CHAPTER-3
EXPERIMENTAL METHODOLOGY

3.1 INTRODUCTION

This chapter provides a detailed step-by-step procedure adopted for weighing and mixing of powder particles, compaction processes, determination of compaction characteristics, sintering, determination of sintering characteristics, microscopy of powder particles, green and sintered briquettes. The overall view of the experimental methodology adopted is shown in the flow chart (Fig. 3.1).

Fig. 3.1: Experimental methodology in the form of flow chart
3.2 MATERIALS USED

The materials used to prepare different metal matrix composites through powder metallurgy technique are fly-ash, aluminum and lead powder particles.

The fly-ash has been collected from Dr. Narla Tata Rao Thermal Power Station, Ibrahimpatnam, Vijayawada, Krishna District, Andhra Pradesh, India. The chemical analysis (constituents shown in Table-3.1) of the fly-ash has been carried out at M/s Natural Resource Development Cooperative Society Ltd. (NRDCS), Hyderabad, Andhra Pradesh, India.

Table- 3.1: Constituents of the fly-ash

<table>
<thead>
<tr>
<th>S.No</th>
<th>Constituents</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Loss on Ignition (LOI) %</td>
<td>0.52</td>
</tr>
<tr>
<td>2</td>
<td>Silica as SiO₂ %</td>
<td>61.75</td>
</tr>
<tr>
<td>3</td>
<td>Iron Oxide as Fe₂O₃ %</td>
<td>01.06</td>
</tr>
<tr>
<td>4</td>
<td>Aluminum as Al₂O₃ %</td>
<td>27.79</td>
</tr>
<tr>
<td>5</td>
<td>Titanium oxide as TiO₂ %</td>
<td>0.95</td>
</tr>
<tr>
<td>6</td>
<td>Manganese as MnO %</td>
<td>0.14</td>
</tr>
<tr>
<td>7</td>
<td>Calcium oxide CaO %</td>
<td>4.36</td>
</tr>
<tr>
<td>8</td>
<td>Magnesium oxide MgO%</td>
<td>0.73</td>
</tr>
<tr>
<td>9</td>
<td>Sodium oxide Na₂O %</td>
<td>0.15</td>
</tr>
<tr>
<td>10</td>
<td>Potassium oxide K₂O %</td>
<td>0.64</td>
</tr>
<tr>
<td>11</td>
<td>Phosphorus as P₂O₅ %</td>
<td>0.83</td>
</tr>
<tr>
<td>12</td>
<td>Sulphate as SO₄ %</td>
<td>0.98</td>
</tr>
</tbody>
</table>

The aluminum powder having the specifications mentioned in Table-3.2 has been supplied by M/s S.D.Fine Chem. Ltd, Mumbai-400030 and the lead powder comprising the specifications listed in Table-3.2 has been obtained from M/s Loba Chemicals, Mumbai-400002, India.
Table-3.2: Specification of the powders

<table>
<thead>
<tr>
<th>S.No</th>
<th>Powder</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Aluminum</td>
<td>Purity- 99.0%, Atomic weight-26.98, The maximum limits of impurities as specified by the supplier are Iron (Fe)0.5%, Copper (Cu) 0.05%, Manganese (Mn) 0.01%, Lead (Pb) 0.03%, and Substances insoluble in HCl-0.05%, Mesh size 200, Type of powder-LR.</td>
</tr>
<tr>
<td>2.</td>
<td>Lead</td>
<td>Purity- 99.5%, Atomic weight-207.20, Mesh size-325, Melting point-327°C</td>
</tr>
</tbody>
</table>

Scanning electron microscopy (SEM) model JEOL-JSM5410 is used to study the size, size distribution and morphology of fly-ash, aluminum and lead powders. The particle size distribution of fly-ash is also determined by the sieve analysis.

### 3.3 WEIGHING AND MIXING OF POWDERS

The following five wt% compositions of Al-15wt% Pb with 0 to 20wt% fly-ash powder mixes are prepared to study the effect of fly-ash on compacting and sintering characteristics of Al-Pb metal matrix composites.

(1) Al-15wt% Pb-0wt% fly-ash
(2) Al-15wt% Pb-5wt% fly-ash
(3) Al-15wt% Pb-10wt% fly-ash
(4) Al-15wt% Pb-15wt% fly-ash
(5) Al-15wt% Pb-20wt% fly-ash

To study the effect of Pb in Al/fly-ash metal matrix composites, the following four powder mixtures are prepared.

(1) Al-10wt% fly-ash
(2) Al-10wt% fly-ash-5wt% Pb
(3) Al-10wt% fly-ash-10wt% Pb

(4) Al-10wt% fly-ash-20wt% Pb

A batch total weight of around 200 grams powder is prepared by weighing individual powder particles. An electronic weighing machine with an accuracy of 0.001 gram least count is used for this purpose. The weighed powder is transferred into a plastic container, which in turn is placed in a powder mixing chamber after closing it with a plastic lid.

The mixing chamber is a rectangular box fabricated with mild steel sheet and a mild steel rod is welded to its one corner eccentrically. The end of the rod is placed in the chuck of the lathe machine (fig. 3.2) and is rotated at 32 rpm for duration of one hour. To ensure proper mixing of the powder particles, the direction of mixing is changed from clockwise to counter-clockwise for every five minutes. After mixing, the powder mixture is carefully transferred into a conical flask. The conical flask is closed with a rubber cork having glass tube and flexible rubber tube. The rubber tube is folded and tied with a rubber band to minimize the oxidation of the powder mix.

Fig. 3.2: Mixing processes on a lathe machine
3.4 PREPARATION OF BRIQUETTES

This section explains the experimental set up used and the compaction process employed for the preparation of specimens.

3.4.1 Experimental Setup

The briquettes of cylindrical shape are prepared using the cylindrical die (Fig.3.3). Engine valve guide and puppet valve are used as die and punch respectively for compressing the powders. To compact and confine the powder mix in the die, punch and lower punch supports are used. While ejecting the compact, the die assembly is placed in the ejecting block. The compaction process is carried on an electronic universal testing machine (UTM) of 40 ton capacity. Silicone spray is used as a die wall lubricant to reduce the sticking of powders to the surface of the die bore and also to eject the finished component from the die easily. It is sprayed over both on inner walls of the die and on the punch.

Fig. 3.3: Die, punch, supporting block and ejection block
3.4.2 Compaction Process

Initially the briquettes are prepared without die wall lubricant. This results in scored and rough surfaces due to high frictional forces between internal die wall and external surface of the briquettes. Also the green briquettes are damaged at the edges. The ejection forces are also high. Use of silicone spray as die wall lubricant has enabled smooth ejection of green briquettes and also at lower pressures. Hence, the silicone spray is used as the die wall lubricant for preparing the green briquettes in the present investigation.

The green briquettes are prepared using single action die compaction process. The die and punch are cleaned with cotton. The silicone spray is applied on the internal walls of the die and on the punch. The lower punch support is placed at the bottom of the die. The die is filled with constant volume of powder mix and the upper punch inserted into the die. The die assembly is placed on the lower platform of UTM as shown in Fig. 3.4. Then compaction is carried out by setting the UTM for the compression test and the maximum capacity loads of 12.72KN (200MPa), 19.08KN (300MPa) and 25.44KN (400MPa) are applied hydraulically. Thereafter the hydraulic pressure is released and lower platform is brought down for ejecting the compact from die. An ejection block especially fabricated for this purpose is placed between the die assembly and the lower platform of the UTM. The load is again applied by the UTM for ejecting the green compact out of the die cavity and the corresponding maximum load is noted down.
After removing the compact from the die, it is packed in the polythene packet to avert oxidation of the specimen. Three different loads are applied for each composition. At each composition, thirty briquettes are prepared by applying the same load.

### 3.5 Determination of Compacting Characteristics

The prepared green briquettes are characterized by determining ejection pressure, green density, porosity, spring back, green hardness and green strength. The length and diameter of the briquettes are measured with digital micrometer having a least count of 0.001mm and are designated as ‘d<sub>g</sub>’ and ‘l<sub>g</sub>’ respectively. The various compacting characteristics are determined by using the equations mentioned in the following sub-sections.

#### 3.5.1 Ejection Pressure

During the compaction process, the powder particles are deformed and considerable amount of stress is developed inside the green compact. As a result of this, some external pressure is required to eject the compact from the die. This pressure (so called ejection
pressure) is required to eject the compact from the die. The ejection pressure is an indication of the frictional forces between the die wall and the surface of the compact. Higher is the ejection pressure, greater are the frictional forces. The frictional forces are likely to cause damage (to the surface and edges of the compact) to the compact during ejection. Highly polished die and the use of good lubricant can reduce the ejection forces. Hence, the measurement of ejection force may help in the evaluation of die design and finding efficiency of the lubricant used. The ejection pressure is calculated from the ejection load using the following relation:

\[
P_e = \frac{F_e}{\pi d_g l_g} \text{ MPa}
\]  

(3.1)

where, \( F_e \) = Ejection load in Newtons (N)
\( d_g \) = Diameter of green compact in mm and
\( l_g \) = Length of green compact in mm

3.5.2 Green Density

It is very difficult to obtain uniform density throughout the green compact. The size and shape of the compact during the sintering processes are affected by the degree of compactness. Non-uniform green density may result in non-uniform shrinkage during sintering causing distortion of shape and deviation from the specified dimensions. Increase in the specimen height may result a greater variation in the green density whereas increase in diameter of the specimen may result in uniform distribution of density. For regular
shaped briquettes, the green density can be easily determined by measuring their weight and dimensions. The Green density is calculated by using the following relation:

\[ \text{Green Density, } \rho_g = \frac{9.81 \times 4000 \times w_g}{\pi d_g^2 l_g} \text{ KN/m}^3 \quad (3.2) \]

where, \( w_g = \) Weight of the green compact in grams

3.5.3 Porosity

Due to compaction of the powder particles, deformation and cold welding of the individual particles occur at the interface of the particles, but complete elimination of voids is extremely difficult. It is very difficult to get a powder metallurgical component without any porosity and this is advantageously used in the production of self-lubricating bearings. An account of porosity is required to completely understand the behavior of the product since it may affect the mechanical properties of the compact.

Picnometry procedure is used to determine the relative density of the fly-ash. Fly-ash is dried in an oven for 24 hours at 100°C and the relative density is calculated by using the following relation:

\[ \text{Relative Density} = \frac{0.8 \times (W_2 - W_1)}{(W_2 - W_1) - (W_3 - W_4)} \quad (3.3) \]

where, \( W_1 = \) Mass of empty picnometer

\( W_2 = \) Mass of picnometer + fly-ash

\( W_3 = \) Mass of Picnometer + fly-ash + Kerosene

\( W_4 = \) Mass of picnometer + Kerosene
The theoretical densities for different compositions are calculated using rule of mixture from the individual densities of the powders. The use of rule of mixture is universally used for calculating the densities of various composites/alloys. Hence the same is adopted in the present investigation.

The volume percentage of the individual element is calculated by using the following equation.

\[
\% \text{ Volume of } x = \left( \frac{W_x}{\rho_{x}} \right) \times 100
\]  

where \( W = \text{weight\%} \), \( \rho = \text{density} \) and \( x = \text{Fly-ash or Pb} \).

The theoretical density of the composite is calculated by using the following equation.

\[
\text{Theoretical density} = \frac{\left(100 - V_{\text{Fly-ash}} - V_{\text{Pb}}\right)\rho_{\text{Al}} + V_{\text{Fly-ash}} \rho_{\text{Fly-ash}} + V_{\text{Pb}} \rho_{\text{Pb}}}{100}
\]  

Where \( V_{\text{Fly-ash}} = \text{Volume \% of fly-ash} \)

\( V_{\text{Pb}} = \text{Volume \% of Pb} \)

\( \rho_{\text{Al}} = \text{Density of aluminum (2.7 grams/cm}^3\text{)} \)

\( \rho_{\text{Fly-ash}} = \text{Density of fly-ash (1.973 grams/cm}^3\text{)} \)

\( \rho_{\text{Pb}} = \text{Density of lead (11.34 grams/cm}^3\text{)} \)

The sample calculation of the theoretical density is given in appendix-I and the theoretical densities of different composites are tabulated in Table-3.3.
Table- 3.3: Theoretical densities of the composites

<table>
<thead>
<tr>
<th>Composition</th>
<th>Volume % of fly-ash</th>
<th>Volume % of Pb</th>
<th>Theoretical density g/cc</th>
<th>Theoretical density KN/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-15% Pb-0% fly-ash</td>
<td>0.0</td>
<td>4.03</td>
<td>3.04</td>
<td>29.89</td>
</tr>
<tr>
<td>Al-15% Pb-5% fly-ash</td>
<td>7.56</td>
<td>3.95</td>
<td>2.98</td>
<td>29.28</td>
</tr>
<tr>
<td>Al-15% Pb-10% fly-ash</td>
<td>14.83</td>
<td>3.87</td>
<td>2.92</td>
<td>28.70</td>
</tr>
<tr>
<td>Al-15% Pb-15% fly-ash</td>
<td>21.81</td>
<td>3.79</td>
<td>2.86</td>
<td>28.13</td>
</tr>
<tr>
<td>Al-15% Pb-20% fly-ash</td>
<td>28.52</td>
<td>3.72</td>
<td>2.81</td>
<td>27.59</td>
</tr>
<tr>
<td>Al-10% fly-ash-0% Pb</td>
<td>13.19</td>
<td>0.0</td>
<td>2.60</td>
<td>25.53</td>
</tr>
<tr>
<td>Al-10% fly-ash-5% Pb</td>
<td>13.70</td>
<td>1.19</td>
<td>2.70</td>
<td>26.51</td>
</tr>
<tr>
<td>Al-10% fly-ash-10% Pb</td>
<td>14.24</td>
<td>2.47</td>
<td>2.81</td>
<td>27.56</td>
</tr>
<tr>
<td>Al-10% fly-ash-20% Pb</td>
<td>15.47</td>
<td>5.38</td>
<td>3.05</td>
<td>29.93</td>
</tr>
</tbody>
</table>

The percentage porosity is calculated from the following equation:

\[
\% \text{ True Porosity} = \left(1 - \frac{\rho_{\text{th}}}{\rho_g}\right) \times 100
\]

where, \(\rho_{\text{th}}\) = Theoretical density of the briquettes

The true density for pure aluminum = 26.458KN/m³

True density of Fly-ash = 19.355KN/m³

True density of lead = 111.245KN/m³

### 3.5.4 Spring Back

During the compaction process the plastic deformation of the powder particles occur because of the external pressure. The plastic deformation may cause work hardening and an increase in the elastic limit. After the compact is ejected from the die, these elastic forces may act on the compact and may have tendency to expand the green compact considerably. This phenomenon is known as spring back. During the ejection process the elastic recovery of the residual stresses may occur when the compact left the die. When the residual
stress exceeds the green strength of the compact, cracking may occur in the compact. Hence, it is necessary to determine the extent of the spring back for manufacturing the components with close dimensional tolerances.

The diameter of the die bore is measured with inside micrometer having the least count of 0.01mm and the % spring back (diametrical) is calculated from the following equation:

\[
\text{% Spring Back} = \frac{d_s - d_d}{d_d} \times 100
\]  

(3.7)

where, \(d_d\) = the diameter of the die bore in mm (9.00 mm).

### 3.5.5 Green Hardness

The hardness of the green compact is usually affected by its compaction pressure, density, grain size and shape. Since single action compaction process is employed, the briquettes are tested for hardness only on the top surface for uniformity. TH130 integrated hardness tester shown in Fig. 3.5 is used to measure the green hardness of the briquettes as per ASTM A 956-02. The instrument is distinguished by its compact size, low load application, high accuracy, wide measuring range, quick and simple to operate. It automatically computes the hardness values and the statistical mean value. It is set for the normal direction (+90°), for required hardness scale HB (Brinell hardness) and for the required material (aluminum alloy).
3.5.6 Green Strength

The green strength of the compact primarily depends on the compaction pressure and it arises mainly from cold welding, mechanical interlocking of the neighboring particles and particle shearing. Particle size, shape and structure may also affect the green strength. In the present study, the compression test is used to determine the green strength of the briquettes as per ASTM E9-89a.

A 2000kg capacity electronic tensometer model PC 2000 supplied by M/s Kudale Instruments Private Limited- Pune, shown in the Fig. 3.6, is used to find the compressive strength of the briquettes. The test speed is chosen as 0.2mm/minute. The electronic tensometer is fitted with load and extension indicators having the least count of 10N and 0.01mm respectively. The compression attachment is fixed to the tensometer and the specimen is held between the flat grips of the compression attachment as shown in Fig. 3.7. The rack is then engaged with the wheel. The load and extension are adjusted to read
zero by pressing the tare buttons. The test is started by pressing the test button. The maximum crushing load $F_c$ at which the specimen crushed is noted down. The tests are conducted at room temperature. Three identical specimens are tested for each compaction pressure. The green compressive strength is calculated from the equation:

$$\text{Green Strength} = \frac{4 \times F_c}{\pi d_g^2} \text{ MPa} \quad (3.8)$$

where, $F_c =$ Crushing load in Newton

Fig. 3.6: Electronic tensometer model PC2000

Fig. 3.7: Compression attachment on tensometer
3.6 SINTERING OF GREEN BRIQUETTES

The green briquettes prepared are very brittle and cannot be used as such. Hence these green briquettes are sintered in order to develop sufficient strength and density. In a multi component powder system, sintering is usually carried out at a temperature ranging from 0.7 to 0.9 times the absolute melting point of the base metal. During sintering there may be possibility of numerous processes like removal of absorbed gases, liquids, solids; recrystallization and grain growth, movement of atoms by diffusion and change in density. The briquettes obtained are sintered at four different temperatures 500, 530, 560 and 590°C in an argon gas atmosphere in order to study the effect of sintering temperature on sintering characteristics. As the effect of temperature is more predominant than the effect of time, the sintering time is fixed as 45 minutes.

3.6.1 Sintering Furnace

An electric resistance-horizontal tubular furnace having openings at both ends is used for sintering the briquettes. The furnace as shown in Fig. 3.8 is fitted with a digital indicator cum controller with an accuracy of ±1°C. The maximum temperature of the furnace is 1000°C.

3.6.2 Sintering Atmosphere

The sintering furnace atmosphere may depend on the type of material and the properties required in the sintered product. The commonly used sintering atmospheres are either the reducing or the neutral
atmosphere. Among the inert gases, argon and helium are widely used for sintering reactive metals. In the present work, the components are sintered in an argon gas for the neutral atmosphere for duration of 45 minutes.

3.6.3 Sintering Procedure

A transparent quartz tube with 13mm and 16mm of internal and outer diameters in that order is used for sintering the briquettes. The ends of the quartz tube are fitted with cork and a glass tube. A flexible tube is connected to each of the glass tubes. One tube is connected to the argon gas cylinder and another tube connected to the conical flask containing water. Five briquettes separated by thin asbestos discs are placed in the quartz tube. The tube is inserted into the furnace such that the briquettes are located exactly at the center of the furnace. The gas flow is adjusted for about 30 bubbles per minute. The sintering is carried out at four constant temperatures of 500, 530, 560 and 590°C for 45 minutes. After 45 minutes of sintering, the power is switched-
off and the quartz tube is slide so that the briquettes are brought completely outside the furnace, keeping the gas flow inlet connection intact. The briquettes are allowed to cool keeping the rate of gas flow same during the cooling of the briquettes. After cooling, the gas flow is stopped and the sintered briquettes are taken out and packed in the polythene cover.

3.7 DETERMINATION OF SINTERING CHARACTERISTICS

The sintered briquettes obtained as per the procedure mentioned in sub-section 3.6.3 are weighed using the electronic weighing machine having least count of 0.001 grams. The length $l_s$ and diameter $d_s$ of the sintered briquettes are measured with digital micrometer having least count of 0.001 mm. The sintering characteristics viz., sintered density, sintered porosity, sintered hardness and sintered strength are determined.

3.7.1 Determination of Sintered Density

The sintered density may give information regarding porosity which may affect indirectly the mechanical properties like strength and hardness. The sintered density is determined by measuring the weight and dimensions of the component and is calculated using the following relation:

$$\text{Sintered Density, } \rho_s = \frac{9.81 \times 4000 \times w_s}{\pi d_s^2 l_s} \text{ KN/m}^3 \quad (3.9)$$

where, $w_s = \text{Weight of sintered compact in grams}$
3.7.2 Determination of Sintered Porosity

For the powder metallurgical products it is inevitable to avoid porosity and therefore, the theoretical density can not be obtained. The porosity present in these components may act as stress risers and may influence the elongation, impact and fatigue strength. Hence, the knowledge on the sintered porosity is vital to determine the mechanical behavior of the powder metallurgical components. For self-lubricating bearings, the amount of porosity is required to assess their performance. The sintered porosity is obtained from the following equation:

\[ \% \text{Sintered Porosity} = \frac{\rho_{\text{th}} - \rho_s}{\rho_{\text{th}}} \times 100 \]  

(3.10)

where, \( \rho_{\text{th}} \) = the theoretical density of the briquettes

3.7.3 Determination of Sintered Hardness

The sintered hardness may influence the wear and abrasion resistance of the briquettes. The sintered hardness of the compact is tested using TH130 Integrated Hardness Tester in the same way as that used for measuring the green hardness.

3.7.4 Determination of Sintered Strength

The sintered strength is the ultimate quality control characteristic for the final powder metallurgical product. In the present study, the compression test is used to determine the sintered strength. A 2000kg capacity electronic tensometer PC 2000 model supplied by M/s Kudale Instruments Private Limited- Pune, shown in the Fig. 3.6 is
used to determine the compressive strength of the briquettes. The maximum reading of crushing load and load required for 20% deformation is noted down. Three identical specimens are tested for each compaction pressure and temperature. The sintered compressive strength is calculated from the following equation:

\[
\text{Sintered Strength} = \frac{4 \times F_c}{\pi d_s^2} \text{ MPa.} \quad (3.11)
\]

where, \( F_c \) is the crushing load or load required for 20% deformation in Newtons and 
\( d_s \) is the diameter of the sintered compact in mm.

### 3.8 WEAR TEST OF BUSH

To test the material used in the present investigation for its practical application, a bush is manufactured from Al-Pb/0, 5, 10wt% fly-ash composite compacted at 400MPa and sintered at 560°C. The weight loss due to wear of the bush is tested experimentally (Fig.3.9). The bush is kept in the cast iron holder and the holder is rotated at 890rpm for duration of 5 hours. After completion of the test, the weight of the bush is measured and the weight loss is calculated by using the equation:

\[
\% \text{ Weight loss} = \frac{W_f - W_i}{W_i} \times 100 \quad (3.12)
\]

where, \( W_i \) is the initial weight, grams 
\( W_f \) is the final weight, grams
Fig. 3.9: Experimental set up for testing bush wear