CHAPTER 2

MATERIALS AND METHODS

2.1 INTRODUCTION

This section of the research work describes the method of production of ductile iron and carbidic ductile iron, its heat treatment process and standards of different characterization. The experimental work and characterization is done in two phases. An austempered ductile iron is produced and analyzed in the first phase of the work.

The production of austempered ductile iron is done in two steps. One is the production of ductile iron castings and the second is the austempering heat treatment of the specimens. Then the austempered specimens are subjected to various mechanical tests like tensile, hardness, impact and abrasion wear test. Microstructure analysis is carried out and SEM analysis is also carried out on the impact and the wear test specimens.

In the second phase of the research, the carbidic ductile iron is produced by melting route. Different levels of chromium alloyed carbidic ductile iron are produced with high chromium ferro-chrome as alloy addition. Tensile, impact, hardness and wear test specimens are machined from the casted Y-blocks. The carbidic ductile iron specimens are austempered to form the carbidic austempered ductile iron. Mechanical properties of the CADI specimens are measured, SEM analysis of impact fracture and wear surfaces are also carried out.
2.2 PHASE I – PRODUCTION AND CHARACTERIZATION OF DI AND ADI

2.2.1 Introduction

In the first phase of the experimental work ductile iron is casted and austempered to get the austempered ductile iron. The base composition of ductile cast iron is hypereutectic, where the carbon and silicon contents are typically 3.7 and 2.5 respectively with a carbon equivalent (CE) of 4.5. High silicon content is to be retained to get the spherical graphite because silicon is a graphite stabilizer. The graphite nodules increase the mechanical properties of the materials. Steel scraps, foundry returns and pig iron are melted in a medium frequency induction furnace; composition is adjusted by adding shell coke as the carburizer, Fe-Si for silicon increase and superheated to 1590°C. Thus the first constituents appear during solidification are graphite nodules, which nucleate and grow, without any martensite, but eventually with austenite enclosing the graphite nodule.

The processing scheme utilized in the production of ductile iron using a magnesium ferro-silicon alloy of 6-8% Mg (Fe-Si-Mg) includes the following steps. The step by step process of the ductile iron production process is explained in Figure 2.1.

1. Build a charge from steel scrap, foundry returns (risers, gates, etc.) and pig iron.
2. Melt the charge and superheat to 1590°C.
3. Verify the composition and adjust the carbon and silicon levels.
4. Pour into magnesium treatment ladle covered with Fe-Si-Mg alloy.
5. Transfer into pouring ladle and inoculate with ferrosilicon based inoculants.
6. Pour the melt into the prepared CO₂ mould.
2.2.2 Raw Material Selection

The raw materials are important for the production of ductile iron. Final composition of the melt mainly depends on the raw materials used and the final properties of the material. The charge consists of low manganese steel scrap, foundry returns like runners, gates, shell coke, and pig iron.

2.2.3 Melting and Composition Control

An electric induction furnace is used for melting the base metal. The basic melting processes are furnace operations including charging, melting, composition analysis, composition adjustment, slag removal and superheating. The raw materials are added to the melting furnace directly and heated. The molten metal is tapped by tilting and pouring through the spout for the magnesium treatment.
2.2.4 Magnesium Treatment

It is the critical step in the ductile iron making. The amount of residual magnesium present in the melt during solidification is in the range of 0.03 to 0.05 weight percent. Magnesium contents less than this amount will result in flake graphite, and the amount more than this results in the appearance of exploded graphite. Either of the type contributes to degradation of the ductility of the cast iron.

The tundish cover ladle method is suitable for better magnesium recovery. Figure 2.2 shows the design of a tundish cover ladle suitable for the magnesium treatment. The use of a refractory dividing wall to form an alloy pocket in the bottom of the ladle gives an improved Mg recovery. The diameter of the filling hole is chosen to minimize the generation of fume while allowing the ladle to be filled quickly without excessive temperature loss. It is essential that the Fe-Si-Mg alloy is not exposed to the liquid iron until quite late in the filling procedure, so the filling hole is positioned to introduce liquid iron away from the alloy pocket in the ladle bottom.

![Figure 2.2 Magnesium treatment process](image)
The calculated amount of magnesium alloy is kept in the alloy pocket and covered with steel turnings, Fe-Si pieces of size 25 × 6 mm. When the melt level in the ladle reaches the dividing wall, iron flows over and forms a semi-solid mass with the covered material allowing the ladle to be almost filled before the reaction starts, thus ensuring good recovery of Mg. This is done primarily to reduce the violence of the reaction that occurs when the molten iron contacts the magnesium.

In order to minimize temperature losses during treatment, the ladle and cover should be separately heated with gas burners before assembly. The common magnesium treatment (Fe-Si-Mg) master alloy which is used in this process contains approximately 6-wt% Mg, about 45 wt% Si, with the balance Fe. About twice as much as magnesium is to be added during treatment and is required in the casting (this represents a 50% recovery) because of the oxidation losses during the violent treatment reaction.

Amount of melt = 50 kg
Magnesium content of the master alloy = 6 wt%
Expected recovery = 50%
Magnesium master alloy required = 1.5 kg/100 kg of metal.

During this time the magnesium reaction involves production of bubbles of magnesium vapour which proceeds to rise up through the molten iron bath which is now covering the pockets in the chamber. For successful treatment results, there should be a significant portion of the magnesium is dissolved into the molten iron, so that the correct conditions for graphite nodule formation are met in the solidifying melt. Typical “recoveries” of magnesium for the Tundish cover treatment facility are in the range of 50 - 60 percent. Successful nodularization requires a composition of about 0.03 to 0.05 weight percent elemental magnesium in the iron. It is necessary that sulphur content should be kept below 0.015% for successful treatment,
because ability of the sulphur to react with the magnesium (forming Mg₂S) removes elemental magnesium from the melt. Often the melt needs to be desulphurized before the treatment begins. It usually involves additions of Ca (calcium) to combine with the sulphur. The calcium sulphide will rise to the slag layer and be skimmed.

Tapping time is usually around 40 seconds. The temperature loss during magnesium treatment is around 50°C, so the tapping temperature must be adjusted accordingly; treatment temperatures around 1540°C are commonly used. After treatment, the tundish cover is removed; the metal is transferred to a pouring ladle where inoculation takes place. The liquid metal must be poured within a short period of time after treatment, usually less than 5 minutes. Longer time may fade the magnesium in the liquid metal and lead to the formation of vermicular cast iron with poor mechanical properties.

2.2.5 Inoculation

Immediately after magnesium treatment, the iron must be inoculated. Graphitizing inoculant BACAL 25 is used. The inoculant manufactured and supplied by M/s SNAM alloy is used. The BACAL contains 25% barium and remaining calcium. Normally 0.3 wt percentage of inoculants is added into the melt. Inoculation treatment is not permanent. The inoculants effect starts to fade from the time it is added. Significant fading occurs within five minutes of inoculation. As the inoculating effect fades, the number of nodules formed decreases and the tendency to produce chill and mottled iron increases. In addition, the quality of the graphite nodules deteriorates and quasi-flake nodules occur.

2.2.6 Moulding, Pouring and Knockout

CO₂ mould with designed runners and risers is prepared for the Standard Y-block pattern as per the ASTM A 370 standards and the
dimensions are shown in Figure 2.3. Casting trials are done by filling the molten metal through the designed runners and risers and checked for its filling performance. This trial shows good filling performance of the mould. The same design of runner and riser is used for the Y-block casting production. The molten metal after treatment is poured into the mould within a short span of time to avoid the fading of magnesium. The mould is allowed to cool for a period of 12 hours and the casting is knocked off from the mould. The casting is shot blasted to remove the sand particles on it. The runner and the risers are removed from the casting using arc cutting. After cleaning, the visual inspection is carried out on the Y-block that reveals defect free cast surface. Cracks, blow holes, porosities are not observed on the surfaces.

![Figure 2.3 Standard dimensions of Y-block casting](image)

### 2.2.7 Composition Analysis

The final composition of the specimen is analyzed using a vacuum spectrometer. The results of the composition are shown in Table 2.1. Specimens are machined from the lower part of the Y-block (Hatched lines shown in Figure 2.3) for the characterization. Melt 1 is the common unalloyed ductile iron (500/7) casting taken for the experimentation. Nodule count 80
should be the minimum requirement for effective austempering. Standard wire cutting, machining and grinding operations are employed for specimen preparation.

Table 2.1 Composition of melt 1 by wt%

<table>
<thead>
<tr>
<th>Melt Identification</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Mg</th>
<th>Cr</th>
<th>Cu</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melt 1</td>
<td>3.6840</td>
<td>2.5332</td>
<td>0.4841</td>
<td>0.029</td>
<td>0.010</td>
<td>0.0408</td>
<td>0.0267</td>
<td>0.0341</td>
<td>0.00</td>
</tr>
</tbody>
</table>

The microstructure of the melt 1 specimen is analyzed after austempering. The microstructure does not reveal any formation of ausferrite matrix. It contains graphite nodules in the pearlite and ferrite matrix. Literatures show that small quantities of alloying are necessary for best austempering. Hardenability elements are to be alloyed for the formation of ausferrite matrix. Copper, nickel, chromium, titanium and molybdenum are some of the hardenability agents used in ductile iron. In this study, copper and molybdenum are added to get the austempered ductile iron.

Table 2.2 Composition of alloyed DI

<table>
<thead>
<tr>
<th>Composition</th>
<th>Melt 2</th>
<th>Melt 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>3.5862%</td>
<td>3.3652%</td>
</tr>
<tr>
<td>Si</td>
<td>2.4957%</td>
<td>2.8112%</td>
</tr>
<tr>
<td>Mn</td>
<td>0.4681%</td>
<td>0.2657%</td>
</tr>
<tr>
<td>P</td>
<td>0.0240%</td>
<td>0.0410%</td>
</tr>
<tr>
<td>S</td>
<td>0.008%</td>
<td>0.0070%</td>
</tr>
<tr>
<td>Mg</td>
<td>0.0490%</td>
<td>0.0320%</td>
</tr>
<tr>
<td>Cr</td>
<td>0.0256%</td>
<td>0.0410%</td>
</tr>
<tr>
<td>Cu</td>
<td>0.3145%</td>
<td>0.3600%</td>
</tr>
<tr>
<td>Mo</td>
<td>0.0000%</td>
<td>0.4200%</td>
</tr>
</tbody>
</table>
Selection of the raw material and the melting is carried out as in the previous case. Calculated amount of copper for melt 2, copper and molybdenum for melt 3 are added in the raw materials. Recovery of 60% of the alloying elements is considered. Weight of the melt is 50 kg. Addition of copper turnings for the melt 2 is 300 grams. 300 grams of copper turnings and 700 grams of HCFeMo are added to the melt 3 to increase the content of copper and molybdenum. The composition of the Y-block castings is checked using spectrometer and the same has been given in Table 2.2.

Microstructures of the melt 2 and 3 are analyzed after austempering treatment. The microstructure reveals ausferrite matrix. The fact sheet of complete experimentation and characterization of austempered ductile iron is shown in Figure 2.4.

2.2.8 Specimen Preparation

Specimens are machined from the lower part of the Y-block for the tensile test, hardness test, wear test and impact toughness test. The positions of the specimens in the Y-block are marked as hatching lines in Figure 2.3. 10mm x 10mm size bars are cut from the Y-block casting by hacksaw cutting. These bars are milled to the Charpy impact test specimen of dimensions 10mmx10mmx55mm as per ASTM A370 standard. The standard tensile test specimen, hardness test specimen and the wear test specimens are turned in lathe.

2.2.9 Austempering Heat Treatment Process

Austempered ductile iron is produced by heat-treating the cast ductile iron alloyed with small amounts of copper and molybdenum. The final properties of the material are determined by careful choice of heat treatment parameters. The austempering process improves the strength of the specimens with minimal distortion and stresses. Austempering heat treatment process is a two stage process. During the first stage the specimens are heated to
austenitizing temperature and held for two hours. In the second stage, the specimens are rapidly cooled from the austenitizing temperature to austempering temperature and soaked for a long time.

Figure 2.4 Flow chart for I-phase investigation
The steps followed in the austempering process are:

1. Heating to the austenitizing temperature (A to B) – 920°C.
2. Austenitizing (B to C) at 920°C for two hours.
3. Rapid cooling to the austempering temperatures (C to D) like 250°C, 275°C, 300°C, 325°C and 350°C.
4. Isothermal heat treatment at the austempering temperature for two hours (D to E).
5. Air cooling to room temperature (E to F).

Figure 2.5 shows a schematic of the austempering process.

![A Typical Austempering Cycle](image)

**Figure 2.5 Schematic of the austempering process**

Two electrical resistance type salt bath furnaces (Figure 2.6) are used for the austempering process. One is for austenitizing and another is for austempering. Furnace system and process are driven via a state-of-art human machine interface, which also provides access to the heat treatment programmes. Flexibility in the furnace and controller configurations allows the austempering process to be tailored to the part.
The specimens are immersed in the crucible containing the molten salt bath. The mode of heat transfer to the work piece is by convection through the liquid bath. These salt bath offers certain advantages over other quench medium.

- All work pieces are at uniform temperature and have identical surroundings. Such a condition results in better surface conditions and consistent and reproducible results.

- Since work piece is in direct contact with the molten bath, there is no danger of oxidation and/or decarburization.

- Salt bath also reduces the fluctuations of temperature.

The commonly used salts are nitrates, carbonates, chlorides, cyanides and caustic soda. The composition of salt bath used for the austenitization and the austempering process are tabulated in Table 2.3. The salt is managed and reused to obviate environmental damage.

Table 2.3 Salt bath composition and working temperature

<table>
<thead>
<tr>
<th>Working Temperature</th>
<th>Composition of Salt Bath</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Austenitization</strong></td>
<td></td>
</tr>
<tr>
<td>Working temperature range:</td>
<td>Sodium Nitrate = 50%</td>
</tr>
<tr>
<td>650°C - 1050°C</td>
<td>Potassium Nitrate = 50%</td>
</tr>
<tr>
<td><strong>Austempering</strong></td>
<td></td>
</tr>
<tr>
<td>Working temperature range:</td>
<td>Sodium Nitrate = 13%</td>
</tr>
<tr>
<td>175 – 540°C</td>
<td>Potassium Nitrate = 50%</td>
</tr>
<tr>
<td></td>
<td>Sodium nitrite = 37%</td>
</tr>
</tbody>
</table>
The choice of austenitizing temperature depends on the chemical composition of the ductile iron. The time of austenitizing is as important as the choice of temperature. The austenitizing temperature should be so chosen that the component is in the austenite + graphite ($\gamma + G$) phase.

Elements like silicon raise the upper critical temperature (UCT) while manganese will lower it. If the austenitizing temperature is below the UCT or in the subcritical range ($\gamma + \alpha + G$), then proeutectoid ferrite will be present in the final microstructure, resulting in a lower strength and hardness of the material. Once the proeutectoid ferrite is formed, the only way to eliminate is reheat it above the UCT. The ductile iron components should be held for a sufficient time to create an austenite matrix saturated with carbon. This time is additionally affected by the alloy content of the ductile iron. Iron with heavily alloyed material takes longer time to austenitizing. In this study, the samples are austenitised at 920°C for a time of 2 hours and then austempered.

Cooling from the austenitizing temperature to the austempering temperature (Figure 2.5 C to D) must be completed rapidly enough to avoid
the formation of pearlite. The austenitized specimens are transferred to austempering salt bath within five seconds. If pearlite is formed, the strength, elongation and toughness will be reduced.

The formation of pearlite can be caused by several things, most notably a lack of quench severity or a low hardenability for the effective section size. The traditional quench media, oil and water are not used so that the load does not reach the $M_s$ temperature and brittle martensite cannot develop. It is possible to increase the quench severity of molten salt quench baths by making water additions. By inoculating the salt bath with water, quenching rates are enhanced to allow treatment of larger section parts without adjustment of cast composition. The range of the austempering temperature for the production of ADI and CADI is 250 to 400°C. Austempering time also varied between one and four hours in a time interval of one hour for CADI production. The austempering temperature and time decides the final properties of the austempered ductile iron and carbidic austempered ductile iron. Higher grades of ADI are produced at lower quench temperatures.

Once the austempering process is completed and the ausferrite has been produced, the components are cooled to room temperature. The cooling rate will not affect the final microstructure and final properties of ADI as the carbon content of the austenite is high enough to lower the martensite start temperature to a temperature significantly below room temperature.

2.2.10 Characterization

2.2.10.1 Metallographic examination

It remains the most important tool for the study of micro-constitutions in the material. The metallographic sample preparation is carried
out by using standard techniques. The specimen for microscopic examination is made flat using grinding wheel and then polished with various grades of emery sheets, followed by disc polishing using diamond paste to reveal the microstructure. The polished samples are etched with 5% nital. The etched specimen microstructure is analyzed and then photograph is taken using the Nikon Epiphot-Dx optical microscope (shown in Figure 2.7) equipped with high resolution digital camera at various magnifications.

![Metallurgical microscope](image)

**Figure 2.7 Metallurgical microscope**

### 2.2.10.2 Hardness test

Brinell hardness is determined by forcing a hard steel ball of specified diameter under a specified load on the surface of the specimen and measuring the diameter of the indentation left after the test. The Brinell hardness number is obtained by dividing the load used, in kilograms, by the actual surface area of the indentation in square millimeters. The result is a pressure measurement, but the units are rarely stated. The size of the specimen used for the hardness test is shown in Figure 2.8.
The Brinell hardness tester is shown in Figure 2.9. The Brinell hardness test is conducted as per the ASTM E10 standard and the method consists of indenting the test material with a 10 mm (D) diameter hardened steel ball subjected to a load (P) of 3000 kgf. The full load is applied for 10 to 15 seconds on the specimens. The diameter of the indentation (d) left in the test material is measured using powered microscope with a least count of 0.01 mm. The Brinell harness number is calculated by using Equation (2.1).

\[ HBN = \frac{2P}{\pi D(D - \sqrt{D^2 - d^2})} \]  

(2.1)

Figure 2.9 (A) Indentation (B) Brinell hardness testing machine
The diameter of the impression is the average of two readings at right angles of an impression and four impressions per specimen are carried out. Compared to the other hardness test methods, the Brinell ball makes the deepest and widest indentation, so the test averages the hardness over a wider amount of material, which will more accurately account for multiple grain structures and any irregularities in the uniformity of the material. This method is the best one for achieving the bulk or macro-hardness of a material, particularly those materials with heterogeneous structures.

2.2.10.3 Impact toughness test

Impact toughness is a measure of the energy absorbed during the fracture of a specimen of standard dimensions and geometry when subjected to impact loading. The Charpy impact test is a standardized high strain-rate test, which determines the amount of energy absorbed by the material during fracture. This absorbed energy is the measure of toughness of the given material.

![Charpy impact testing machine](image)

**Figure 2.10 Charpy impact testing machine**

Charpy impact toughness test is conducted as per ASTM E 23 standard at room temperature using a charpy impact testing machine with 300
joules hammer capacity and 4.5ms\(^{-1}\) striking velocity. The pendulum type charpy impact test equipment set up is shown in Figure 2.10. Tests are conducted on unnotched test samples of size 10mm x 10mm x 55mm and the average of the two impact values is considered. The standard charpy impact test specimen is shown in Figure 2.11. It is widely applied in industry, as it is easy to prepare and conduct and results can be obtained quickly and cheaply.

![Figure 2.11 ASTM standard charpy test specimen](image)

The qualitative results of the impact test can be used to determine the ductility and strength of the material. Materials with high strength and high ductility exhibits high toughness and the materials of low strength and high ductility have low impact toughness. If the material breaks on a flat plane, the fracture is brittle, and if the material breaks with jagged edges or shear lips, then the fracture is ductile.

### 2.2.10.4 Ultimate tensile strength test

The ultimate tensile strength is the maximum stress that can be sustained by a structure in gradually increasing tensile load that is applied uniaxially along the long axis of the specimen. If the stress is applied and maintained, fracture will be the result. Tensile specimens are machined as per the standards of ASTM A 370 and heat treated. The standard dimensions of the tensile test specimen are shown in Figure 2.12. The tensile test is conducted in a micro tensile testing machine of 2000 N capacity and the ultimate tensile strength of the specimen is calculated. Two tensile samples in
each condition are prepared and tested. Average of these two readings is considered for discussion.

![Figure 2.12 Tensile test specimen](image)

2.2.10.5 Wear test

Pin-on-disk abrasive test is a commonly used technique for investigating abrasive wear of the material. Pin-on-disk is an apparatus that consists essentially of a “pin” in contact with a rotating disk. The pin can be the test piece of interest. The contact surface of the pin is flat. A predetermined load is also applied to the specimen. The disk revolves in a particular speed, and an appropriate track on the disk is selected based on the velocity of the travel required. In a typical pin-on-disk experiment, the material removed is determined by weighing and/or measuring the weight of the specimen. The schematic of the abrasive wear test is shown in Figure 2.13.

![Figure 2.13 Schematic diagram of abrasive wear test](image)
Figure 2.14 Pin-on-disk machine setup

Figure 2.14 shows the pin-on-disk machine setup. The main variables that affect friction and wear are the velocity and the normal load. In addition, specimen orientation can be important if retained wear debris affects the wear rate. Most commercial pin-on-disk testers use high loads (e.g. 100-1000N) obtained with dead weight and large areas of contact.

Figure 2.15 Wear test specimen

Abrasion wear resistance of the material is measured as per ASTM standard G99-05 using Pin-on-disk wear testing machine. The material to be tested is made in the form of a pin with dimensions 10mm diameter and
20mm long cylinder as shown in Figure 2.15. The pin is weighed using the balance. It is fixed in the specimen holder and kept pressed against the wear disc in the machine using predetermined load. Disk hardness of HRC – 65, Load of 98.1N is applied to the specimen, with a travel velocity of one m/s and a distance of 10,000m is considered for the measurement of weight loss. The specimen is removed and weighed. The weight loss is noted. The weights are measured by means of 0.01 mg precision scale.

2.2.10.6 SEM analysis

The scanning electron microscope (SEM) is shown in Figure 2.16, which provides a valuable combination of high resolution imaging. The big advantage of SEM is that the sample surface can be examined directly with a depth of field very much greater than that of the optical microscope at high magnifications (>100,000x) and in some cases with better resolution.

Figure 2.16 Scanning electron microscope setup

The impact fractured surface is analyzed using Scanning Electron Microscope of JEOL MODEL JSM 6360 with magnification minimum of 25x and maximum of 2 lakhs. The result is a television-type image of the portion
of the sample surfaces being scanned by the beam. By studying the fracture surface the mode of fracture is whether ductile or brittle has to be established.

Ductile Fracture: Ductile fracture has been defined rather ambiguously as fracture occurring with appreciable gross plastic deformation. Another important characteristic of ductile fracture is that it occurs by a slow tearing of the metal with the expenditure of considerable energy. Ductile fracture can take several forms of single crystals of HCP metals and may slip on successive basal plans until the final crystal separates by shear. A shear fracture occurs as a result of extensive slip on the active plan. A fractured surface that is caused by shear appears at low magnifications to be grey and fibrous. Dimpled rupture is characterized by cup like dispersions. This type of fractured surface denotes a ductile fracture.

Brittle Fracture: Cleavage fracture represents brittle fracture that occurs along crystallographic planes. The characteristic feature of cleavage fracture is flat face. The flat faces exhibit river marking. The river marking are caused by crack moving through the crystal along a number of parallel planes which forms a series of plateaus and connecting edges. The direction of river pattern represents the direction of crack propagation.

2.3 PHASE II - PRODUCTION AND CHARACTERIZATION OF CADI

2.3.1 Introduction

This phase of the experimental work describes the production and the characterization of Carbidic Austempered Ductile iron. Carbidic ductile iron is a ductile cast iron containing carbides in the pearlite and ferrite matrix (they are either thermally or mechanically induced), and that is subsequently austempered to produce an ausferrite matrix with an engineered amount of carbides. The final material is called as carbidic austempered ductile iron.
Carbides in the ductile iron is induced by

- Reducing the graphitizing elements such as silicon.
- Increasing the cooling rate of solidification by using chills.
- Introducing carbide stabilizing elements like chromium, manganese, molybdenum and titanium.

The melt with suitable carbide stabilizing elements is treated with magnesium and/or rare earths result in spheroidal graphite with carbides in the microstructure. Carbides are stable and tend to retain their as-cast volume fractions after austenitizing (Hayrynen et al 2003). The carbides produced from this technique cannot be “dissolved” by subsequent austempering heat treatment. The carbidic austempered ductile iron is formed by austempering the produced chromium alloyed ductile iron and characterized. Impact fractured surface and wear surfaces are analyzed using SEM.

**2.3.2 Production of CADI**

The charge for the production of carbidic ductile iron consists of foundry returns like runners, gates, shell coke, ore with 3 to 4.5 % carbon, 50% pig iron ingot and low manganese steel scrap. An electric induction furnace is used for the melting of base metal. The basic melting process are furnace operations including charging, melting, composition analysis, composition adjustment and superheating. The raw materials and fluxes are added into the melting furnace directly. After melting the slag is removed from melt.

Now the high-carbon ferro-chromium is added into the melt to increase the chromium level. The expected amount of chromium in the high carbon ferrochromium alloy is around 60 %. The amount of chromium alloy
added for each 50 kg melt is given in Table 2.4. After the addition of chromium the molten metal is tapped for magnesium treatment. Now the melt is magnesium treated by following the standard procedure and then inoculated. The amount of inoculants added to this melt is reduced by 10% compared to the regular ductile iron. The inoculants are used as much the formation of alloy carbide is not effective.

Table 2.4 Amount of chromium alloy added to the melt for CADI

<table>
<thead>
<tr>
<th>Melt Identification</th>
<th>Planned Chromium Content</th>
<th>Alloy used</th>
<th>Amount of chromium alloy added (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1% Cr</td>
<td>1 wt%</td>
<td>HCFeCr</td>
<td>0.83</td>
</tr>
<tr>
<td>0.8% Cr</td>
<td>0.8 wt%</td>
<td>HCFeCr</td>
<td>0.67</td>
</tr>
<tr>
<td>0.6% Cr</td>
<td>0.6 wt%</td>
<td>HCFeCr</td>
<td>0.50</td>
</tr>
<tr>
<td>0.4% Cr</td>
<td>0.4 wt%</td>
<td>HCFeCr</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Standard Y-block carbon di-oxide mould is made by machine moulding. Magnesium treated and inoculated melt is poured into the mould. After solidification and curing, Y-block is knocked out from the mould, runners and risers are removed from the casting and afterwards shot blasted. The material in the as-cast condition is called as carbidic ductile iron. Y-blocks are macro examined for porosity and blow holes by the naked eye. Castings without porosity are used for further specimen preparation and austempering heat treatment. Some of the specimens are tested in the as-cast condition.

The composition of the Y-blocks after casting is measured using a vacuum spectrometer. The amount of the other elements is kept constant except the content of chromium. The amount of chromium is varied from 0.4% to 1.0%. Specimens for metallographic examination, hardness test,
impact toughness test, tensile test and wear test from all the compositions of defect free Y-blocks are machined. Austempering heat treatment is carried out by following the procedures explained under section 2.2.9. The specimens are austenitized at 920°C for two hours and austempered at different temperatures of 250°C, 300°C, 350°C and 400°C. The time of austempering is varied from one hour to four hours with an interval of one hour. The heat treatment process and characterization of the carbidic austempered ductile iron is shown as fact sheet in Figure 2.17.

2.3.3 Characterization of CADI

Mechanical properties of the carbidic austempered ductile iron like tensile strength, hardness, impact toughness and wear loss are measured. The methods of measurement of these mechanical properties are done by following the standard procedures.

2.3.3.1 Metallographic examination

The metallographic examinations on the carbidic ductile iron and the carbidic austempered ductile iron are carried out using optical microscope. The standard specimen preparation procedures are explained in section 2.2.10.1. The specimens are polished, etched in 10 % ammonium persulphate and the volume percentage of carbide is measured using Neophot microscope and “Metal Plus Version-1.0” image analysis software. The ASTM designation E562-89 describes systematically manual point counting procedure for statistically estimating the volume fraction of an identified phase from sections through the microstructure. Ammonium persulphate etchant is used to find the amount of carbides. This ammonium persulphate tints the matrix dark and leaves the carbides as white. The micro-photographs of CADI are taken by using 5% nital etching.
2.3.3.2 Hardness test

Brinell hardness test is conducted on the as-cast carbidic ductile iron and the carbidic austempered ductile iron. The standard dimensions of the specimen and the experiment producers have been explained in section 2.2.10.2.

2.3.3.3 Impact toughness test

Charpy impact toughness test is conducted on the as-cast carbidic ductile iron and the carbidic austempered ductile iron. The standard dimensions of the specimen and the experiment producers have been explained in section 2.2.10.3.

2.3.3.4 Ultimate tensile strength test

Ultimate tensile strength test is conducted on the as-cast carbidic ductile iron and the carbidic austempered ductile iron. The standard dimensions of the specimen and the experiment producers have been explained in section 2.2.10.4.

2.3.3.5 Wear test

Pin-on-disk wear abrasion test is conducted on the as-cast carbidic ductile iron and the carbidic austempered ductile iron. The standard dimensions of the specimen and the experiment producers have been explained in section 2.2.10.5.
Figure 2.17 Fact sheet of CADI heat treatment and characterization

2.3.3.6 SEM analysis

The SEM analysis of impact fracture surface and the wear surface are examined using scanning electron microscope to find out the type of
2.4 TAGUCHI METHOD

Taguchi method provides a systematic and efficient approach for conducting experimentation to determine the optimum settings of design parameters for performance and cost. The Taguchi method utilizes orthogonal array to study a large number of variables with a small number of experiments. It can reduce development cost by simultaneously studying a large number of parameters. Using orthogonal arrays, the method can significantly reduce the number of experimental configurations. The conclusions drawn from small scale experiments are valid over the entire experimental region spanned by the control factors and their settings.

2.4.1 Design of Experiments

Two major steps are involved in the production of carbidic austempered ductile iron, one is casting and the other one is austempering heat treatment. Composition of the material is the most important factor when compared to the other factors like hardness of the mould, the filling velocity of the melt, gating design etc. So the composition, particularly the chromium content affects the properties of CADI.

In the heat treatment process, austenitizing temperature and time, as well as austempering temperature and time are the controllable parameters. Austempering temperatures and time are taken into account for the present study. Therefore, three major factors have influence over the production of the CADI. They are chromium content, austempering temperature and austempering time. Four levels in each process parameter are selected and the level values are shown in Table 2.5.
Table 2.5 Factors and levels – L_{16}(4^3)

<table>
<thead>
<tr>
<th>Code</th>
<th>Factors</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% Cr Chromium Percentage</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>T_A Austempering Temperature (ºC)</td>
<td>250</td>
</tr>
<tr>
<td></td>
<td>t_A Austempering Time (Hours)</td>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Cr</td>
<td>0.4</td>
<td>0.6</td>
<td>0.8</td>
<td>1.0</td>
</tr>
<tr>
<td>T_A</td>
<td>250</td>
<td>300</td>
<td>350</td>
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<td>t_A</td>
<td>1</td>
<td>2</td>
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<td>4</td>
</tr>
</tbody>
</table>

The Taguchi method provides the laying out of the experimental conditions using specially designed tables called Orthogonal Array (OA). Taguchi orthogonal array is used for the selection of experiments at different levels among the entire parameter space. An appropriate orthogonal array of L_{16}(4^3) is selected using the Minitab -15 software based on the number of factors and its levels. This orthogonal array shown in Table 2.6 gives the number of experiments to be conducted with their factor levels.

This array consists of 16 rows, each representing an experiment with factor level; the columns are assigned to the factors. The plan of experiments is made of 16 tests in which the first column is assigned to the chromium content (% Cr), second column for the austempering temperature (T_A) and the third column for the austempering time (t_A).

After completion of all the experiments, the data are considered as per the orthogonal array and S/N ratio of individual performance is computed. A grey relational co-efficient and grey relational grade for all S/N ratios is calculated. The major influencing parameters on the mechanical properties are assessed by using ANOVA and optimal parameters are predicted for the CADI production.
Table 2.6 $L_{16}$ Orthogonal array

<table>
<thead>
<tr>
<th>Trial No</th>
<th>Levels of input parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% Cr</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
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</tr>
</tbody>
</table>

2.5 FIELD TEST

Ploughing conditions often cause excessive wear of agriculture ploughshares. The hard particles like SiO$_2$ and quartz present in the soil cause high degree of wear on the ploughshares. The hardness of these particles are beyond 900 HV. The ploughshares are also subjected to very highly complicated dynamic stresses during ploughing. So far, forged steel (EN 45 steel) has been the material used as ploughshare in Tamilnadu. Often this forged steel withstands the dynamic stresses developed during ploughing rather than the wear. The wear resistance of the forged steel is very less.
One of the major expected applications of ADI and the CADI is agricultural equipments. As infield test, the developed CADI material is used as plough point and its wear behavior is checked. The used plough point is shown in Figure 2.18. The plough points are attached to the ploughshare of a tractor using threaded fasteners. Three types of plough points are prepared. One is 1% chromium as cast carbide ductile iron and the other is the 1% chromium CADI heat treated at 920°C, two hours as austenitizing and 300°C, 2 hour time as austempering conditions. The third is the regularly used forged steel plough point as reference. The test is conducted at Mettupalayam area of Coimbatore district. The as-cast material and CADI worked well as plough point. The material loss is calculated after 40 hours of ploughing.