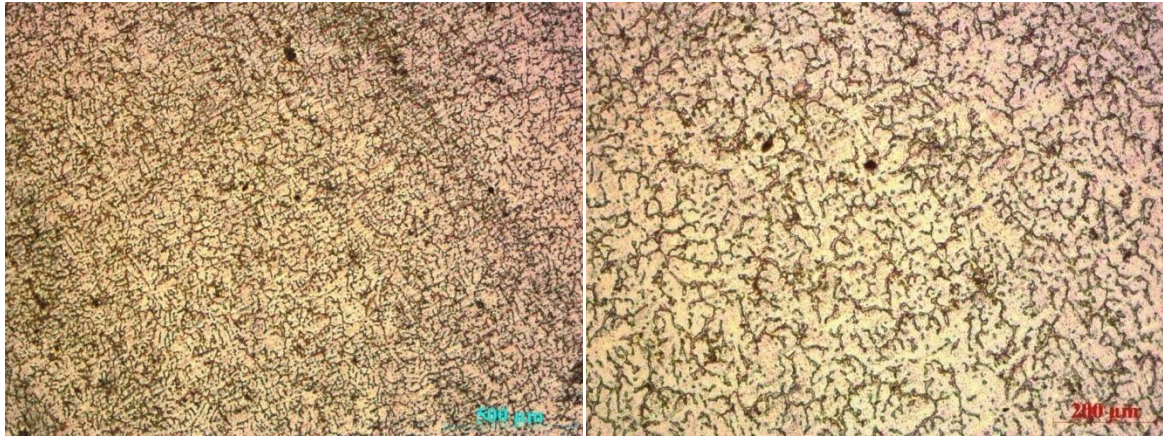
Fig 4.1.3 Microstructure of Squeeze cast ingot (un-etched):



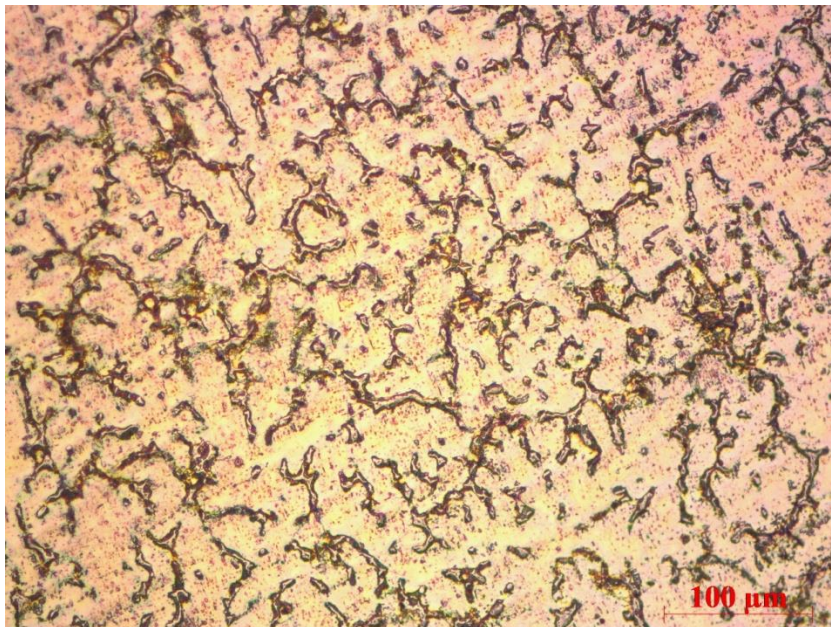
4. Microstructure of Squeeze cast ingot (etched):

a. Magnification – 50X

b. Magnification – 100X



b. Magnification – 200X



c. Magnification – 500X

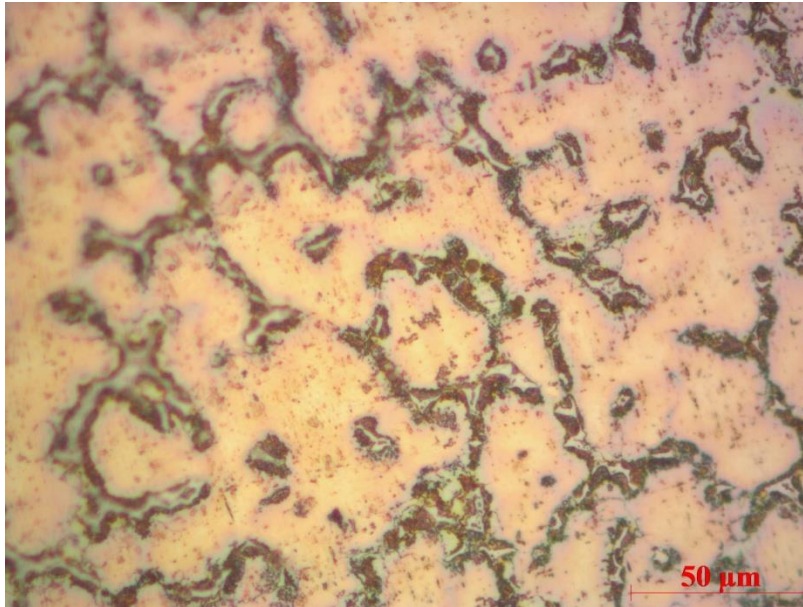


Fig 4.1.4 Microstructure of Squeeze cast ingot (etched):

The microstructure observed contains a dark phase and a light phase. The light phase is the  $\alpha$ -phase of magnesium. The dark phase is the  $\beta$ -phase precipitates of Mg17Al12 are formed due to the addition of alloying elements. Some dark black spots also observed. These spots can be inclusions or oxide precipitates formed due to reaction with air.

The observed microstructure shows finer grain structure of the squeeze casted sample than the gravity casted sample. The high pressure applied leads to increase in solidification rate so this grain structure is observed.



## 4.2. Vickers Micro-hardness observations:

Load applied = 500gmf

Dwell time = 10 seconds

### 4.1. Hardness of Gravity casted sample:

Sl. No.	Hardness Value
1	61.3 HV
2	63.9 HV
3	69.2 HV
4	68.3 HV
5	64.8 HV

$$\begin{aligned}\text{Mean Hardness Value} &= (61.3 + 63.9 + 69.2 + 68.3 + 64.8)/5 \\ &= \mathbf{65.5 HV}\end{aligned}$$

### 4.2. Hardness of Squeeze casted sample:

Sl. No.	Hardness Value
1	78.6 HV
2	82.7 HV
3	78.0 HV
4	81.1 HV
5	85.6 HV

$$\begin{aligned}\text{Mean Hardness Value} &= (78.6 + 82.7 + 78.0 + 81.1 + 85.6)/5 \\ &= \mathbf{81.2 HV}\end{aligned}$$

The higher hardness value observed for the squeeze casted sample is due to the high pressure applied during solidification of squeeze casted sample. The high pressure compacts the squeeze casted sample which leads to increase in bond strength of the material. Moreover, the fine grain size achieved increases the resistance to dislocation movement which leads to strain hardening of the material increasing the hardness of the sample.

### 4.3. Wear Test Observations:

The test was conducted at a constant speed of 25 rpm.

Time – 360 seconds.

The linear velocity (V) is calculated by using the formula:

$$V = \text{RPM} * \text{track radius} * 1.10472$$

Table 4.3. Wear test Parameters.

Track Diameter	2mm	4mm	6mm	8mm
Load Applied	5N	10N	15N	20N
Linear Velocity	0.00262	0.00524	0.00786	0.01048

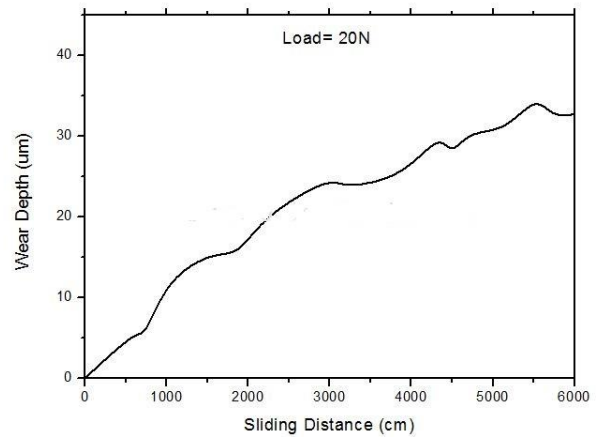
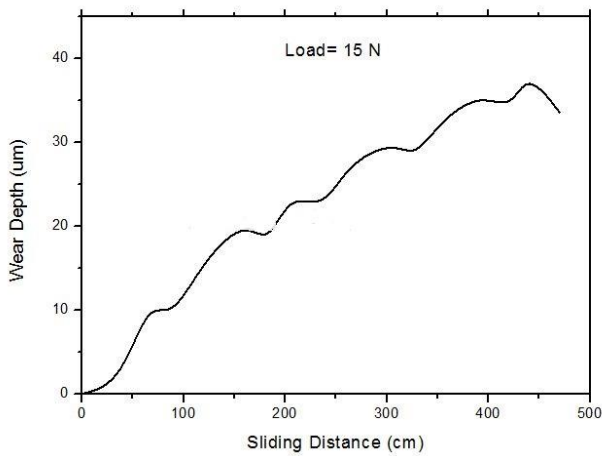
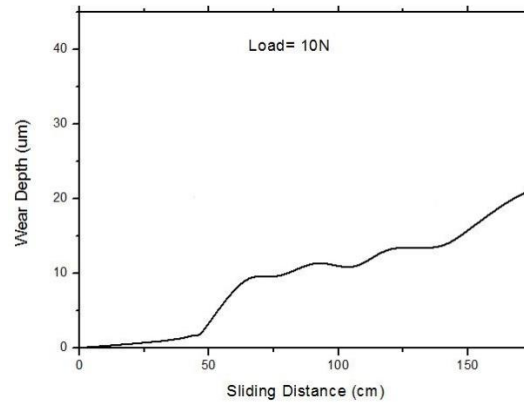
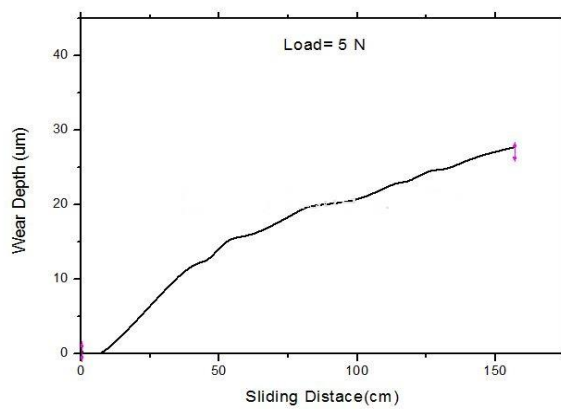


Fig. 4.2.1 Graphical observations for different track diameters in the Gravity Cast Sample:

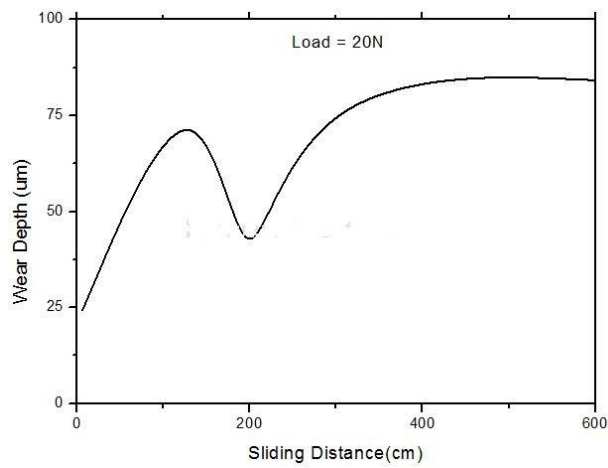
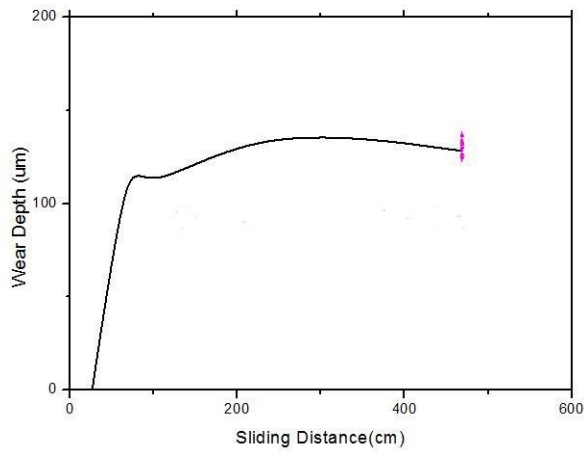
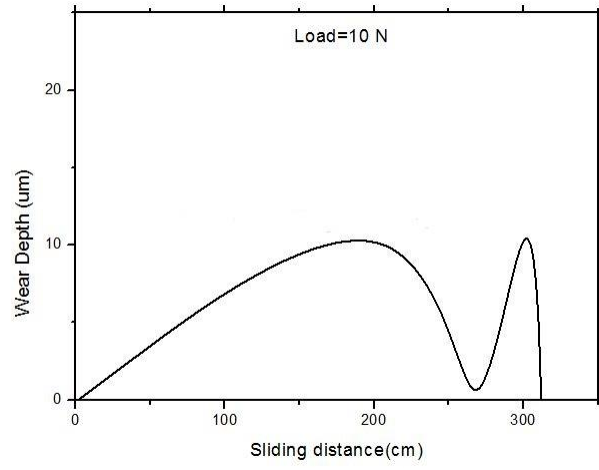
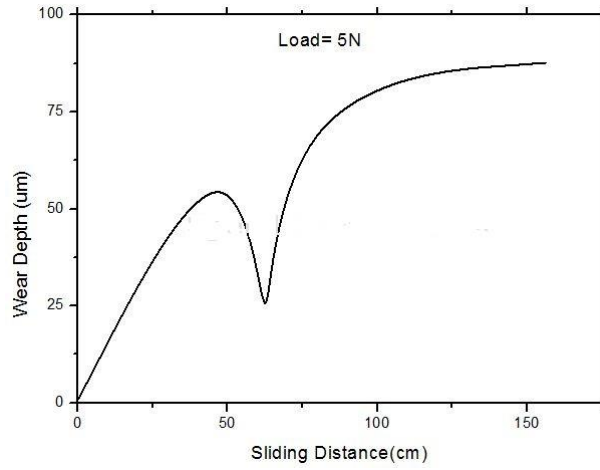


Fig. 4.2.2 Graphical observations for different track diameters in the Squeeze casted sample:

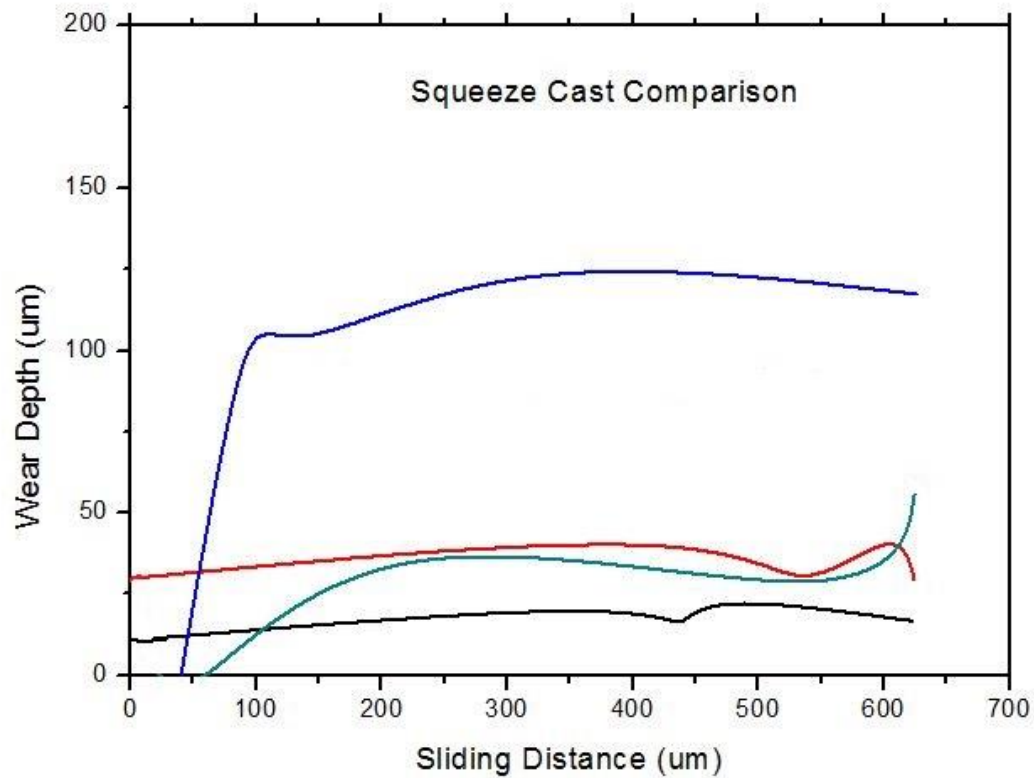


Fig. 4.2.3 Graphical observations for superimposing different track diameters in the Squeeze casted sample:

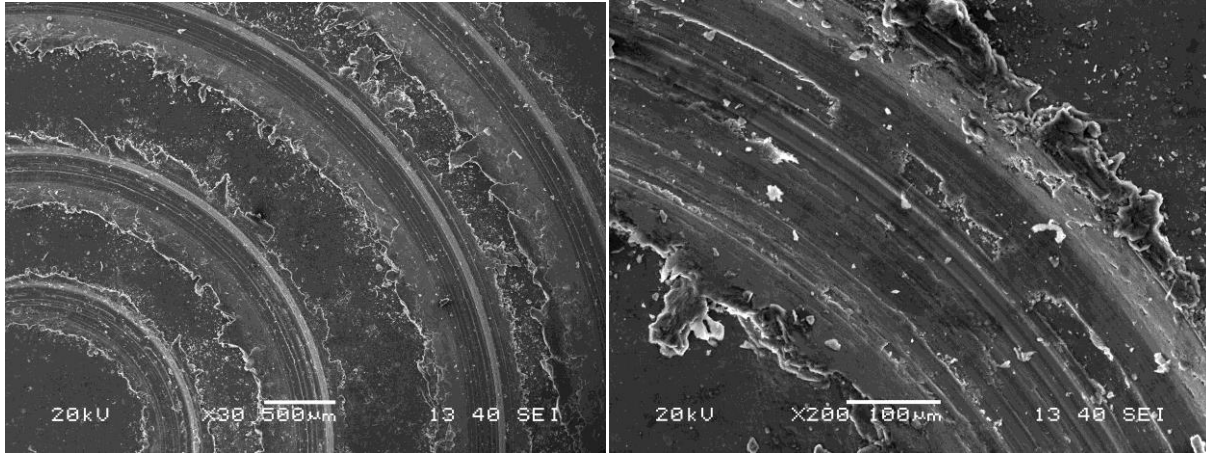
The wear test observations show that as the load increases, the wear depth increases. The scatter in the lines depicts the unevenness of the sample surface. This may be due to presence of surface cracks or voids. The uneven height of the sample can also be a governing factor.

A lot of variation in the wear depth with increasing load of 10N and 15N is not observed. This may be due to the increase in diameter with increase of load.

## 4.4. Scanning Electron Microscope Observations

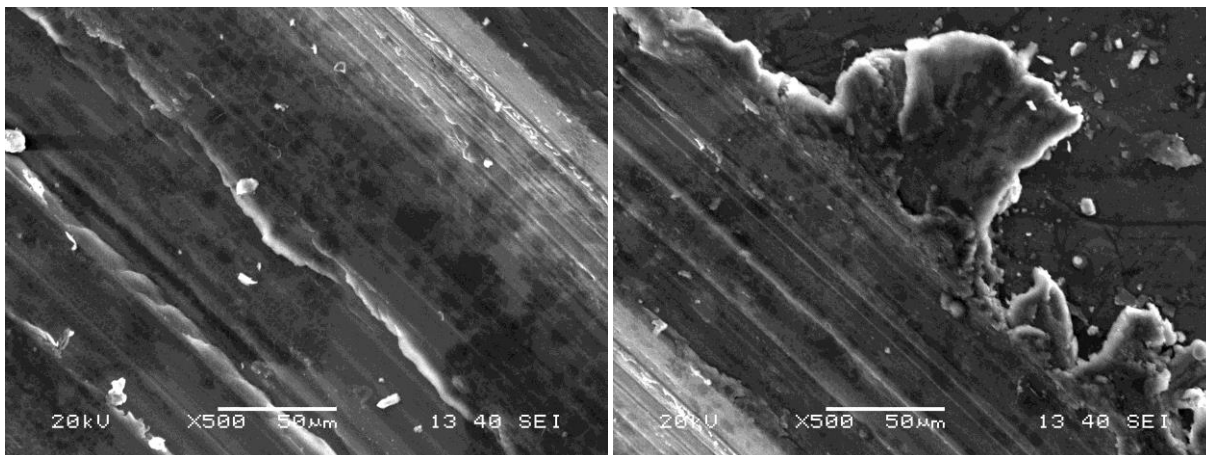
### 4.4.1. SEM of Gravity casted sample

- a. Wear cycles at 5N, 10N, 15N & 20N load    b. Wear cycle at 5N load



- c. Grooves observed at 20N load

- d. Area affected due to wear at 20N load



e. Microstructure of the grinded gravity casted sample.

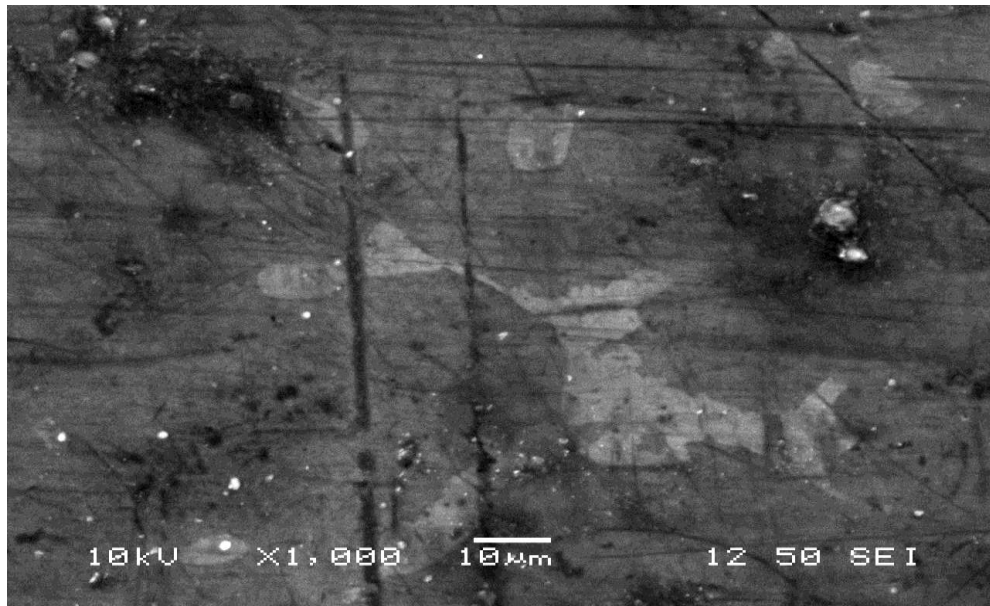
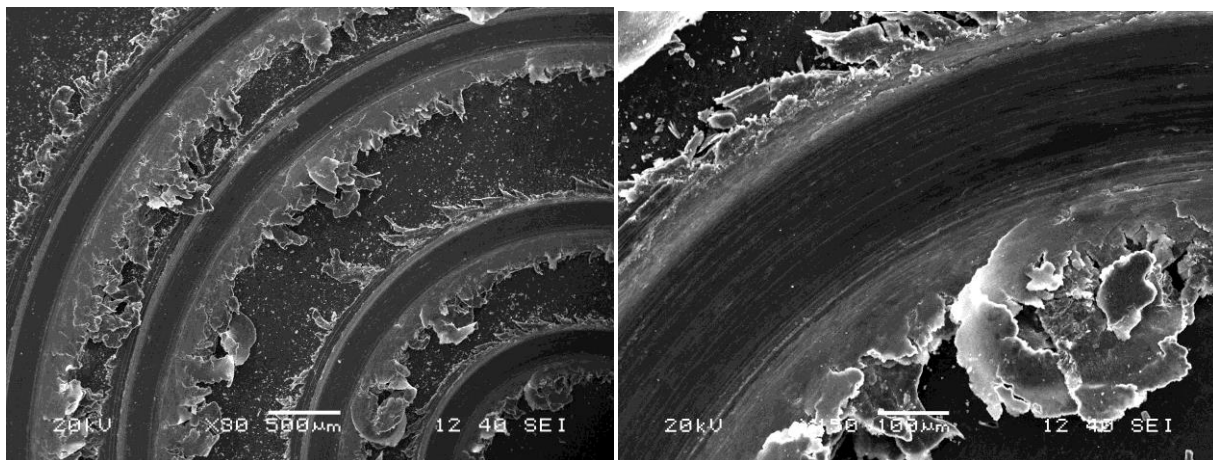


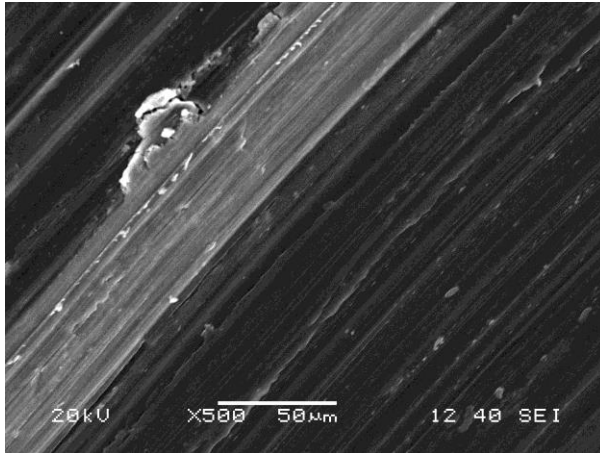
Fig. 4.4.1 SEM observations of Gravity casted sample

#### 4.4.2. Squeeze casted sample:

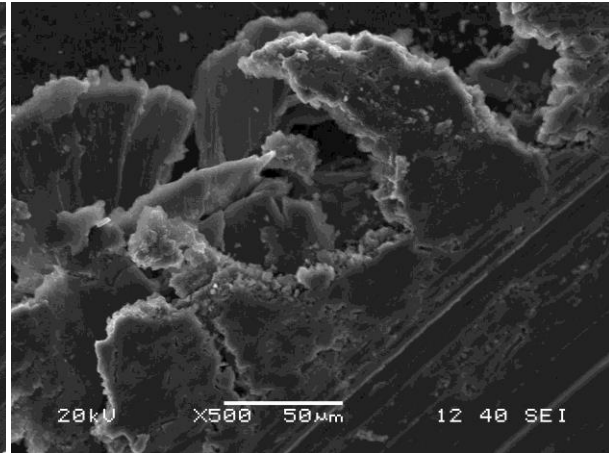
a. Wear cycles at 5N, 10N, 15N & 20N load    b. Wear cycle at 5N load



c. Grooves observed at 20N load



d. Area affected due to wear at 20N load



e. Microstructure of polished Squeeze casted sample.

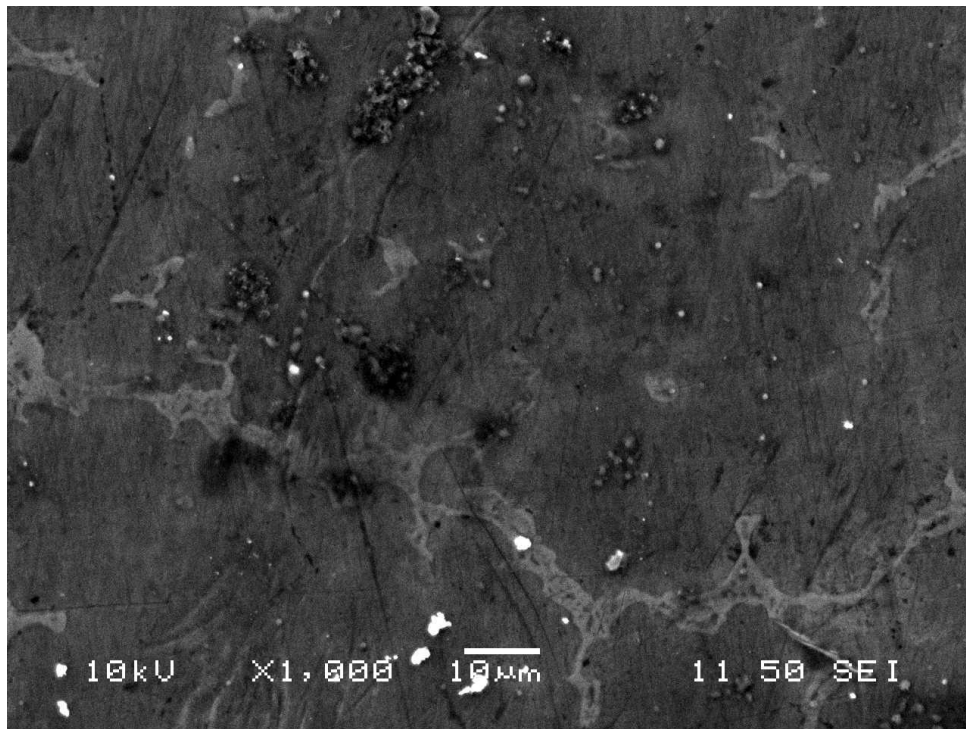


Fig. 4.4.2 SEM observations of Squeeze casted sample

Comparison of different tracks can be made with the help of the observations from the SEM. These observations show that as the load is increased, the area affected due to wear testing increases. The intensity and the number of grooves increase due to increase in linear velocities of the sample. Different phases in the microstructure are observed which are similar to that observed under optical microscope. Scratches and other surface defects are also observed.

## 4.5. Observations for Immersion/Corrosion Test:

Time for testing = 24 hours = 86400 seconds

Table 4.4. Immersion/Corrosion Test observations

	Gravity casted sample	Squeeze casted sample
Initial Surface area (approx.)	8.55 sq.cm.	5.32 sq.cm.
Initial weight	2.9332 gm	1.1170 gm
Weight after 24 hours in NaCl solution	2.7110 gm	0.7233 gm
Weight after dipping in Chromium trioxide	1.5504 gm	0.2154 gm

Percentage weight loss is calculated as

$$= (\text{initial weight} - \text{final weight}) / \text{initial weight} * 100\%.$$

So, percentage loss in weight of gravity casted sample in NaCl solution = 7.57%

Percentage loss in weight of squeeze casted sample in NaCl solution = 35.25%

Percentage loss in weight of gravity casted sample in chromium trioxide solution = 42.81%

Percentage loss in weight of squeeze casted sample in chromium trioxide solution = 70.22%

Corrosion Rate can be calculated as:

$$= \Delta W / (\rho * A * T)$$

Where,

$\Delta W$  is the weight loss of the sample.

A is the initial total surface area of the sample.

T is the Time interval for which the experiment is conducted.

$\rho$  is the density of the sample.

Therefore, the corrosion rate for gravity casted sample is = 0.0000167 mg/sec

And the corrosion rate for squeeze casted sample is = 0.0000476 mg/sec



Note: The corrosion rates observed is an approximate value. The exact value of the surface area and density of the sample are unknown. Therefore an approximate value is used for calculation of corrosion rate.

The percentage weight loss in each sample and the corrosion rates show that the gravity casted sample shows more corrosion resistance than the squeeze casted sample. This is observed because of the finer grain size of the squeeze casted sample which provides more surface area for corrosion reaction to occur and hence increases the rate of corrosion.

The chromium trioxide solution is used to remove the entire corrosion product formed on the surface of the samples. So the loss in weight of the samples after dipping in chromium trioxide solution is due to removal of almost the entire corrosion product. Even after dipping in chromium trioxide solution, the percentage weight is more in case of squeeze cast product. This shows that the amount of corrosion product formed is more in case of squeeze casted sample.

## **5. CONCLUSION**

The casting technique applied for squeeze casting of magnesium alloy AZ91 occurs at a much faster rate than that of the gravity casting process. More care has to be taken while squeeze casting process because the die is also pre-heated. Moreover, due to the application of pressure, there is a chance of splatter of the molten metal which has very high risks.

The microstructure observations with the help of optical microscope are done which shows the presence of  $\beta$ -phase in the Magnesium alloy. The different grain size of the different samples can be observed.

The hardness value for gravity casted and the squeeze casted sample is measured. The observed value shows the difference in hardness of the two types of casting. The hardness value of squeeze casted alloy is more than that of the gravity casted alloy.

Wear testing of the sample showed the difference in wear depth and sliding distance of the material when tested with a ball on plate wear tester. As the load and diameter increases the wear depth increases. It is also observed that squeeze casted sample shows more wear resistance than the gravity casted sample.

Scanning Electron Microscope observations show the effect on the area near the wear tracks because of changing load and track diameter during wear testing is done. The increased density of grooves is also observed. The microstructure of the specimen is also observed, the dark and light phases are visible.

The immersion/corrosion testing shows the comparison between the corrosion rates between the squeeze casted and gravity casted samples. The corrosion rate which theoretically should be higher in case of squeeze casted sample is confirmed.

## **FUTURE SCOPE**

Magnesium alloy AZ91 shows good properties which can be used in automobile industries and aeronautical applications. The increase in usage of the alloy will increase the fuel efficiency of the vehicle. The major concern is the high temperature applications of the alloy. If the material can be made usable for high temperature applications without increasing the cost of the alloy then it can become a replacement for aluminum and steel in many areas, providing similar strength with lesser weight. Therefore comparison of different techniques and alloys are required to get the optimum property required for a given scenario.

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