



# STUDY AND THERMO-ANALYSIS OF FERRO TITANIUM

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## ABSTRACT

The present dissertation is titled “**Study and Thermo-Analysis of Ferrotitanium**”. As a developing nation there’s an increase in manufacturing industry which leads to demand of high performance materials such as high grade steels. In order to increase the performance of steel different types of raw materials are added to steel according to requirement. The report helps us to understand the properties of Ferrotitanium, its manufacturing process, analysis of its composition. All new standards have been taken into consideration as well as the results of individual research studies. This project has been guided by **MINEX MEATLLURGY, NAGPUR.**

**KEYWORDS** – *Ferrotitanium, LECO TC 230, LECO TC 500, WDXR, ICP*

## 1. INTRODUCTION

Humans have been making iron and steel for centuries. Steel fueled the industrial revolution and remains the backbone of modern industrialized economies.

Steel’s versatility, in terms of its composition and properties, its strength-to-weight ratio and its ability to be infinitely multicycle into new products sets steel apart from other materials and has been instrumental in its ongoing success.

Various raw materials like Ferro Titanium, Ferro Aluminium, Ferrosilicon Magnesium are used to vary the properties of steel as per requirement of the customers.

The proportions of raw material used vary according to the process and the type of steel required. Steel can be described in general terms as iron with most of the carbon removed, to make it tougher and more ductile. There are many forms (grades) of steel, each with its own specific chemical composition and properties to meet the needs of the many different applications.

About Ferrotitanium:

Ferrotitanium is a ferroalloy, an alloy of iron and titanium with between 10–20% iron and 45–75% titanium and sometimes a small amount of carbon. It is used in steelmaking as a cleansing agent for iron and steel; the titanium is highly reactive with sulfur, carbon, oxygen, and nitrogen, forming insoluble compounds and sequestering them in slag, and is therefore used for deoxidizing, and sometimes for desulfurization and denitrification. In steelmaking the addition of titanium yields metal with finer grain structure. Ferrotitanium can be manufactured by mixing titanium sponge and scrap with iron and melting them together in an induction furnace. Ferrotitanium powder can be also used as a fuel in some pyrotechnic compositions.

### 1.1 Problem statement

Steels of same grade cannot be used for various applications so, there is need of addition of various ferroalloys to the steel while smelting.

Precipitation of chromium carbides occur on grain boundaries at elevated temperature.

High nitrogen content in gray cast iron can promote pearlite formation and may lead to white iron structure.

### 1.2 Objective

- To manufacture high grade steel with different Titanium composition.
- To manufacture and test the raw material according to requirement of the customer.
- To enhance the properties of steel by adding Titanium to molten steel.

Example: RAW MATERIAL: FERRO TITANIUM

IRON AND OTHER ELEMENTS (35-30% by weight)

TITANIUM (65-70% by weight)

Traces of (CARBON, OXYGEN, NITROGEN, SULPHUR)

### 1.3 Scope

Ferro Titanium has low density, high strength and excellent corrosion resistance. Hence it is used in military and commercial aircrafts, paints, special steel and flux cored wire. Ferro Titanium is also used in stainless steel because it acts as a carbide stabilizer to prevent the precipitation of chromium carbides on grain boundaries during extended holding at elevated temperature. Without the presence of Titanium, chromium would be depleted at grain boundaries.

Ferro Titanium is also used to protect Boron in hardenability steel because Boron reacts with Oxygen, Nitrogen, and Carbon present in the steel hence loss of Boron takes place and to neutralise the effect strong nitride formers are added such as (Titanium, Aluminium).

### 1.4 Methodology

#### 1.4.1 Manufacture of ferrotitanium

To manufacture 1 ton of Ferrotitanium in MINEX METALLURGY following steps are performed by skilled chemist and operators under highly controlled facility.

- ❖ STEP 1 - Melting of steel scrap (300 kg) in INDUCTION MELTING FURNACE at 1200°C.
- ❖ STEP 2 - Addition of Titanium Scrap (700 kg) to molten steel and increasing the furnace temperature up to 1700°C.
- ❖ STEP 3 – Addition of Ferro silicon (2 kg) to maintain fluidity.
- ❖ STEP 4 – Removal of slag
- ❖ STEP 5 – After removal of slag, the mixture is then poured into a rectangular slab for solidification at room temperature.
- ❖ STEP 6 – Break solidified Ferrotitanium into lumps.

#### 1.4.2 Test and analysis of ferrotitanium

After manufacturing the pieces, ferrotitanium is then tested for its composition in different machines.

- ❖ STEP 1 - Sample reduction by quarter cornering
- ❖ STEP 2 - Taking 10-12 pieces of 8-10 mm size (100 gm) in VIBRATING CUP MILL for grinding.
- ❖ STEP 3 – Grinding of sample up to 200 mesh.
- ❖ STEP 4 – Forming of pallet from reduced sample in PRESS PALLET MACHINE at 25 ton pressure.
- ❖ STEP 5 – placing pallet in LECO TC 230 to determine the composition of CARBON and SULPHUR.

### 1.5 Organization of dissertation

This report shall be presented in a number of chapter, starting with introduction and end with conclusion and future scope. Each of the other chapter will have precise title reflecting the content of the chapter. A chapter can be subdivided into sections, subsections and sub-subsections so as to present the content discreetly and with due emphasis.

## 2. LITERATURE REVIEW

**M.Pourabdoli et.al [1]** This paper studies about ilmenite concentrate of Iran region which was successfully smelted by Electro-Slag Crucible Melting (ESCM) process to produce TiO<sub>2</sub>-rich slag as primary product and pig iron as by-product. Moreover, effects of reductant (carbon) amount and smelting time on TiO<sub>2</sub> and FeO content of slag and iron recovery were studied. It is found that addition of carbon excess to stoichiometric amount decreases the iron recovery and TiO<sub>2</sub> content in slag. If the smelting time keeps between 0 to 17 min, the TiO<sub>2</sub> content and iron recovery will be increased, whereas smelting time in excess of 17 min will cause products decline significantly. At optimum conditions, TiO<sub>2</sub> content in slag and the iron recovery are 70 wt.% and 84%, respectively. Kinetic studies proved that the reduction of equivalent FeO was first-order reaction. The studies made by X-ray diffraction analysis showed that formation of the titanium sub-oxides (Ti<sub>3</sub>O<sub>5</sub> and Ti<sub>2</sub>O<sub>3</sub>) causes the decrease in iron recovery and TiO<sub>2</sub> content.

In this paper author has worked on lowering ilmenite and carbon concentration by using below stated process. Titania-rich slag was prepared by smelting of ilmenite in an a.c. ESCM furnace. This furnace uses the graphite electrode (45 mm diameter) and graphite melting crucible (115 mm internal diameter) for

melting. In this process, the chemical separation of titanium and iron is based on the large difference between the stability of their oxides and physical separation of titania-slag and metallic iron is based on the difference between their densities .

Ilmenite reduction process has been performed in an electric arc furnace, rotary reduction kiln and plasma arc furnace. Recently, Electro-Slag Crucible Melting (ESCM) is used for smelting of ilmenite concentrate. The ESCM method melts the charge by slag layer heated by graphite electrode in refractory lined crucible. The conductivity of ESCM slag is low and when the slag is added to high titania slag leads to reduce the conductivity. Therefore ,during the melting, the slag acts as a resistance and most of the electrical current converts to the heat that is consumed for heating and melting the slag. In the first stage of process when arc was struck between the graphite electrode and the base of graphite crucible, ESCM slag was poured to the crucible gradually; then, after melting the ESCM slag, mixture of ilmenite and petroleum coke was poured in the crucible in five min. The time of reduction was accounted after pouring all of the charge in the crucible. During the melting process, temperature was controlled by infrared pyrometer and Pt–Pt/Rh thermocouple at 1600–1650 C. The amount of ESCM slag in all experiments was 5 wt.% of ilmenite. The charge was prepared by mixing 2kg ilmenite and the certain amount of petroleum coke depending on each experiment. The voltage and current of process was about 20 V and 400 A, respectively. In all experiments, after certain time, the melt was poured in metallic mould, and then the solidified iron and the slag were separated and weighed. In few experiments, which metallic iron could not be separated simply, the mixture of slag and residual iron was crushed and separated magnetically. The slag was analyzed for titanium and iron by standard wet chemical methods.

The author concluded that ,

- (1) The iron recovery and TiO<sub>2</sub> increase when the carbon amount increases up to stoichiometric amount, but when the carbon amount exceeded the stoichiometric amount, the iron recovery and TiO<sub>2</sub> content decrease significantly.
- (2) If the smelting time keeps between 0 and 17 min, the TiO<sub>2</sub> content and iron recovery are increased.
- (3) The formation of titanium sub-oxides leads to decrease the TiO<sub>2</sub> content of slag and iron production in the metallic phase.

**Vivek.r.gandhewar et.al [2]** indicated the study of various furnaces manufactured world wide. This paper shows the manufacturing and working principle of different types of furnaces and what material is used for combustion in the particular furnace. Induction melting furnaces in India were first installed to make stainless steel from imported SS Scrap. But in years 81-82 some entrepreneurs, who were having small size induction furnaces making stainless steel, experimented in making mild steel from steel melting scrap, they succeeded. More firms in northern India produced steel (Pencil Ingots) by using 500 kg to 1 tonne induction furnaces. Bigger size Induction furnaces were then installed first in North India and then in other states of India. By 1985-86, the technology of making mild steel by Induction Furnace route was mastered by Indian

Technicians. Induction furnace manufacturers saw the potential and started manufacturing bigger size/capacity furnaces. By 1988-89 period 3 tonne per charge induction furnaces were installed (became standard) all over India. The chemistry of melt was adjusted by adding mill scale, if opening carbon of bath was more. Good quality of steel melting scrap was used. In 1991-92, the Government license and control on steel making and rolling was removed. Then more induction furnaces were installed all over India. The use of sponge iron made it possible to adjust chemistry of melt. Thus good quality of Mild Steel pencil ingots are being produced with no tramp elements. Different types of induction furnaces are also mentioned in this paper.

In this paper the author is focusing on improving the efficiency of steel melting processes. After actually watching all the steel melting process, we came to know that, what are the various losses and where heat is lost.

Hence for improving its efficiency and for reducing the losses we have made recommendation like Scheduling of operations, Molten metal delivery, Preheating, No time delay in holding the molten metal, Reuse of hot gases, Using the good quality raw material, Proper charging practice. If this comes in regular practice obviously it helps to increase its efficiency. Material and energy losses during these process steps represent inefficiencies that waste energy and increase the costs of melting operations. Modifying the design and/or operation of any step in the melting process may affect the subsequent steps. It is, therefore, important to examine the impact of all proposed modifications over the entire melting process to ensure that energy improvement in one step is not translating to energy burden in another step.

Although these technologies require little or no capital for deployment, engineering assistance is needed for the facility to reap its maximum benefits. For example steel melting energy efficiency can be improved if there is no time delay in holding the molten metal where additional energy is required to maintain the temperature of the molten metal until it is poured. If a furnace can be scheduled so that it can operate at continuous full power, the energy requirements for melting steel can be reduced substantially. While reviewing the literature, it is observed that till this time an attempt is not made by the concerns to study the impact of variation of one parameter on others. Through the planned design of experimentation, the effect of individual parameters can be studied and its overall effect on productivity can be found out.

**B.R.Nijhwana et.al [3]** This paper starts with an outline of established methods of manufacture of different ferro-alloys required for making alloy steels with a comparison of their merits as judged by the products made.

The ferro-alloys discussed include those based on manganese, chromium, silicon, tungsten, vanadium and phosphorus and of different qualities and grades. The position of the production of the individual Ferro-alloys in India is discussed and the great importance emphasized of expanding production of those required in relation to the expansion of the Indian iron and steel industry. A program of Ferro-alloy production is outlined, with discussion of availability of raw materials, manufacturing capacity and economic factors.

Different methods of producing Ferro alloys are discussed in this paper.

The functions of a Ferro-alloy are threefold:

- (i) To deoxidize and clean the molten bath
- (ii) To control the solidification of the metal, and
- (iii) To give the end product the desired physical characteristics such as, tensile strength toughness, ductility, hardenability, corrosion resistance, etc.

The maximum consumption of ferroalloys in the iron industry is for the purpose of deoxidizing and cleaning the steel.

There are three methods of manufacturing Ferro-alloys discussed in this paper:

- (1) Blast furnace method,
- (2) Electric furnace method, and
- (3) Alumino thermal method.

Also the manufacturing of different Ferro alloys is discussed in this paper.

**R.W.Bebbington et.al [4]** this paper indicates some of the reasons why boron and titanium are added to steels, especially the stainless varieties. It mentions the addition techniques for introducing the elements into the steel, and highlights the production routes for the two ferroalloys. Boron markedly affects the mechanical properties of stainless steels, specifically their hot shortness, creep resistance, intergranular corrosion resistance, neutron-absorption capacity. Similarly, Titanium is highly reactive in steelmaking processes with elements such as carbon, oxygen, nitrogen and Sulphur. While this accounts for some of the desirable properties, it also leads to some of the difficulties. Titanium additions can be made in the form of metal scrap, sponge or, more normally, as a ferrotitanium alloy. Originally, ferrotitanium was supplied as a 40 per cent titanium alloy made by the alumino thermic process. With the increasing use of titanium metal in the aerospace industry, large volumes of turnings and other forms of titanium scrap became available at relatively low prices. This led to the present production route, which converts the titanium scrap into a eutectic grade containing 70 per cent titanium.

Production of Ferrotitanium:

Raw materials for the manufacture of ferrotitanium are purchased from around the world from approved sources. They can be in many forms, such as turnings, sheets, heavy sections, Careful sampling is required; this is not easy in the case of mixed lots of turnings and small solids, but is relatively easy on large plate. The materials are subjected to crushing, degreasing for turnings, or cutting to size for solids. Blends are made up and charged into the coreless induction furnace with ferrous scrap. The reaction is extremely vigorous

and aggressive on furnace linings. After alloying, the melt is poured into a large cast-iron pan and, after solidification, the material is crushed, sieved, blended, and packed.

Addition of boron to steel:

Boron is generally added to steel in the form of Ferro boron. It combines aggressively with oxygen and nitrogen dissolved in steel, and it is thus necessary to exercise care in steelmaking to prevent such reactions; otherwise, the effectiveness of the boron will be irretrievably lost.

A standard practice is to reduce the oxygen levels to a minimum, probably with a calcium silicon treatment, and then to add ferroboration to the ladle between the time the ladle is 1/4 to 3/4 full, but certainly after all the other alloying additions have been made. Precautions against re-oxidation of the heat are recommended. Recoveries of 60 to 65 per cent are normal, and up to 80 per cent can be achieved with carefully controlled practice.

The author concluded that, how a ferroalloy producer can be of service to the steel industry, the key areas for both the alloy producer and the steelmaker being those of

Improving the quality and developing new and better products.

In line with these aims, it is felt that a greater dialogue between the customer and the producer can be of mutual benefit both commercially and technically.

**Ari Saastamoinen et.al [5]** has studied about the effect of titanium and nitrogen content on the austenite grain structure, hardenability and mechanical properties of laboratory-scale direct-quenched and tempered martensitic 0.16C-0.2Si-1.1Mn-0.40Mo-0.60Cr-0.04Al-16B steels have been studied. Titanium (0.01-0.03 wt.%) and nitrogen (30-60 ppm) contents have been varied resulting different levels of excess titanium above the stoichiometric amount for TiN.

Furthermore, two tempering temperatures (210 and 600 °C) were applied for laboratory hot rolled and direct-quenched steels. Austenite grain structure investigations have been performed with light optical microscopy and general microstructure investigations using field emission scanning electron microscope.

It was found that Ti/N ratios close to stoichiometry, i.e. 2.4-4.2, provided both the finest austenite grain structure and the best impact toughness after hot rolling and direct quenching. Excess of titanium was shown to contribute to secondary hardening during high-temperature tempering, while excess of nitrogen had a significant negative effect on the hardenability. In this study, the effect of titanium and nitrogen contents on the hardenability, prior austenite grain structure and mechanical properties of direct-quenched martensite will be investigated. Furthermore, the effects of both low-temperature tempering (LTT) and high-temperature tempering (HTT) on the properties of direct-quenched and tempered martensite with several different titanium and nitrogen contents will be investigated.

To investigate the hardenability of compositions, continuous cooling transformation (CCT) diagrams were determined using dilatometric data obtained with a Gleeble 1500 thermo-mechanical simulator. In the Gleeble, the specimens were heated to 1100 °C with 20 °C/s heating rate and held for 4 minutes. After



soaking time samples were cooled at 2 °C/s until 850 °C and held for 10 seconds. After the holding time three compression were performed with strain of 0.2 and strain rate of 1 s<sup>-1</sup>. Compressions were performed with 25 seconds intervals. After the final compression, samples were held at 850 °C for 25 seconds and cooled into room temperature with various cooling rates ranging from 1.5 to 96 °C/s. The diameter of cylinder specimens was measured using a strain gauge during the compression tests and cooling. Specimens were later cut into half along the compression axis for hardness measurements and microstructural characterization.

Hot rolling experiments were performed with laboratory scale hot rolling and direct quenching equipment. After reheating at 1080 °C for 2 hours, a 60 mm thick slabs to a final thickness of 10 mm and direct quenched immediately after rolling to room temperature at about 50–100 °C/s. The pass schedule was designed so that the last passes were below the austenite recrystallization temperature to obtain pancaked Austenite, tempering trials were performed using two different tempering processes: LTT (peak temperature of 210 °C) and HTT (peak temperature of 600 °C). The soaking time at the peak temperature in both tempering regimens was 30 minutes. Average heating rate during tempering was 0.5 °C/s. After tempering, specimens cooled freely in air. Actual peak temperatures and heating rates were measured with thermocouples.

Hardnesses (HV5 or HV10) were measured using a Duramin-A300 (Struers). Charpy-V impact tests were performed at -40 °C and -60 °C (2 specimen/temperature) using 10 x 10 x 55 mm<sup>3</sup> sized specimens according to standard ISO 148-1:2010 [1]. Longitudinal tensile tests were performed using round bar specimens according to standard ISO 6892

The prior austenite grain structure was revealed using picric acid etching. After etching, light optical microscope (LOM) was used to determine prior austenite grain structure parameters with the mean linear intercept method on transverse sections containing the rolling direction (RD) and plate normal direction (ND) at the quarter-thickness position. Furthermore, general microstructure were studied using nital etching and field emission scanning electron microscope (ZEISS SIGMA FESEM).

**J.PIWNIK et.al [6]** this paper states that In order to improve the tribological properties of titanium alloy Ti6Al4V composite surface layers Ti/TiN were produced during laser surface gas nitriding by means of a novel high power direct diode laser with unique characteristics of the laser beam and a rectangular beam spot. Microstructure, surface topography and micro hardness distribution across the surface layers were analyzed. Ball-on-disk tests were performed to evaluate and compare the wear and friction characteristics of surface layers nitrided at different process parameters, base metal of titanium alloy Ti6Al4V and also the commercially pure titanium. Results showed that under dry sliding condition the commercially pure titanium samples have the highest coefficient of friction about 0.45, compared to 0.36 of titanium alloy Ti6Al4V and 0.1-0.13 in a case of the laser gas nitrided surface layers. The volume loss of Ti6Al4V samples under such conditions is twice lower than in a case of pure titanium. On the other hand the composite surface layer

characterized by the highest wear resistance showed almost 21 times lower volume loss during the ball-on-disk test, compared to Ti6Al4V samples.

The specimens of titanium alloy Ti6Al4V were cut from a hot-rolled rod with a diameter of 50.0 mm. Next the disks were machined by milling and turning to a nominal diameter of 46.0 mm and a thickness of 3.0 mm. In turn, the specimens of commercially pure titanium were cut from a flat sheet 3.0 mm thick and then the specimens were machined to a predetermined dimension of test disks. Prior to the laser nitriding the test disks were mechanically ground and rinsed with acetone. Disks were mounted in a rotary drive and set horizontally. The rectangular laser beam was focused on the top surface of disk specimens and set transversely to the travel direction. Pure gaseous nitrogen was fed through a cylindrical nozzle with a diameter of 12 mm. The nitrogen flow rate was kept at 18 l/min. The nitrided single beads had a diameter of 30 mm. The test nitrided surface layers were produced at constant scanning speed of 200 mm/min and different laser output power from 400 to 1000 W, thus different heat input.

The test surface layers produced during laser gas nitriding of the titanium alloy Ti6Al4V were examined by visual testing, surface topography analysis and tribological analysis. The surface topography was measured and analyzed by an optical, non-contact Profilograph Micro Prof 100 FRT. After visual examinations and surface topography analysis some of the samples were sectioned and polished by 180, 400, 600, 1200, 1500 grit SiC abrasive papers and 0.5  $\mu\text{m}$  diamond paste. The metallographic samples were etched by Kroll reagent. The microstructure of base metal and surface layers were examined and analyzed by an optical and also by scanning electron microscopy (SEM).

The tribological characteristic of the titanium alloy substrate Ti6Al4V, commercially pure titanium and the test nitrided surface layers was evaluated by a ball-on-disk tribometer T-01M under room temperature of 23°C, according to the ASTM G99 standard. The relative humidity was about 55% $\pm$ 5%. Steel balls with a diameter of 10.0 mm were used as the counterface material. The normal load was set as 30 N. The sliding distance was 188.4 m, while the sliding speed was 0.13083 m/s. The tangential force of friction and displacement value were continuously measured and recorded during tests using a data acquisition system with PC computer.

This study has shown that the laser gas nitriding of titanium alloy Ti6Al4V by the HPDDL (high power direct diode laser) laser leads to a significant increase in wear and friction characteristic. Surface layers produced during HPDDL nitriding are cracks free. The tribological characteristics of the nitrided surface layers are related to the processing parameters and thus to the surface topography, morphology and phase composition. Surprisingly the wear resistance of nitrided surface layers during ball-on-disk test is not directly correlated to micro hardness. The highest wear resistance showed the test surface layer produced at the minimum laser power (thus minimum heat input) and characterized by the lowest Vickers microhardness. The mechanism of wear is combined adhesion and abrasion.

**Kandula Ankamma et.al [7]** this paper has highlighted on the effort that has been made to correlate the "Effect of Titanium in trace level on the tensile strength, hardness and microstructure of gray cast iron". Titanium in trace level has a significant effect on the properties and microstructure of gray cast iron.

Titanium is deliberately added to study its effect on properties and microstructure. Titanium up to 0.05% has shown a decrease in tensile strength and hardness values, while beyond this amount the properties are slightly improved.

To avoid trace element contamination in charge materials, all bought in scrap should be examined prior to the stockpiling so that undesirable charge materials can be removed. In the present work, the effect of Titanium on the mechanical properties and graphitic structure at trace level on gray cast iron are studied. Titanium is deliberately added to the gray cast iron to know its effect on the properties of gray cast iron. The experimental work was carried out at TELCO, Jamshedpur.

Gray Cast iron Composition: On the basis of elements present in gray cast iron can be divided into three categories

- Major elements,
- Minor elements
- Trace elements.

The experimental procedure carried throughout is stated as:

- Molding
- Melting
- Charging sequence
- Inoculation
- Titanium addition
- Casting
- Specimen preparation

Further, they also performed few tests for testing validation:

- Tensile strength test: This test was carried out on a Universal testing machine of 60 ton capacity in mechanical testing laboratory at TELCO, Jamshedpur. The load on the test specimen was applied steadily till fracture occurs.
- Hardness test: Brinnels hardness test was carried out using a standard hardness testing machine. The steel ball diameter of the indenter is 10 mm and the load applied was 3000 Kg. hardness values are measured at three different places across the cross section of test piece and the average of three values are noted.
- Metallography: The samples for photo micro graphs are prepared according to the standard procedure. Around 20 mm thick sample was taken from the fractured tensile test bars. It is polished first on a belt grinder, emery papers of varying specifications from 01 to 03 and finally on a cloth grinder to get a mirror finish to the sample. The microstructure was observed using microscope at 100X magnification without etching and with etching with 2% Nital.

The author concluded that the microstructure of base gray cast iron consists of type A graphitic structure, with pearlitic matrix. With increasing additions of titanium, both the tensile strength and hardness values show decreasing trend upto 0.05% titanium addition. Beyond 0.05% titanium these values have again

revealed improvement in tensile strength and hardness. So care should be taken to avoid the contamination of titanium in trace level from pig iron, non-ferrous metal scrap, vitreous enameled scrap, leaded steel scrap, purchased scrap containing lead or coated with lead based paint. To avoid trace element contamination in charge materials, all bought in scrap should be examined prior to the stockpiling so that undesirable charge materials can be removed.

### 3. EXPERIMENTAL MACHINES

List of Machines were used:-

- 3.1 Induction Furnace
- 3.2 Vibrating cup mill
- 3.3 Press Pellet Machine
- 3.4 LECO TC 230
- 3.5 LECO TC 500
- 3.6 WDXRF

#### 3.1 Induction Furnace

An induction furnace is an electrical furnace in which the heat is applied by induction heating of metal. Induction furnace capacities range from less than one kilogram to one hundred tonnes, and are used to melt iron and steel, copper, aluminium, and precious metals. The advantage of the induction furnace is a clean, energy-efficient and well-controllable melting process compared to most other means of metal melting.

Most modern foundries use this type of furnace, and now also more iron foundries are replacing cupolas with induction furnaces to melt cast iron, as the former emit lots of dust and other pollutants.

Since no arc or combustion is used, the temperature of the material is no higher than required to melt it; this can prevent loss of valuable alloying elements. The one major drawback to induction furnace usage in a foundry is the lack of refining capacity; charge materials must be clean of oxidation products and of a known composition and some alloying elements may be lost due to oxidation (and must be re-added to the melt).



Figure1. Ref. Minex metallurgy, Nagpur

### 3.2 Vibrating Cup Mill

In the Vibrating Cup Mill, the grinding is performed by horizontal circular oscillations of the grinding set on a vibrating plate. The grinding set consisting of ring and puck comminute the grinding sample with extremely high pressure, impact forces and friction. In this form of grinding, the transmission of forces onto the grinding sample is much more important than the pure motor power. A special motor and now equipped it with an especially interference-resistant, torque-optimised frequency converter which fulfils all the relevant safety standards worldwide. It ensures that the motor output is precisely matched to the grinding material and grinding set - which optimises the energy consumption.



Figure2.Ref. <https://lavallab.com/products/crushers/vibratory-disc-mill/>

### 3.3 Press Pellet Machine

A pellet mill, also known as a pellet press is a type of mill or machine press used to create pellets from powdered material. Pellet mills are unlike grinding mills, in that they combine small materials into a larger, homogeneous mass, rather than break large materials into smaller pieces.

The powder is introduced to the top of the die and as the die rotates a roller presses the powder through the holes in the die. A cutter on the other side of the die cuts the exposed pellet free from the die. In the ring die there are radial slot throughout the die. Powder is fed into the inside of the die and spreaders evenly distribute the powder. Two rollers then compress the powder through the die holes. Two cutters are used to cut the pellets free from the outside of the die.



Figure 3 .Ref. Minex metallurgy, Nagpur



Figure 4 .Ref. (PELLET) Minex metallurgy, Nagpur

### 3.4 LECO TC (C/S) 230

When LECO CS230 carbon sulfur analyzer works, its operational principle is first to put a certain quality of sample into a high-frequency magnetic field which has been passed through an oxygen stream. The sample and flux would be induced and heating, combusting in oxygen, carbon and sulfur of sample reacts with oxygen to produce carbon dioxide and sulfur dioxide which will accompany with the carrier gas into the gas system. They will first to reach the sulfur dioxide detector testing pool to determine sulfur, and then using thermal oxidation of copper to convert carbon monoxide into carbon dioxide. Next, sulfur dioxide will be converted into sulfur trioxide, and then it would be absorbed by the plastic which has a strong absorbability. Then, the sample gas would go through the high and low content of carbon dioxide of the infrared detector cell to detect carbon.



Figure 5 .Ref. Minex metallurgy, Nagpur

### 3.5 LECO TC (O/N) 500

The TC500 is a microprocessor-based, software-controlled instrument that measures both nitrogen and oxygen in a wide variety of metals, refractories, and inorganic materials. The inert gas fusion principle is employed. A weighed sample, placed in a high-purity graphite crucible, is fused under a flowing helium gas stream at temperatures sufficient to release oxygen, nitrogen, and hydrogen. The oxygen in the sample, combines with the carbon from the crucible to form carbon monoxide. The nitrogen present in the sample releases as molecular nitrogen, and any hydrogen present is released as hydrogen gas. The helium carries

the sample gases through heated rare earth copper oxide which converts carbon monoxide to carbon dioxide (CO) and hydrogen to water (H<sub>2</sub>O). The nitrogen passes through unchanged. The gases are then passed through a CO infrared (IR) cell where the oxygen is measured as CO. CO and H<sub>2</sub>O are then removed by a Lecosorb /Anhydron trap, while the nitrogen passes through to a thermal conductivity (TC) cell for determination.



Figure 6 .Ref. Minex metallurgy, Nagpur

### 3.6 WDXRF (WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE).

Wavelength Dispersive X-ray Fluorescence (WDXRF) is one of two general types of X-ray Fluorescence instrumentation used for elemental analysis applications. In WDXRF spectrometers, all of the elements in the sample are excited simultaneously. The different energies of the characteristic radiation emitted from the sample are diffracted into different directions by an analyzing crystal or monochromator (similar to the action of a prism dispersing different colors of visible light into different directions). By placing the detector at a certain angle, the intensity of X-rays with a certain wavelength can be measured. Sequential spectrometers use a moving detector on a goniometer to move it through an angular range to measure the intensities of many different wavelengths. Simultaneous spectrometers are equipped with a set of fixed detection systems, where each system measures the radiation of a specific element. The principle advantages of WDXRF systems are high resolution (typically 5 – 20 eV) and minimal spectral overlaps.





Figure 7 .Ref. Minex metallurgy, Nagpur

#### 4. EXPERIMENTAL RESULT ASSUMPTION

The pallet formed and tested shows the following result when tested in various machines for 1 gm of ferrotitanium

LECO TC 230

Carbon- 4 ppm or up to 4% by weight

Sulphur - 0.4 ppm or up to 0.4% by weight

LECO TC 500

Nitrogen - 0.5 ppm or maximum up to 2% by weight

Oxygen - 0.5 ppm or up to 0.4 % by weight

WDXRF

ELEMENT	Titanium (Ti)	Silicon (Si)	Aluminium (Al)	Carbon (C)	Vanadium (V)	Iron (Fe)
COMPOSITION WEIGHT %	65-70%	1-1.5%	6% max	0.2 %max	3% max	Remaining percentage.

Table 1



Figure8. Ferrotitanium lumps - Ref. Minex metallurgy

## 5. EXPERIMENTAL ANALYSIS OF FERROTITANIUM

As we obtained the lumps of ferrotitanium through above given manufacturing methodology now we analyse the composition of individual elements.

Analysis is done in either ways in order to increase the accuracy of tracing elements

First analysis is done on WDXRF (WAVELENGTH DISPERSIVE XRAY FLUORESCENCE) and simultaneously other is done on ICP (INDUCTIVE COUPLED PLASMA OPTICAL EMISSION SPECTROMETER)

### 5.1 Analysis on WDXRF

Analysis is done through following these procedures:-

### ❖ **STEP 1 : Breaking of Lumps**

After obtaining solidified ferrotitanium it is further broken down into lumps through quarter cornering. Now these lumps of random shapes are then forwarded for grinding in vibrating cup mill machine.

### ❖ **STEP 2 : Grinding of lumps in Vibrating cup mill**

These lumps are grinded up to 240 mesh that is in powder form. Now this powder is taken in Press pellet machine.

### ❖ **STEP 3 : Making pellets**

In press pellet machine the powder form of Ferrotitanium is put under 25 ton of pressure for molding it into pellet form and is then forwarded to different machines for tracing the composition of element.

### ❖ **STEP 4 : Final analysis in WDXRF**

Now the pellet is placed in tracing platform of WDXRF machine where the software is used to trace the elements by weight analysis of ferrotitanium.

## **5.2 Analysis through ICP (inductive coupled plasma optical emission spectrometer)**

### ❖ **STEP 1 : Acid fuming chamber**

In this step the grind (240 mesh) lumps are directly taken to acid fume chamber where it is cleaned by acid for accurate tracing of individual elements.

### ❖ **STEP 2 : Tracing through ICP**

The grind lumps are dried at 105 degree Celsius and placed in ICP for accurate tracing of individual elements.

As we know that there are two ways to identify traces but Minex metallurgy mostly use WDXRF for analysis of its lumps so we also tested the lumps in WDXRF. Below are the results of the analysis.

For obtaining accurate results analysis from batch of 1 ton of ferrotitanium, lumps were randomly selected and placed in four different in loose pouch packing bags.

Lumps were then made in pellet formation and then tagged as BAG-1, BAG – 2, BAG – 3, BAG – 4.

After placing in WDXRF consecutively these compositions were traced

**RESULTS BY WEIGHT%****Test result from BAG 1**

Sample ID	% Ti	%Si	%Al
Bag-1	66.03	1.20	2.20
Test Method	ISO-7692-1983(E)	SOP/CLMM/XRF/01-03	SOP/CLMM/XRF/01-03

%V	%C	%S	%Fe
0.576	0.117	0.012	Balance
SOP/CLMM/XRF	SOP/CLMM/CS/01	SOP/CLMM/CS/01	balance

**Test result from BAG 2**

Sample ID	% Ti	%Si	%Al
Bag-2	66.09	1.26	2.91
Test Method	ISO-7692-1983(E)	SOP/CLMM/XRF/01-03	SOP/CLMM/XRF/01-03

%V	%C	%S	%Fe
0.614	0.131	0.013	Balance
SOP/CLMM/XRF	SOP/CLMM/CS/01	SOP/CLMM/CS/01	balance

**Test result from BAG 3**

Sample ID	% Ti	%Si	%Al
Bag-2	66.12	1.18	2.23
Test Method	ISO-7692-1983(E)	SOP/CLMM/XRF/01-03	SOP/CLMM/XRF/01-03

%V	%C	%S	%Fe
0.631	0.134	0.014	Balance
SOP/CLMM/XRF	SOP/CLMM/CS/01	SOP/CLMM/CS/01	balance

**Test result from BAG 4**

Sample ID	% Ti	%Si	%Al
Bag-2	66.14	1.01	2.21
Test Method	ISO-7692-1983(E)	SOP/CLMM/XRF/01-03	SOP/CLMM/XRF/01-03

%V	%C	%S	%Fe
0.650	0.155	0.006	Balance
SOP/CLMM/XRF	SOP/CLMM/CS/01	SOP/CLMM/CS/01	balance

## Average Test Result

Sample ID	% Ti	%Si	%Al
	66.10	1.16	2.39
Test Method	ISO-7692-1983(E)	SOP/CLMM/XRF/01-03	SOP/CLMM/XRF/01-03

%V	%C	%S	%Fe
0.618	0.134	0.011	Balance
SOP/CLMM/XRF	SOP/CLMM/CS/01	SOP/CLMM/CS/01	balance

## COST ESTIMATION

The cost estimation under is approximate and may vary according to different taxes and demands.

### 1. MACHINES

#### 1.1 PRODUCTION MACHINE

SR NO.	MACHINE	COST(INR)
1.	INDUCTION FURNACE	92 LAKH
2.	VIBRATING CUP MILL	15 LAKH
3.	PRESS PALLET	20 LAKH

**1.2 TESTING MACHINE**

SR NO.	MACHINE	COST (INR)
1.	ICP (INDUCTIVE COUPLED PLASMA OPTICAL EMISSION SPECTROMETER)	1 CRORE
2.	LECO TC 230	60LAKH
3.	LECO TC 500	65 LKH
4.	WDXRF (WAVELENGTH DISPERSIVE X-RAY FLUROSCENCE)	80 LAKH

**2. RAW MATERIALS**

SR NO.	RAW MATERIAL	COST (PER KG INR)	TOTAL COST
1.	MILD STEEL SCRAP (300 KG)	25	7500
2.	TITANIUM SCRAP (700 KG)	290	2,03,000
3.	FERRO SILICON (2 KG)	75	150

**3. FINAL MATERIAL**

1 TON OF FERRO TITANIUM = INR 2, 80,000

## CONCLUSION

- After testing pallet in LECO TC 230 and LECO TC 500 we conclude that negligible amount of traces of Nitrogen, Sulphur, Oxygen and Carbon are found.
- Ratio of titanium to iron is 70:30
- Sample weight is inversely proportional to carbon composition.
- The ferrotitanium manufactured is suitable to increase the strength and ductility of steel.

## REFERENCE

### Research papers

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### Figures

1. Induction furnace- Minex Metallurgy Nagpur
2. Vibrating cup mill – Google Images
3. Press Pallet – Minex Metallurgy Nagpur
4. LECO TC 230- Minex Metallurgy Nagpur
5. LECO TC 500- Minex Metallurgy Nagpur
6. WDXRF - Minex Metallurgy Nagpur
7. FERROTITANIUM LUMPS – Google Images