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FINAL REPORT ON TESTS ON IRON ORE AND COAL
SAMPLES RECEIVED FROM THE GOVERNMENT OF VIETNAM
THROUGH UNIDO

REF: CONTRACT NO.84/98 (PROJECT NO.SI/VIE/84/801)

DECEMBER 1985

ENGINEERING AND PROJECTS DIVISION
SPONGE IRON INDIA LIMITED
HYDERABAD

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FINAL REPORT ON TESTS ON IRON ORE AND COAL SAMPLES
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1.0

INTRODUCTION

United Nations Industrial Development Organisation (UNIDO) vide their P.O.No.15-4-0098 (Contract No.84/98) dated 8.3.1985 awarded to Sponge Iron India Limited (SIIL) the work relating to investigations on the feasibility of production of sponge iron suitable for steelmaking using iron ore and coal from Vietnam. The Contract was awarded in response to the proposal for undertaking such a study, submitted by SIIL vide their reference No.SI/E&P/8168/279/84 dated 12.9.1984.

1.2

In terms of the Contract the scope of work is as follows:

- a) Beneficiation and upgrading of iron ores wherever required, to high grade green concentrate consistent with optimum recovery/yield figures.
- b) Pelletizing of the green concentrate to yield high quality heat hardened pellets.
- c) Investigation work using pellets (vide 'b' above) and local non-coking coals to produce highly metallised sponge through direct reduction in rotary kiln operations.
- d) Preparation of a comprehensive self-contained detailed report covering 'a' to 'c' above.

1.3

Iron Ore and Coal samples of approximate quantity of 100 Kg of iron ore and 70 Kg of coal were received from the Institute of Ferrous Metallurgy, Ministry of Mechanic and Metallurgy, SRV for undertaking the above tests. The samples were received at the SIIL Test Centre in the second week of May'85 and soon after, the test work was undertaken. On the basis of chemical investigation carried out on the samples, it was established that no beneficiation or upgradation of the samples particularly for iron ore, is necessary and they could be utilised for test work, as received. Accordingly, the following programme of test work was drawn up.

- i) Chemical Analysis of Iron Ore and Coal.
- ii) Determination of physical characteristics of iron ore and coal.
- iii) Study of ash fusion characteristics of reductant viz , coal.
- iv) Determination of reactivity of coal.
- v) Reducibility Tests.

2.0 TEST OBJECTIVES

- 2.1 The prime objective of the Bench Scale Tests was to determine the suitability of iron ore and coal supplied by the Government of Vietnam under Project No.SI/VIE/84/801 for production of highly metallised sponge iron suitable for steelmaking in Electric Arc Furnaces. The tests carried out and their objectives are summarised below:
- 2.1.1 To analyse the iron ore chemically in order to determine its suitability with regard to Fe-total, gangue and other constituents, for sponge iron production and for subsequent processing in Electric Arc Furnaces for steelmaking.
- 2.1.2 To determine the cold strength of the iron ore and its resistance to abrasion, impact and other handling methods using the shatter test apparatus.
- 2.1.3 To determine the decrepitation and reduction behaviour of the iron ore during reduction under standard test conditions.
- 2.1.4 Chemical Analysis of coal to determine fixed carbon, ash and volatile matter and also detailed analysis of ash in the coal.
- 2.1.5 To determine the reactivity of the coal and to predict the optimum material bed temperature to ensure the desired degree of reduction.

2.1.6 To study the ash softening and fusion characteristics of the coal to predict optimum operating temperature in the rotary kiln.

3.0 TECHNICAL REQUIREMENT OF RAW MATERIALS

3.1 Iron Ore, Coal and Limestone are the basic raw materials required for the production of sponge iron in the rotary kiln process based on 100% coal operation. As the manufacture of sponge iron in rotary kiln is sensitive to the characteristics of the raw materials with regard to size distribution and other chemical parameters, bench scale testing forms the first essential step in determining the suitability of any ore, coal and limestone combination for the production of sponge iron suitable for Electric Arc Furnace steel-making.

3.2 Iron Ore

Iron Ore should have as high iron content as possible, preferably above 62% with low gangue components and low levels of impurities such as sulphur and phosphorus. The requirement of high iron content in ore arises from the needs of the process of conversion of sponge iron into steel in Electric Arc Furnace. The higher iron level in the ore gives rise to higher total iron in the product and consequently higher liquid metal yield in Electric Arc Furnaces and minimum iron losses in the slag. Low levels of gangue are required as there is no removal of gangue in the direct reduction process. Further gangue requires additional melting power and appropriate limestone additions to remove it as slag in the steelmaking stage. Based on the operating experience, total gangue below 6% would be desirable in the iron ore with silica being less than 3%. The Sulphur and Phosphorus contents are also of utmost importance while selecting

the ores keeping the specification of the steels to be produced in view.

Suitable precautions are to be taken for desulphurisation in the rotary kiln, as sponge iron tends to pick up the sulphur from coal in the absence of desulphuriser. Phosphorus level in ore assumes importance as the phosphorus in the ore is retained in the sponge iron without any change during the reduction process. Eventhough it is desirable to limit the phosphorus below 0.06% in the ore, it has been observed that phosphorus levels could be tolerated upto 0.12% in sponge iron. It has been reported that phosphorus is in the oxide form in the gangue matrix and is absorbed by the slag at lower oxidising potential and at lower basicities in Electric Arc Furnaces. Apart from the chemical characteristics as above, the iron ore 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 iron ores should not be less than 25%. In addition to the physical strength, the decrepitation behaviour of the ores during reduction is of specific importance for rotary kiln operations as the fines generated during reduction form low melting compounds with the coal ash and stick to the refractory lining of the rotary kiln.

It would be desirable to have less than 5 to 7 percent -1mm fines in the product. The ore should also have good reducibility with coal to be suitable for use at the kiln operating temperatures. Reducibility index of the order of 94 to 96 per cent tested at a temperature of 1000°C is found to be optimum to get metallisation level of 90 to 92 per cent at the kiln operating temperature. Reducibility index lower than the above value will adversely affect the throughput of the kiln.

3.3

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 lower temperatures and at high throughput rates. In general coal reactivity should be of the order of 2.2 cm³ of CO/gC Sec. 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 ash content in coal can be tolerated upto a level of 25 per cent for use in rotary kilns and any increase beyond this level will reduce the throughput capacity. The volatile matter in coals should be of the order of 30 per cent so as to heat the iron ore to the reduction temperature within the shortest possible time. The fixed carbon should be of the order of 40 to 45 per cent.

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 sponge iron fines, it is desirable to have softening point of coal ash in excess of 1160°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 $100 - 150^{\circ}\text{C}$ 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 the inorganic 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 sponge iron in the absence of desulphuriser. In short the total sulphur in coals should be low, preferably below 1%. However, coals having high percentage of fixed carbon like anthracite, could also be considered as reductant through blending with bituminous or sub-bituminous coal so that the reactivity of the coal blend improves. 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.

3.4

Limestone

Limestone is used in the process as a desulphurizer as the coal used for reduction contains sulphur. Limestone containing an average of 45 per cent 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.

4.0 TEST RESULTS AND EVALUATION

4.1 Chemical Analysis of Iron Ore Samples

4.1.1 The iron ore samples were analysed for the constituents such as Fe-total, FeO, SiO₂, Al₂O₃, CaO, MgO, S, P and LOI. The chemical analysis was carried out as per standard procedures. The results of the chemical analysis of the iron ore samples are presented in Table - 1. The analysis of the iron ore indicates the presence of higher percentage of Fe⁺⁺ and consequently the presence of magnetite. This would however require to be reconfirmed by conducting mineralogical studies which can be performed on the samples to be received for tonnage scale tests subsequently.

4.1.2. Fe-Total:

Chemical analysis of iron ore sample indicated that the iron content is in general high of the order of 67% and the ore as such does not require any beneficiation or upgradation.

4.1.3 Gangue content

The gangue content in the ore sample is 2.62% in which of SiO₂ accounts for 0.7% only the balance being Al₂O₃. It is considered that the low SiO₂ level would be beneficial in Electric Arc Furnace steelmaking.

4.1.4 Impurities

The impurities normally present in iron ore like sulphur and phosphorus are negligible in the samples received and as such the ore is extremely suitable for producing high quality and special steel.

4.1.5 LSI

The loss in ignition is also on the lower side which further substantiates that the ore is of superior quality and suitable for sponge iron manufacture by Direct Reduction Process in a rotary kiln.

4.2 Physical Tests

4.2.1 Shatter Tests:

The shatter tests indicate the cold strength of the material for its resistance to abrasion, impact and handling. The shatter index of 98.3% establishes that the ore is of hard type and can withstand multiple handling.

4.2.2 Bulk Density: The Bulk Density of the iron ore is 2.1 T/M³.

4.2.3 Screen Analysis:

The screen analysis of iron ore as received is at Table-2.

4.2.4 Thermal Degradation Tests

The Thermal Degradation of ore is defined as the fragmentation and breaking up of the ore when subjected to heating to a temperature of 700°C and cooling in the presence of air. The thermal degradation index of the

iron ore under dynamic conditions is of greater significance in the rotary kiln process. The test conditions for the dynamic thermal degradation test are presented at Annexure-1 and the test results are furnished in Table-3.

The thermal degradation index is 9.7% which further confirms the suitability of the iron ore for use in rotary kiln for direct reduction.

4.3 Coal

4.3.1 Proximate Analysis

The proximate analysis of the coal samples was carried out as per the standard procedure. The average results of the analysis are presented in Table-4. From the table it could be seen that the volatile matter is only 4.21 per cent, fixed carbon is 90.19% and ash is 5.6%. From this, it is observed that the coal is of the anthracite variety.

4.3.2 Sulphur content

The sulphur content of the coal is one of the important characteristics for evaluating its suitability 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 sulphur pickup in sponge iron in the absence of desulphuriser. The total sulphur in the coal samples was analysed by gravimetric method as per standard procedure. The different forms of sulphur in the

coal were also analysed and the results are presented in the Table-4. The total sulphur in coal was less than 0.60 per cent which is well within the normal range indicated for coals for direct reduction purpose.

4.3.3 Calorific Value

The average net calorific value of the coal samples was observed to be of the order of 9700 K cal/Kg which is considered more than adequate for the specified purpose. The calorific value of the reductant was determined in an Adiabatic Bomb Calorimeter.

4.3.4 Chemical Analysis of the coal ash

The chemical composition of the coal ash influences the softening characteristics of the coal ash. Higher concentrations of silica and alumina are generally associated with higher softening temperatures. However, higher concentration of iron oxide in the ash lowers the softening point considerably. The analysis of coal ash for the determination SiO_2 , Al_2O_3 , Fe_2O_3 , CaO and MgO was carried out and the results are presented in Table-4. The results of the chemical analysis of the coal ash indicate that the ash is acidic in nature, with silica accounting for 49%.

4.3.5 Melting Characteristics of the coal ash

In the Rotary Kiln Process the kiln is to be operated normally at a temperature of 1050°C to get the desired degree of reduction and this requires coals with relatively

higher ash softening temperatures of the order of 100-150°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 the coal ash was found to be extremely good and the softening point itself was observed to be more than 1500°C. The test results of the melting behaviour of the coal ash are given in Table-4. The photographs of the Leitz Heating Microscope and the various stages of the sample during ash fusion test is shown at Plate 1,2 & 3.

4.3.6 Reactivity of the Coal

Reactivity of the coal refers to the amount and the rate of carbon monoxide generation through the well known Boudouard reaction. This is an important factor in the rotary kiln operations since the generation of carbon monoxide required for the reduction of iron ore is formed in situ and is a function of the temperature in the kiln. The reactivity of the coal is determined by the weightloss method. The test results carried out on the coal give the reactivity as 0.8 cc of carbon monoxide per gram of carbon per second. This value as compared to normal bituminous coals is low and therefore, the use of this coal for reduction calls for a higher operating temperature so that metallisation of the product is satisfactory. This aspect has been amply confirmed in the reducibility tests described in the next chapter of the report where higher operating temperature and longer retention time gave better levels of metallisation.

Alternatively the reactivity of coal can be improved by suitably blending with bituminous or sub-bituminous coals.

4.4 REDUCIBILITY TESTS

4.4.1 The reducibility tests were conducted in an electrically heated laboratory rotary furnace. The photograph of which is shown at Plate - 4. The results of tests on Vietnam Ore in combination with Vietnam Coal at standard test conditions i.e. at a reduction temperature of 1200°C with retention time 3 hours and at a reduction temperature of 1050°C with retention time 4 hours is shown at Table - 5 & 6. Comparative tests carried out on the iron ore sample from vietnam with local coal (Manuguru coal of Singareni Collieries) is at Table - 7. It can be seen from the reducibility test results that for Vietnam coal it is necessary to maintain higher temperatures to achieve the required levels of metallisation whereas with local coals, lower operating temperature of the order of 1000°C was sufficient for attaining the required levels of metallisation. The tests establish that the iron ore is highly reducible and therefore suitable for direct reduction.

4.4.2 Decrepitation Behaviour

The decrepitation behaviour with regard to generation of -1 mm fines in the product was extremely good (only of the order of 4%). The 1-3 mm fines fraction was of the order of 15-20% and as such the ore can be considered ideal from the operation point of view for direct reduction in a rotary kiln.

4.4.3 Process Degradation Index

The process degradation index of iron ore which is about 30% is extremely satisfactory. In respect of coal it is 26.5% which is reasonable.

4.4.4 Grain Size wise analysis

The grain size analysis of the magnetic product for Fe-Total, Fe-Metallic and degree of metallisation are indicated at Table - 8. From the table it can be seen that the degree of metallisation for higher size fraction is reasonably good which is indicative of the characteristic of the ore and it also confirms that higher size fractions of iron ore could be used in the Direct Reduction Process.

4.4.5 Chemical Analysis of the Product

The detailed chemical analysis of product is at Table - 9. It can be seen from the table that sulphur and phosphorus levels are negligible and there is no appreciable pickup of sulphur during the reduction process.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 From a review of the bench scale test results, it is observed that the ore possesses good reducibility characteristics. This has been confirmed when reducibility tests were carried out with local coals when it was possible to achieve good metallisation levels averaging 95% (with only 3 hrs. retention time at a temperature 1000°C). However, this could also be on account of better reactivity of local coals. However, in respect of the test results carried out with Vietnam coal, it is observed that higher operating temperatures are required to be maintained to achieve the desired levels of metallisation.

5.2 Coal: Coal samples are of anthracite type and comparatively less reactive. In order to achieve better reduction it is necessary to operate at higher temperature of the order of 1100°C with longer retention time and also increased quantity of coal. This is confirmed in the test results where with a reduction temperature of 1050°C, retention time of 4 hrs. a carbon(fix)/iron ratio of 0.7, optimum results were obtained. As ash fusion temperature of Vietnam Coal is considerably high (above 1500°C) there is no danger of ash fusion and consequent operational problems when higher operating temperatures are aimed at for achieving the desired level of reduction. In view of this, it is possible to use anthracite coal satisfactorily for reduction purposes except that a higher carbon fix/iron ratio would have to be maintained for achieving the desired metallisation. Alternatively, through blending bituminous coal to an extent of about 20-30% the desired results

could be obtained. This can be confirmed in a tonnage scale test in which injection coal from the discharge end of the kiln could be of bituminous variety the feed coal being similar to the sample supplied for the Laboratory Scale Tests.

Table-1CHEMICAL ANALYSIS OF IRON ORE

Constituent	-----		
	1st %	2nd %	Ave %
Fe-Total	67.02	67.02	67.02
Fe ⁺⁺	9.77	7.26	8.52
FeO	12.57	9.34	10.95
Fe ₂ O ₃	81.85	85.44	83.64
SiO ₂	0.65	0.75	0.70
Al ₂ O ₃	1.79	2.05	1.92
CaO + MgO	Traces	Traces	Traces
Sulphur	<.01	<.01	<.01
Phosphorus	0.042	-	0.042
L.O.I	1.14	1.55	1.35

Table-2

PHYSICAL DATA OF VIETNAM ORE & COAL
AS RECEIVED

<u>Size</u>	<u>Ore</u> <u>%</u>	<u>Coal</u> <u>%</u>
+20	12.80	Nil
+15	40.48	0.98
+10	46.40	43.93
+8	0.32	28.85
+5	-	18.36
+3	-	4.93
+1	-	2.62
-1	-	0.33
Total	100.00	100.00

Table - 3

PHYSICAL DATA OF AS RECEIVED SAMPLES

	<u>Vietnam Ore</u>	<u>Vietnam Coal</u>
1. Bulk Density T/M ³	2.1	0.475
2. Shatter Index	98.30%	
3. Thermal Degredation Index	9.70%	

PROPERTIES OF REDUCTANT - VIETNAM COAL

Proximate Analysis (% Dry)

Ash	:	5.60
V.M	:	4.21
F.C	:	90.19
		<hr/>
Total		100.00
		<hr/>

Forms of Sulphur

Organic Sulphur	:	0.523
Pyritic Sulphur	:	0.050
Sulphate Sulphur	:	0.017
Total Sulphur	:	0.59

Net Calorific Value : 9703.14 Kcal/Kg

Ash Analysis

SiO ₂	49.00
CaO	2.35
MgO	Traces
Al ₂ O ₃	33.02
Fe ₂ O ₃	5.59
K ₂ O, Na ₂ O and others	10.04

Melting behaviour of coal ash

Softening point °C	:	above - 1500°C
Melting point °C	:	"
Flow point °C	:	"
Reactivity	:	0.8 cc of CO/g of C

Table -5

SUMMARY OF SALVIS KILN TEST RESULTS

Ore ;VIETNAM		Reduction Temp. 1000 ^o C Reductant: Vietnam Coal			Retention Time - 3hrs. C/Fe: 0.5		Kiln discharge		
Sl.No.	Met %	R.I.	Decrepitation Behaviour			Process Degradation		Mag %	Non-mag %
			-1mm	-3mm	-5mm	Oxide feed	Reductant		
1.	below 50	-	16.03	30.53	37.39	37.19	42.42	55.27	44.7
2.	-do-	-	15.15	29.54	35.60	43.70	42.02	58.15	41.8
3.	-do-	-	12.39	26.55	35.40	42.08	24.71	47.88	52.1

Table-6

RESULTS OF TESTS CARRIED OUT IN LABORATORY ROTARY FURNACE

Ore: Vietnam

Reductant : Vietnam Coal

C/F = 0.7

Sl.No.	Temp. °C	Time (hrs.)	Meta- llisa- tion %	Redu- ction Index %	<u>Decrenititation behaviour</u>			<u>Process degra- dation</u>		% of Magne- tics	% of non- magne- tics
					-1mm %	-3mm %	-5mm %	Oxide feed %	Reductant %		
1.	1050	4	93.00	95.08	1.71	13.68	18.81	21.08	30.32	60.94	39.06
2.	1050	4	86.00	90.17	4.46	19.64	26.78	32.81	29.43	55.56	44.44
3.	1050	4	89.00	92.28	4.08	24.47	34.69	38.50	20.13	56.00	44.00
Average 1050		4	89.33	92.51	3.42	19.26	26.76	30.79	26.63	57.5	42.50

Table-7

RESULTS OF TESTS CARRIED OUT IN LABORATORY ROTARY FURNACE

Ore : Vietnam

Reductant : Manuguru Coal

C/Fe = 0.5

Sl.No.	Temp. °C	Time (hrs.)	Meta- llisa- tion %	Reduc- tion Index %	Decrepitation behaviour			Process Degradation		% of Magne- tics	% of non- magne- tics
					-1mm %	-3mm. %	-5mm %	Oxide feed %	Reductant %		
1.	1000	3	96.00	97.19	4.62	21.44	30.79	36.40	40.25	56.32	43.68
2.	1000	3	96.00	97.19	4.24	20.34	28.82	33.96	45.86	59.90	40.10
3.	1000	3	94.26	95.97	6.35	19.09	26.90	32.98	40.13	63.32	36.68
Average	1000	3	95.42	96.78	5.07	20.29	28.86	34.41	42.08	59.85	40.15

Table-8

GRAIN SIZEWISE ANALYSIS (1050°C 4 hrs.)

Vietnam Ore + Vietnam Coal

	<u>Fe(T)</u> %	<u>Fe(Net.)</u> %	<u>Metallisation</u> %
-1 mm	86.01	75.35	87.00
+ 1-5 mm	90.48	83.69	92.50
+ 5-10 mm	92.15	83.89	91.04
+ 15 mm	87.13	74.93	86.00

PROXIMATE ANALYSIS OF COMPOSITE NON-MAGNETICS SAMPLE

<u>VM</u> %	<u>Ash</u> %	<u>FC</u> %
1.31	24.44	74.25

Table-9

CHEMICAL ANALYSIS OF SPONGE IRON

Fe-Total	:	94.11
Fe (metallic)	:	87.88
Metallisation	:	93.39
FeO	:	8.01
SiO ₂	:	1.02
Al ₂ O ₃	:	2.25
Phosphorus	:	0.046
Sulphur	:	0.01
CaO + MgO	:	Traces

THERMAL DEGRADATION OF IRON ORE (DYNAMIC)

(A) Raw Materials Requirements:

- i) Chemical Analysis of iron ore
- ii) Screen Analysis of iron ore

(B) Test Conditions

Charge

Weight of ore : 1000 gm
Size of ore : 6-20mm

Furnace Conditions

Temperature of furnace of feeding : Room Temp. 30°C
Heat up time : 120 minutes
Test Temperature : 700°C
Testing of period : 120 minutes
Sample Cooling : Furnace Cooling
Furnace Atmosphere : Air

- (C) Thermal Degradation Index expressed as percentage of (-5mm) fraction produced in heat sample.

SPONGE IRON INDIA LIMITED
TEST CENTRE

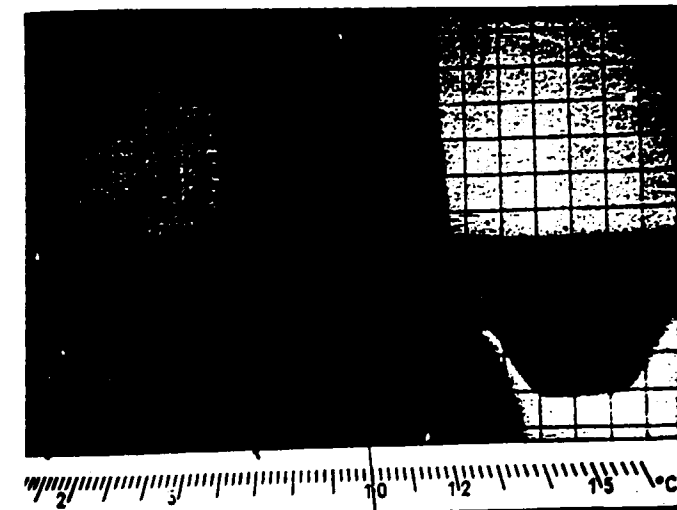
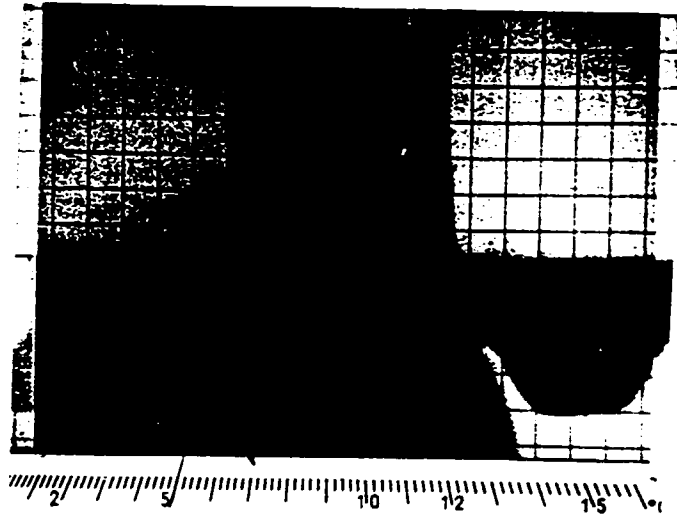
LIETZ HEATING MICROSCOPE



SPONGE IRON INDIA LIMITED

TEST CENTRE

ASH FUSION CHARACTERISTICS - VIETNAM COAL

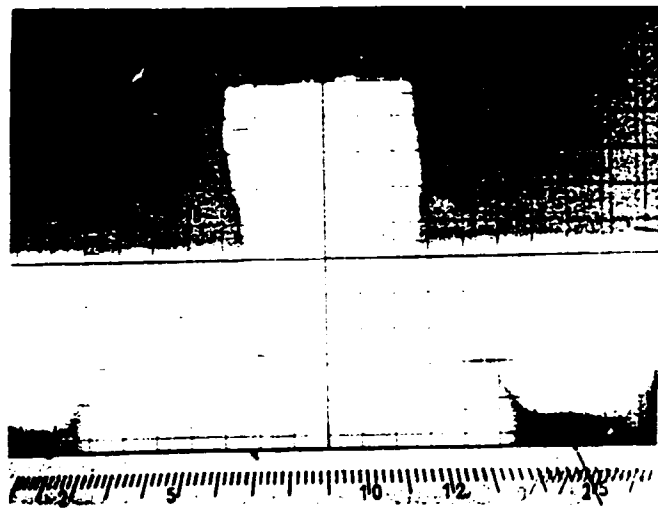


SIL

PLATE-2

SPONGE IRON INDIA LIMITED
TEST CENTRE

ASH FUSION CHARACTERISTICS - VIETNAM COAL



SII

PLATE-3

SPONGE IRON INDIA LIMITED

TEST CENTRE

LABORATORY ROTARY FURNACE



SII

PLATE-4