OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

FAIR USE POLICY

CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

19968

39p
tables
diagrams

DEVELOPMENT OF TECHNOLOGY FOR PRODUCTION OF SYNTHETIC RUTILE THROUGH DIRECT REDUCTION - MAGNETIC SEPARATION AND ELECTROSMELTING TECHNOLOGY

UC/IND/87/146/11-51

INDIA

Technical Report: Direct Reduction and Electrosmelting

Techniques for Synthetic Rutile Production

Prepared for the Government of India

by the United Nations Industrial Development Organization
acting as executing agency for the
United Nations Development Programme

Based on the work of Osmo Vartiainen,
Consultant

Backstopping Officer: Y. Grebtsov,
Metallurgical Industries Branch

United Nations Industrial Development Organization
Vienna

XI-1992

EXPLANATORY NOTES

During the period of the mission the following exchange rate and prices were effective:

Exchange rate	1 USD = Rs 28.00
Furnace oil	Rs 4-5.00/l
Electric power	Rs 2.00/kWh, for industry
	Rs 1.10/kWh, domestic
Petrol for car	Rs 20.00/l
Labour salary, average	Rs 3000.-/month

I. ABSTRACT

Number of project:
UC/IND/87/146/11-51

Title of project:
DEVELOPMENT OF TECHNOLOGY FOR PRODUCTION OF SYNTHETIC RUTILE THROUGH DIRECT REDUCTION - MAGNETIC SEPARATION AND ELECTROSMELTING TECHNOLOGY.

Purpose of project:
Technical assistance in the investigation of a process for production of synthetic rutile from ilmenite by adopting direct reduction - magnetic separation and smelting techniques.

Implementation:
The project was implemented between the 7th October and 2nd November, 1992 in India.

Main conclusions and recommendations:

In the project the results of the previous studies were thoroughly analyzed, some check-up tests made and pre-reduction and smelting trials carried out.

Production of synthetic rutile was investigated in addition to a prolonged (up to 12 hrs) treatment also by using "flash reduction" i.e. very short (< 1h) reduction time in order to form mainly magnetic iron rather than metallic iron. The aim was to remove a bulk of iron magnetically and the rest by leaching or smelting.

In the electrosmelting tests of the ilmenite concentrate and the pre-reduced ilmenite the effects of reductant quantities, basicity and viscosity of slag were the objects of the research work as well as operational technique.

Although there is still room for laboratory investigations to study certain details of processing it is suggested to proceed to the pre-feasibility study phase with larger scale test runs.

II CONTENTS

III. INTRODUCTION

Sponge Iron India Limited (SIIL) has set a target to develop a technology to increase the rate of TiO_2 in Indian Ilmenite concentrate from the present level of ab. 55 % to > 90 %. The iron content should be recovered as saleable product.

The aim of the present project is to investigate the production possibilities of synthetic rutile especially concentrating on the direct reduction - magnetic separation - smelting technologies. To carry out this investigation the UNIDO, Metallurgical Industries Branch, Vienna, Austria made a contract for one month with the undersigned, Mr. Osmo Vartiainen, Dr.Eng., OV-Eng Oy, Helsinki, Finland to assist the research personnel of SIIL at Paloncha plant. The project was implemented between the 7th October and 2nd November, 1992 (8-29.10. at Paloncha).

During the project work the previous investigations made by SIIL with various contractors were thoroughly studied. Some results of the investigations in question were rechecked, processing possibilities surveyed and future development lines planned. A detailed study of magnetic separation has been carried out earlier as a separate work by Mr. Moon Kim (see VI.3.1).

A serie of pre-reduction tests on conditions which have not been studied earlier were made. Also some reduction smelting trials in the newly installed submerged arc furnace were carried out by using ilmenite concentrate and pre-reduced ilmenite. The amount of the latter material available was only ab. 10 kgs, enough for one smelting test only. As a conclusion of the completed project work it is been suggested to proceed to the pre-feasibility study phase based only on a few processing alternatives.

IV. PROJECT WORK

IV.1 Summarized conclusions from investigations carried out previously

IV.1.1 Ilmenite concentrate

- The composition of the ilmenite concentrate used in the SIIL's Paloncha plant corresponds to the Indian ilmenite concentrates described in literature, see Annex VI.4.

- "Indian ilmenites" are very well comparable in composition with those of USA and Australian ilmenite concentrates though ZrO_2-content is a bit high in Indian ilmenites.

- Rather high TiO_2-content, ab. 56 % is due to occurrences of rutile, pseudorutilo and pseudo-brookite, oxidation of Fe^{2+} to Fe^{3+} resulting to e.g. pseudorutile $Fe_2Ti_3O_9$ and leucoxene minerals in ilmenite concentrate.

- Ilmenite concentrate has gone through the magnetic-electrostatic separation processes in the Indian Rear Earths Limited plant.

- When TiO_2-content in Ti-concentrates mined in Australia is mostly as rutile and ilmenite,in China as titani-ferrous magnetites, so Indian ilmenites are found to contain pseudorutile, pseudobrookite and various other weathered products.

Ilmenite beach sand concentrate from Malaysia is known for its fair amount of radioactive minerals.

IV.1.2 Pre-reduced ilmenite

- Practically all grains in pre-reduced ilmenite are magnetic containing various amounts of metallic iron (5-50 vol.%)

- Iron reduction by using low value coal has brought reasonable good results although optimum conditions have to be determined further. The laboratory scale tests have proven that the degree of reduction to metal can reach a level of ab. 90 % at a temperature of 1100 oC.

IV.1.3 Production of TiO_4-slag and pig iron from ilmenite concentrate

- The preliminary trial tests in an arc furnace gave low iron slag with 87.5 % of TiO_2. This is thought to be suitable for $TiCl_4$-processing. High-iron (ab. 9.4 %) slag with 78.9 % of TiO_2 was obtained. This product is suited for use in manufacturing of pigment grade titanium dioxide.

- More investigations are needed to specify suitabilities of the above products for different uses.

- The metal phase from smelting contained 1.2-1.5 %C and 0.55 - 1.02 % TiO_2 sulphur amount being 0.3-0.5 % and silicon 0.07-0.75 %

- More refining tests about iron metal being suitable as raw material in cast iron and steel industry is suggested to be made.

IV.1.4 Production of TiO_2-slag and pig iron from pre-reduced ilmenite

- Low-iron (2.9 %) slag with 87.5 % TiO_2 and high-iron (8.5 %) slag with 81.3 % of TiO_2 were obtained in test runs.

- The metal in the tests above contained 0.39 and 0.72 % Ti depending on Fe-content of the slag. Si-contents were 0.54 % and 0.13 % correspondingly and Mn 0.07 % and 0.08 % the carbon content being 0.3 %.

- The total amount of impurities in pre-reduced ilmenite is rather low being less than 10 %. However, the suitability of Ti-slag for the manufacturing of Ti Cl_4 needs more systematic investigations.

IV.1.5 Magnetic concentration of pre-reduced ilmenite

- Reduction of ilmenite concentrate has been tested in variable conditions against temperature (up to 1100 oC), reaction time (3-7 hrs) and amount of reductant (over 2 x theoretical).

- Because practically all particles of pre-reduced ilmenite lot are found to contain metallic iron, thus being highly magnetic, the magnetically removed metallic iron will not increase the degree of TiO2 more than removal of ash and nonburnt carbon might cause.

IV.1.6 Electric energy consumption in smelting of ilmenite concentrate

It is estimated that an industrial scale application (ref. USSR technology) for the production of slags from Indian ilmenite concentrate needs 2980 kWh/t slag of electric energy and 5 MVA furnace output is 10800 t/a when producing low-iron (4 % FeO) slag. The corresponding figures with high-iron (10 % FeO) slag are 2650 and 12100.

IV.1.7 Electric energy consumption in smelting of pre-reduced ilmenite

With pre-reduced ilmenite (80 % reduction) the electric energy consumption is estimated to be 1720 kWh/t slag and the annual capacity of 18650 t. When high-iron slag is produced the values are 1450 kWh/t slag and 22150 t/a correspondingly.

IV.2 Preliminary check-up tests

To become convinced of the results of the previously made trials a few elementary tests were carried out.

IV.2.1 Ilmenite concentrate

- When using an electromagnetic laboratory drum type rotating separator (Paloncha R/D) with 1000 gauss no magnetic portion was recovered
- Handmagnet (ferrite magnet) in close contact to material brought 99 % of the concentrate sample into the magnetic portion.

IV.2.2 Pre-reduced ilmenite

- Hand magnet test showed the material being highly magnetic; only ash and unburnt carbon were found in non-magnetic portion.

- By leaching the material in 1 % NH4Cl solution at temperature of 95oC in non-oxidizing conditions only ab. 1 % of the total iron was leached out during 4 hrs.

- 100 gpl H2 SO4 solution correspondingly gave similar results.

- The NH4 Cl test above was repeated in oxidizing conditions, T 40oC, 1h, with the same results as mentioned earlier.

- But a test with 100 gpl H2SO4 in oxidizing conditions, T 40oC, 1h, gave the following results:

ab. 82 % of total iron and 97 % of metallic iron went into solution the residue having 1.12 % Fe; TiO2-content in residue was 83.2 % in figures.

The test above showed, that

- There are no possibilities to remove iron either from ilmenite concentrate or pre-reduced ilmenite by magnetic separation .

This concerns the available test material in Paloncha R/D.

- Pre-reduced ilmenite has not been after-oxidized significantly.

- Iron can be leached out effectively in oxidizing conditions using acid like H4SO4 (or HCl).

IV. 3 Pre-reduction tests

The pre-reduction tests were carried out by using the laboratory rotary furnace (Salvis kiln) in the R/D department of Paloncha plant, see VI.3.7

The main goal of these tests was to find out the impact trends of the following main factors:
- reduction time from ab. 1/3h to 12 hrs (3-7hrs in previous trials)
- reduction temperature, 1000oC/1100oC
- effect of iron sulfide in reduction.

The amount of carbon used was constant being C/Fe 0.6 equal to ab. 2.1 x theoretical.

The results obtained are given in Table VI.3.8 They indicate that
- the amount of Fe2+ decreases by increasing reduction time
- the amount of Fe3+ was ab. 2.5-3 % already from 20 min. (20+171/2) to 12hrs

- by increasing the reduction temperature from 1000oC to 1100oC the Fe^{3+} content decreased to 1.6%

- by using a small amount of iron sulfide Fe^{3+} content drops to 0.8 % and amount of Fe^{2+} doubles correspondingly
- Fe met., metallization reached the level of 70 % already in 20 (+171/2) min. at 1000oC increasing to 75 % at temperature of 1100oC
- the degree of metallization was to 79 % by using 12hrs reduction time at 1000oC
- addition of iron sulfide has effected reduction of Fe^{3+} to Fe^{2+} very strongly before Fe met. is formed
- reduction - oxidizing process obviously reoxidizes metallic iron to Fe^{2+} quite effectively as assumed.

IV.4 Smelting tests

The smelting tests were conducted on ilmenite by using the laboratory submerged arc furnace, 50 kVA, at Paloncha R/D, see VI.3.9.

The aim of the trials was to investigate tentatively effects of the following main factors in smelting process as well as to practise the operation of the furnace being installed recently:
- amount of reductant, 1.0-2.0 x theoretical (+ carbon from electrodes)
- basicity of slag 1.5-2.1
- viscosity of slag, fluorspar 3-5.5 % of amount of slag.

The amount of reductant was calculated basing on the following chemical reactions:

$$FeO + C = Fe + CO$$
$$Fe_2O_3 + 3C = 2Fe + 3CO$$
$$3TiO_2 + C = Ti_3O_5 + CO$$

The further reduction of Ti_3O_5 was not taken into account.

The first trial runs were carried out as batch tests emptying the furnace by tilting after each heat. It was not possible to measure the temperature accurately; the optical pyrometer available gave measurements only to 1200oC max. The estimate of the temperature visually in the tests was ab. 1600-1700oC. The matte and slag were separated from each other when pouring out the material, the slag crushed to -1mm, magnetic part was separated by using low magnetic drum separator and combined with matte; non-magnetic portion formed slag still contained ash and unburnt coal. Matte was to be analyzed for C and Ti and slag for Fe and TiO_2.

The amount of pre-reduced ilmenite available was ab. 10 kgs which was enough for one test run only.

A second test series was planned thus that the material (100 kgs) should be charged continuously into the furnace at the pace of melting. When the furnace has become full of melt it is emptied to the half through the lower outlet, melt recovered, charging of the furnace continued, and so on. This type of investigation will give more relevant information about the distributions of iron and titanium, power and electrode consumption, slag composition, etc. This test serie had to be canceled because of the relining of the furnace.

The test results are given in Table VI.3.10
They show that:

- the average amount of slag in tests 211-215 was ab. 5.5 kgs per heat amounting to ab. 78 % of the discharge material

- by using the figures above the iron recovery in matte is 90.8 % in average in all the smelting tests completed.
- a good fluid slag was obtained by using ab. 3 % of fluorspar with the basicity of 1.6-2.1
- the carbon content of the matte varied between 1.2 and 1.8 %
- the iron content of the slag (with ash and unburnt coal) was between 3.3 and 5.5 %

- by using pre-reduced ilmenite smelting test results were well comparable to the use of ilmenite concentrate.

IV.5 Alternative methods for recovery of metal values from Indian ilmenites

The following examinations concern the ilmenite concentrates (Indian ilmenite) investigated at SIIL's Palancha plant. There are the following essential alternatives to treat the concentrates keeping in mind the goals set.

IV.5.1 Upgrading of reduced original concentrate by physical methods

IV.5.1.1 Iron converted into magnetic iron and then magnetic separation either

- 1.1 by accurate control of reduction process or
- 1.2 by bringing reduced iron to magnetic iron by oxidation and then magnetic separation

IV.5.2 By hydro-and pyrometallurgical methods

- 2.1 Removal of iron by acid leaching from pre-reduced ilmenite
- 2.2 Chloridizing of ilmenite directly to $TiCl_4$ by using fluidized bed technology
- 2.3 Oxidizing - reduction - acid leaching treatment of ilmenite

IV.5.3 By smelting reduction

- 3.1 (Submerged) arc furnace process, iron separation
- 3.2 Arc furnace process with slag purification and
- 3.2.1 Removal of iron in slag by acid-leaching or
- 3.2.2 Oxidation of iron in slag to Fe_3O_4 and removing it by magnetic separation

IV.5.4 Stationary requirements and their conditions; effect on various alternative production routes

In the following the alternatives listed above are examined more in detail.

IV.5.1 Upgrading of reduced original concentrate by physical methods.

The ilmenite concentrate has been originally processed by magnetic-electrostatic separation. The purity of the produced concentrate is rather high the content of magnetite being very low (< 5 %). Thus further concentration by the methods mentioned does not bring any significant improvement.

1.1 Accurate control of the reduction process.

It appears that the reduction of weathered ilmenite proceeds in two main steps. The first step, which occurs during the first few minutes, is the reduction of Fe^{3+} to Fe^{2+}. It is found in some laboratory tests (ref. literature) that over half of the Fe^{3+} is reduced during this initial period. After 12 hrs Fe^{3+} forms only 2-3 % of the total iron. If it is assumed that Fe^{3+} is contained in the pseudorutile the reaction can be written as follows:

$$Fe_2Ti_3O_9 + CO = 2FeTiO_3 + TiO_2 + CO_2.$$

The second step being much slower than the first one, corresponds to the reduction of Fe^{2+} to metallic iron as follows:

$$FeTiO_3 + CO = Fe + TiO_2 + CO_2$$

Thus metallic iron formation starts first after a certain period, when magnetic iron formation has reached its peak values. Controlling the reactions accurately might give, at least theoretically, a possibility to remove a significant amount of iron by using low magnetic separation. The optimum reduction time is probably between a few minutes and a half an hour.

1.2 Bringing reduced iron to magnetic iron by oxidation and then magnetic separation. The aim is to oxidize the reduced iron to magnetite as follows:

$$3Fe + 2O_2 = Fe_3O_4$$

Fe_3O_4 can be separated by magnetic means. And the rest of iron staying in upgraded ilmenite could be removed by using acid leaching.

IV.5.2 By hydro- and pyrometallurgical methods

2.1 Removal of iron by acid leaching from pre-reduced ilmenite.

Leaching of metallic iron from pre-reduced ilmenite where metallization of iron is 85-92 %, could be expected to produce TiO_2-recidue containing nearly 90 % of TiO_2.

2.2 Chloridizing of ilmenite directly to $TiCl_4$ by using fluidized bed technology.

The tests of using ilmenite concentrates instead of rutile for manufacturing $TiCl_4$ by the chloridizing process in the fluidized bed reactor have been quite promising. Though about some corrosion and operational difficulties have been reported in the literature. The chloridizing treatment with coal as reductant is done at the temperature of 800-1000oC. Iron will be mainly in the form of $FeCl_3$. The reactions are highly exothermic.

2.3 Oxidizing - reduction - acid leaching treatment of ilmenite.

This method is so called MURSO-process, where the iron of ilmenite is changed into such a composition that it can be leached out with acid. Oxidation is done at a temperature of ab. 950oC e.g. in a fluidized bed reactor, where Fe^{2+} oxidizes to Fe^{3+}. After that, in the reduction stage temperature being ab. 900oC, Fe^{3+} is reduced to Fe^{2+}. In this treatment the structure of ilmenite mineral becomes weaker and iron will be converted into leachable form. The synthetic rutile as a final product is reported to contain ab. 96 % TiO_2 and 1.5 % Fe_2O_3.

IV.5.3 By smelting reduction

As only iron should be reduced thus the conditions have to be determined by such a way that titanium stays in oxide form in slag. However, the following reduction reactions are possible

$$3\ TiO_2 + C = Ti_3O_5 + CO$$
$$2\ Ti_3O_5 + C = 3\ Ti_2O_3 + CO$$
$$Ti_2O_3 + C = 2\ TiO + CO$$

In the arc fernace conditions titanium is mostly in the form of Ti_2O_3 and Ti_3O_5. To avoid further reduction the temperature is recommended to be kept lower than 1525oC. Also CaO/SiO_2 - ratio in slag is suggested to be ab. 2.2 - 3.5 .

3.1 Arc furnace process, iron separation.

Reduction of Indian ilmenites brings iron into metallic form with a rate of ab. 89 %. The arc furnace treatment of this pre-reduced ilmenite gives rather pure iron matte with good separation from TiO_2-rich slag when its composition (viscocity, temperature) is kept correct during smelting.

Smelting ilmenite concentrate directly needs electric energy ab. twice as much as starting from pre-reduced ilmenite. Also the unit capacity of the furnace is only about 50-60 % of the last mentioned.

Price of energy, production requirements, market prices and conditions, etc. effect the selection of the production route.

One of the latest developments concerning ilmenite and titaniferrous magnetites by smelting reduction is a technology called "the melt circulation" technology. In this method coal ash being used as reducer, is kept separate from the titanic slag. Thus it does not matter what quality of coal is used in the process. However, the method is still under an intensive basic research.

3.2 Arc furnace process with slag purification

The slag received from the smelting-reduction process normally contain still some iron which can be removed either

3.2.1 by using acid (sulphuric/hydrochlorid) leaching or .

3.2.2 by oxidizing iron to Fe3 O4 and removing it magnetically.

IV.5.4 Stationary requirements and their conditions: effect on various alternative production routes

The following facts effective for SIIL and India have to be taken into notice when selecting a technology for synthetic rutile production and its refining:

- There are large and high quality ilmenite raw material resources in suitable grain size and easily to be mined
- There are large coal resources though low grade in consistency
- SIIL has long time experience in production of sponge iron from granular hematite ore
- Cheap and abundant labor force
- Expensive electric energy
- Environmental protection requirements; difficulties accumalating on industries.

Taking the above into account there appear to be the following processing routes which should get the high priorities when planning the future development work.

4.1 Smelting reduction of ilmenite concentrate by using (submerged) arc furnace process, matte and slag separation and either

4.1.1 Oxidation of iron in slag to be removed magnetically

or

4.1.2 Removal of iron left-over in slag by acid leaching

4.2 Pre-reduction of ilmenite concentrate and then smelting and either

4.2.1 Oxidation of iron in slag and then to be removed magnetically

or

4.2.2 Removal of iron in slag by acid leaching

IV.6 Conclusions

The refining of Indian ilmenites has been studied intensively during the last 5-6 years by SIIL. It was found necessary in the present project work to analyze thoroughly the previous results obtained , to check over and run a few pre-reduction and smelting trials to define preliminarily the effects of the main parameters in the technologies concerned.

The chemical Ti-analyses supposed to be made at the Paloncha plant were not completed before this report was revised. Thus the conclusions are based mainly on the iron analyses.

It was found that to increase the TiO_2-content of ilmenite concentrate or that of pre-reduced ilmenite produced earlier at Paloncha is not possible by using magnetic separation. Only ash and unburnt coal can be recovered as non-magnetic product when treating pre-reduced ilmenite material.

There are laboratory results based on the literature that when using fairly short reduction time, from 5 to 30 minutes, in the pre-reduction process a certain part of the iron is forming magnetic iron before it is in the metallic form. Thus it should be possible to remove a part of the iron content of ilmenite magnetically. A couple of test runs with the reduction time of 37 1/2 min. (20 + 17 1/2) showed that the metallization of the iron had reached already the level of ab. 70 %. Although the question of the reduction time (flash-reduction?) is quite interesting it is probably rather difficult to control the required conditions well enough on an industrial scale rotary kiln type furnace.

In the tests the metallization degree of iron has been slightly less than 90 % .

To remove iron from the ilmenite concentrate on an industrial scale there are two main routes. Viz. the direct electrosmelting - reduction process where the most of iron is recovered as matte and the rest in slag by magnetic separation and/or acid leaching. The iron in slag can be oxidized to magnetite by using hematite ore or oxygen and then be removed by magnetic separation.

Another processing route is to pre-reduce ilmenite and then to carry out the smelting process and iron removal as described above. Also the pre-reduced ilmenite can be upgraded by removing iron directly by leaching, of course.

The re-oxidizing processes of iron mentioned above need more laboratory tests. Whereas the acid leaching of iron from pre-reduced ilmenite seems to work well. In this project work 97 % of metallic iron went into solution the residue having 1.12 % Fe and 83.2 % TiO_2.

Removal of ash and unburnt carbon effectively in industrial scale operations has to be looked after.

To find out an optimum technic-economic solution for processing of Indian ilmenite concentrate one has to take into account the local conditions considering various prices and costs as well as marketing possibilities of the final products.

V. RECOMMENDATIONS

In order to find out the optimum technic - economic processing solution for upgrading Indian ilmenite concentrate by SIIL it is necessary to carry out a pre-feasibility study with larger scale tests. Meanwhile it is recommended that both the pre-reduction and smelting reduction tests should continue systematically.

Limiting the reduction time to be between 5 minutes and half an hour at various temperature levels in the ilmenite reduction process with the magnetic separation of the pre-reduced material is an interesting subject.

Removal of ash and unburnt carbon has to be investigated more closely.

The differencies between laboratory and production scale tests when treating hematite ore to produce sponge iron should be cleared up. This can be important in designing the continuous pre-reduction process for ilmenite concentrate, too.

The operation of the submerged arc furnace should be practised and improved by the research personnel thus that the conditions in the furnace could be controlled well and exactly. Some extra auxiliaries should be purchased, like a pyrometer with a scale up to ab. 1750oC. (present to 1200o C max.). When the matters above are in order it is recommended that a systematic factorial experimentation procedure should be carried out to find out the optimum processing conditions. Experience thus obtained could be utilized later on when running the process on a larger scale. Of course, the results of the laboratory tests mentioned are necessary also for basic engineering work.

Helsinki, Finland

Osmo Vartiainen, consultant

ANNEX VI.1

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

INDIA

Development of Technology for Production of Synthetic Rutile through Direct Reduction-Magnetic Separation and Electrosmelting Technology

JOB DESCRIPTION UC/IND/87/146

11-51

Post title Metallurgical Consultant/Expert in Rotary Kiln Reduction of Ilmenite Concentrate

Duration One month

Date required As early as possible

Duty station Kothagudem/Hyderabad with travel within the country.

Purpose of project Technical assistance in the investigation of a process for production of synthetic rutile from ilmenite by adopting direct reduction – magnetic separation and smelting techniques.

Duties The Metallurgical Consultant/Expert will be required to work in collaboration with a Consultant/Expert in Magnetic separation techniques (11-52) and undertake the following:

(1) Examine various methods of and selection of an optimum method for the preparation of ilmenite and coal to render them suitable for direct reduction tests in a rotary kiln.

(2) Conduct various tests in reduction of ilmenite with coal in a rotary kiln for determining optimum parameters for reduction.

(3) Examine suitable methods for the liberation of the product of reduction as in (2) above in order to separate individual mineral entities by differential magnetic separation.

(4) Conduct various tests in differential magnetic separation on the product of reduction and determine the optimum parameters for separation.

(5) Train the local technical staff in conducting tests as in (1) to (4) above.

..../..

VI.2 People met

VI.2.1 Counterpart staff

SPONGE IRON INDIA LIMITED, Khanij Bhavan (6th Floor) 10-3-311/A, Castle Hills, Masab Tank Hyderabad 500 028 (A.P.)

Mr. Chiranjiv Singh, Chairman and Managing Director
Mr. K.P.Patnaik, Chief General Manager
Mr. J.M. Reddy, Manager (Engg.&.Proj.)
Mr. Mr. K. Hari Kishan, Assistant Engineer

SPONGE IRON INDIA LIMITED, SIIL Campus-507154, Paloncha, Dist. Khammam, Andhra Pradesh

Mr.K.S.N. Murthy, Chief Works Manager
Mr. K. Suryanaryana, Personnel Manager
Mr. G.V. Subramanyam, Assistant Manager (R&D)
Mr. Chirra L. Nath, Metallurgist (R&D)
Mr.V.B.S. Sastry, Chemist (R&D)
Mr. S. Vijayanand, Sr. Chemist
Mr. L.V. Sastry, Sr. Personnel Officer (IR)
Mr.P.Ankineedu, DM (END)
Mr. N.Ananda Rao, Senior Supervisor
Mr. L.V. Sastry, Sr. Personnel Officer (R)
Mr. P. Satyanarayana, Manager (Constructions)
Mr. K. Sreenivasa Reddy, Assistant Engineer
Mr. D. Venkateswarh, Assistant Manager, Power plant
Mr.P.V. Ramana, Assistant Engineer(Mechanical)

VI.2.2 Other People

UNIDO, 55, Lodi Estate, New Delhi-110003

Mr. MD. Matiul Islam, UNIDO Country Director for India and Bhutan

Mr. Lasse Möller, Ph.D., IPO, UNIDO

ELKEM Metallurgy (P) Limited, 66/67 Mahavir Centre, Plot No. 77, Sector 17, Vashi, New Bombay-400 703

Mr. G.R. Shama Rao, Technical Representative

VI.3 Tables and figures

ANNEX VI.3.1

VI.3.1 The previous reports available to the undersigned during the mission (8.10.-2.11.1992)

o "Test Results of Ilmenite Fines of Indian Rare Earths Limited", SIIL Test Centre, 1986 (report not dated)

o Technical report of Mr. Valery P. Pechenkin, UNIDO SSA Expert to India, on the work fulfilled in India for "Sponge Iron India Ltd." Company, ab. 1987-88 (report not dated)

o "Laboratory and Bench Scale Testing of OSCOM Ilmenite Concentrate For Electrosmelting to Titania Slag and Pig Iron Products", by Technoexport USSR, All-Union Research and Design Titanium Institute, UNIDO Contract 90/062, 1991

o as above but concerning processing of Abu-Chalaga (Egypt) deposit ilmenite, by Technoexport, UNIDO 90/063, 1991

o "Final Report on Laboratory Reduction Tests Conducted on Aku-Chalaga (Egypt) Ilmenite Samples", SIIL Test Centre, Hyderabad, June 1991

o Technical Report: "Magnetic Separation of Ores/Minerals", UC/IND/87/146/11-52, UNIDO, by Mr. Moon Kim, June 1992
re: Development of technology for Production of Synthetic Rutile Through Direct Reduction-Magnetic Separation and Electrosmelting Technology

VI.3.2 Chemical analyses of Indian ilmenites in comparison with others

Element	Paloncha Ilmenite Conc. %	Redused ilmenite %	Indian ilmenites, generally %	Ilmenite concentrate USA %	Australia %
TiO2	55.98	58.49	54-61	44-65	47-57
Fe tot.	28.48	34.63			
FeO	18.67	4.31	9-27	4-40	14-27
Fe2O3	19.96	0.80	14-25	1-26	11-26
Fe met.	-	30.72	-	-	-
SiO4	1.80	2.34	0.4-1.4	0.3-4.6	0.1-1.5
CaO	0.25	0.35	0.05-0.2	0.05-1	0.02-0.2
MgO	0.45	0.55	0.6-1.0	0.05-2.4	0.2-2
Al2O3	1.00	1.20	0.9-1.3	0.1-3	0.5-4
MnO	0.56 x	0.55 x	0.3-0.4	0.05-1.4	0.5-1.8
Cr2O3	0.09 x	0.08 x	0.05-0.25	<0.01-0.3	0.02-4
P2O5	0.05 x	0.03 x	0.1-0.3	0.01-1.0	0.02-0.04
V2O5	0.24 x	0.26 x	0.1-0.6	<0.01-0.20	0.10-0.22
ZrO2	0.13 x	0.09 x	0.5-2.2	<0.01-0.6	<0.01-0.1
S	0.01 x	0.06 x			

x = OSCOM-ilmenite

ANNEX VI.3.3-4

VI.3.3 Screenanalysis of ilmenite concentrate

Screen Br.St. mesh	u	wt. % x %	y %
22	710		100.00
30	500	0.15	99.85
52	300		
72	212	57.04	42.81
100	150	24.89	17.92
150	106	17.33	0.59
200	75	0.59	-
		100.00	

S2900120

VI.3.4 Screenanalysis of pre-reduced ilmenite

		wt %			
Br.ST.	u	Product 1		Product 2	
mesh		x %	y %	x %	y %
16	1000	19.02	80.98	14.09	85.91
18	850	0.98	80.00	1.78	84.13
22	710	1.64	78.36	2.70	81.43
30	500	2.30	76.06	2.78	78.65
52	300			6.64	72.01
72	212	56.72	19.34	22.61	49.40
100	150	10.66	8.68	36.07	13.33
150	106	7.05	1.63		
200	75	0.65	0.98	11.80	1.53
-200	-75	0.98	-	1.53	-
		100.00		100.00	

ANNEX VI.3.5

VI.3.5 Analyses of various additives used in test work

Lime:

CaO	85 %
SiO2	2-3
R2O3	2-3
LOI	10

Fluorspar:

CaF2	92 %
SiO2	2-3

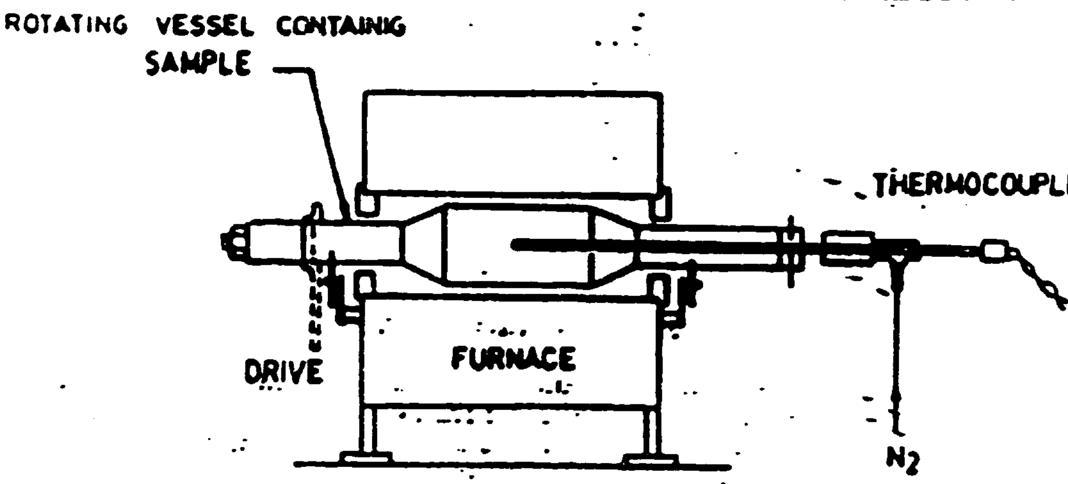
Leco coal:

Fixed carbon	62 %
Ash	15
Volat.m.	23

VI.3.6 Reduction of ilmenite concentrate.
Summarized results of previous tests

Selvis-test	ToC	Reduction time, hrs	% Fe.tot	Fe met.	Metallization	C/Fe
1986	1000	3	36.8	28.3	76.9	0.7
1986	1050	4	39.0	32.4	84.3	0.7
1986	1100	4	38.6	31.2	81.3	0.65
1986	1100	4	37.07	30.9	83.3	0.7
1986	1050	5	38.5	33.1	85.9	0.7
1991	1100	6	37.3	21.1	56.7	Theoretical
1992	1060	7	34.6	30.7	88.7	0.6

LABORATORY ROTARY FURNACE (SALVIS KILN)



Rotary Tube for Salvis Test

Rotary drum dia	:	150/90 mm
Reactor length	:	650 mm
Connected load	:	18 KW
Maximum operating temp.	:	1100°C
Connected voltage	:	3 x 415V AC 50 HZ
Thermocouple	:	pt-Rh-pt
Heating of tube	:	5°C/min
RPM of tube	:	8

[illegible]

VI.3.8 Reduction of ilmenite concentrate

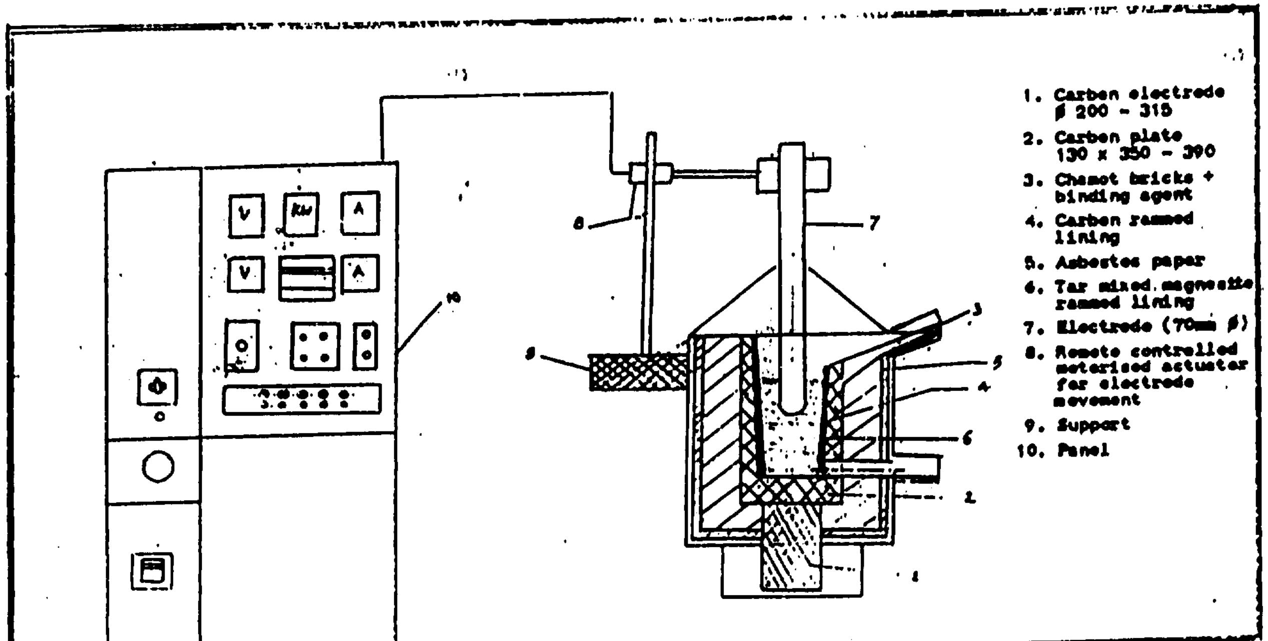
Test No. OVE-	Red.time min./h	Oxidat. time	Temp. oC av.	Discharge mater. WT g	% magn.	% n-magn.	Analysis of magnetics, % Fe total	Fe2+	Fe3+	Fe metallic	Metallization	Analysis of discharge material, % Fe total	Fe2+	Fe3+	Fe met.	Metallization
204	20	-	1001	1042	75.83	24.17	35.18	10.78	2.39	25.13	71.43	27.93	13.64	1.60	16.20	58.00
203 x	20	-	1001	1135	67.57	32.43	36.30	20.10	0.80	20.11	55.40	31.28	18.67	2.80	14.80	47.31
207	45	-	1000	1031	72.05	27.95	35.74	9.34	3.19	26.25	73.44	27.92	20.11	9.58	5.58	19.98
205 x	45	-	1002	1090	76.36	23.64	36.30	22.26	0.80	18.43	50.77	32.39	4.31	20.76	14.57	44.82
206	60	-	1000	1057	77.14	22.86	35.74	18.31	2.79	19.55	54.70	29.04	9.70	5.19	17.87	61.53
202	120	-	1003	1023	77.89	22.11	36.30	7.19	3.99	27.92	76.91	31.83	17.95	2.39	16.2[illegible]	50.89
201	12h	-	1000	984	83.40	16.60	35.36	5.75	4.79	29.04	78.76	32.95	12.56	5.59	19.27	53.48
208	20	-	1095	994	75.76	24.24	34.63	9.43	1.60	26.25	75.00	31.83	10.06	6.38	19.55	61.42
209	4h	10h	1000	1079	68.37	31.63	33.57	20.11	11.76	9.49	28.27	27.93	6.82	25.96	4.47	16.00
210	4h	10h	1001	1073	69.37	30.63	27.92	17.95	3.99	11.17	40.00	29.04	25.13	6.30	5.03	17.32

x) sulphide addition

Procedure: heating - loading - heating - cooling to ab. 200oC - disharge
Total treatment time = reduction time + av. 17 1/2 min.
Temperature drop during loading: av. 28oC
Reductant: Leco coal (lignite), Fixed C 62 %; ash 15 %, Volatile matters 23 %
C/Fe = 0.6
Nitrogen atmosphere
Oxidation by oxygen gas
Sulphide: iron sulfide calcine with 2.8 % S = 0.1 % S of iron in charge

TECHNICAL DATA OF SAF

1.	Transformer rating	50 KVA
2.	Rated current	667 A max.
3.	Weight	419 Kg.
4.	Overall dimensions	
	Width	615 mm
	Depth	560 mm
	Height	868 mm
5.	Electrode dia	70 mm
6.	Distance between tap hole & hearth.	100 mm
7.	Tap hole diameter	40 mm
8.	Internal depth	440 mm
9.	Electrode stroke	600 mm
10.	Electrode feed speed	0.21 m min^{-1}



GENERAL ARRANGEMENT OF LABORATORY SAF, 50 KVA CAPACITY

VI.3.10 Smelting tests on ilmenite

Test No DVS-	Charge, kgs Ilmenite conc.	Leco coal	Lime	Fluorspar	Basicity	Products Matte kg	C %	Slag kg	Fe tot. %	Remarks
211	10.0	1.30	0.6	-	2.1	0.746	1.78	5.200	5.58	Crust formation observed. Same test repeated. Metal temp. good. slag viscous. Slag could contain coke ash left in F/C.
212	10.0	1.50	0.6	0.25	2.1	2.124	1.71	5.118	4.47	Metal temp. good, slag still viscous.
213	10.0	2.00	0.6	0.25	2.1	0.326	1.83	5.062	4.05	CaF2 added. Metal and Slag temp. good.slag fluid Some metal in the F/C
214	10.0	2.50	0.6	0.40	2.1	1.160	1.5	2.344	5.30	Metal and Slag temp. low. Slag is viscous. Considerable metal quantity in F/C.
215	10.0	2.50	0.45	0.25	1.6	1.604	1.32	9.668	3.32	Slag is fluid. Good yield of previous heat.
216	10.0	2.50	0.25	-	1.5	1.366	1.20	4.470	5.50	All metal drained. Metal and Slag fluid. 1.0 Kg Iron ore added to oxidise Fe to Fe3O4.
	Pre-reduced ilmenite									
217	08.5	0.24	0.64	0.16	2.2	2.388	1.6	2.932	5.02	Pre-reduced Ilmenite used for melting. Metal and Slag fluid.

VI.4 Bibliography

The following articles and papers were given by the undersigned to the SIIL's Head office in Hyderabad and R/D department in Paloncha. The content of the documents was thoroughly discussed with the persons concerned.

- "Arc-furnace research and development",K.J. Irvine, Ironmaking and Steelmaking, 1977 No.2

- Discussion: Special technique and the future, Chairman: Dr H.W. Kirkby, CBE (Firth Brown Ltd) "A new constituent for slags used in Steelmaking", Ironmaking and Steelmaking, 1977 No.2

- "Industrial process for recovery of metal values from alloy scraps - pretreatment of alloy scrap", Osmo Vartiainen, OV-Eng Oy(Osmo Vartiainen Engineering Co.), Helsinki, Finland, MMIJ/IMM, Kyoto 1989

- "Factorial design and analysis to obtain optimum operation conditions", Osmo Vartiainen, MSc., Lic.Sc., etc. IFAC Symposium, Helsinki, Finland, 1983

- "Process for recovering metal values from alloy scraps", Osmo Vartiainen, Helsinki, Finland, USA-pat., 1984

- "Westralian Sands", International Mining, June 1987