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**FINAL REPORT ON  
LABORATORY REDUCTION TESTS CONDUCTED  
ON ABUGHALAGA ILMENITE SAMPLES  
AT  
SIIL TEST CENTRE**

**JUNE – 1991**



**ENGINEERING AND PROJECTS DIVISION  
SPONGE IRON INDIA LIMITED  
HYDERABAD - 500 028.**

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ON ABUGHALAGA ILMENITE SAMPLES  
AT  
SILL TEST CENTRE

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**FINAL REPORT ON LABORATORY REDUCTION TESTS**  
**CONDUCTED ON ABUGHALAGA ILMENITE SAMPLES AT SIIL TEST**  
**CENTRE**

**1.0 INTRODUCTION**

- 1.1 United Nations Industrial Development Organization (UNIDO) under the Project No.XP/EGY/90/018. "Laboratory and Bench Scale testing of Abughalaga Ilmenite ore for metallurgical processing to titania slag and low phosphorous Pig Iron" awarded the test work to sponge Iron India Limited (SIIL) vide their Contract No.90/118. The contract was awarded in response to the proposal submitted by SIIL vide their letter No.SI/E&P/RO/129/90 dt.02.05.1990.
- 1.2 The total project work is divided into two parts viz. Part-I Production of Pre-reduced Ilmenite and Part-II Production of Titania rich slag by electrosmelting. The first part of the work i.e. production of pre-reduced Ilmenite was entrusted to SIIL (contractor-I) and the Second part electrosmelting of the prereduced ilmenite for production of titania rich slag and raw ilmenite was awarded to M/s. Titanium Institute, Zaporozhya, USSR (Contractor -II).
- 1.3 In terms of the contract the scope of work of SIIL (Contractor - I) is as follows:
- 1.3.1 To carry out laboratory/bench scale pre-reduction testing (with coal as solid reductant) of one characteristic ilmenite sample (about 600 Kg.) to ilmenitic sponge iron (degree of metalization at least 80%) suitable for consequent electrosmelting to titania rich slag and pig iron.
- 1.3.2 To determine the technical parameters of the pre-reduction process, specific consumption data and quality/yield of the final report.
- 1.3.3 To prepare a Progress Technical Report of the results from the tests with assessment and conclusion about suitability of the raw material for direct reduction.

In realization of the above scope of services, the Contractor is required to carry out the following main activities:

a) Carrying out laboratory physico-chemical and mineralogical analysis of the characteristics ilmenite ore sample (600 Kgs.) according to Contractor's methodology. Technical and chemical analysis of the solid reductant (coal) to be used in the direct reduction tests.

b) Carrying out laboratory/bench scale Direct Reduction testing with the ilmenite ore sample according to Contractor's technology and methodology. the contractor has to be in possession of appropriate laboratory and bench scale facilities proven for determination of all technical process parameters, specific consumption data and product yield and quality.

c) Preparation and transportation of the pre-reduced ilmenite ore sample (about 550 Kgs. ilmenite sponge iron) has per "b" above to the testing facilities of contractor No.2 for electrosmelting. The sample has to be accompanied by appropriate technical characteristics certificate.

d) Preparation and submission of progress technical reports with all tests results, techno-economic calculations, appraisals and conclusion.

e) Participation in final evaluation and review of testing results and discussion on progress reports in Egypt together with consultants from contractors No.2 (Electrosmelting tests) and UNIDO's back-stopping professional staff.

1.4 600 Kgs. of raw ilmenite was received from Government of Egypt for undertaking the reduction tests and producing pre-reduced ilmenite. The samples were received at SIIL Test Centre in 3rd week of May, 1990. As per the directions of UNIDO suitable non-coking coal was used as reductant for the tests. After receipt of ilmenite sample, detailed schedule of test work was drawn and accordingly the work was undertaken.

1.5 This report discusses the details of tests conducted on Abughalaga Ilmenite ore in combination with Manguru coal of Singareni Collieries, as reductant. The report also indicates the optimum test conditions as also the test results of both laboratory rotary furnace and short

rotary kiln tests conducted at SILL Test Centre on  
this set of raw materials.



## 2.0 TEST OBJECTIVE

- 2.1 The prime objective of the test work is to determine by laboratory and bench scale testing the suitability of Abughalaga ilmenite ore and available coal (Manuguru) as reductant for production of pre-reduced ilmenite by Direct Reduction Process and to assess the main technical parameters and product quality for industrial processing.
- 2.2 Keeping in view the above prime objective and the scope of test work, a detailed schedule of test work was drawn up which is enumerated below:
  - 2.2.1 To analyse the Abughalaga ilmenite sample chemically to determine its various constituents like Fe, TiO<sub>2</sub>, and other tramp elements.
  - 2.2.2 To determine physical properties such as petrological studies of sample, screen analysis, Bulk Density, shatter and tumbler indices etc.
  - 2.2.3 To carry out reduction tests in Laboratory rotary furnace to ascertain the reducibility characteristics of the ilmenite ore and to determine optimum test parameters.
  - 2.2.4 To produce pre-reduced ilmenite in short rotary kiln and to determine the technical parameters and product quality.

### **3.0 TEST MATERIAL**

#### **3.1 Ilmenite**

500Kgs. of Raw Ilmenite Ore was received from Government of Egypt. The chemical analysis and physical properties of the as received samples of Ilmenite ore, as analysed at SIIL test centre are given at table 1 & 2.

#### **3.2 COAL**

Coal from Manguru Mines of Singareni Collieries Company Limited which is being used in SIIL plant for production of Sponge Iron has been considered as reductant in the proposed test work. The chemical and physical properties of the coal sample used in the tests is presented in table 3 & 4.

#### **3.3 Desulphariser (Lime Stone)**

Small quantity of desulphariser is to be used in the reduction tests to restrict pickup of sulphur from coal by the pre-reduced Ilmenite during reduction tests. Lime stone used in the SIIL plant has been considered as desulphariser for the tests. The screen analysis and the chemical analysis of the lime stone used in the tests are presented at Table-5.

#### 4.0 DESIRED TECHNICAL QUALITY REQUIREMENTS OF RAW MATERIALS TO BE USED IN DIRECT REDUCTION PROCESS USING ROTARY KILN:

4.1 Iron bearing ore, Coal and Limestone are the basic raw materials required for the production of pre-reduced material in the rotary kiln process based on 100% coal operation. As the processing of iron bearing material for reduction in rotary kiln is sensitive to the characteristics of raw materials with regard to size distribution and other chemical parameters, bench scale testing forms the first essential step in determining the suitability of the raw materials and deciding about the process parameters.

#### 4.2 **IRON BEARING MINERAL (Ilmenite)**

For better reduction and enrichment of the desired elements in the reduced product, the basic ore should have iron content of the order 50% with low gangue components and low levels of impurities such as sulphur and phosphorus. The requirement of iron content in the mineral arises from the fact that higher the iron content, it is better the reduction. Low level of gangue are required as there is no removal of gangue in the direct reduction process is envisaged. Further gangue requires additional melting power and appropriate limestone additions to remove it as slag in the smelting stage. After reduction of iron oxide the weight of the mineral reduces to the extent of removal of oxygen the other mineral present improves proportionality.

The sulphur and phosphorus content are also of utmost importance while selecting the ores keeping in view the specification of the final metal to be produced. Suitable precautions are to be taken for desulphurisation in the rotary kiln, as the reduced material tends to pick up the sulphur from coal in the absence of desulphuriser. Apart from the chemical characteristics as above, the iron bearing mineral has to satisfy certain minimum requirements with regard to physical strength. Shatter, Tumbler and Abrasion indices give an indication of the physical strength.

Based on bench scale tests of different ores and studies on the decrepitation behaviour in the rotary kiln of the SILL plant, it is noted that shatter index of ores should not be less than 90%.

#### 4.3 Coal

The main chemical characteristics of the coal which influence its suitability as the reductant are reactivity, proximate analysis comprising of fixed carbon, ash and volatile matter, melting characteristics of coal ash under reducing conditions, the total sulphur and the different forms of sulphur present. Coals of higher reactivity are preferable as they permit the operation of the kiln at the lower temperatures and at high throughput rates. The ash in coal should be as low as possible as it occupies the effective kiln volume reducing the space available for iron bearing materials. The volatile matter in coals should be of the order of 30% so as to heat the ore to the reduction temperature within the shortest possible time. The fixed carbon should be of the order of 40 to 45 percent. The melting characteristic of coal ash is of utmost importance while evaluating coals for direct reduction application. As the coal ash forms low melting compounds with fine reduced material, it is desirable to have softening point of coal ash in excess of 1200 deg.C under reducing conditions. The kiln operating temperature in the reduction zone is so chosen that it is lower than the ash softening point by 100 - 150 deg.C to minimise the formation of accretions. The sulphur content in the coal, in the form of organic and inorganic compounds, also merits careful consideration. Part of organic sulphur gets volatilised in the pre-heating zone of the kiln and increases the sulphur load in the waste gas system. Organic sulphur tends to get released in the reduction zone along with the utilisation of carbon and gives rise to sulphur pick up in the reduced material in the absence of the desulphuriser. In short the total sulphur in coals should be low, preferably below 1%. However, coals having high percentage of fixed carbon with low volatile matter like anthracite, could also be considered as effective reductant through blending with bituminous or sub-bituminous coal or in combination with oil or natural gas. Alternatively, such coals could also be used by maintaining higher operating temperatures when the reactivity of the coal improves. The related problems of ash softening and subsequent fusion at higher operating temperatures are absent if the ash content is low.

#### 4.4 Limestone

Limestone is used in the process, as a desulphuriser as the coal used for reduction contains sulphur. Limestone containing an average

of 45 percent of CaO has been observed to be adequate for this requirement. The size distribution of limestone also needs to be considered as it is observed that the desulphurising ability of finely granulated limestone is very good. The size range of 1 to 3 mm is found to be very effective in rotary kilns. The property of limestone used in the tests are given in table-5.

## 5.0 LABORATORY AND BENCH SCALE TEST RESULTS

### 5.1 Chemical Analysis of Abughalaga ilmenite Sample

The ilmenite sample was analysed as per the standard analytical procedure. Various constituents such as Fe (Total), Fe (Met.), FeO, Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, MgO, P, S, Cr<sub>2</sub>O<sub>3</sub>, MnO, BaO, Cu & Na were determined and the results are presented in Table-1.

#### 5.1.1 Iron content

The total iron content present in the Abughalaga ilmenite sample was found to be 39.70%. The detailed analysis to find different forms of iron present in the ore revealed that the iron in the mineral is present in the two forms i.e. Fe<sup>+2</sup> and Fe<sup>+3</sup>. This was also revealed in the petrological study conducted on the ore. The analysis results shows that FeO content is 24.69% and Fe<sub>2</sub>O<sub>3</sub> content is 29.32%. As can be seen from the chemical analysis of the ore, the iron is associated with the oxygen in magnetite form. Generally magnetite type ores are poor in reducibility character. This was also revealed in the reduction tests conducted on the ilmenite ore at an operating temperature of 1090 deg.C with six hours reduction time only, a metallization of 66% was obtained. Whereas for normal type of ores that are being used in SILL for production of Sponge Iron a metallization of 90% can be achieved at an operating temperature of 1000 deg.C and 3 hours reduction time. From the point of view of reduction, the iron content in the ilmenite can be said to be on the lower side.

#### 5.1.2 Titanium content

Titanium is the fourth most abundant structural metal and ninth most abundant element in earth crust. Titanium normally occurs in natural minerals as its oxides and mostly forms the stable TiO<sub>2</sub> compound. The ilmenite sample was analysed as per standard procedure to find the Ti content in the ore. The chemical analysis presented in table - 1 shows that TiO<sub>2</sub> content in the Abughalaga ilmenite sample is of the order

37.20%.

#### 5.1.3 Gangue content

The gangue content in the ore constitutes SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> which are undesirable in the recovery of prime metal. The gangue content in the ilmenite as analysed is of the order 3.93%, in which SiO<sub>2</sub> constitutes 2.62% and Al<sub>2</sub>O<sub>3</sub> constitutes 1.31%. The total gangue content in the ore is on lower side compared to other prime iron ores.

#### 5.1.4 Impurities

The impurities normally present in the ore is phosphorous and sulphur. As can be seen from the chemical analysis the impurities level in the ilmenite is on lower side.

### 5.2. **Physical tests on ilmenite**

5.2.1 The physical tests like petrological studies, bulk density, shatter and tumbler tests have been conducted on the ilmenite. The petrological studies reveals the nature and the form of the ore. While shatter and tumbler tests determines the cold strength of the material for their resistance to abrasion impact handling. The tumbler and shatter indices give an indication of probable extent of fines likely to be generated during the reduction process in the kiln and while handling the product.

#### 5.2.2 Petrological studies

Petrological studies were conducted on Abughalage ilmenite sample at Research and development laboratories of National Mineral Development Corporation, Hyderabad. The petrological studies reveal that the presence of ilmenite as major mineral where as quartz formed the major gangue. Goethite, limonite, hematite and pyrite were present. The ilmenite samples observed to be feebly magnetic inferring that the iron oxide present is in magnetite form. The detailed petrological study report is presented at Annexure-I.

#### 5.2.3 Shatter tests

The shatter index of ilmenite ore is 93.40%. The shatter index result indicates that the

ilmenite ore can withstand well to normal handling and is suitable to process in the rotary kiln.

#### 5.2.4 Tumbler index

The tumbler index of Abughalaga ilmenite ore is observed to be 88.50%. This result indicates the ore could be classified as medium hard and considered suitable for direct reduction process using rotary kiln technology.

#### 5.2.5 Abrasion index

The abrasion index of the ilmenite ore sample is 11.83%. The result indicates that fines generation in the rotary kiln operation would be of the order 25 to 30% which was also confirmed in the reduction tests. From the experience of the SIIL operating plant the abrasion index is considered to be good.

#### 5.2.6 Bulk density

The bulk density of the ilmenite sample is 2.10 tons/M<sup>3</sup>. The bulk density is less compared to the iron ores which is due to the less iron content in the ilmenite sample. However this is not of much significance or constraint for processing the material in the rotary kiln for production of reduced material.



## 5.3 Properties of Reductant used (Manguru Coal, Singareni Collieries Co.Ltd, India)

### 5.3.1 Proximate Analysis:

The proximate analysis of the coal samples for the determination of the fixed carbon, volatile matter and ash was carried out as per standard procedure. The average results of the analysis carried out are presented in the table-3.

From the table it could be seen that the volatile matter is 27.90%, fixed carbon is 46.40% and ash is 25.70%.

### 5.3.2 Sulphur Content:

The sulphur content of the coal is one of the important characteristics for the evaluation of the suitability of the coal to be used as reductant for direct reduction of iron ores. The sulphur in coal is normally composed of inorganic/pyritic sulphur, Sulphate sulphur and organic sulphur. The presence of organic sulphur contributes to the sulphur pickup in the sponge iron in the absence of desulphariser. The total sulphur in the coal sample was analysed by gravimetric method. The different forms of the sulphur in Manguru coal were also analysed and the results of these chemical analysis are presented in the table-3. The total sulphur in the coal was 0.88%. With the organic sulphur of the order of 0.48% , it could be observed that the sulphur content in product could be controlled well below acceptable limit with usage of limestone (Blast Furnace grade) as desulphariser.

The average gross calorific value of the Manguru coal sample was observed to be of the order of 5320 Kcal/Kg which is considered optimum for the specified purpose. The calorific value of the reductant was determined in an adiabatic Bomb Calorimeter.

### 5.3.3 Chemical Analysis of the coal ash

The chemical composition of the coal ash influence the softening characteristics of the coal ash. Higher concentration of silica

and alumina are generally in line with higher softening temperature. However, higher concentration of the iron oxides in the ash lowers the softening point considerably. The analysis of the coal ash for the determination of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO and MgO was carried out and the results are tabulated in the table-3. The results of the chemical analysis of the coal indicate that the ash is acidic in nature and predominantly of silica with a value of more than 50%.

#### 5.3.4 Melting Characteristics of coal ash

In the rotary kiln process the kiln is to be operated at a temperature of 1050 deg.C to get the desired degree of reduction and this requires coals with relatively higher ash softening temperatures of the order of 100 - 150 deg.C above the kiln operating temperatures. The melting characteristics of the coal ash is determined in a LEITZ HEATING MICROSCOPE and the points of important observation are the softening or the initial deformation point, the melting or hemispherical point and the flow point. The behaviour of Manguru coal was found to be extremely good and the initial softening point itself was observed to be 1220 deg.C. It is felt that this could be attributed to lower concentration of sulphate sulphur in the coal ash. The test results of the melting behaviour of the coal ash are indicated in the Table-4.

#### 5.3.5 Reactivity of coal

Reactivity of the coal refers to the amount and the rates of carbon monoxide generation through the well known Boudouard reaction. This is an important factor in the rotary kiln operation since the generation of the carbon monoxide required for the reduction of the iron ore is formed in situ and is a function of the temperature in the kiln. The reactivity of the coal is determined by weightloss method. The test results carried out on Manguru coal indicate the reactivity as 2.10 c.c. of carbon monoxide per gram of carbon per second.

## 5.4 REDUCIBILITY TESTS IN LABORATORY ROTARY FURNACE

Reducibility tests are carried out in order to determine the behaviour of iron bearing ore and coal during the reduction and to predict the behaviour of raw materials in rotary kiln for commercial operation. In the laboratory rotary furnace reduction tests, operating conditions of the rotary kiln are simulated. The test results indicate the behaviour of the ores and coal under reducing conditions and establishes the reducing properties based on which the suitability of the ores and coals for use in rotary kilns could be determined and the operating parameters are found to achieve the desired levels of product quality in the actual operations.

### 5.4.1 LABORATORY ROTARY FURNACE TEST RESULTS

The reducibility tests are conducted in an electrically heated rotary furnace normally at a reduction temperature of 1000 deg.C and for a retention time of 3 hours. Based on the comparative study of Bench and Demonstration scale tests on known ores and coals, the test parameters/conditions for the laboratory rotary furnace have been developed by SILL. Number of laboratory rotary furnace tests have been conducted on Abughalaga ilmenite ore in combination with Manguru coal to determine the optimum operating parameters for achieving 80% metallization in the kiln.

The summary of the laboratory rotary furnace test results conducted on the Abughalaga Ilmenite ore in combination with Manguru coal are presented in tables.6-10.

### 5.4.2 DEGREE OF METALLIZATION

The degree of metallization aimed was atleast 80%. The first experiment was done at normal operating condition viz a reduction temperature of 1000 deg.C and retention time 3 hours and a C/Fe ratio of 0.5. For these test conditions a metallization of only 22.56% was achieved with Abughalaga ilmenite ore. In order to improve the metallization levels the tests conditions were varied. Table - 11 shows the degree of metallization achieved during the laboratory furnace tests conducted at different tests conditions. Normally higher

degree of metallization is possible with higher reduction temperatures and longer retention time. But higher reduction temperatures are limited by the ash fusion characteristics of coal used as reductant and condition of the furnace refractories. Keeping in view these conditions the final test parameters were established. At a reduction temperature of around 1100 deg.C and six hours retention time it was possible to achieve a degree of metallization of 66.00%.

#### 5.4.3 THE DECREPITATION BEHAVIOUR OF ILMENITE ORE

The process degradation index of the ilmenite ore during the reduction tests is in the range of 39% to 42% which shows that the ore decrepitates during reduction. The - 1 mm fines generation during the laboratory furnace tests is of the order 10% indicating that the ilmenite ore generates fines during the reduction.

#### 5.4.4 GRAINSIZE WISE ANALYSIS

The detailed grainsize chemical analysis presented in the tables-6 to 10 shows that the metallization level is almost uniform in all the size ranges and from point of reducibility this uniform metallization is considered ideal.

## 5.5 SHORT ROTARY KILN TESTS

The short rotary kiln is an oil fired furnace lined with high alumina refractory bricks and is designed for batch operations. The short rotary kiln is equipped with a charging door and a sampling port through which samples are drawn at definite intervals of time. It is also equipped with an oil burner whose flame is of horseshoe shape passing over the material bed inside the kiln. In view of this, actual conditions prevailing inside a commercial kiln can be simulated precisely i.e. reducing material bed and oxidizing free board gases, the waste gases pass through the exhaust duct provided at the top of burner stand. The rate of flow of waste gases and the kiln pressure are controlled by the damper provided in the waste gas line. The samples drawn at various intervals of time are cooled in inert (Nitrogen) atmosphere and analysed for rate of reduction and carbon consumption.

5.5.1 In all six short rotary kiln tests were conducted to process 420 Kg. of Abughalaga ilmenite in combination with Manguru coal. Since the prime aim of the bench scale tests were to produce prereduced ilmenite of reasonably higher degree of metallization for further processing the same to produce Titania rich slag in submerged arc furnace. The original as received samples was crushed to the size of 3 to 15 mm and this material was used in the short rotary kiln for production of prereduced material. The as received Abughalaga ilmenite sample was of the size -50mm, the detailed size analysis of the as received sample is given in table-2. In order to process these material in rotary kiln the as received Abughalaga material was crushed and prime size product of 3 to 15 mm. size was used in the final tests.

After conducting the required number of tests, a net of 420 kg. of ilmenite was processed in the short rotary kiln in six batches to produce 305 kg. of prereduced ilmenite. The standard test conditions worked out in laboratory rotary furnace was maintained for the short rotary kiln tests. The results of the short rotary kiln are presented in table-12 to 17.

The chemical analysis of samples drawn at various intervals of time have been analysed for Fe(T), Fe(Mat), Carbon and sulphur in the case of magnetic products and for non-magnetic products the fixed carbon, ash and volatile matter have been analysed. The results of the short rotary kiln tests shows that a metallization of about 65% could be achieved. The analysis of non-magnetic product reveals that carbon is properly utilized during the tests. The product from the all rotary short rotary kiln tests were mixed and a composit sample of 300 kg. prereduced ilmenite sample was despatched to USSR for further testing. The detailed chemical analysis of the composit sample as despatched to USSR is presented in table-18.

## 6.0 FINDINGS

- 6.1 From the laboratory and bench scale tests conducted on Abughalaga ilmenite sample, it is observed that ilmenite ore is suitable in combination with a non\_coking coal to produce prereduced ilmenite in a rotary kiln.
- 6.2 The reducibility tests conducted on the Abughalaga ilmenite ore reveals that the Abughalaga ilmenite ore is reducible in character as compared to the other similar ores and it requires higher time to obtain higher degree of metallization.
- 6.3 Sub\_bituminous coals having a fixed carbon of 45% is considered suitable as reductant.
- 6.4 From the extensive tests conducted on Abughalaga ilmenite ore in combination with Manguru coal following operating parameters are suggested for reducing Abughalaga ilmenite with suitable coal in commercial operation for production of prereduced ilmenite.

- i) Reduction Temperature : 1100 deg.C
- ii) Retention time : 6 hours
- iii) C/Fe ratio : 0.65

### iv) Consumption norms

Quantity of material required to produce 1.0 kg. of prereduced ilmenite.

- |              |   |               |
|--------------|---|---------------|
| Raw ilmenite | : | 1.38 kg.      |
| Coal         | : | 0.75 kg.      |
| Limestone    | : | Not required. |

## 7.0 Conclusions

Total 600 kg. of Abughaiega ilmenite sample was received from Govt. of Egypt. After conducting number of tests for determining chemical, physical properties and optimum test conditions finally 420 kg. of ilmenite in the size range of 3 to 15 mm. was processed in the short rotary kiln to produce prereduced material. From these tests 305 kg. of prereduced material of 66.11% metallization was produced.

The Screen and chemical analysis of the composite prereduced Ilmenite sample produced in number of test runs in the short rotary kiln is given in the table-18. This material was despatched to M/S. Titanium Institute, Zaporozhy, USSR, through UNDP, Delhi on 20.9.1990.



RESEARCH AND DEVELOPMENT CENTRE  
NATIONAL MINERAL DEVELOPMENT CORPORATION LIMITED

PETROLOGICAL LABORATORY

PETROGRAPHIC CHARACTERISTICS AT A GLANCE

**PHYSICAL PROPERTIES**

**MICROSCOPIC PROPERTIES**

**COLOUR** : Dark grey to blackish grey.

**LUSTER** : Metallic to sub-metallic.

**STREAK** : Brownish black.

**FRACTURE** : Uneven.

**HARDNESS** : 5.5-6.0.

**CLEAVAGE** : Absent.

**SP. GR. OF LUMPS** : 4.23 (Average).

**SP. GR. OF FINES** : ----

**MAGNETISM** : Feebly magnetic.

**ESSENTIAL MAJOR  
MINERAL** :

ILMENITE

**ACCESSORY MINOR  
MINERAL** :

Goethite, Limonite, Hematite  
and pyrite.

**MAJOR GANGUE  
MINERAL** :

Quartz.

**MINOR GANGUE  
MINERAL** :

Goethite, Limonite, Hematite  
and pyrite.

**NAME OF THE ROCK/DRE** : Ilmenite rock

**TEXTURE** :

Medium to coarse grained.

**LOCALITY SOURCE** : Abughalaga (SIII)

**GRAIN SIZE** :

Ilmenite (5-860 microns).  
Other constituents were amorphous  
in nature.

**REMARKS** : The report pertains to  
the samples received.

**ALTERATION PRODUCTS** : Goethite, Limonite, Hematite  
and pyrite.

PETROGRAPHIC REPORT ON ILMENITE SAMPLE RECEIVED FROM  
SPONGE IRON INDIA LIMITED (SIIL)

INTRODUCTION:

A sample of ilmenite rock weighing about 300 grams was received in R&D Centre of NMDC on 4th June, 1990 from M/s. Sponge Iron India Limited (SIIL) for mineralogical studies.

PURPOSE

The purpose of the study was to find out the mineralogical composition, nature of gangue mineral and the size of the ilmenite grains.

MEGASCOPIC CHARACTERISTICS

Colour : Dark grey to blackish grey.  
Streak : Brownish black.  
Lustre : Metallic to sub-metallic.  
cleavage : Absent.  
Hardness : 5.5-6.0.  
Fracture : Uneven.  
App.Sp.gravity : 4.23  
Magnetism : Feebly magnetic.

Microscopic characteristics

The microscopic studies revealed the presence of ilmenite as major mineral whereas quartz formed the major gangue. Goethite, limonite, hematite and pyrite were present in minor quantities. The texture was medium to coarse grained. The description of the individual minerals are given below:

ILMENITE:

It was noticed as a major mineral. The grains were euhedral and subhedral. It was mostly light grey in colour with brownish tint. The cleavage was absent. Reflectivity was moderate. Pleochroism was very weak. Under crossed nicols it showed uniaxial extinction. Twinning was rarely observed. At few places ilmenite was seen altered to hydrous iron oxides. The ilmenite grains were tightly embedded in the groundmass composed of

quartz. Few minute grains of pyrite were seen within the ilmenite grains. The size of the ilmenite grains ranged from 5 to 880 microns.

#### QUARTZ:

It occurred as major gangue mineral. The ground mass was comprised of quartz which was generally amorphous in nature. Occasionally it showed crystalline structure. It was colourless to grey in colour. At places admixture of hydrous iron oxide were seen within the intergranular spaces and the cracks.

#### GOETHITE:

It occurred as one of the minor constituents in the sample. It was greyish white in colour with reddish brown internal reflection. It was mainly observed in cracks and cavities. It was found replacing the ilmenite grains as well but such occurrences were not very common. Goethite was amorphous in nature.

#### LIMONITE

It also occurred as minor constituent after goethite. It was seen in association with goethite and quartz specially in cracks and cavities. It was cryptocrystalline in nature.

#### HEMATITE:

Hematite exsolution were occasionally seen in the section. This was observed mostly in the margin of the ilmenite grain. At places it was seen altered to Goethite. The size ranged from 4 microns to 260 microns.

#### PYRITE:

Pyrite was noticed as scattered minute grains within the ilmenite. It was present as alteration product of ilmenite. The detailed properties could not be observed due to its fineness of grain size as it was beyond the scope of the microscope. The size range was from 5 microns to 32 microns.

#### CONCLUSION:

The sample contained ilmenite as major constituent. Quartz was the major gangue mineral.

Goethite, limonite, hematite and pyrite was seen as minor constituents. The ilmenite grains generally appeared quite fresh except few grains which were altered to/replaced by hydrous iron oxides. The grain size of ilmenite ranged from 5 to 880 microns. Other constituents were amorphous in nature. Mainly alteration and replacement textures were observed.

TABLE - 1

Chemical analysis of Abuqhalaga Ilmenite sample

<u>Constituent</u>	<u>Assay (%)</u>
Fe (t)	39.70
TiO <sub>2</sub>	37.20
Fe+2	19.20
FeO	24.69
Fe+3	20.50
Fe <sub>2</sub> O <sub>3</sub>	29.32
SiO <sub>2</sub>	2.62
Al <sub>2</sub> O <sub>3</sub>	1.31
CaO	0.19
MgO	3.55
P	0.015
S	0.021
Cr <sub>2</sub> O <sub>3</sub>	0.15
MnO	0.03
BaO	0.020
Cu	0.060
Na	0.005

TABLE - 2

**Physical properties of Abughalaga Ilmenite Sample****I. Screen analysis of as received sample**

Size Range	Wt. fraction %
+ 30 - 50 mm	25.43
+ 25 mm	25.07
+ 20 mm	13.03
+ 15 mm	17.61
+ 10 mm	10.43
+ 8 mm	02.61
+ 5 mm	02.61
+ 3 mm	0.78
+ 1 mm	0.13
- 1 mm	1.30

**II Other Properties**

Bulk density	2.10 gm/cc
Shatter index	93.94 %

TABLE - 3

Chemical analysis of reductant used in the tests (Manuguru Coal)I. Proximate analysis (Dry basis)

Ash	25.70 %
Volatile matter	27.90 %
Fixed Carbon	46.40 %
Moisture	4.80 %

II. Ash chemical analysis

SiO <sub>2</sub>	59.50 %
Al <sub>2</sub> O <sub>3</sub>	25.40 %
Fe <sub>2</sub> O <sub>3</sub>	8.20 %
CaO	3.40 %
MgO	0.85 %

III. Sulphur analysis in Coal

Organic sulphur	0.4821 %
Pyritic sulphur	0.38 %
Sulphate sulphur	0.016 %
Total sulphur	0.8781 %

TABLE - 4

Physical properties of reductant used in the tests (Manuquru Coal)I. Screen Analysis

<u>Screen size</u>	<u>Weight fraction %</u>
+ 12 - 15 mm	12.80
+ 10 mm	26.87
+ 8 mm	15.45
+ 5 mm	20.40
+ 3 mm	22.48

II. Gross calorific value            5320 K-cal/kg

III. Ash softening characteristics

Softening point	1220	C
Melting point	1335	C
Flow point	1380	C

IV. Reactivity                             $2.1 \text{ cm}^3 \text{ co/gm carb sec.}$



Properties of Limestone used in the testsI. Chemical analysis

<u>Constituent</u>	<u>Assay (%)</u>
CaO	47.26
Fe(t)	1.37
MgO	1.51
S	0.04
SiO <sub>2</sub>	8.23
Al <sub>2</sub> O <sub>3</sub>	1.26
LOI	38.27

II. Screen analysis

+ 3 mm	14.04 %
+ 0.5 mm	42.38 %
- 0.5 mm	43.57 %

## LABORATORY ROTARY FURNACE TEST RESULTS

Test No. 1

Standard Conditions

I. Input Raw Materials

Iron bearing mineral : EGYPT Ilmenite  
 Reductant : Manguru Coal  
 Desulphuriser : SILL Limestone

II. Test conditions

C/Fe : 0.5  
 Reduction Temp. : 1000 C  
 Retention time hrs. : 3 Hrs.

III. Test Results

Average Metallization	Decrepitation Behaviour
(+1 mm) % : 22.56	<u>Size</u> %
	- 1 mm : 9.57
Process Degradation Index :	- 3 mm : 28.96
	- 5 mm : 38.03
- Oxide feed % : 41.38	
- Reductant % : 34.53	

IV. Grainsizewise Analysis of Magnetics (%)

size	Range	Fe(T)	Fe(Met.)	Metn.	Carbon	sulphur
	-1mm	39.00	9.75	25.0	0.45	0.024
1	- 3 mm	39.50	9.09	23.0	0.40	0.020
3	- 5 mm	40.00	9.00	22.50	0.38	0.020
5	- 10mm	41.00	9.22	22.50	0.35	0.018
10	- 15mm	41.00	9.22	22.50	0.35	0.019
	+15mm	-	-	-	-	-

V. Proximate Analysis of Non-magnetic product (char)%

Fixed Carbon : 23.00  
 Ash : 76.00  
 Volatile Matter : 1.00

## LABORATORY ROTARY FURNACE TEST RESULTS

Table-7

Test No. 2

### I. Input Raw Materials

Iron bearing mineral : EGYPT Ilmenite (Standard Size)  
Reductant : Manguru Coal ( -do- )  
Desulphuriser : SIIL Limestone ( -do- )

### II. Test conditions

C/Fe : 0.6  
Reduction Temp. : 1000 C  
Retention time hrs. : 3 Hrs.

### III. Test Results

Average Metallization (+1mm)%	:	26.13	Decrepiation Behaviour	
Process Degradation Index	:		Size	%
- Oxide feed %	:	39.38	- 1 mm	: 10.35
- Reductant %	:	35.46	- 3 mm	: 26.53
			- 5 mm	: 37.46

### IV. Grainsizewise Analysis of Magnetics (%)

size	Range	Fe(T)	Fe(Met.)	Metn.	Carbon	sulphur
	-1mm	39.20	9.41	24.00	0.38	0.020
1	- 3 mm	39.35	9.26	23.55	0.35	0.019
3	- 5 mm	40.32	10.33	25.63	0.28	0.019
5	- 10mm	40.80	10.98	26.92	0.26	0.017
10	- 15mm	40.03	10.34	25.83	0.26	0.017
	+15mm	-	-	-	-	-

### V. Proximate Analysis of Non-magnetic product (char)%

Fixed Carbon : 25.00  
Ash : 73.50  
Volatile Matter : 1.5

LABORATORY ROTARY FURNACE TEST RESULTS

Table-8

Test No. 3

I. Input Raw Materials

Iron bearing mineral : EGYPT Ilmenite (std size)  
Reductant : Manguru Coal ( -do- )  
Desulphuriser : SILL Limestone ( -do- )

II. Test conditions

C/Fe : 0.65  
Reduction Temp. : 1050 C  
Retention time hrs. : 4 Hrs.

III. Test Results

Average Metallization (+1 mm)%	: 47.23	Decrepitation Behaviour	
Process Degradation Index	:	Size	%
- Oxide feed %	: 41.38	- 1 mm	: 9.25
- Reductant %	: 36.32	- 3 mm	: 30.35
		- 5 mm	: 38.47

IV. Grainsizewise Analysis of Magnetics (%)

size	Range	Fe(T)	Fe(Met.)	Metn.	Carbon	sulphur
	-1mm	39.36	17.75	45.10	0.42	0.024
1	- 3 mm	41.13	19.41	47.20	0.36	0.021
3	- 5 mm	42.33	19.57	48.35	0.35	0.017
5	- 10mm	42.56	20.91	49.12	0.33	0.016
10	- 15mm	40.32	19.49	48.35	0.28	0.017
	+15mm	-	-	-	-	-

V. Proximate Analysis of Non-magnetic product (char)%

Fixed Carbon : 28.00  
Ash : 70.90  
Volatile Matter : 01.10

LABORATORY ROTARY FURNACE TEST RESULTS

Table-9

Test No. 4

I. Input Raw Materials

Iron bearing mineral : EGYPT Ilmenite std  
Reductant : Manguru Coal std  
Desulphuriser : SILL Limestone std

II. Test conditions

C/Fe : 0.65  
Reduction Temp. : 1080 C  
Retention time hrs. : 5 Hrs.

III. Test Results

Average Metallization (+1 mm)%	:	64.26	Decrepitation Behaviour		
Process Degradation Index	:		Size	%	
- Oxide feed	%	42.66	- 1 mm	:	8.34
- Reductant	%	33.78	- 3 mm	:	25.26
			- 5 mm	:	37.47

IV. Grainsizewise Analysis of Magnetics (%)

size	Range	Fe(T)	Fe(Met.)	Metn. Carbon	Carbon	sulphur
	-1mm	42.32	24.69	58.35	0.40	0.020
1	- 3 mm	43.56	27.16	62.36	0.35	0.020
3	- 5 mm	43.95	28.66	65.38	0.28	0.018
5	- 10mm	43.56	27.55	63.23	0.27	0.016
10	- 15mm	42.46	26.36	62.15	0.28	0.014
	+15mm	-	-	-	-	-

V. Proximate Analysis of Non-magnetic product (char)%

Fixed Carbon : 34.00  
Ash : 62.00  
Volatile Matter : 04.00

LABORATORY ROTARY FURNACE TEST RESULTS

Table-10

Test No. 5

I. Input Raw Materials

Iron bearing mineral : EGYPT Ilmenite std  
Reductant : Manguru Coal std  
Desulphuriser : SIIL Limestone std

II. Test conditions

C/Fe : 0.65  
Reduction Temp. : 1100 C  
Retention time hrs. : 6 Hrs.

III. Test Results

Average Metallization (+1 mm)%	:	66.50	Decrepitation Behaviour	
Process Degradation Index	:		Size	%
- Oxide feed	%	43.78	- 1 mm	9.65
- Reductant	%	34.64	- 3 mm	26.72
			- 5 mm	39.15

IV. Grainsizewise Analysis of Magnetics (%)

size	Range	Fe(T)	Fe(Met.)	Metn.	Carbon	sulphur
	-1mm	42.38	25.62	60.45	0.41	0.023
1	- 3 mm	45.03	28.91	64.20	0.38	0.021
3	- 5 mm	45.33	30.24	65.70	0.25	0.019
5	- 10mm	43.78	28.94	66.10	0.24	0.017
10	- 15mm	43.68	27.85	65.76	0.22	0.015
	+15mm	-	-	-	-	-

V. Proximate Analysis of Non-magnetic product (char):

Fixed Carbon : 33.00  
Ash : 64.00  
Volatile Matter : 03.00

**Degree of Metallization obtained at various test conditions**

S.No.	Reduction temp.deg.C	Retention time hrs.	C/Fe Ratio	Metallization %
1	1000	3	0.5	22.50
2	1000	3	0.6	25.63
3	1050	4	0.65	48.20
4	1080	5	0.65	64.50
5	1100	6	0.65	66.50

## SHORT ROTARY KILN TEST RESULTS

Table-12

Expt No. 1

Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 %)

Reductant : Manguru Coal

Temp. : 1100 C

C/Fe ratio : 0.65

### I. Screen Analysis

Screen Size (mm)	<u>Feed Material</u>		<u>Products</u>	
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %
- 15 !		12.00	-	-
+ 10 !	50	30.50	14.29	-
+ 8 !		12.80	23.21	16.15
+ 5 !	50	19.30	32.14	20.85
+ 3	-	25.40	21.43	23.69
+ 1	-	-	3.57	20.25
- 1	-	-	5.36	14.06

### II. Chemical Analysis of Product (Magnetics)

Sample No.	Sample Reference	Temp. deg. C	Fe (T) %	Fe (M) %	Metn. %	C %	S %
(1)	After 4th hour	1080	43.36	23.99	55.32	0.35	0.020
(2)	After 5th hour	1090	44.36	24.72	59.71	0.32	0.020
(3)	After 6th hour	1100	45.32	26.74	63.43	0.29	0.018

### III. Chemical Analysis of Product (Non-magnetics)

Sample No.	Volatile matter %	Ash %	Fixed Carbon %
(1)	4.50	64.97	30.53
(2)	3.60	68.04	28.36
(3)	1.60	76.27	22.13



## Short Rotary Kiln Test Results

Table-13

Expt No. 2

Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 kg.)      Reductant : Manguru coal

Temp. : 1100 C      C/Fe : 0.65

### II. Screen Analysis

Screen Size (mm)	<u>Feed Material</u>		<u>Products</u>		
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %	
- 15 :		06.91	-	-	+15
+ 10 :	50	29.32	19.87	-	+10
+ 8 :		15.56	18.02	15.34	+8
+ 5 :	50	23.73	29.72	23.17	+5
+ 3 :	-	24.45	18.02	20.61	+3
+ 1 :	-	-	07.21	25.32	+1
- 1 :	-	-	07.21	15.54	-1

### I. Chemical Analysis of Product (Magnetics)

Sample No.	Sample Reference	Temp. deg. C	Fe (T) %	Fe (M) %	Metn. %	C %	S %
(1)	After 4th hour	1090	43.86	23.49	53.56	0.42	0.020
(2)	After 5th hour	1100	43.86	25.49	58.13	0.36	0.019
(3)	After 6th hour	1100	44.84	29.26	65.23	0.29	0.017

### III. Chemical Analysis of Product (Non-magnetics)

Sample No.	Volatiles matter %	Ash %	Fixed Carbon %
(1)	4.20	66.35	29.35
(2)	3.60	73.45	24.35
(3)	1.10	80.75	18.15

### Short Rotary Kiln Test Results

Table-14

Expt No. 3

Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 kg.)

Reductant : Manguru Coal

Temp. : 1100 C

C/Fe : 0.65

#### I. Screen Analysis

Screen Size (mm)	<u>Feed Material</u>		<u>Products</u>	
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %
- 15 !		07.53	-	-
+ 10 !	50	32.34	13.80	-
+ 8 !		17.36	12.10	18.35
+ 5 !	50	28.57	24.10	25.38
+ 3	-	14.20	28.45	20.31
+ 1	-	-	12.95	24.59
- 1	-	-	08.60	11.37

#### II. Chemical Analysis of Product (Magnetics)

<u>Sample No.</u>	<u>Sample Reference</u>	<u>Temp. deg.C</u>	<u>Fe(T) %</u>	<u>Fe(M) %</u>	<u>Metn. %</u>	<u>C %</u>	<u>S %</u>
(1)	After 4th hour	1080	42.86	21.49	50.16	0.51	0.019
(2)	After 5th hour	1090	43.36	23.64	54.53	0.42	0.020
(3)	After 6th hour	1100	43.86	29.11	66.36	0.32	0.018

#### III. Chemical Analysis of Product (Non-magnetics)

<u>Sample No.</u>	<u>Volatile matter %</u>	<u>Ash %</u>	<u>Fixed Carbon %</u>
(1)	4.20	67.10	28.70
(2)	2.10	74.70	23.20
(3)	1.15	81.55	17.30

### Short Rotary Kiln Test Results

Table-15

Expt No. 4  
Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 kg.)      Reductant : Manguru Coal  
Temp. : 1100 C      C/Fe : 0.65

#### I. Screen Analysis

Screen Size (mm)	<u>Feed Materials</u>		<u>Products</u>		
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %	
- 15 !		95.90	-	-	+15
+ 10 !	50	25.92	12.32	-	+10
+ 8 !		21.58	11.36	13.13	+8
+ 5 !	50	30.34	20.35	20.32	+5
+ 3	-	16.36	22.72	25.79	+3
+ 1	-	-	22.89	28.38	+1
- 1	-	-	10.36	12.38	-1

#### II. Chemical Analysis of Product (Magnetics)

Sample No.	Sample Reference	Temp. deg.C	Fe(T) %	Fe(M) %	Metn. %	C %	S %
(1)	After 4th hour	1090	43.36	21.41	49.38	0.43	0.019
(2)	After 5th hour	1090	44.13	23.67	53.64	0.38	0.018
(3)	After 6th hour	1100	44.86	29.41	65.56	0.29	0.018

#### III. Chemical Analysis of Product (Non-magnetics)

Sample No.	Volatile matter %	Ash %	Fixed Carbon %
(1)	3.50	68.15	28.35
(2)	2.05	78.60	19.35
(3)	1.15	82.35	14.50

Short Rotary Kiln Test Results

Table-16

Expt No. 5  
Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 kg.)

Reductant : Manguru Coal

Temp. : 1100 C

C/Fe : 0.65

I. Screen Analysis

Screen Size (mm)	<u>Feed Materials</u>		<u>Products</u>	
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %
- 15 !		06.66	-	-
+ 10 !	50	30.88	14.33	-
+ 8 !		22.78	12.38	09.18
+ 5 !	50	25.36	22.36	26.79
+ 3	-	14.32	22.68	22.98
+ 1	-	-	18.42	22.73
- 1	-	-	09.83	18.32

II. Chemical Analysis of Product (Magnetics)

Sample No.	Sample Reference	Temp. deg. C	Fe(T) %	Fe(M) %	Metn. %	C %	S %
(1)	After 4th hour	1080	43.36	22.69	52.33	0.39	0.019
(2)	After 5th hour	1090	44.78	25.03	55.89	0.29	0.018
(3)	After 6th hour	1100	44.78	29.53	66.38	0.29	0.018

III. Chemical Analysis of Product (Non-magnetics)

Sample No.	Volatile matter %	Ash %	Fixed Carbon %
(1)	4.10	69.55	26.35
(2)	2.15	77.75	20.10
(3)	0.95	82.75	16.30

## Short Rotary Kiln Test Results

Table-17

Expt No. 6

Reference: Abughalaga Ilmenite

Ore : Ilmenite (70 kg.)

Reductant : Manguru Coal

Temp. : 1100 C

C/Fe : 0.65

### I. Screen Analysis

Screen Size (mm)	<u>Feed Material</u>		<u>Product</u>	
	Ilmenite %	Coal %	Magnetics %	Non-Magnetics %
- 15 !		07.13	-	-
+ 10 !	50	33.37	11.36	-
+ 8 !		24.48	14.93	09.36
+ 5 !	50	23.69	24.32	21.38
+ 3	-	11.33	25.91	26.87
+ 1	-	-	16.04	27.72
- 1	-	-	07.36	14.67

### II. Chemical Analysis of Product (Magnetics)

Sample No.	Sample Reference	Temp. deg.C	Fe(T) %	Fe(M) %	Metn. %	C %	S %
(1)	After 4th hour	1080	43.86	23.84	54.36	0.38	0.020
(2)	After 5th hour	1090	44.13	25.55	57.89	0.33	0.019
(3)	After 6th hour	1100	44.86	30.65	68.32	0.27	0.019

### III. Chemical Analysis of Product (Non-magnetics)

Sample No.	Volatile matter %	Ash %	Fixed Carbon %
(1)	3.50	65.00	31.50
(2)	2.15	70.35	27.50
(3)	0.90	79.75	19.35

**Detailed Analysis of Prerduced Ilmenite**

Material : Composite sample of prerduced Ilmenite produced in six short rotary kiln tests

Source of Material : Raw Ilmenite from Abughalaga, Egypt -  
Reduced at SIIL Test Centre.

Chemical Constituent	Assay %	Screen Analysis	Weight %
Fe(Total)	45.20	+10mm	14.32
TiO <sub>2</sub>	40.40	+ 8mm	15.33
Fe(Met.)	29.92	+ 5mm	25.50
Metallization +3	66.19		
Fe	4.99		
FeO	12.19	+ 30mm	23.20
Fe <sub>2</sub> O <sub>3</sub>	7.12	+ 1mm	13.50
SiO <sub>2</sub>	2.83	- 1mm	08.15
Al <sub>2</sub> O <sub>3</sub>	1.98		
CaO	0.31		
MgO	3.90		
P	0.02		
S	0.023		
C	0.29		
Cr <sub>2</sub> O <sub>3</sub>	0.17		
MnO	0.035		
BaO	0.024		
Cu	0.007		
Na	0.006		