# CHAPTER-3

## MATERIALS AND METHODOLOGY

Table of Contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.0</td>
<td>MATERIALS AND METHODOLOGY</td>
<td>39</td>
</tr>
<tr>
<td>3.1</td>
<td>Materials</td>
<td>39</td>
</tr>
<tr>
<td>3.2</td>
<td>Methodology</td>
<td>39</td>
</tr>
<tr>
<td>3.3</td>
<td>Production of sand castings using coke fired furnace</td>
<td>40</td>
</tr>
<tr>
<td>3.3.1</td>
<td>Pattern making</td>
<td>40</td>
</tr>
<tr>
<td>3.3.2</td>
<td>Mould making</td>
<td>41</td>
</tr>
<tr>
<td>3.3.3</td>
<td>Melting &amp; Pouring</td>
<td>42</td>
</tr>
<tr>
<td>3.3.4</td>
<td>Sodium modification</td>
<td>44</td>
</tr>
<tr>
<td>3.3.5</td>
<td>Strontium modification</td>
<td>45</td>
</tr>
<tr>
<td>3.4</td>
<td>Production of permanent mould castings and modification</td>
<td>45</td>
</tr>
<tr>
<td>3.4.1</td>
<td>Permanent mould</td>
<td>45</td>
</tr>
<tr>
<td>3.4.2</td>
<td>Melting &amp; Pouring</td>
<td>46</td>
</tr>
<tr>
<td>3.4.3</td>
<td>Sodium modification</td>
<td>48</td>
</tr>
<tr>
<td>3.4.4</td>
<td>Strontium modification</td>
<td>48</td>
</tr>
<tr>
<td>3.5</td>
<td>Heat treatment of Al-Si Alloys</td>
<td>48</td>
</tr>
<tr>
<td>3.5.1</td>
<td>Solutionization and age hardening</td>
<td>49</td>
</tr>
<tr>
<td>3.6</td>
<td>Machining of specimen as per ASTM standards for various tests.</td>
<td>50</td>
</tr>
<tr>
<td>3.7</td>
<td>Tests conducted</td>
<td>51</td>
</tr>
<tr>
<td>3.7.1</td>
<td>Microstructural study</td>
<td>52</td>
</tr>
</tbody>
</table>
3.7.2 : Hardness test 53
3.7.3 : Tension test 55
3.7.4 : Impact test 57
3.7.5 : Dry sliding wear test 58
3.7.6 : Machinability test 59
3.7.7 : Fog corrosion test 61
3.0 MATERIALS AND METHODOLOGY

3.1: MATERIALS

A357.0 (Al-7.0Si-0.6Mg) alloys ingots as shown in Plate 3.1 were taken and the chemical composition of the alloy was obtained using Optical Emission Spectrometer (Baird-Dv6E) is shown in Table 3.1.

Plate 3.1: A357.0 alloy ingots

3.2. METHODOLOGY

The experimental procedure involved:-

i) Production of A357 Castings

ii) Heat treatment of alloy specimen

iii) Machining of specimen as per ASTM standards for various tests

iv) Conduction of various tests and comparison of results.
3.3 Production of Sand Castings Using Coke Fired Furnace

Sequence of operations: Pattern making, mould making, melting, modification, degasification, pouring and solidification.

3.3.1. Pattern making

In this present investigation, wooden pattern of cylindrical tapered cross section (Fig. 3.1 & Plate 3.2) was used to produce tensile test specimen and split pattern (Plate 3.3) was used for producing specimen for other tests.

Fig. 3.1: Wooden pattern
(All dimensions are in mm)

Plate 3.2. Wooden pattern
(Used to produce tensile specimen).

Plate 3.3. Split pattern
3.3.2 Mould making:

The next step in the sand casting process is to create the mould for the casting. A sand mould was formed by packing sand around the pattern. Green silica sand of AFS grain fineness number 25 with the following ingredients was used as mould material.

- Bentonite (Clay binder) : 11.2%
- Moisture : 3.25%
- Additives (to improve surface finish) : Coal dust 2.5%

The above ingredients were mixed thoroughly for about 3-5 minutes. Chalk powder was sprinkled on the patterns for easy stripping. Mould boxes and moulds used for producing various specimens are shown in the Plate 3.4 and plate 3.5.

Plate. 3.4. Mould boxes
(PVC pipes for tensile specimen) (Cope and drag flask set-up for other tests)
3.3.3 Melting and pouring.

Melting of A357 was carried out in a crucible coke fired furnace (plate 3.6). The cross section of the furnace was circular. In the furnace the crucible with the ingots was kept on the pedestal, coke lumps were packed around it and ignited. A357 ingots were heated to 850° C for 3 to 4 hr.
100g of 1T cleaner (Plate 3.7 and 3.9), a mixture of magnesium and potassium chlorides was added to remove all the impurities in the molten metal. Then 50g of Hexachloro ethane (degassifier, Plate 3.8 and 3.10) was added to remove the gases. After 5 minutes the molten metal was poured into the moulds at 720°C and allowed to solidify (Plate 3.11a and 3.11b) to produce A357 specimen in as cast condition.

Plate 3.7. 1T Cleaner

Plate 3.8. Degassifier

Plate 3.9. Addition of 1T cleaner

Plate 3.10. Degasification
The specimens were left to cool for 1 hr. Then the moulds were broke opened and the to remove the specimen.

**3.3.4. Sodium Modification**

Modification is the process of changing the shape of the second phase elements, by controlling its growth and enhancing the growth of primary phase. This improves the mechanical properties of the alloy. For that certain solid agents are used.
The percentage of these elements is very small and the effect is remarkable. Modification is carried out extensively in aluminum silicon alloy.

4kg of A357 alloy was taken in a crucible and heated in a coke furnace to 850°C for 3hr. 0.01% of sodium [88% (NaCl and KCl) and 12% NaF] (Plate 3.12) was added. 100g of 1T cleaner was added to remove impurities. Then 50g of degassifier was added to remove the gases and after 5 min the molten metal was poured into moulds at 720°C.

Plate 3.12.Na modifier          Plate 3.13.Sr modifier

3.3.5. Strontium Modification

4kg of A357 alloy was taken in a crucible and heated in a coke furnace to 850°C for 3hr. 0.01% Sr [Al-10% Sr alloy] (Plate 3.13) was added. 100g of 1T cleaner was added to remove impurities. Then 50g of degassifier was added to remove the dissolved gases. After 5 min the molten metal was poured into moulds at 720°C.

3.4. Production of Permanent Mould Castings and Modification

3.4.1. Permanent mould: The process makes the use of a metallic mould (Cast-iron) to produce the castings. The cast-iron moulds of diameter 60mm and height 175mm
were used to cast the metal. Two die halves shown in the plate 3.14 were used in this present study. The die halves were cleaned and coated with a refractory mould coat. Then the dies were preheated, closed and clamped.

Plate3.14. Permanent mould die halves

Plate3.15. 3-Phase arc furnace

Plate3.16. Crucible with molten metal

3.4.2 Melting & Pouring:

The melting operation was carried out in an electric arc furnace shown in the fig 3.15. The specification of the furnace used is given in Table 3.2. The charge (A357 ingots) were taken in crucible of capacity 5kg and heated to 850°C for 4hr. Then the molten metal was degassed using Hexa-chloroethane (C₂Cl₆) tablets (0.5%
weight of the metal approx. 50g). The tablet was powdered and plunged into the metal and held at the bottom to enable chlorine gas to purge through the melt and remove the dissolved gases. Then the slag was removed. After 5 minutes the molten metal was poured into the cavity via sprue/pouring cup and allowed to solidify under gravity shown in the Plates 3.17 to 3.19.

Plate 3.17. Degassifier

Plate 3.18. Degassification

Plate 3.19. Pouring of molten metal

After solidification casting was removed by opening the mould/die halves and specimen were taken out and left to cool in air.
3.4.3. Sodium Modification:

0.01% Sodium salt [88%NaCl and KCl (1:1) and 12%NaF] which was in the tablet form was added to molten A357 alloy after removing the furnace, mixed well with a stirrer. After 5 min degasification was carried out then the slag is removed. After 5 minutes the molten metal was poured into the cavity at 720°C and allowed to solidify under gravity or atmospheric pressure.

3.4.4. Strontium Modification:

0.01% of Sr in the form of Al-10%Sr master alloy was added to molten Al-Si alloy and the same procedure was repeated.

3.5. Heat Treatment of Al-Si Alloys

Aluminium-silicon alloys containing copper or magnesium are heat treated to obtain desirable properties such as strength and ductility. Al-Si alloys are treated to higher temperatures of 480 to 540°C to improve their toughness. The thermal treatment results in the modification of the primary angular silicon particles to spherical shape.
3.5.1 Solutionization and age hardening:

The conventional heat treatment involving solution treatment, followed by quenching and age hardening was carried out in an electric furnace (fig 3.18) as follows. Solutionization temperature was selected from Al-Mg$_2$Si Pseudo Phase diagram (Fig.1.1). Sand cast and die cast A357 specimen in as cast condition, sodium modified condition and strontium modified condition were solutionized at temperatures of 540°C for 8 hr, water quenched (at 60°C) and then age hardened at 155°C for 6 hr, 8 hr and 10 hr.

These specimen were designated as S-540-8hr-155-6hr, S-540-8hr-155-8hr, and S-540-8hr-155-10hr where, S represents sand casting method, 540 represent solutionizing temperatures, 8hr represents solutionization time, 155 represents age hardening temperature and 6hr, 8hr, 10hr indicate aging times.

Similarly permanent mould specimens were designated as P-540-8h-155-6hr, P-540-8hr-155-8hr, and P-540-8hr-155-10hr where, P represents permanent mould casting method. During this process very fine particles of Mg$_2$Si are precipitated resulting in dramatic increase in strength and hardness by precipitation hardening.
3.6. Machining of Specimen as per ASTM Standards for Various Tests:

Casting was first sectioned using milling cutter and then machined to the desired shape and size using center lathe (plate 3.22).
3.7. TESTS CONDUCTED:

The following tests were conducted to assess the properties of the A357 alloy. Five specimen were studied for each testing condition and the average value was recorded for all the mechanical tests.

- Microstructural examination - To assess the distribution of Si particles in the Al matrix, dendritic cell size etc.
- Hardness test - To determine the BHN of different specimen.
- Tension test - To determine yield strength, ultimate strength and % elongation of the specimen.
- Impact test – To determine the toughness of the material.
- Dry wear test - To determine rate of removal of material.
- Machinability test - To determine the machinability characteristics.
- Impact test - To determine toughness of the material.
- Corrosion test - To determine corrosion resistance of the material.
3.7.1. Microstructural analysis:

The primary objective of metallographic examination is to reveal the constituents and structure of metals and their alloys by means of a microscope usually “metallurgical Microscope”. The metallographic specimen were prepared in accordance with ASTM standards ASTM E 340 & ASTM E407. Since the specimen gives the interpretation of the material that is being studied, here a section perpendicular to the surface was chosen. The perpendicular cross section reveals much information on variations in structure from center to surface and distribution of non-metallic throughout the section.

- The specimens for microstructure analysis were machined in the form of cylindrical pieces of 20 mm diameter, 20 mm thickness as shown in plate3.23.
- These samples were ground with a sequence of abrasive papers of ranging in the order of 220, 400, 600, and 1000.
- The specimen were then polished using a polishing machine (Plate3.24) in which the specimen is held against the velvet cloth covered on a rotating disc and diamond paste is used as an abrasive and kerosene as lubricant to obtain mirror finish.
- The samples were washed and dried and observed under a metallurgical microscope (Plate3.25) in unetched condition and photographed.
- Then specimen were etched with 0.5% HF solution and washed under running water.
- Dried and etched specimen were again observed under microscope and photographed.
3.7.2. Hardness test (BHN)

Brinell hardness test is a simple indentation test for determining the hardness of variety of materials. A constant load between 500-3000 kgf will be applied for 10 to 30 seconds, using a hardened steel ball or tungsten carbide ball indenter.

In this present investigation Brinell hardness tester -INDENTEC, (plate3.27) was used to measure the hardness of the specimen.
Procedure

Specimens were machined to required dimensions (cylindrical specimen of 20 mm diameter, 20 mm thickness as per ASTM E10-08 standards) and surface of specimen was leveled with abrasive papers. A constant load of 500 kg is applied on the surface of specimen for about 30 seconds using 10 mm hardened steel ball indenter which results in an indenter on the surface.

The diameter of indentation was measured using Brinell microscope.

For each sample at least four readings were taken and average values were found. BHN was calculated using the formula given below.

![Ball indentation on specimen](image)

Fig. 3.3: Ball indentation on specimen
Brinell hardness number (BHN) = $$\frac{2P}{\pi D (D - \sqrt{D^2 - d^2})}$$

Where $P = \text{Load applied in kgf}$ and $D = \text{Ball indenter diameter in mm}$

### 3.7.3 Tension test

Tensile test is one of the most common mechanical tests performed to determine several mechanical properties of a material. This test was conducted using ‘Universal Testing Machine'. The specimen were machined as per ASTM D8 Standards as shown in the Fig 3.4, Plate 3.28

![Tensile specimen](image)

as per ASTM where $L=280 \text{ mm}$, $G=110 \text{ mm}$, $D=12.6 \text{ mm}$
Procedure:

- The tensile testing was conducted using a 450000N capacity “Universal Testing Machine” (Plate.3.29) A load range of 200000N was selected for the current experimentation.
- By holding the test bar in the appropriate shackles; the calibrated extensometer is fixed to it by leaving at least 30 mm clearance between upper & lower portions of the jig.
- The test bar was then strained to attain 0.1-0.2% permanent set of gauge length in the extensometer by turning on the strain knob slowly.
- After attaining the permanent set; the load was noted down & the extensometer was removed. Finally the sample was loaded till failure. The ultimate load and fracture load were noted down. The results of the tensile tests were averaged from four determinations.
### 3.7.4 Impact Test:

Impact is a measure of the energy absorbed during the fracture of a specimen of standard dimensions and geometry when subjected to very rapid (impact) loading. Impact test was conducted using Izod impact tester (Plate3.30), with specimen (plate3.31) as per ASTM D256 standards to measure impact energy which is important in assessing the ductile-to-brittle transition behavior of a material.

#### Procedure

- The specimen was inserted in the shackles and the shackles with the specimen were fixed in the vice provided at the bottom using the gripper and the gauge such that the notch faces the pendulum.
- The swinging weight of the pendulum was lifted to maximum height and locked.
- The pointer of the scale was brought to the zero position.
- The pendulum was then released by operating the lever to strike the specimen and fracture it.
- The impact energy of the specimen was noted down on the graduated scale.
3.7.5 Dry Sliding Wear Test

A computerized pin on disc wear testing machine with data acquisition system was used for recording the linear wear of the specimen in microns. The specimen (Plate 3.32.) were machined to the required dimensions as shown in the Fig 3.5

Tests were conducted as per ASTM-G99 norms using pin-on-disc wear testing machine (Plate 3.33) driven by a D.C motor with counter face disc of the machine having diameter of 190 mm and thickness 30 mm. A pivoted steel lever supporting a loading pan on one end has a universal clutch provided at the other end to hold cylindrical specimen. The specimen presses against the counter face when weights are placed in the pan.

The track radius of specimen was varied by sliding the support of the pivoted lever along the guide ways provided on the table top of machine. The pressure was varied by placing the required load in pan. A new specimen and freshly prepared counter face were used in every test.

Fig. 3.5 and Plate.3.32 Wear testing specimen
Specimen was fixed in holder of lever at one end. At another end of the lever a known weight was applied, so that specimen was in contact with disc plate. Wear indicator has been calibrated to zero and motor was switched on so that disc plate rotates at constant speed. The test was carried out for 20 min for each testing condition. The above procedure was repeated for different speed and load conditions. The speeds selected for the present study were 300, 400, 500 rpm. and the loads selected were 5N, 10N and 15 N.

3.7.6. Machinability test:

Machinability is the ability of the material by which it permits easy removal of material so that it can be imparted with different shapes. It is a complex property which depends on many factors like material composition and homogeneity, machining parameters like cutting speed, feed, and depth of cut, tool geometry, and type of operation.
Procedure

Machinability test was carried out by fixing the specimen (plate 3.35) to the lathe chuck and the dynamometer (plate 3.34) to the tool post to measure the cutting forces (fig 3.6) on the tool. Lathe was switched on and dynamometer readings were taken by varying cutting parameters speed, feed and depth of cut.

- Effect of modification, heat treatment, cutting parameters on cutting forces was studied and compared for various testing conditions by plotting graphs.

Fig. 3.6 Directions of cutting Forces Plate3.34 Lathe tool dynamometer

Plate 3.35: Machinability test specimen
3.7.7 Fog Corrosion test

Fog corrosion test was carried out using fog corrosion testing machine (plate3.36). The surfaces of the specimen (plate3.37) were cleaned using the emery sheets of 400, 600 and 800 grades and initial weight of Specimen was noted down. The specimens were hung inside the chamber (plate3.38) containing NaCl solution with salt concentration of 28.935g/l of water. The temperature of the salt solution was maintained at 60°C. A constant blow of compressed air is provided in to the solution to ensure the proper stirring.
After every 24hr the specimen were cleaned in distilled water, dried thoroughly, after removing the corroded layer using emery paper they were weighed in an electronic balance. The test was conducted for 10 days.

Corrosion rate was calculated using the formula

\[
\text{Corrosion rate (mm/yr)} = 86.7 \times \frac{W}{d \times A \times t}
\]

Where \( W \) is weight loss (mg), \( d \) is density (g/cm\(^3\)) of test specimen, \( A \) is exposure surface area of test specimen (cm\(^2\)), and \( t \) is time in hr.