CHAPTER 4
EXPERIMENTAL INVESTIGATIONS

4.1 MATERIALS

Commercially available standard cast Al alloy LM 13 (shown in Fig. 4.1) was used as the matrix material for the research work. LM13. Its chemical and physical composition is as given in the Table 4.1 and 4.2 respectively. The specific alloy was chosen because the material has attracted increasing interest in automotive applications, especially in heavy wear components such as piston, cylinder blocks.

4.1.1 Reinforcement

In this research work, the reinforcement material chosen was nano-sized zirconium oxide (ZrO\textsubscript{2}) in particulates form as shown in the Fig 4.2. The mesh size of ZrO\textsubscript{2} ranges from 70 to 100nm. Its chemical and physical properties are as shown in the Table 4.3 and Table 4.4 respectively.

4.2 COMPOSITE PREPARATION (NMMCs)

The method employed for development of nanocomposites involved two processes. The primary processing consists of synthesis of nano-sized ZrO\textsubscript{2} reinforced aluminum alloy based nanocomposites (NMMCs) containing five different weight percentage of ZrO\textsubscript{2} developed via conventional stir cast route (vortex). The secondary process involved, the deposited developed NMMCs were hot extruded in a hydraulic press at 250 °C (thickness
of the samples was reduced from 25mm to 23mm in the hydraulic press).

4.2.1 Primary process (synthesis of nanocomposites)

The synthesis of aluminium alloy based nano-metal matrix composite (NMMCs) were developed through vortex route. Cleaned metal ingots (matrix alloy-LM 13) were melted to the desired temperature up to 650 °C in graphite crucibles under the coverage of flux in order to minimise the oxidation of molten metal; 3-phase electrical resistance furnace with mechanical stirring attachment was used for melting the matrix alloy as shown in the Fig 4.3 & 4.4. The electrical resistance furnace temperature was pre-set to 650 °C and was switched on; once the desired temperature was reached, the molten metal was degassed using hexachloroethane tablets. ZrO₂ particulates of mesh size 70 to 100nm was preheated separately in an oven up to 600 °C and it was transferred to the molten alloy using graphite spoon and stirred continuously with mechanical stirrer.

The stirring time was maintained between 1 to 2 minutes at an impeller speed of 400rpm, to create vortex in order to distribute the reinforcement particulates more uniformly in the molten alloy. After the complete injection of the reinforcement particles, the molten metal was once again stirred for few seconds just before it was poured into the dry sand mould. The matrix-reinforcement mixture after being poured into dry sand mould was allowed to solidify from one end of silicon carbide chill which was pre-set in
the mould as shown in the Fig. 4.5 and 4.6. The amount of ZrO$_2$ incorporated in the matrix alloy was varied from 3 to 15 Wt.% in the step of 3%. The moulds is shown in the Fig. 4.6 to produced plate type of casting (225 X 150 X 25mm (AFS standard)) which was prepared using silica sand with 5% Bentonite as binder and 5% moisture and dried in the warm air. The as cast matrix alloy and nanocomposites thus obtained are as shown in Fig.4.7.

**Table 4.1 Chemical composition of aluminium silicon alloy (LM 13).**

<table>
<thead>
<tr>
<th>Elements</th>
<th>Zn</th>
<th>Mg</th>
<th>Si</th>
<th>Ni</th>
<th>Fe</th>
<th>Mn</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt.%</td>
<td>0.5</td>
<td>1.4</td>
<td>12</td>
<td>1.5</td>
<td>1.0</td>
<td>0.5</td>
<td>Bal</td>
</tr>
</tbody>
</table>

**Table 4.2 Physical properties of aluminium silicon alloy (LM 13).**

<table>
<thead>
<tr>
<th>Material</th>
<th>Coefficient of thermal expansion (per °C at 20 – 100°C)</th>
<th>Thermal conductivity (w/mK at 25°C)</th>
<th>Electrical conductivity (% copper std at 20 °C)</th>
<th>Density (g/cm$^3$)</th>
<th>Sp. heat (J/kgK at 100 °C approx.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LM 13</td>
<td>0.000019</td>
<td>154.91</td>
<td>29</td>
<td>2.7</td>
<td>963</td>
</tr>
</tbody>
</table>
Table 4.3 Chemical composition of ZrO₂.

<table>
<thead>
<tr>
<th>Elements</th>
<th>SiO₂</th>
<th>CaO</th>
<th>MgO</th>
<th>Fe₂O₃</th>
<th>Al₂O₃</th>
<th>TiO₃</th>
<th>ZrO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt.%</td>
<td>0.1</td>
<td>0.2</td>
<td>3.3</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>Bal</td>
</tr>
</tbody>
</table>

Table 4.4 Physical properties of ZrO₂.

<table>
<thead>
<tr>
<th>Elements</th>
<th>ZrO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coefficient of thermal expansion (per °C) x 10⁻⁶</td>
<td>11</td>
</tr>
<tr>
<td>Thermal conductivity (W/mK)</td>
<td>1.7 – 2.7</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>5 – 6.15</td>
</tr>
<tr>
<td>Sp.Heat (J/kgK at 100 °C approx)</td>
<td>420 – 540</td>
</tr>
<tr>
<td>Melting point °C</td>
<td>1860</td>
</tr>
<tr>
<td>UTS Mpa</td>
<td>425</td>
</tr>
<tr>
<td>VHN</td>
<td>150</td>
</tr>
<tr>
<td>Young’s Modulus Gpa</td>
<td>98</td>
</tr>
</tbody>
</table>

Fig: 4.1 Ingots of matrix alloy (LM 13).

Fig: 4.2 Nano-sized ZrO₂ particulates (70 to 100nm).
Fig: 4.3 Stir-casting experimental set-up.

Fig: 4.4 Uniform stirring of matrix alloy with reinforcement.

Fig: 4.5 Schematic diagram of mould with end chill to produce plate type casting (AFS standard).

Fig: 4.6 Mould box with wooden pattern and silicon carbide as end chill.

Fig: 4.7 As cast matrix alloy and NMMCs containing various wt.% of ZrO₂.
4.2.2 Secondary Process

In the secondary process, the developed NMMCs and aluminium alloy were heated uniformly up to 250 °C in furnace followed by hot extrusion. The samples were placed on the platform of hydraulic press having 150 ton capacity and were pressed uniformly by applying pressure till its thickness was reduced from 25mm to 23mm. The hydraulic press and the extrusion process are as shown in the Fig 4.8.

![Hydraulic press of 150 ton capacity](image)

Fig: 4.8 Hydraulic press of 150 ton capacity.

4.3 HEAT TREATMENT PROCESS (AGEING)

The mechanical properties of as cast aluminum composites are inferior due to presence of residual stresses and porosity incurred during solidification process. In order to obtain optimal mechanical properties of the developed nanocomposites and microstructural uniformity various heat treatments are applied to the cast composites. The developed NMMCs and matrix alloy were
subjected to heat treatment (ageing) process carried out in heat treatment furnace after the hot extrusion process.

The samples were placed inside the heat treatment furnace in batch of two and the furnace temperature was pre-set to 360 °C ± 5 °C. On reaching the pre-set temperature, the samples were soaked for period of four hours followed by ambient cooling. The heat treatment process is as shown in the Fig 4.9. The primary aim of this heat treatment (ageing) process was to relieve residual stresses developed during solidification and extrusion process.

![Fig: 4.9 Heat treatment (aging process).](image)

### 4.4 METALLOGRAPHIC SAMPLE PREPARATION

Samples were cut from the chill end of the final cast components for microstructural characterization. Samples are prepared as per standard metallographic procedures as explained below:

- Polishing
- Etching
- Examination
Polishing

- Plane surface was obtained by rubbing on a medium mill file.
- The specimens were rubbed successively on No. O/1, O/2, O/3, and O/4 metallographic emery papers. Polishing operation was performed using velvet cloth wrapped on the disc rotating at about 300 rpm using water suspension of aluminium oxide as abrasive.
- The final wet polishing operation was performed on a disc rotating at 150 to 200 rpm covered with ‘kitten’s ear’ broadcloth with 0.25 µm diamond paste, after polishing was completed the specimens were washed in lukewarm water and dried.

Etching

The keller’s reagent was prepared by mixing 5ml of concentrated HNO₃, 3ml HCl and 2ml of HF acid in and 95 ml of distilled water. Each sample was soaked in the keller’s reagent for about 30 seconds. The samples were washed with ethanol and dried.

Examination

- The etching samples were placed on the platform of the microscope and the microstructures of the specimen were observed under desired magnification and final images were captured.
4.4.1 Optical microscopy

NIKON-Metallurgical microscope LV150-Japan make with clemax image analyzer as shown in Fig 4.10 was used for the OM observation. Micrographs were captured using computer interface equipped with high resolution CCD camera in presence of bright field mode.

![Metallurgical optical microscope equipped with image analyzer and camera.]

Fig: 4.10 Metallurgical optical microscope equipped with image analyzer and camera.

4.5 EVALUATION OF HARDNESS

The hardness can be defined as resistance offered by virtue of material against plastic deformation, usually measured by indentation. It can be related or referred to resistance against scratch, abrasion and cutting. However, it can also be said as the internal property of a material which offers resistance to permanent
deformation like change in shape, bent etc., against applied load. Higher the hardness of the material greater force is required to cause deformation and for simplicity in metallurgy, hardness is the resisting force offered by material against indentation. Usually hardness is evaluated by pressing a pointed or round shaped indenter on a surface under static load and its hardness is expressed in terms of the load per unit area of indentation. In this research work, the hardness of developed NMMCs and matrix alloy were evaluated at both macro and micro levels as explained below:

4.5.1 Macrohardness

The macrohardness of the developed NMMCs and the matrix alloy were evaluated by using Brinell hardness testing machine as shown in the Fig 4.11. The test was conducted according to AFS standards, by pressing a hardened steel ball of 10mm diameter on the polished surface of the specimen by applying a load of 1000 kg maintained for duration of 15 seconds. The values reported in the Table 5.1 are the average of three readings taken at different locations on the surface of the specimen. The equation as mentioned below was used to evaluate the macrohardness of the samples in terms of BHN. The test samples are shown in the fig 4.13.

\[
BHN = \frac{2F}{\pi \times D[D - (D^2 - d^2)^{1/2}]} \quad \text{-------- 4.1}
\]
Where, \( F \) = Force applied in N.

\( D \) = Indentor diameter in mm.

\( d \) = Indentation diameter in mm.

**4.5.2 Microhardness**

The micro-hardness of developed NMMCs and the matrix alloy were evaluated on the polished specimens by using digital Vickers hardness tester as shown in the Fig 4.12 in accordance with ASTM E18-94 standards. Each sample was placed on the platform of the testing machine and a load was applied in order of 25gf indentation for a dwell time of 15 sec. The microhardness of all the test samples was recorded in terms of HV and tabulated as shown in the Table 5.1. The test samples are also shown in the Fig 4.14.

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**Fig: 4.11** Brinell hardness test Equipment.

**Fig: 4.12** Vickers hardness testing equipment.
4.6 TENSILE TEST

The tensile property evaluation of developed NMMCs and matrix alloy was performed on AFS standard in accordance with IS 1608:2005/ISO 6892:1998 standards at ambient temperature conditions by using universal material testing machine (Instron-8516) as shown in the Fig 4.16. The dimensions of test specimen are given in the Table 4.5 & Fig 4.15 respectively. The Instron-8516 universal tensile testing machine was connected to a computer for recording and calculating data such as yield stress, ultimate tensile strength, percentage elongation and young’s modulus during the test. In the course of testing, steady rate of tensile loading on the test specimen was maintained with cross-head movement set at the rate of 0.254mm/min. The yield strength was measured at 0.2% offset of stress-strain curve. All the broken specimens are as shown in the Fig 4.17.
Table 4.5 Dimensions of the tensile test specimen.

<table>
<thead>
<tr>
<th>SL.NO.</th>
<th>Diameter (d) (mm) with tolerance</th>
<th>Gauge length mm ( \text{Lo}=k\sqrt{\text{So}} )</th>
<th>Min. Parallel length (mm)</th>
<th>Grip dia (mm) ‘D’</th>
<th>Grip length (mm)</th>
<th>Radius R</th>
<th>Total length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20 ±0.15</td>
<td>100 ±1</td>
<td>110</td>
<td>25</td>
<td>55</td>
<td>15R</td>
<td>250</td>
</tr>
</tbody>
</table>

Fig: 4.15 Drawing of tensile test specimen.

Fig: 4.16 Standard Instron (8516) universal testing machine with specimen on load
4.7 FRACTURE TOUGHNESS TEST PROCEDURE

Fracture toughness test was performed on the test samples of developed NMMCs and matrix alloy in accordance with ASTM E399-1990 standards using a closed loop Instron servo-hydraulic material testing system as shown in the Fig 4.20. The test samples were subjected to 3-point bending of machined specimen which was pre-cracked by fatigue. Among the various kinds of available specimen configurations, compact test specimen (CT) was selected due to simplicity in its fabrication, requires less material and does not demand any kind of special fixtures during mounting in the jaws of testing machine.

The CT specimen prepared and its dimensions are as shown in the Fig 4.18 and Fig 4.19 respectively. A straight through notch of length ‘\(a_0=10\)mm’ is introduced by CNC-operated wire cutting machine. Thereafter, at the notch vicinity, plastic zone is generated through fatigue loading by using dynamic testing machine.
**Specimen specifications**

- Total width = 31.75mm
- Load width, W = 25.40mm
- Thickness, B = 12.67mm
- Initial Crack, \( a_0 \) = 10mm
- Fatigue Pre-crack, \( a_p \) = 2.99mm
- Total crack, \( a \) = 12.99mm

A maximum load of 2KN and minimum load of 0.6KN was maintained throughout the test during fatigue loading. The process of introducing a crack with a plastic zone at the vicinity of the notch with the influence of fatigue load is called fatigue pre-cracking. In pre-cracking, a crack of length \( a_p = 2.99 \text{mm} \) is introduced. For the ratio \( a/W = 0.511 \), \( f(a/W) \) is noted down as 9.96 for further validations. The fractured test samples are as shown in the Fig. 4.21.

- For the evaluating the fracture toughness parameters, it is important that fracture toughness test satisfies three important criteria: Firstly, the specimen geometry by which \( K_{IC} \) can be evaluated with significant accuracy. Secondly, at onset of cracking, load value and crack length must be accurately measured and finally, pre-cracking should ensure that the crack introduced is a shape one.
Fig: 4.18 Fracture toughness test Specimens.

Fig: 4.19 Dimension of CT Specimen.

Fig: 4.20 Instron servo-hydraulic material testing machine with specimen on load.
4.8 DRY SLIDING WEAR TEST

The Dry sliding adhesive wear characterization was evaluated on the specimens of developed NMMCs and matrix alloy by using standard computerized pin on disc (POD) friction and wear monitoring test rig as shown in the Fig 4.24. The schematic representation of dry sliding wear is also shown in the Fig 4.22 and its technical specifications are given in the Table 4.6. It consists of a driving spindle which can provide multiple spindle speeds that inturn revolve the disc. A lever arm with a pivot arrangement provides a stationary arrangement at one end of it for holding the specimen-pin on the wear track; and also provides force propositional to the applied load on the wear track. The other end of it has a carrier for mounting load.

The machine automatically monitors the number of pre-selected disc revolutions and shuts off the machine automatically, once pre-selected numbers of revolutions are completed. A weight
added on the hanger generates proportional force exerted by specimen-pin on the circular wear track and the wear track is a circle which allows multiple passes on the same track. This system is equipped with frictional force monitoring load cell with computer interface which records the coefficient of friction of the sample during the test.

**Table 4.6 Technical specifications of the (POD) wear testing machine.**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pin dimensions</td>
<td>3 to 12 mm diameter and 10 to 50 mm length</td>
</tr>
<tr>
<td>Disc dimensions</td>
<td>160mm diameter and 8 to 12 mm thickness</td>
</tr>
<tr>
<td>Wear track radius</td>
<td>8mm to 125 mm diameter</td>
</tr>
<tr>
<td>Disc rotating speeds</td>
<td>100 to 1000 rpm</td>
</tr>
<tr>
<td>Sliding speed range</td>
<td>6 m/s to 26 m/s</td>
</tr>
<tr>
<td>Normal load</td>
<td>200 N (maximum)</td>
</tr>
<tr>
<td>Frictional force</td>
<td>0 to 200 N (digital readout)</td>
</tr>
<tr>
<td>Wear height loss measurement</td>
<td>LVDT of 1.0 µm Least Count</td>
</tr>
<tr>
<td>Input power</td>
<td>230 V, 5 A, I Phase, 50 Hz</td>
</tr>
</tbody>
</table>

**4.8.1 Test Procedure**

The wear tests specimens prepared from the developed NMMCs and matrix alloy were of dimensions $\varnothing 6$ mm X 40 mm length as shown in Fig 4.23. The dry sliding adhesive wear test was conducted in accordance with ASTM-G99. The disc chosen for carrying out wear test is made of high carbon EN31 steel having a hardness of HRC 60. Before the test, samples were cleaned with spirit and known quantity of mass was added to the load hanger (say 10 N). The motor was started and the desired rotational speed
of 600rpm is selected; once the desired speed is indicated, the lever arm with specimen-pin attachment is brought in contact with the disc (wear track radius (90mm) pre-selected). The test was carried out for 12 minutes of duration and after the test, then specimen is removed and wear debris present on the disc were cleaned. Same procedure is repeated for varying loads of 30N, 40N and 50 N.

During the course of testing, a computer interfaced with the machine records wear and friction coefficient data with least count of 1.0 µm. As the wear takes place from the specimen-pin surface it pushes down on the wear track to establish contact, this downward linear movement of pin-surface with the counterface gives the wear height loss of the pin and is being recorded by the LVDT attached to the wear monitoring test-rig. This wear height loss of the pin measured during the test was used to evaluate the volumetric wear loss. From the POD experiment the height loss experienced by the pin is obtained in microns.

- Volumetric loss in mm³ = Area of the Pin × Height loss.
- Wear rate in mm³/N-m = Volumetric Loss/(Sliding Distance × Load).
- Following graphs were plotted to study the dry sliding wear behaviour of the developed NMMCs and matrix alloy:
  - Specific wear rate Vs Load.
  - Specific wear rate Vs Sliding distance.
  - Coefficient of friction Vs Sliding distance.
Fig: 4.22 Schematic diagram of dry sliding wear test.

Fig: 4.23 Wear test Specimens.

Fig: 4.24 Ducon-make computerized pin-on-disc friction and wear monitoring test-rig.
4.9 EVALUATION OF THERMAL PROPERTIES

In present research work associated with investigations on mechanical properties and wear characterization of developed NMMCs and matrix alloy, an attempt was also made to evaluate the thermal properties via thermal conductivity of the developed NMMCs and matrix alloy in order to extend its applications towards thermal capabilities. The thermal conductivity of these materials was evaluated by applying guarded comparative longitudinal heat flow technique in accordance with ASTM E1225-04 standards involving measurement of thermal conductivity of solids. An experimental set-up was established in Heat and Mass Transfer Lab.

- **Experimental procedure**

  Evaluation of thermal conductivity of developed NMMCs and matrix alloy were determined by establishing an experimental setup as per the schematic diagram as shown in the Fig 4.25 & 4.26. The components includes thermocouples (6 NO's), heater, heat-sink with cooling fan, digital voltmeter, dimmerstat, digital temperature indicator and insulating materials as shown in the Fig (4.33 to 4.38). The experimental set up as shown in Fig 4.30 consists of placing the test specimen in between two pure aluminum bars of standard dimensions. The test specimens includes matrix alloy (LM 13) and developed NMMCs that are fabricated using liquid metallurgy route as explained in section 4.2.1 and their related photographs are as shown in the Fig 4.27 to 4.29. All the test
specimens were prepared to standard dimensions measuring 50 mm diameter and 50mm height (cylindrical shape).

On the cylindrical surface of all the specimens, a cylindrical hole of diameter 5mm was drilled at distance of 7mm from the top end and 7mm from the bottom end for inserting thermocouples. Initially the matrix alloy bar was centrally placed with pure Al bar at the top and bottom sides as shown in the Fig 4.38. Six thermocouples were then inserted into the previously drilled holes as shown in the Fig 4.31. A thin layer of Si-grease was applied in between each bar to decrease contact resistance followed by the tightly wrapping asbestos tape (primary insulating material) around the three bars. The top bar represents the heat source with heater tape being tightly wrapped as shown in the Fig 4.31. Care was taken to wrap the heater tape with asbestos tape & with plaster of paris to prevent heat losses diametrically as shown in the Fig 4.32. Finally, another round of asbestos thread was wrapped tightly over the asbestos tape for better insulation (secondary insulation) as shown in the Fig 4.33.

Some dead weight was also placed on top of heating bar to establish close contacts between each other. A heat sink was placed at the bottom of setup to ensure one dimensional heat flow from top to bottom. A 12 volts fan was used to circulate air through the fins of heat sink. All electrical connections were made as per the circuit diagram and the power source was switched on and a constant voltage was maintained through the dimmerstat. Temperature
readings were recorded at regular intervals of time at steady state conditions. The above procedure was repeated by replacing the central matrix Al-alloy bar with the developed NMMCs containing varying wt.% of nano-ZrO₂ particulates (varied from 3wt.% to 15% insteps of 3%) and corresponding temperature readings were recorded. The thermal conductivity of the developed NMMCs and matrix Al-alloy bar were evaluated using Fourier relations as given below:

\[ Q = k A \Delta t/\Delta x \] ------ 4.2

Where,
\( Q \) = Rate of heat transferred in Watts.
\( k \) = Thermal conductivity of the material in W/mK.
\( A \) = Cross sectional area across which heat is transferred in m²
\( \Delta t \) = Difference in temperature along the direction of heat flow in K.
\( \Delta x \) = The length between the 2 points where temperature is measured in m.

Fig: 4.25 Schematic diagram of Guarded-Comparative Longitudinal Heat Flow technique (ASTM- E1225-04 standard).

Fig: 4.26 The Circuit Diagram for the experimental setup.
Fig: 4.27 Dry sand mould (Ø 60mm X 60 mm).

Fig: 4.28 Casting of specimen (for thermal conductivity).

Fig: 4.29 Cast specimen (for thermal property).

Fig: 4.30 Experimental set up (guarded comparative heat flow technique).

Fig: 4.31 The experimental setup with heater on top.

Fig: 4.32 Fist level of insulation.
Fig 4.33 Setup with complete insulations.

Fig 4.34 The Cooling fan.

Fig 4.35 The temperature indicator.

Fig 4.36 The Voltmeter indicator.

Fig 4.37 The Heat sink.

Fig 4.38 The Dimmerstat.