Chapter 3

EXPERIMENTAL WORK

The main aim of present research work is to improve the properties of commercially pure aluminium (CPA) by varying different parameters like amount of particulate reinforcement and its sequence of addition. Selected reinforcement is MnO_2 because it is stable up to the temperature at around 500 ^oC, above which decomposition take place [134] at 535 ^oC and release pure manganese and oxygen. Optimization of Mg was also checked for commercially pure aluminium because Mg acts as wetting agent for generated in-situ phases. The process parameters are as reported in table 3.1. To check the effects of additions, detail analysis has been carried out in three different phases of experiments:

1. **Phase I:** Optimization of magnesium content into CPA
2. **Phase II:** Effect of variation of MnO_2 content by changing its addition sequence into CPA and
3. **Phase III:** Effect of variation of MnO_2 by changing its addition sequence along with optimised magnesium metal from phase I into CPA.

In phase I experiments, the amount of Mg was optimised through various trials. The amount of magnesium was varied from 0.05 to 7 wt % by keeping all other parameters constant. In phase II, amount of reinforcing phase (MnO_2) was optimised without addition of Mg as wetting agent. Phase II was conducted in two different approaches. In first approach (sequence A), MnO_2 reinforcing phase was added after melting of aluminium as conventionally practiced. In second approach (sequence B), reinforcing phase MnO_2, was added before the melting of the aluminium, i.e. MnO_2 powder was mixed with the charging materials with solid CPA. During sequence B, due to gradual heating of MnO_2 particles and more time involvement in various reactions, it increased the recovery Mn from MnO_2 as indicated in chapter 04.

In phase III, combined effects of reinforcing phase MnO_2 along with wetting agent Mg were studied. In this phase amount of Mg was kept fixed as 3 wt.% which was optimised from phase I results while amount of MnO_2 was varied like 1, 2.5 and 4 wt.% for both sequences of addition. Addition of MnO_2 reinforcing phase was kept limited upto 4 wt.% because beyond this limit, it was observed that the liquid CPA rejected the excess powder of MnO_2. These experiments were carried out in two different approaches as indicted in phase II. In first approach (sequence A), MnO_2 added after melting of commercially pure aluminium whereas in second approach (sequence B), reinforcing phase was added before the melting of CPA. The results of both approaches were analysed in the next chapter. Formations of in-situ phases were confirmed which are improving various properties.

Table 3.1: Process Parameters

Sr. No.	Process Parameter	Process Variables		
		Phase I	*Phase II*	*Phase III*
1	Amount of Commercially Pure Al	700 gm	700 gm	700 gm
2	Processing temperature	720 ^oC	720 ^oC	720 ^oC
3	Time of processing	15 min	15 min	15 min
4	Stirrer speed	100 RPM	100 RPM	100 RPM
5	Amount of Magnesium addition (wt %)*	0.05, 0.15, 0.5, 1, 1.5, 2, 3, 4, 5, 6 and 7	–	3
6	Amount of MnO_2 addition (wt %)*	–	0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4	1, 2.5 and 4
7	Sequence of MnO_2 addition#	–	A and B	A and B

*All compositions are in weight percent unless stated otherwise

#Sequence A: MnO_2 added after melting of commercially pure aluminium and

Sequence B: MnO_2 added before melting of commercially pure aluminium

3.1 Experimental set up and its description

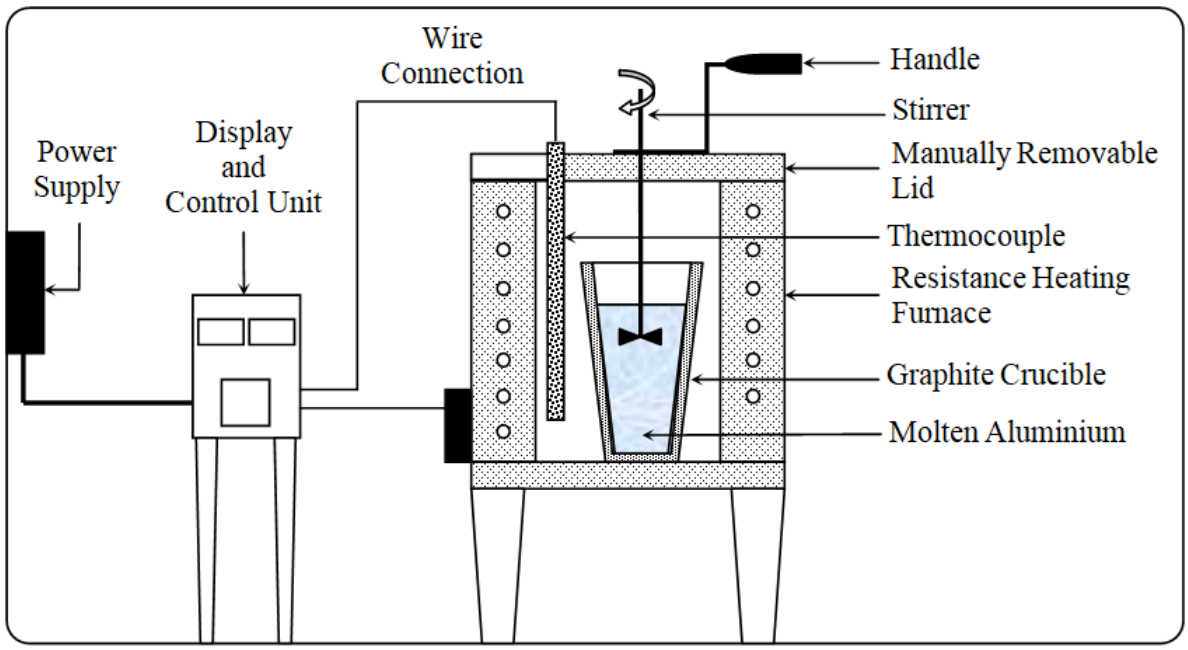


Figure 3.1: The schematic diagram of the experiment set-up used.

3.1.1 Electrical resistance Al melting furnace:

The aluminium melting furnace used in this work is consists of the followings:

1. **Main melting zone:**
   It is essentially square cross sectioned zone consisting outer mild steel wall and inner stainless steel wall having hole at the bottom side. In between outer and inner wall, heating coils are arranged as to provide resistance heating for the melting. Heating coils are made up of Kanthal wire.

2. **Thermocouple:**
   It is Chromel-Alumel type thermocouple inserted into the melting zone to predict the temperature of the melt by displaying on to the temperature display panel in the degree Celsius unit.

3. **Temperature controller:**
   It is connected to the heating coil as well as to the thermocouple. The automatic rely regulates the temperature to the preset limit and helps to maintain optimum temperature.

4. **Stirrer:**
   It is typical turbine type four blade stainless steel stirrer which was used during stirring

to get uniform distribution of MnO_2 particles within the matrix. Before using the stirrer, it was coated with slurry of (MnO_2 + Kerosene) followed by drying. The purpose of this coating is to prevent dissolution of iron from the stainless steel into the molten composite at processing temperature. Stirrer was connected to the motor which provides the rotation to the stirrer at certain RPM.

Figure 3.2: Four blade turbine type stainless steel stirrer used in present work.

5. **Speed and temperature controller:**
   It is essentially used to control the speed of stirrer and the temperature of the bath via controlling the speed of motor. It is also used to indicate the actual speed of stirrer in working condition. This can be achieved by putting sensor near the bottom of the motor. Speed controller display shows the speed in the form of rpm and temperature of the bath.

6. **Metallic die:**
   It is a metallic split die having 5 rod shape cavities in which liquid composite bath was poured and solidified into rod shape to prepare the samples for various characterizations. Overall dimension of two part metallic die are of 23 x 23 x 6 cm as shown in figure 3.3. It contains runner, risers, gating system and central pouring system. Final size of cast rod is of 2 cm diameter and 17 cm in length.

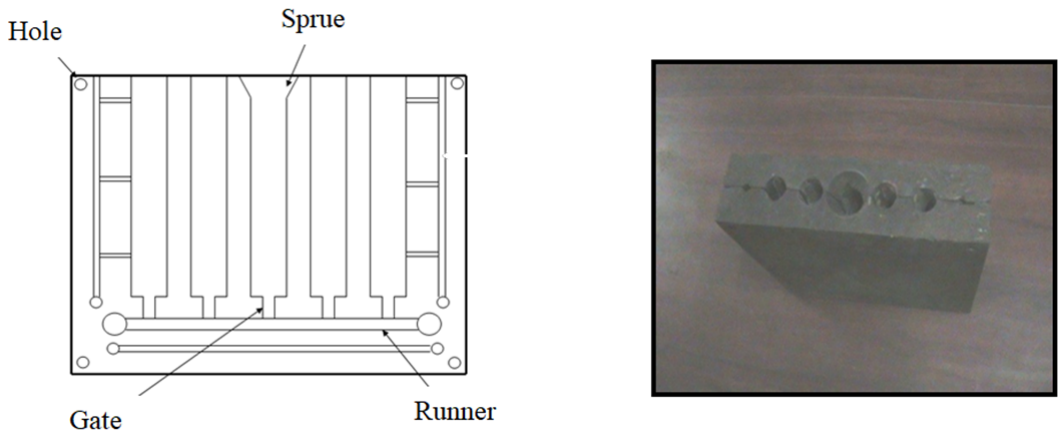
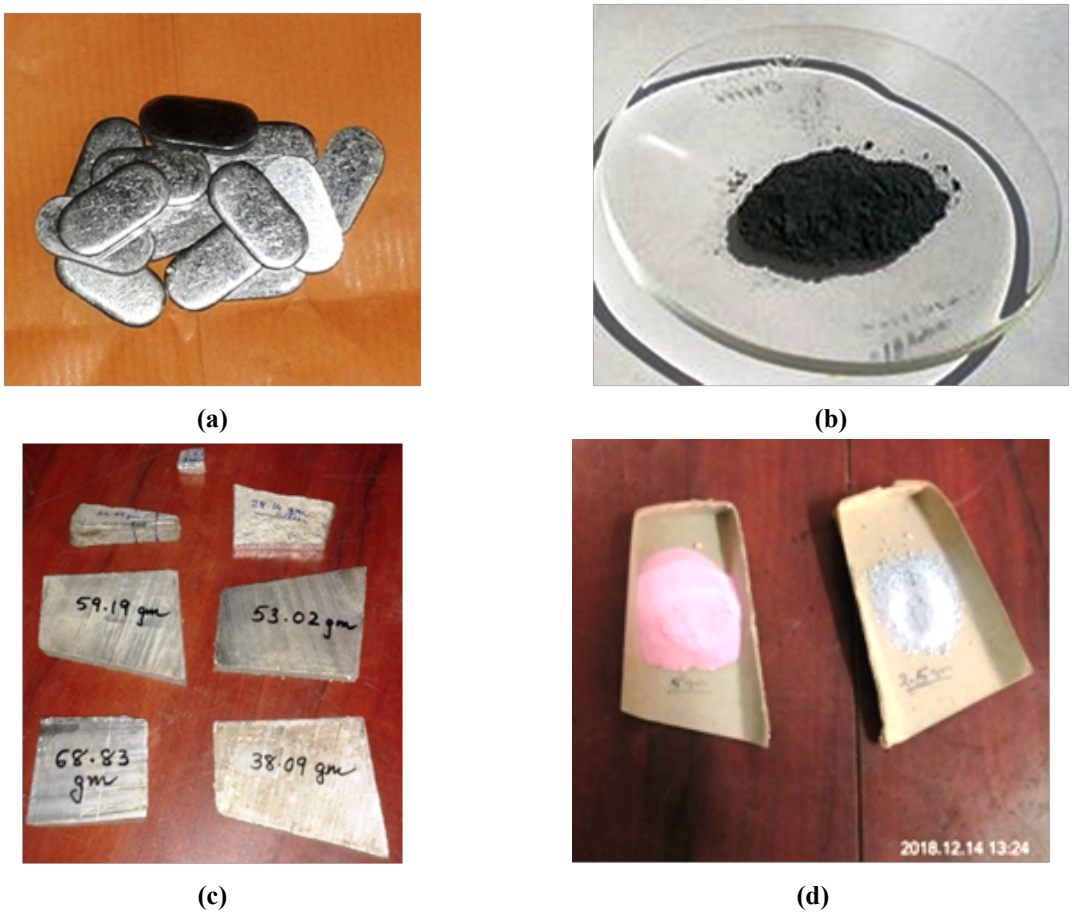


Figure 3.3: Metallic die used in present work.

7. **Raw materials:**



(a) **(b)**

(c) **(d)**

Figure 3.4: (a) Raw CPA, (b) MnO_2 powder, (c) solid magnesium block and (d) fluxing powder and degassing tablet powder.

8. **Auxiliary Equipments:**

(a) *Al rod:* It was used for dipping Mg block into the melt because Mg is light in weight and floats on the molten aluminium surface. It also reduce the oxidation problem and helps in better recovery of Mg metal.

(b) *Toggle:* It was used to handle hotter equipments for safety point of view. For example preheated MnO_2 particles, the crucible of molten metal, etc.

(c) *Strobometer or Techometer:* It was mainly used to measure the speed of the stirrer if digital speed indicator not worked correctly.

(d) *Muffle furnace or oven:* It was used to preheat the MnO_2 particles and Mg block.

(e) *Drossing basket:* It was used to hold dross which was taken out from the upper layer of the melt before pouring.

(f) *Spoon type rod:* It was used to taken out dross from the upper layer of melt.

(g) *Glows:* It was used to protect hands while poring.

(h) *Oxy acetylene flame:* It was used to preheat the die before pouring the composite melt.

(i) *Hammer:* It was used to open split die after solidification of the casting.

(j) *Hacksaw blade:* It was used to cut raw materials and cast rods into desired size for characterization.

Figure 3.5: Placing and setting up the crucible filled with charge materials in the resistance heating furnace.

(a) **(b)**

Figure 3.6: Resistance heating furnace before and after experimentation.

3.2 Experimental Procedure

Experiments have been performed in 3 stages as mentioned earlier. Detail discussion of each phase is presented below.

3.2.1 Phase I: Optimization of Magnesium content in CPA

Phase I study involved the addition of commercially pure magnesium into commercially pure aluminium. As shown in table 3.1 of process parameters, there were 11 runs of experiments carried out to check the effect of magnesium. One extra run of only CPA without magnesium addition and using similar processing conditions was also taken for comparison purpose.

Experimental Steps:

Experimental steps in the form of flow chart is shown in figure 3.7. These experiments are performed to determine the optimum level of magnesium for present CPA which used as matrix material.

700 gm of CPA, 720 ^oC processing temperature and 15 minutes processing time were kept same for all the experiments. Magnesium amount was varied as 0.05, 0.15, 0.5, 1, 1.5, 2, 3, 4, 5, 6 and 7 wt %.

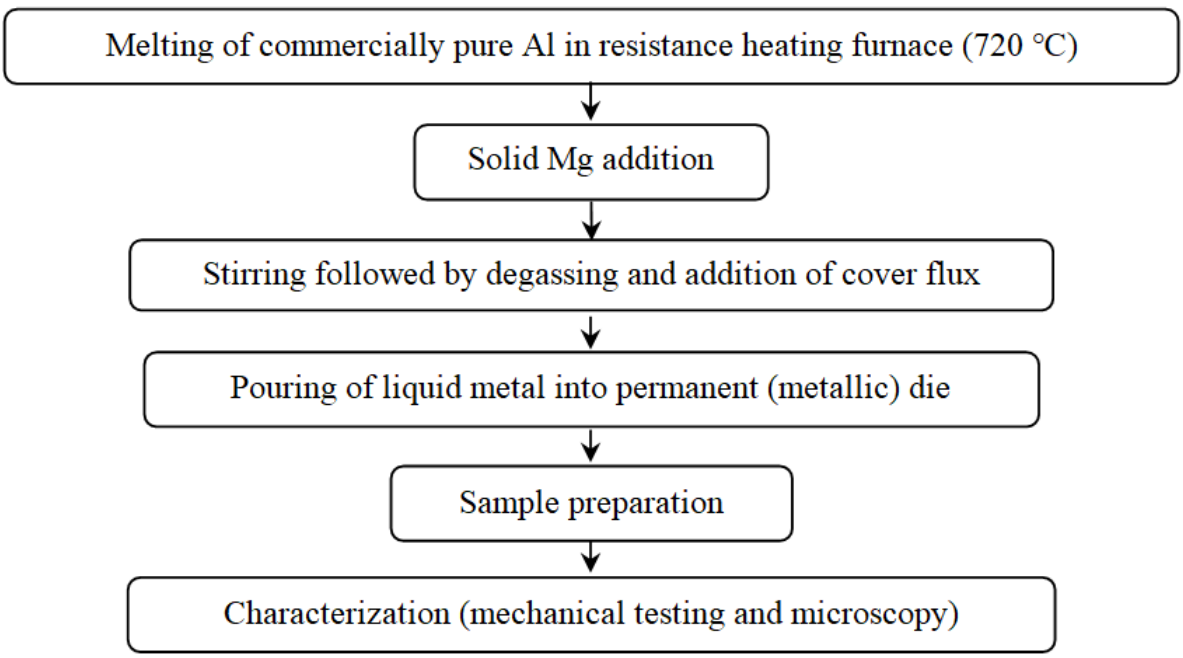
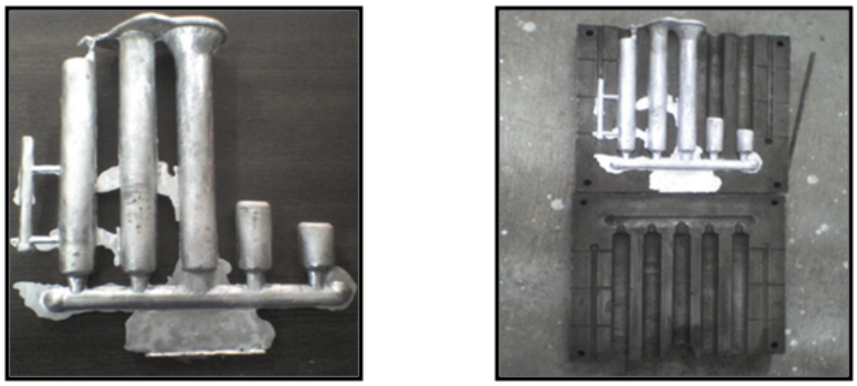


Figure 3.7: Flow chart of experimental steps followed in phase I.

Steps:

1. 700 gm CPA was kept in graphite crucible and it placed inside the resistance heating furnace for melting.
2. Furnace was switched on with 720 ^oC set temperature.
3. To utilize time during melting, following activities were performed:
   (a) Weighing of commercially pure Mg block as per the requirement.
   (b) Metallic die was cleaned and prepared for the casting.
   (c) To prevent die chilling while pouring, preheating of die was carried out with the help of oxy-acetylene flame. It also helps to increase solidification time and prevent various casting defects.
   (d) The stirrer speed was set to 100 RPM using speed controller knob.
   (e) Regular monitoring of the furnace temperature was done.
4. Once the melting was achieved, predetermined amount of magnesium was added to the melt. LM 0 Al rod was used to dip light magnesium metal piece into the liquid aluminium bath until it melted.
5. Immediately after melting of Mg, stirring was carried out at constant speed of 100 RPM for 5 minutes. Formation of vortex was avoided during stirring to prevent pick up of the gases by molten bath from surrounding. Stirring ensures uniform mixing of CPA and Mg in liquid bath.
6. During stirring, the temperature of the slurry was maintained within ± 10 ^oC of the processing temperature.
7. To remove unwanted impurities from the liquid bath and to prevent molten bath from environmental contamination, melt treatments (i.e degassing and fluxing) were given to the bath at 720 ^oC temperature.
8. Immediate after melt treatment, melt cleaning was carried out. After superheating melt was poured into preheated metallic die by ensuring the pouring temperature should not drop below 710 ^oC because the pouring temperature plays very crucial role.
9. The cast metal was allowed to cool in air within the die.
10. The furnace was then turned off and cleaned once it cooled.

11. After 5 minuets time, solidified metal was removed from the die with the help of hammer (as shown in the figure 3.8).



(a) **(b)**

Figure 3.8: Solidified AMMC in metallic die.

12. Identification marking was done on the cast rods.
13. Casting weight was checked to determine yield as shown in figure 3.9

Figure 3.9: Checking final yield after solidification of the AMMC (weight measurement).

14. For characterization of various properties such as hardness, tensile strength, density, microstructures, etc., sample cutting was done manually. See figure 3.10, 3.11 and 3.12.

Figure 3.10: Manual sample cutting by hacksaw.

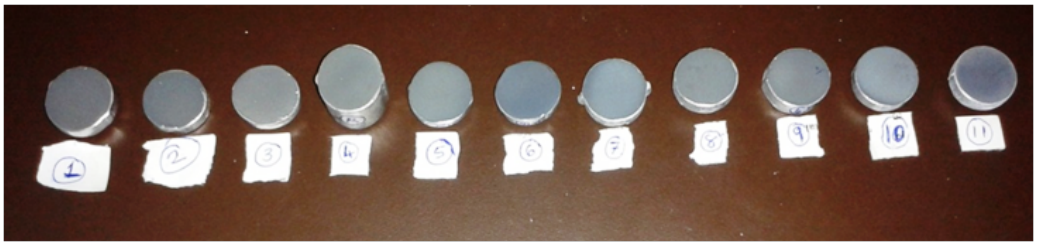


Figure 3.11: Preparation of hardness and microstructure samples.

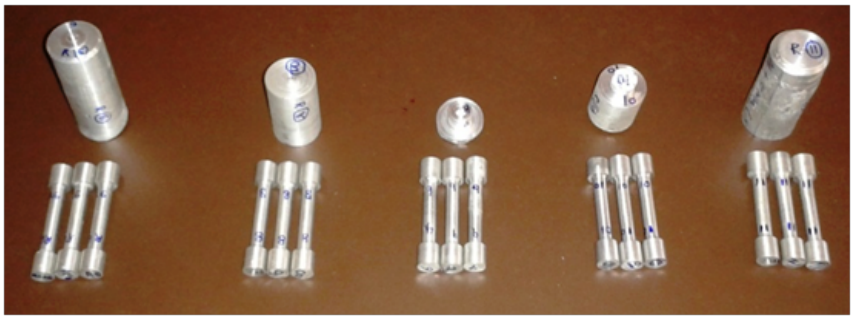


Figure 3.12: Preparation of tensile specimens.

3.2.2 Phase II: Effect of variation of MnO_2 content by changing its addition sequence into CPA

Phase II study involved the addition of MnO_2 into commercially pure aluminium (CPA) without addition of commercially pure magnesium. As shown in table 3.1 of process parameters, there were 16 runs of experiments carried out to check the effect of MnO_2 by changing the sequence of addition. 0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4 wt % MnO_2 in two different sequences (A and B). Below flow chart explain experiments for phase II.

Sequence A: *MnO_2 addition in commercially pure aluminium after melting*

Steps:

1. 700 gm CPA was kept in graphite crucible and it placed inside the resistance heating furnace for melting.
2. Furnace was switched on with 720 ^oC set temperature.
3. To utilize time during melting, following activities were performed:
   (a) Weighing of commercially pure MnO_2 powder as per the requirement.

(b) Metallic die was cleaned and prepared for the casting.

(c) To prevent die chilling while pouring, preheating of die was carried out with the help of oxy-acetylene flame. It also helps to increase solidification time and prevent various casting defects.

(d) The stirrer speed was set to 100 RPM as required by using speed controller knob.

(e) The stirrer rod and blades were coated by applying slurry of fine MnO_2 powder in kerosene and dried, so that there was no significant dissolution of iron from the stirrer into molten aluminium.

(f) Regular monitoring of the furnace temperature was done.

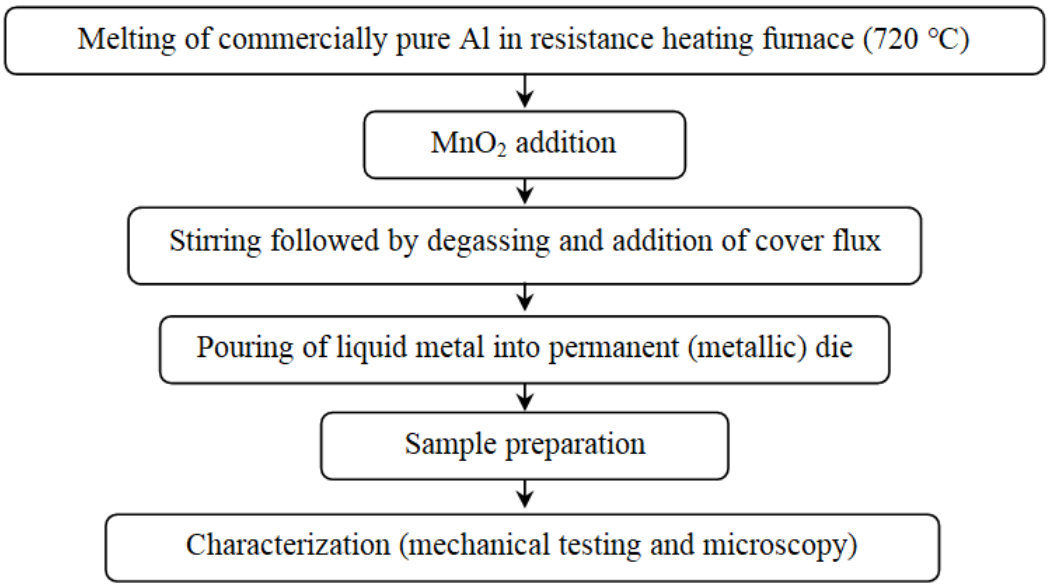


Figure 3.13: Flow chart of experimental steps followed in Sequence A of phase II.

4. Once the melting was achieved, predetermined amount of MnO_2 particles was added manually at the approximately 100 gm/ min rate of addition.

5. Immediately after the addition of MnO_2 , stirring was carried out at constant speed of 100 RPM for 5 minutes. Formation of vortex was avoided during stirring to prevent pick up of the gases by molten bath from surrounding. Stirring ensures uniform dispersion of MnO_2 particles in melt otherwise they may tend to float on molten aluminium.

6. During stirring, the temperature of the slurry was maintained within ± 10 ^oC of the processing temperature.

7. To remove unwanted impurities from the liquid bath and to prevent molten bath from environmental contamination, melt treatments (i.e degassing and fluxing) were given to the bath at 720 ^oC temperature.

8. Immediate after melt treatment, melt cleaning was carried out. After superheating melt was poured into preheated metallic die by ensuring the pouring temperature should not drop below 710 ^oC because the pouring temperature plays very crucial role.
9. The cast in-situ composite was allowed to cool in air within the die.
10. The furnace was then turned off and cleaned once it cooled.
11. After 5 minuets time, solidified metal was removed from the die with the help of hammer (as shown in the figure 3.8).
12. Identification marking was done on the cast composite rods.
13. Casting weight was checked to determine yield as shown in figure 3.9
14. For characterization of various properties such as hardness, tensile strength, density, microstructures, etc., sample cutting was done manually. See figure 3.10, 3.11 and 3.12.

Sequence B: *MnO_2 addition in commercially pure aluminium before melting*

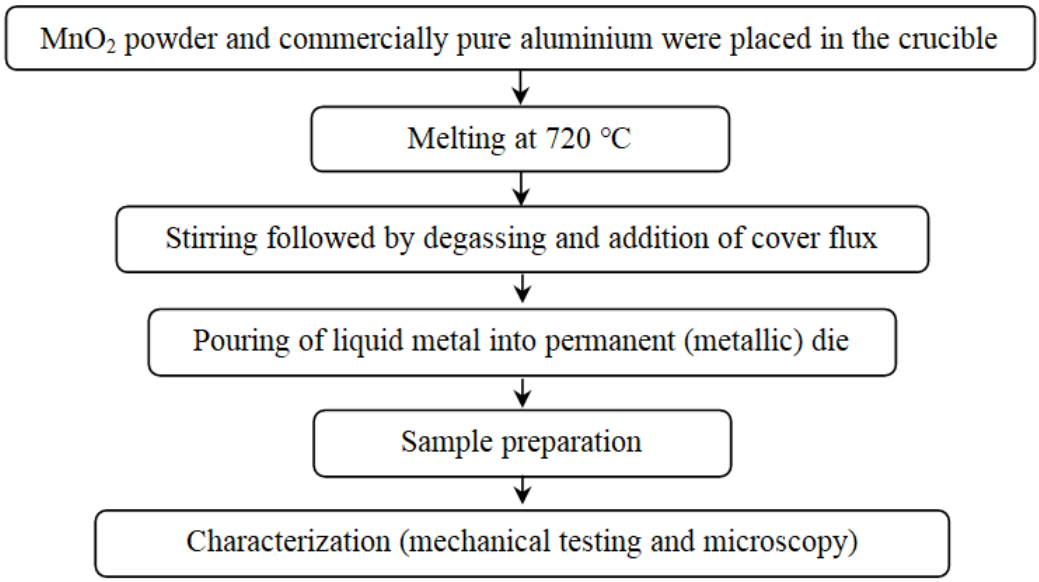


Figure 3.14: Flow chart of experimental steps followed in Sequence B of phase II.

Steps:

1. 700 gm of CPA and predetermined wt % of MnO_2 powder was mixed and kept in the graphite crucible. Crucible was placed inside the resistance heating furnace. for melting upto 720 ^oC.
2. Melting takes some time so in between melting following practice was carried out.

(a) Metallic die was cleaned and prepared for the casting.

(b) To prevent die chilling while pouring, preheating of die was carried out with the help of oxy-acetylene flame. It also helps to increase solidification time and prevent various casting defects.

(c) The stirrer speed was set to 100 RPM using speed controller knob.

(d) The stirrer rod and blades were coated by applying slurry of fine MnO_2 powder in kerosene and dried, so that there was no significant dissolution of iron from the stirrer into molten aluminium.

(e) Regular monitoring of the furnace temperature was done.

3. Once the melting was achieved, stirring was carried out at constant speed of 100 RPM for 5 minutes. Formation of vortex was avoided during stirring to prevent pick up of the gases by molten bath from surrounding. Stirring ensures uniform dispersion of MnO_2 particles in melt otherwise they may tend to float on molten aluminium.

4. During stirring, temperature of the slurry was maintained within ± 10^oC of the processing temperature.

5. To remove unwanted impurities from the liquid bath and to prevent molten bath from environmental contamination, melt treatments (i.e degassing and fluxing) were given to the bath at 720 ^oC temperature.

6. Immediate after melt treatment, melt cleaning was carried out. After superheating melt was poured into preheated metallic die by ensuring the pouring temperature should not drop below 710 ^oC because the pouring temperature plays very crucial role.

7. The cast in-situ composite was allowed to cool in air within the die.

8. The furnace was turned off and cleaned once it cooled.

9. After 5 minutes, solidified composite was removed from the die (as shown in the figure 3.8).

10. Identification marking was done on the cast composite rods.

11. Casting weight was checked to determine yield as shown in figure 3.9

12. For characterization of various properties such as hardness, tensile strength, density, microstructures, etc., sample cutting was done manually. See figure 3.10, 3.11 and 3.12.

3.2.3 Phase III: Effect of variation of MnO_2 by changing its addition sequence along with optimised magnesium metal from phase I into CPA

Phase III study involved the addition of Mg and MnO_2 both into commercially pure aluminium in two different sequences (A and B as mentioned in table 3.1). There were total 06 runs of experiments, three in each sequence, performed to check the effect of magnesium and MnO_2 by changing its sequence of addition on various properties on CPA matrix. From phase-I results, optimum value of magnesium in commercially pure aluminium was 3 wt%. Hence, keeping this result in mind, we have fixed the magnesium content at 3 wt %, whereas MnO_2 concentration was varied as 0.5, 2.5 and 4 wt % in two different sequences (A and B). As mentioned below flow-sheet, experiments have been followed.

Sequence A: *MnO_2 addition in commercially pure aluminium after melting*

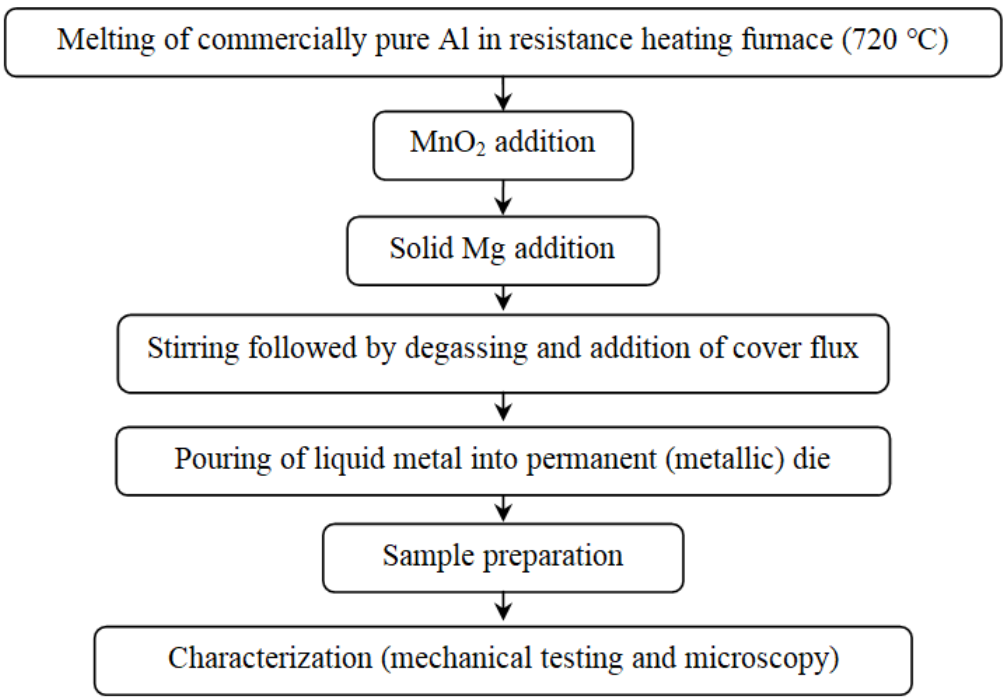


Figure 3.15: Flow chart of experimental steps followed in Sequence A of phase III.

Steps:

1. 700 gm CPA was kept in graphite crucible and it placed inside the resistance heating furnace for melting.
2. Furnace was switched on with 720 ^oC set temperature.

3. To utilize time during melting, following activities were performed:
   (a) Weighing of commercially pure MnO_2 powder and commercially pure Mg block as per the requirement.
   (b) Metallic die was cleaned and prepared for the casting.
   (c) To prevent die chilling while pouring, preheating of die was carried out with the help of oxy-acetylene flame. It also helps to increase solidification time and prevent various casting defects.
   (d) The stirrer speed was set to 100 RPM using speed controller knob.
   (e) The stirrer rod and blades were coated by applying slurry of fine MnO_2 powder in kerosene and dried, so that there was no significant dissolution of iron from the stirrer into molten aluminium.
   (f) Regular monitoring of the furnace temperature was done.
4. Once the melting was achieved, predetermined amount of MnO_2 particles was added manually at the approximately 100 gm/ min rate of addition.
5. Immediately after the addition of MnO_2, stirring was carried out at constant speed of 100 RPM for 5 minutes. Formation of vortex was avoided during stirring to prevent pick up of the gases by molten bath from surrounding. Stirring ensures uniform dispersion of MnO_2 particles in melt otherwise they may tend to float on molten aluminium.
6. During stirring, the temperature of the slurry was maintained within ± 10 ^oC of the processing temperature.
7. After uniform mixing was achieved, stirrer was removed and 3 wt % of magnesium was added to the melt. Addition of Mg helps to increase the wettability of MnO_2 powder by molten aluminium.
8. Once the magnesium block melted, again stirring was carried out by lowering the stirrer into the liquid bath for another few seconds to achieve uniform mixer.
9. To remove unwanted impurities from the liquid bath and to prevent molten bath from environmental contamination, melt treatments (i.e degassing and fluxing) were given to the bath at 720 ^oC temperature.
10. Immediate after melt treatment, melt cleaning was carried out. After superheating melt was poured into preheated metallic die by ensuring the pouring temperature should not drop below 710 ^oC because the pouring temperature plays very crucial role.
11. The cast in-situ composite was allowed to cool in air within the die.

12. The furnace was turned off and cleaned once it cooled.
13. After 5 minutes, solidified composite was removed from the die (as shown in the figure 3.8).
14. Identification marking was done on the cast composite rods.
15. Casting weight was checked to determine yield as shown in figure 3.9
16. For characterization of various properties such as hardness, tensile strength, density, microstructures, etc., sample cutting was done manually. See figure 3.10, 3.11 and 3.12.

Sequence B: *MnO_2 addition in commercially pure aluminium before melting*

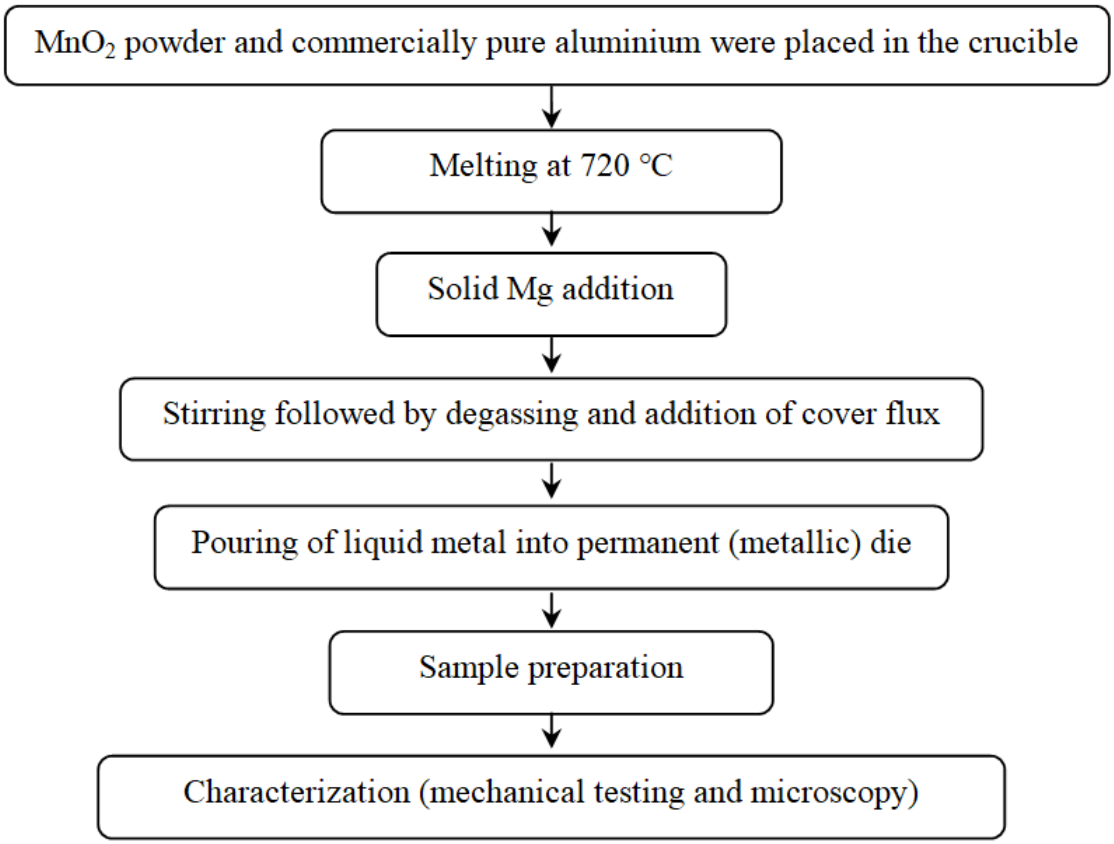


Figure 3.16: Flow chart of experimental steps followed in Sequence B of phase III.

Steps:

1. 700 gm of CPA and predetermined wt % of MnO_2 powder was mixed and kept in the graphite crucible. Crucible was placed inside the resistance heating furnace. for melting upto 720 ^oC.

2. Melting takes some time so in between melting following practice was carried out.
   (a) Weighing of commercially pure Mg block as per the requirement.
   (b) Metallic die was cleaned and prepared for the casting.
   (c) To prevent die chilling while pouring, preheating of die was carried out with the help of oxy-acetylene flame. It also helps to increase solidification time and prevent various casting defects.
   (d) The stirrer speed was set to 100 RPM using speed controller knob.
   (e) The stirrer rod and blades were coated by applying slurry of fine MnO_2 powder in kerosene and dried, so that there was no significant dissolution of iron from the stirrer into molten aluminium.
   (f) Regular monitoring of the furnace temperature was done.
3. Once the melting was achieved, stirring was carried out at constant speed of 100 RPM for 5 minutes. Formation of vortex was avoided during stirring to prevent pick up of the gases by molten bath from surrounding. Stirring ensures uniform dispersion of MnO_2 particles in melt otherwise they may tend to float on molten aluminium.
4. During stirring, the temperature of the slurry was maintained within ± 10 ^oC of the processing temperature.
5. After uniform mixing was achieved, stirrer was removed and 3 wt % of magnesium was added to the melt. Addition of Mg helps to increase the wettability of MnO_2 powder by molten aluminium.
6. Once the magnesium block melted, again stirring was carried out by lowering the stirrer into the liquid bath for another few seconds to achieve uniform mixer.
7. To remove unwanted impurities from the liquid bath and to prevent molten bath from environmental contamination, melt treatments (i.e degassing and fluxing) were given to the bath at 720 ^oC temperature.
8. Immediate after melt treatment, melt cleaning was carried out. After superheating melt was poured into preheated metallic die by ensuring the pouring temperature should not drop below 710 ^oC because the pouring temperature plays very crucial role.
9. The furnace was turned off and cleaned once it cooled.
10. After 5 minutes, solidified composite was removed from the die (as shown in the figure 3.8).
11. Identification marking was done on the cast composite rods.

12. Casting weight was checked to determine yield as shown in figure 3.9
13. For characterization of various properties such as hardness, tensile strength, density, microstructures, etc., sample cutting was done manually. See figure 3.10, 3.11 and 3.12.

3.3 Characterisation

The instruments used to measure metallurgical and mechanical properties are discussed below.

3.3.1 Density Measurement

Density measurement was performed using Pycnometer as shown in figure 3.17.

(a) **(b)**

Figure 3.17: Measurement of density by Pycnometer.

Inert Argon gas was used to measure the volume of the samples. Measurements of the sample weight were carried out separately by four decimal accurate digital weighing machine.

3.3.2 Tensile test

Universal Testing machine is generally used where higher strain rates are involved. In this case, low strain rate is preferred as the present samples are cast samples. Low strain rate gives accurate reading of mechanical properties. For cast non ferrous metals, low strain rate is preferred. Figure 3.18 shows the Monsanto 20 tensile testing machine specifically design for low strain rate testing. The tensile test was carried on the Monsanto-20 tensile testing machine.

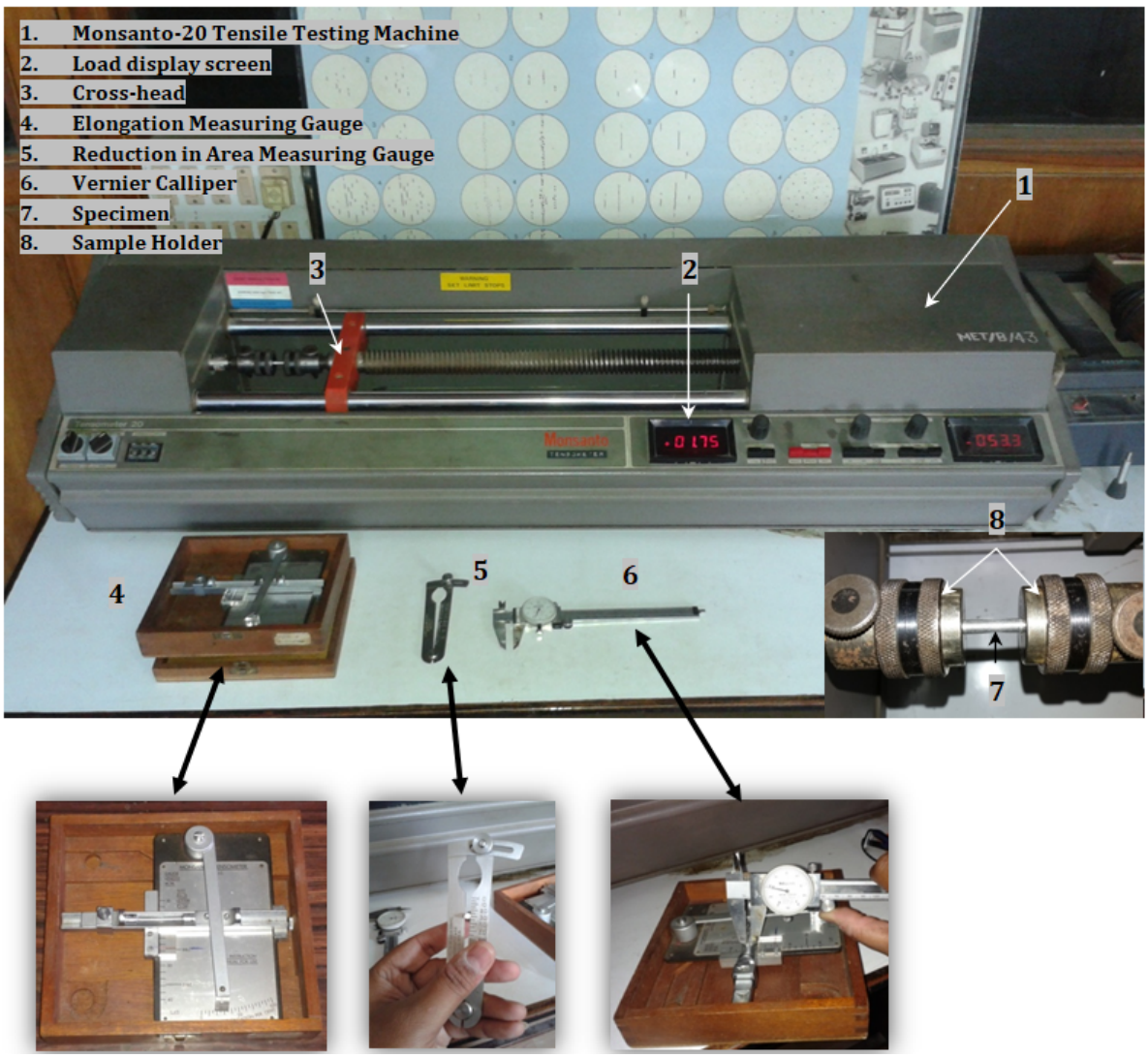


Figure 3.18: Monsanto-20 tensile testing machine and its accessories.

1. **Standard Size of specimen:**
   Following are the dimensions for standard tensile test specimen:
   7.6 mm to 8.1 mm O.D., 42 mm total length, 27 mm gauge length, 25 mm useful gauge length, 5.06 mm gauge diameter, 1 mm radius of fillet. Figure 3.19 indicate the same.

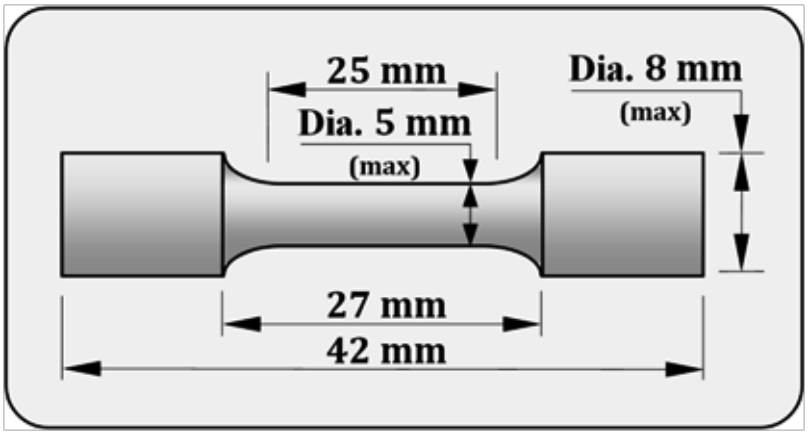


Figure 3.19: Typical dimensions of the specimens for the tensile test.

2. **Available load limit settings:**
   20 kN/2000 kgf-Red, 2000N/200 kgf-White, 200 N/20 kgf-Blue. But we set red.

3. **Strain-Rate:**
   The cross-head speed was adjusted to a value of 0.5 mm/min.

Precautions

1. Misalignment of the specimen in the specimen holder should be avoided.

2. Higher strain-rates should be avoided.

3. Care should be taken that the specimen breaks from the centre rather than from the sides which can be achieved by controlling the porosities generated during casting. Perfect sample preparation is required which can be possible using CNC machine.

3.3.3 Hardness test

The hardness testing was performed on the Brinell Hardness Testing Machine. Generally for testing of Fe and steel, a load of 3000 kg is applied using a ball diameter of 10 mm for about 10 to 30 sec. Similarly for non-ferrous metals and alloys a load of 500 kg or less is applied using ball indenter of 2.5–10 mm.

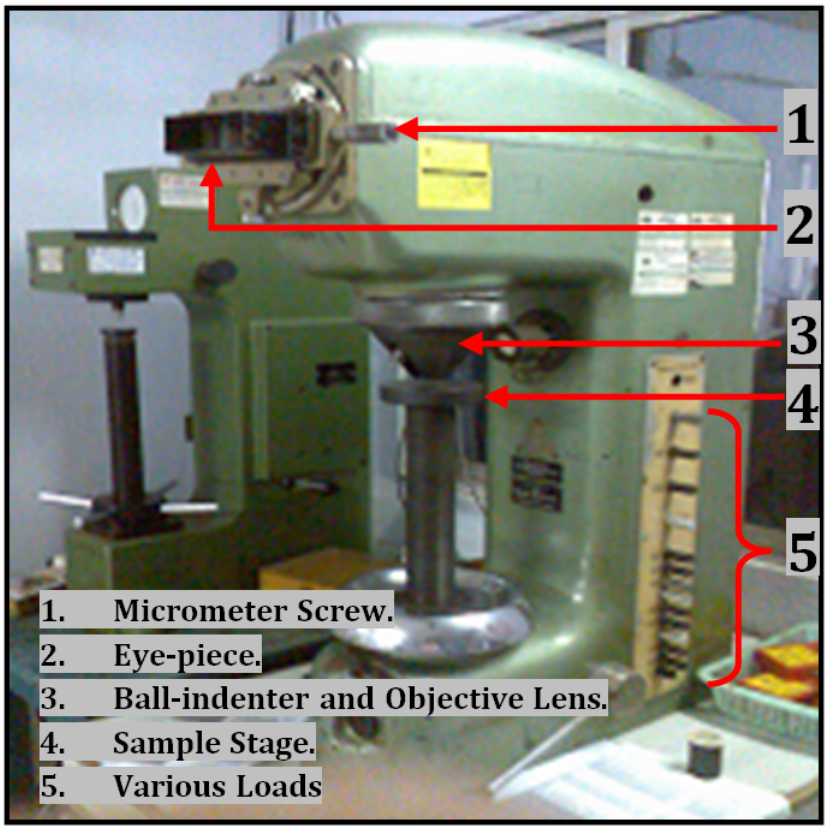


Figure 3.20: Brinell hardness testing machine.

Process Details

The ball indenters are made up of high carbon hardened steel or tungsten carbide. After the load application the indenter was taken out and the diameter of circular impression was measured by a special microscope attached to the machine itself. The microscope magnifies the image and with the calibrations on the eyepiece. Measurement of the diameter of indentation was done with an accuracy of 0.01 mm.

Ball diameter: 2.5 mm
Load Applied: 15.625 kg
Time of Application of load: 10 sec.

Note

1. In this case surface preparation is needed. The surface of the specimen should be flat so as to avoid the errors in the measurement of hardness.
2. In this test we used lower loads as compared to the general loading because if we apply larger loads larger indentations will be obtained which will not be measured with the eyepiece. Low loads leads to smaller impressions and hence it was used.

3.3.4 Microstructure Observation

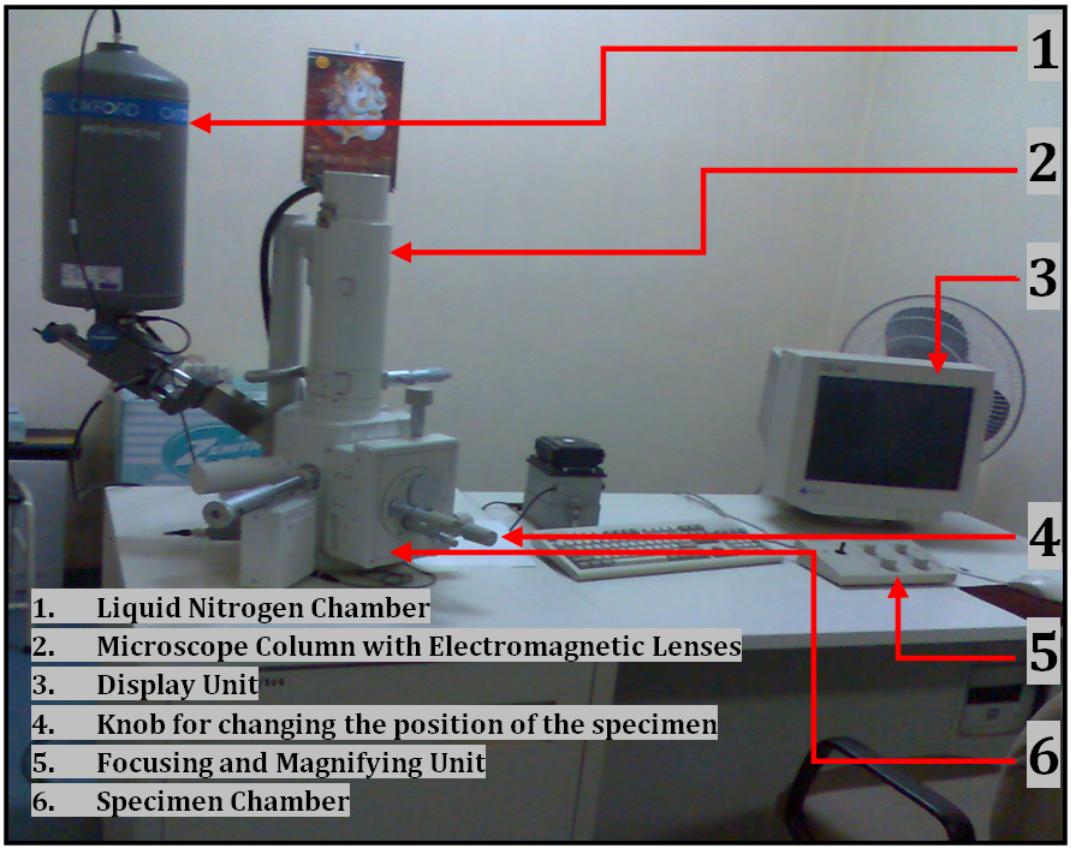


Figure 3.21: Scanning electron microscope.

In present research work, JSM-5610 LV model Scanning Electron Microscope (SEM) was used to carry out microstructure observation and qualitative analysis. Along with SEM, optical microscope also used at various magnification to analyse resulting structure of in-situ MMCs. In figure 3.21, SEM is shown which was used in this work.

Qualitative and quantitative analysis of various elements were performed initially by using the Energy Dispersive X-Ray Spectroscopy (EDS) and then further confirmed by the spectroscopy analysis.

3.3.5 XRD Analysis

The XRD analysis of the composite samples have been performed using PANnalytical X'Pert PRO XRD machine with CuK_α radiation and 2θ range from 20^o to 100^o. Nickel filter was used to suppress the unwanted CuK_β peaks. Figure 3.22 shows the XRD machine used for the characterization.

Figure 3.22: X-ray diffraction machine.

Parameters for XRD Scan:

Scan Axis: Gonio
Start Position [°2Th.]: 20
End Position [°2Th.]: 100
Step Size [°2Th.]: 0.0170
Scan Step Time [s]: 8.2550
Scan Type: Continuous
PSD Mode: Scanning
PSD Length [°2Th.]: 2.12
Divergence Slit Type: Fixed
Divergence Slit Size [°]: 0.8709
Specimen Length [mm]: 10.00
Measurement Temperature [°C]: 25.00
Anode Material: Cu
K-Alpha1 [Å]: 1.54060
Generator Settings: 40 mA, 45 kV
Goniometer Radius [mm]: 240.00
Incident Beam Monochromator: No