CHAPTER – I

INTRODUCTION

1.1 MAGNESIUM ALLOY
Magnesium is well known for its light weight characteristics and abundantly available nature in the earth. In 19th century magnesium was produced and used in large quantity for making aircraft components and automobile applications[1]. In engineering applications magnesium was not used alone but magnesium alloy was normally used for these applications for weight reduction to strength ratio. Also magnesium alloy have superior characteristics compared to other structural metals. Some of the alloying metals used are aluminium, zinc, manganese, calcium, tin, lithium, zirconium, etc.

1.1.1 Advantages
- Magnesium alloys have high strength to weight ratio when properly alloyed.
- Magnesium is a ductile material so it helps for easy forming and machining operations.
- Higher corrosion resistance occurred in some of the magnesium alloys[2].

1.1.2 Applications
Automobile industry uses magnesium alloys for making vehicle parts like steering wheel, wheel rim and seat frame in more quantity [3] and power train applications [4]. Magnesium alloys are used as implants in human body because of its mechanical properties are alike to the human bone [5]. Aerospace structure systems are made by magnesium alloys to reduce the weight of the satellite [6].

1.2 MAGNESIUM COMPOSITES
Composite materials have superior properties than individual components were made by various processing methods for different purposes. Fiber, metal, or ceramic etc are used as reinforcement to improve the mechanical, physical and thermal properties of the composites. These materials specially used for light weight applications with improved properties of its own elements [7]. Various reinforcements in magnesium
matrix like Silicon Carbide (SiC), Aluminium Oxide (Al₂O₃), Carbon Nanotubes (CNT), Titanium Carbide (TiC), and Boron Carbide (B₄C) provides different mechanical properties required for different applications [8].

1.2.1 Advantages
Nowadays magnesium metal matrix composites (MMCs) mainly used in automotive and aerospace applications because of its increased modulus compared to unreinforced alloy. Also the MMC shows better wear properties and low coefficient of thermal expansion thus leads to considerable applications. H. Ferkel et al., prepared Mg composite by reinforcing nano-SiC particles by powder metallurgy (PM) route and followed by hot extrusion were obtained lowest creep rate and largest flow stress composite compared to pure Mg and other composites [9]. S. F. Hassan et al., synthesized a magnesium nano-composite using nano- Al₂O₃ which reveals increased elastic modulus, hardness, 0.2% yield strength, ductility and UTS compared to pure Mg [10].

1.2.2 Applications
Mg MMCs with light weight and high tensile strength characteristics leads to the wide applications of automotive structures and transportation industries. Y. Shimizu et al., developed a Mg composite by reinforcing multi-walled CNT in AZ91D Mg alloy and obtained high tensile strength of 388MPa with light weight characteristics also they suggested that the composite can be utilized in transportation industries to save energy[11]. M. Razavi et al., prepared a composite by reinforcing nano-fluorapatite (FA) in AZ91 Mg alloy matrix for biomedical applications and the composite showed improvement in elastic modulus, corrosion resistance and hardness [12].

1.3 MAGNESIUM MMC PROCESSING
MMC consists of mainly two components which are matrix and reinforcement. A metal is used as matrix and metal or ceramic used as reinforcement. Reinforcements may fall into any one of the following categories which are particulate, whiskers, wires, continuous fibers and discontinuous fibers. MMCs produced for different kinds of applications need suitable mechanical properties which can only be obtained by selecting appropriate manufacturing process. When magnesium exposed to atmosphere leads to atmosphere corrosion because of the oxygen presence in the
atmosphere. Also magnesium is a flammable metal and flame temperature will reach up to 3090°C. Although corrosion and flammable properties affect the processing there are different types of methods were used by the researchers to make magnesium MMC effectively.

### 1.3.1 Types

Generally there are five categories of manufacturing methods used to synthesize Mg MMCs. They are liquid phase processes, solid phase processes, liquid-solid phase processes, deposition techniques, and in situ processes.

- **Liquid phase processes** generally termed as casting and have different methods of mixing the particles with the matrix which are
  - Melt Infiltration
  - Pressure Infiltration
  - Melt Oxidation Processing
  - Liquid Metal Ceramic Particulate Mixing

- **Solid phase processing** generally used metal and ceramic powders processed in number of steps before final preparation. The methods are
  - Powder Metallurgy
  - Diffusion Bonding
  - High Energy High Rate process

- **Liquid-solid phase processes** also termed as two phase processes. Reinforcements are mixed in the region of the phase diagram where the matrix contains both liquid and solid phases. Liquid-solid phase processes are
  - Compo-Casting Process
  - Ospray Deposition Process
  - Variable Co-Deposition of Multiphase Materials

  In deposition process, individual fibers are coated with the matrix material followed by diffusion bonding to form the composite. Some of the deposition processes are
  - Spray Forming
  - Spray Deposition
  - Physical Vapour Deposition (PVD)
  - Chemical Vapour Deposition (CVD)
• Electro Plating
• Immersion Plating [13]

i. Casting process involves pouring of molten metal in a mould cavity and allowing it to solidify to form the product. In MMC casting it is little different. MMCs contain matrix and reinforcement which are different materials and they have different melting points. So either the reinforcement is mixed in the liquid matrix to form the composite or by adding liquid matrix to the arranged fibers. In melt infiltration process, molten metal drawn in to a preform by capillary action. Inert gas pressure is used to force the molten metal in to the preform in case of pressure infiltration process. By oxidation method molten metal is mixed to the preform thus forming composite in melt oxidation process. Particle reinforcements can be mixed directly in to the molten metal in case of conventional casting method [14].

ii. The technology of pressing metal powders into the desired shape called Powder Metallurgy. In this process precision components can be made rapidly and economically in high volume from powders. There are different consolidations techniques available to make sheets, rods and compacted isostatically from powders. Secondary processes like rolling extrusion and be done for sintered compacts to get superior properties of mixed powders. Careful control of process parameters and structure of metal powders results intelligent manipulation of the structure of final sintered materials. High performance alloys with higher density can be manufactured with uniform microstructure which was difficult to synthesize using other methods.

1.4. POWDER METALLURGY PROCESS
PM process starts with production of metal powders by different methods. The steps of PM process are
• Selection of Metal Powders
• Powder Mixing
• Compaction
• Sintering
Secondary Process

Selection of metal powders with particular geometry is an important factor that decides the characteristics of the final compacted product. Especially the particle size, quantity, particle shape and selection of metal type are the most significant factors that give the appropriate metallurgical and mechanical properties of the resulting alloy. While processing metal powders make bonding between each other forms grain boundary and new phase. Also metal powders have more surface area which leads to rapid oxidation in the case of metals like magnesium. Oxides in the metal powders lead to poor mechanical properties and it should be considered while selection of metal powders.

Powder mixing involves mixing of different metal or ceramic powders with additives, lubricants and considering other variables. The variables are:

- Mixing Time
- Type of Mixer
- Volume of Mixer
- Rotational Speed of the Mixer
- Mixing Temperature
- Geometry of the Mixer
- Volume of the Mixer
- Mixing Medium

During mixing segregation of particles may occur. This occurrence due to physical properties of powder, type of handling equipment used, flow rate of the powder and inner surface properties of mixer. Some common types of mixer geometries are rotating cylindrical, rotating cube, double cone and twin shell. Alloying by PM method is very useful because the compositional adjustment can be adopted by readily available powders where which cannot be done by any other methods.

Compaction of metal powders otherwise termed as powder packing starts with the filling of mixed powders in a die cavity of the desired geometry. During compaction powders are squeezed by punch and the powder particle undergoes plastic deformation which produces the compact and the ejected from die for subsequent process. Hydraulic and mechanical presses are used to apply pressure. Compaction pressure is an important factor for deciding the mechanical properties of the compact.
Selection of the press depends on adjustable tonnage, adjustable drawing speeds and constant pressure during entire stroke.

Sintering is the thermal process for consolidating metal powders into coherent structure. This bonding leads to improved properties like strength, electrical and magnetic properties. In this step the compacted preform placed inside a furnace at a constant temperature for a specific time period. During sintering, powder particles make bonding with the neighboring particle and produce a strong bond between them depends on the type of metal. Most of the metals exhibit high temperature corrosion inside the furnace which affects the strength and surface properties of the alloy. This can be avoided by controlling the temperature and furnace atmosphere. An effective way is providing vacuum atmosphere while sintering which eliminates the oxygen contact to the preform.

1.4.1 Advantages
Multiphase composites with wide combination of properties can be economically produced by PM route. Non-equilibrium materials such as microcrystalline and amorphous alloys also produced using PM technique. Also this route is many times more competitive the other fabrication methods like casting, stamping and machining. The technology of powder hot forging can be used to produce precise engineering parts which have properties better than the conventional forging components. New alloying elements can be used regardless of solubility of the metals. Higher density components can be achieved by PM technique compared to conventional casting method. By powder injection moulding (PIM) stronger more uniform and more complex parts can be produced. In various engineering applications a wide range of porosity levels effectively can be utilized.

1.4.2 Applications
In various engineering industries PM products widely used and mainly in automotive industry. The following table 1.1 shows the usage of PM parts in engineering industry [15].

1.5. HOT EXTRUSION
Sintered compact normally exhibits poor strength and mechanical properties. So at this stage the products cannot be used for high strength applications. A
secondary process is mostly needed to improve the mechanical and metallurgical properties.

Table 1.1: Usage of PM Parts in Engineering Industry

<table>
<thead>
<tr>
<th>Application</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Automotive</td>
<td>73</td>
</tr>
<tr>
<td>Recreation, Hand Tools, and Hobbies</td>
<td>10.5</td>
</tr>
<tr>
<td>House Hold Appliances</td>
<td>4.3</td>
</tr>
<tr>
<td>Hardware</td>
<td>3.1</td>
</tr>
<tr>
<td>Industrial Motors, Controls and Hydraulics</td>
<td>1.9</td>
</tr>
<tr>
<td>Business Machines</td>
<td>1.2</td>
</tr>
<tr>
<td>All Others</td>
<td>6</td>
</tr>
</tbody>
</table>

Hot extrusion improves the strength and physical properties of the sintered billet. Hot extrusion is a forming process of obtaining long and straight bars, rods and other shapes by forcing the billet to flow through a die opening when the billet is heated above its recrystallization temperature. The ratio between the cross sectional area of the initial billet to the final extruded rod is called extrusion ratio [16].

Figure 1.1: Extrusion Process

1.6 TAGUCHI METHOD

In PM method, different metal powders mixed in different proportion and the alloy is prepared. The proportion of components of the alloy decides the metallurgical properties and other properties. So finding the correct proportion of alloying powders needs lot of samples and thus increasing the cost of the process. Optimal parameter of
the alloy can be found by different analyzing methods with little data and samples. Taguchi based grey analysis is one of the most efficient method to optimize the alloying elements with different fixed characteristics by analyzing their signal to noise ratio (S/N). By Taguchi method, static and dynamic problem process parameters can be optimized. Static problems focused to find the best control factors which decide the result. Dynamic problem uses Taguchi method to find the process signal which decides the closest expected output required.

1.6.1 Orthogonal Array

Orthogonal arrays used in Taguchi method were selected based on number of levels and number of parameters chosen. Once the levels and parameters are set then the appropriate orthogonal array will be selected. Table 1.2 shows the orthogonal array selection based on the number of parameter sand number of levels. For example three parameters with three level processes can be optimized by using L9 orthogonal array as shown in table 1.3.

<table>
<thead>
<tr>
<th>Number of Parameters (P)</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Levels (L)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>2</td>
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<td>L4</td>
<td>L8</td>
<td>L8</td>
<td>L8</td>
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<td>L12</td>
</tr>
<tr>
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<td>L9</td>
<td>L9</td>
<td>L18</td>
<td>L18</td>
<td>L18</td>
<td>L27</td>
<td>L27</td>
<td></td>
</tr>
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<td>L32</td>
<td>L32</td>
<td></td>
</tr>
<tr>
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<td>L50</td>
<td>L50</td>
<td>L50</td>
<td>L50</td>
<td></td>
</tr>
</tbody>
</table>

Table 1.3: L9 Orthogonal Array for 3 Levels 3 Parameters

<table>
<thead>
<tr>
<th>Number of Runs</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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</tr>
<tr>
<td>2</td>
<td>1 2 2</td>
</tr>
<tr>
<td>3</td>
<td>1 3 3</td>
</tr>
<tr>
<td>4</td>
<td>2 1 2</td>
</tr>
<tr>
<td>5</td>
<td>2 2 3</td>
</tr>
<tr>
<td>6</td>
<td>2 3 1</td>
</tr>
<tr>
<td>7</td>
<td>3 1 3</td>
</tr>
<tr>
<td>8</td>
<td>3 2 1</td>
</tr>
<tr>
<td>9</td>
<td>3 3 2</td>
</tr>
</tbody>
</table>
1.6.2 S/N Ratio

In every static problem, control factors are to be optimized to achieve the expected result. There are three characteristics of the control factors analyzed. They are larger-the-better, smaller-the-better and nominal-the-better. Generally in manufacturing process defects to be eliminated so it was fixed as smaller-the-better characteristic and production rate expected to be higher so higher-the-better characteristics can be taken. Once the characteristics fixed, the number of runs decided based on the control parameters [17].

\[
\text{Higher – the – better: } \frac{S}{N_l} = -10 \log \frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2} \tag{1.1}
\]

\[
\text{Nominal – the – better: } \frac{S}{N_T} = 10 \log \frac{y}{S_y^2} \tag{1.2}
\]

\[
\text{Smaller – the – better: } \frac{S}{N_s} = -10 \log \frac{1}{n} \sum_{i=1}^{n} y_i^2 \tag{1.3}
\]

Where \( n \)-number of observations, \( y \) is the observed data, \( y \) is the mean of the observed data, and \( S_y^2 \) is the variance of \( y \). Though Taguchi method analyzes the output data with individual characteristics it cannot be used to find the multiple performance of alloying elements. The alloying elements will show different performance with the other elements when their percentage varies. At this time grey analysis is used to find related and multiple performance characteristics of compositions in alloy by analyze the obtained data from Taguchi method.

1.7 GREY RELATION ANALYSIS

Grey relation method [18] uses the obtained S/N ratio from the Taguchi analysis to find the grey relation coefficient which is calculated from the normalized S/N ratio. S/N ratio normalized between zero and unity. Then a grade is calculated by averaging the grey relational coefficient related to each performance characteristic. Highest grade shows the optimal parameter level of the composition of the alloy and the significant alloying element can be predicted by using ANOVA method. The steps of grey analysis are generating grey relation, computing grey relation coefficient, and calculating the grey relational grade. For normalizing the obtained S/N ratio the following formulae have been used.
\[Higher - the - better: x_{ij} = \frac{y_{ij} - \min y_{ij}}{\max y_{ij} - \min y_{ij}}\]  
(1.4)

\[Nominal - the - better: x_{ij} = \frac{y_{ij} - y_0}{\max y_{ij} - y_0}\]  
(1.5)

\[Smaller - the - better: x_{ij} = \frac{\max y_{ij} - y_{ij}}{\max y_{ij} - \min y_{ij}}\]  
(1.6)

Where,

\[i = \text{Performance Characteristic},\]

\[j = \text{Experiment Number},\]

\[y = S/N \text{ Ratio of the Parameter},\]

\[x = \text{Normalized S/N Ratio}\]

Grey Relation Coefficient \(\xi_{ij}\)

\[\xi_{ij} = \frac{\min_j \min_i x_i^0 - x_{ij} + \zeta \max_j \max_i x_i^0 - x_{ij}}{x_i^0 - x_{ij} + \zeta \max_j \max_i x_i^0 - x_{ij}}\]  
(1.7)

Where,

\[x_i^0 = \text{Ideal value of normalized S/N ratio for } i^{th} \text{ performance characteristic}\]

\[\zeta = \text{Distinguishing coefficient generally between zero and unity}\]

Grey relational grade \(Y_j\)

\[Y_j = \frac{1}{m} \sum_{i=1}^{m} w_i \xi_{ij}\]  
(1.8)

Where,

\[m = \text{Number of performance characteristics},\]

\[w = \text{Weighting factor}\]

**1.8. CHARACTERIZATION STUDY**

Understanding the behavior of materials required special knowledge and knowing the properties of materials by various characterization studies. For the past couple of decades many characterization methods have been used by researchers to understand the behavior of materials. Analyzing the microstructure of material by various imaging techniques provide the arrangement of grain structure, grain size which reveals the effect in their physical and chemical properties. We will discuss some of the characterization techniques [19] such as microstructure study, X-ray diffraction test (XRD), tensile, compressive, hardness, density and porosity studies.
1.8.1 Microstructure Study
Microstructure study of materials refers the analyzing the image captured using various imaging techniques and the microstructural properties like grain refinement, orientation of grains and crystal structure were studied. These properties strongly influence in the macroscopic properties. Optical microscope used visible light with imaging device used to capture the surface texture of the prepared, polished and etched surface of the material. Different magnification of images can be obtained by changing the eyepieces of the microscope like 50X, 100X, 200X, 400X etc.

1.8.2 XRD
In the characterization of materials, role of X-rays are very important. By diffraction technique the phase of the crystalline materials can be identified. This technique is termed as XRD test. Using this XRD test the compounds of the minerals and powdered metal can be identified. X ray having the property of diffraction when it is focused on a material also it depends on the atomic structure, crystalline phases and compounds. The deflected wave can be identified based on the quantity peak signals which can matched with standard compounds. Figure 1.2 shows an example of XRD image which shows the intensity of diffraction was reached peak position in some angles. These peak signals show the compound of the materials.

![Figure 1.2: XRD Image](image)
1.8.3 Density and Porosity Study

Characteristics of materials also depend on an important physical property which is density. Mathematically, the ratio between the mass and volume is called density. The term porosity is the property reveals the empty space inside the material especially between grains and grain boundaries. There are different methods available to find these properties.

To measure the density, the sample was taken in known shape and linear dimensions of the known sample were taken and the volume will be calculated. The mass of the sample can be found by using precision weighing machine. Using density equation now we can calculate the density of the material. By water displacement method, the sample is immersed in the water and the displaced water volume and the increased mass were used to find the density of the material.

Finding porosity need the bulk volume of the sample which was calculated by linear measurements. The sample weight measured in dry and saturated condition. The difference between the dry and saturated condition masses used to find the pore volume by dividing the differences in mass and density of water. Porosity can be then calculated by the ratio between the pore volume and bulk volume.

\[\text{Porosity } \phi = \frac{\text{Pore volume} (V_p)}{\text{Bulk volume} (V_b)}\]

\[\text{Pore Volume } V_p = \frac{\text{Weight of water in porespace}}{\text{Density of water}}\]

\[\text{Weight of water in porespace } = \text{Weight of sample } \text{wet} - \text{Weight of sample } \text{dry}\]

Bulk volume for a cylindrical sample \(V_b = \pi r^2 h\)

Where,
- \(r\) = radius of cylinder,
- \(h\) = height of cylinder

1.8.4 Hardness Study

Hardness is defined as the property of the material which resists permanent deformation. In other words it is the measure of the resistance provided against indentation and scratching. In Vikers hardness test a square based diamond pyramid was used as indenter. In that indenter the opposite faces included angle is 136°. The Vikers Hardness Number (VHN) is calculated by dividing the applied load by the
surface area of indentation. Area of the indentation was calculated from the diagonal values which were obtained by microscopic measurements.

\[ VHN = \frac{1.854P}{L^2} \]  

(1.9)

Where,
- \( L \) = Average length of diagonals

Micro hardness test uses a diamond shaped indenter with different diagonal lengths for applying load. Long diagonal have the length ratio to the shorter one was near to 7:1 and the indentation loads are very small and down to 25g. The micro hardness number is also called Knoop hardness number (KHN) which is calculated by dividing the applied load to the unrecovered area of indentation.

\[ KHN = \frac{P}{L^2C} \]  

(1.10)

Where,
- \( P \) = Applied Load
- \( L \) = Length of Long Diagonal
- \( C \) = Constant for Indenter

### 1.8.5 Tensile Property Study

Tensile test is conducted to characterize the material in the strength property as well as ductility. In tensile test attest specimen is pulled by increasing load and its deformation with respect to the load is noted. At a particular point the specimen will break which is called breaking point. Stress is the property of the material which resists deformation. Mathematically force divided by its initial cross sectional area is termed as average longitudinal stress. Strain is defined as the ratio between the change in length to the initial gauge length. By conducting tensile test some parameters were found which describes the behavior of metals which are Ultimate Tensile Strength (UTS), yield strength, percentage elongation and reduction of area. The stress value reaches its maximum point during testing before breaking is called UTS. At a particular stress value material undergoes permanent deformation is termed as yield strength. The ratio between the total elongation to the initial gauge length is termed as percentage elongation. Same way the ratio between the final cross sectional area to the initial cross sectional area of the specimen is called reduction in area. UTS and
yield strength are strength parameters and the other two are showing the ductility of the material [20]. An example of stress strain diagram as shown in figure 1.3.

![Stress-Strain Curve](image)

**Figure 1.3:** Stress-Strain Curve

### 1.8.6 Compression Property Study
Compression test is almost same compared to the tension test instead of tensile load compressive load is applied on the specimen. This test is conducted to understand about the behavior of the material when subjected to compressive load.

### 1.8.7 Scanning Electron Microscope Study
Scanning Electron Microscope (SEM) is an imaging microscope which uses focused electrons on the samples to create image. The focused electrons reveal the texture of the surface of the materials in the form of image which helps to understand about the material structure, orientation of grains and other inclusions. High resolution images can be obtained using SEM and it can be focused to nanometer scale. Energy Dispersive x-Ray Spectroscopy (EDS) instruments can be incorporated with SEM and this EDS identifies the abundance of the element.

### 1.9 SUMMARY
Magnesium and its alloys were used in different applications such as automobile parts, space vehicle components and transportation industry. Magnesium MMCs have superior properties than magnesium alloys and exhibits high specific strength. Magnesium MMCs synthesized by different methods like liquid phase, solid phase,
liquid-solid phase, deposition techniques and in-situ method. In solid phase technique, PM method provides more advantages and sintering techniques also used to improve the properties of Mg MMC. A secondary operation widely used after PM steps to obtain increased mechanical and metallurgical properties like hot extrusion. Synthesizing new MMCs required more number of experiments and involves high cost which can be reduced by applying grey based Taguchi method. By Taguchi orthogonal array method less number of experiments performed and from those results optimal parameter can be identified using grey multi performance characteristic analysis method. The properties of the developed MMCs identified by different characterization methods like tension test, compression test, density test, porosity test, hardness test and microstructure study. The increasing demand for magnesium composites with superior mechanical properties used for various applications leads to the further study in new composite development which motivates for this study.