A Thesis
On

PREPARATION AND CHARACTERIZATION OF SPONGE IRON

Submitted in the partial fulfillment of requirement for the
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In

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Submitted by

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CERTIFICATE

This is to certify that the thesis entitled, **"PREPARATION AND CHARACTERIZATION OF SPONGE IRON"** submitted by **Mr. Arun Kumar Singh** in the partial fulfillment of the requirement for the award of the degree of M. Tech in **Materials Science and Engineering** from the **School of Physics and Materials Science, Thapar University, Patiala**, is a record of candidate's own work carried out by him under our supervision and guidance. The matter embodied in this thesis has not been submitted in part or full to any other university or institute for the award of any degree.

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ABSTRACT

Studies on reduction behavior of hematite iron ores with coal procured from different mines were undertaken to achieve better quality of sponge iron. Majority of the iron ores contain high iron and low alumina as well as silica contents. All of these iron ores are free from the deleterious elements (S, P, As, Pb, alkalies etc.). The size of iron ore is very important for reduction reaction. It has been observed that rate of reduction reaction of iron oxide increases with decrease in size of ore. It is observed that for very small size particle, reduction velocity is independent of the diameter whereas for medium size particle it is inversely proportional to (d) diameter of the particle and for larger size it is proportional to $1/d^{2.}$

The reduction of iron oxide is performed under controlled atmosphere. Slowly reductants diffuse inside solid body and oxygen gets removed. It was observed that the iron oxide changes its phases from hematite to magnetite, magnetite to wustite and finally to metallic iron. The degree of reduction is influenced by the coal in the reduction zone area inside the kiln if good quality coal is used. The carbon and oxygen combine together to form carbon monoxide which is used as a reducing gas for the iron ore. The metallic layer thickness gets diminished due to the volatile matter present in coal. An increase in the thickness of product iron layer offers more resistance to the diffusion of carbon and reducing gas and prevents it to interact with the surface of unreduced iron oxide.

Iron ore lumps shows lower degree of reduction than the corresponding fired pellets (small size iron ore). Coal becomes more reactive and occupies less volume in kiln if it has high volume of fixed carbon as well as volatile material in its composition. This results in increase of fusion temperature, good quality and high production of sponge iron. In this work the variables which influence the quality of sponge iron to achieve different grades of sponge iron has been studied and presented in subsequent chapters.

TABLE OF CONTENT

Chapter 1 INTRODUCTION

The conventional route for making steel consists of sintering/pelletization plants, ovens, blast and basic oxygen furnaces. Such plants need huge capital expenses and raw materials as they are the stringent requirements. The integrated steel plants having production less than one million tons annually are generally not considered economically. Along with that the coke ovens and sintering plants in an integrated steel plant are highly polluting. On the other hand installation of highly complex and expensive pollution-control systems make this route highly capital intensive.

Taking into account all the above mentiond demerits a new process known as Direct Reduction of Iron (DRI) process has been developed for iron making. This process is widely accepted by almost all the developing nations. The generation of scrap was less in comparison to the requirement of steel and also steel produced by recycling process is found to follow the more economic pathway than its production via Blast furnace –Basic oxygen furnace (BF-BOF) process. The coal base DRI rotary kiln used for the production of sponge iron with increased quality and produceability needs careful operations throughout the process [1]. Number of DRI proceses came up but rotary kiln process with solid coal as fuel is found to be most acceptable process among others. The rotary kiln DRI process uses non coking coal as major fuel and India is one of the big production house of this fuel. Hence it makes this DRI process to be more economic.

DRI production and consumption are influenced by a range of regional controls which include employment costs, power and gas prices, coal availability and quality, iron ore reserves, infrastructure and the availability as well as prevailing prices of prime scrap.

1.1 RAW MATERIALS: [2]

Following are the key raw materials required for the formation of iron.

1.1.1 Iron Ore: The iron ore used is hematite having Fe content 62-66%. Earlier the initial days the iron ore size was kept at 5-20 mm and was washed in a scrubber, but presently it has become a standard norm to use 5-18 mm ore as feed for a large kiln without scrubbing and/or washing. This has resulted in reducing the cost of iron ore fed to the kiln. The consumption of iron ore has also decreased from about 1600 kg per tonne of sponge iron to 1500 kg levels mainly due to a better understanding of the process, improvements of the equipment and increased levels of automation.

Two main types of iron ore used for iron making are magnetite (Fe_3O_4) and hematite (Fe_2O_3). Common iron ores include:

- **Hematite** - Fe_2O_3 - 70 percent iron
- **Magnetite** - Fe_3O_4 - 72 percent iron
- **Limonite** - $Fe_2O_3 + H_2O$ - 50 percent to 66 percent iron
- **Siderite** - $FeCO_3$ - 48 percent iron

Hematite deposits are mostly sedimentary in origin, such as the banded iron formations (BIFS). BIFS consist of alternating layers of chart (a variety of the mineral quartz), hematite and magnetite. They are found throughout the world and are the most important iron ore in the world today.

It is also marked that reducibility of hematite ore is greater than magnetite as the oxygen removal is 5% more in case of hematite than magnetite. It is because during the reduction the hexagonal hematite gets changed to cubic magnetite and finally to wustite. By this transformation from hexagonal lattice, the volume of solid expands to around 25% more resulting in higher exposure of surface area for reduction where as during transformation of magnetite to wustite the volume expansion is considerably small resulting in poor reducibility of magnetite. It is therefore hematite ore is preferred for sponge iron making.

Agglomeration of Iron Ore

Iron ore fines/blue dust cannot be charged in the blast furnace directly since they block the passage for ascending gas inside the feed. So they are agglomerated (by igniting at lower temperature causing only interfacial fusion) into larger lumpy pieces with or without addition of additives like limestone, dolomite etc. Two types of agglomerated products are commonly used in the industry namely sinter and pellet. Accordingly the processes are known as sintering and pelletising respectively.

(a) **Sinter**: sinter is a clinker like aggregate which is normally produced from relatively coarser fine iron ore (normally –3mm) mixed with coke breeze (-3mm), limestone dolomite fines (-3mm) and other metallurgical return wastes from the plant. Sinter is a much preferred input/raw material in blast furnaces. It improves BF operation and productivity and reduces coke consumption in blast furnace. Presently, more than 70% hot metal in the world (in India 50%) is produced through the sinter.

(b) Pellet: pellets are normally produced in the form of globules from very fine iron ore (normally –100 mesh) and mostly used for production of sponge iron in gas based plants, though they are also used in blast furnaces in some countries in place of sized iron ore

Figure.1.1: Iron ore pellets

High-grade iron ore fines are used for the preparation of pellets (iron content in the ore has got to be very high and gangue low to confirm to the quality of pellets. The content generally is 66% Fe, $SiO_2 + A1_2O_3$ – 3%, sulphur and phosphorus 0.05% each. Pellets are ideal material as a feed to direct-reduction iron (DRI) plants.

Iron Ore Pellet is better Than Iron Ores Lump [2]

1. Very high tumbling index.
2. No disintegration during handling.
3. Low abrasion index compared to lumps.
4. Uniform in size and shape.

5. High compression strength
6. Good porosity.
7. Cannot be contaminated with granite during transportation

1.1.2 Coal: Non-coking coal is being used having certain important parameters considered necessary for the direct reduction of iron ore viz. reactivity, ash softening temperature, caking and swelling indices and sulphur content, etc.

The industry has successfully adopted measures to utilize ‘C’ and ‘D’ grade coals through better process control, installing raw material heating systems, shale picking belts and coal washing plants. With these measures the coal cost has been reduced by nearly 20-30% when compared with the usage of ‘B’ grade coal.

Table 1.1: Classification of coal [3]

Name	Range of Volatile %	Range of Moisture
Anthracite	A_1 3-10	1-3
	A_2 10-15	1-2
Bituminous	B_1 15-20	1.5-5
	B_2 20-32	2.5-5
	B_3 32 over	1-3.0
Lignite	L_1 45-55	10-20
Brown coal	L_2 55-65	10-25

Table 1.2: Grade of coal

Grade	Calorific Value (K. Cal)	Ash+Moisture (%)
A	6200	19.6
B	5600	19.6 – 23.9
C	4940 -5600	23.9 – 28.7
D	4200- 4940	28.7 – 34.1
E	3360 – 4200	34.1 – 40.1
F	2400 – 3360	Nil
G	1300 – 2400 max	Nil

1.1.3 Dolomite: Dolomite is mainly used as a desulphurising agent to prevent the pickup of sulphur by the sponge iron from the sulphur released by the burning of coal inside the furnace. The initial specifications for dolomite were 1-4 mm, later it was found that 4-8 mm dolomite was for more suitable by which the consumption can be reduced by 50%. This was mainly due to the fact that lot of dolomite fines were being lost to waste gases and with 4-8 mm fraction this loss was minimized.

Power: The initial plants were high power consuming units due mainly to the wet waste gas cleaning. The power consumption levels used to be 110-130 units per tonne of sponge iron, with the advent of a dry gas cleaning system (electro-static precipitator), programmable logic operated drives and computers replacing the giant panels, the power consumption has been curtailed to 80-90 units per tonne of sponge iron.

1.2 DEVLOPMENT OF DRI PROCESS AND IS VALUES FOR SPONGE IRON [4]

1.2.1 Coal-Based Process:

This process utilizes non-coking coal as reducing agent along with lumpy rich grade iron ore. The reduction is carried out in an inclined horizontal rotary kiln, which rotates at a predetermined speed. A temperature profile ranging from 800-1050 ^{0}C is maintained along the length of the kiln at different zones and as the material flows down due to gravity and ore is reduced.

The hot reduced sponge iron along with semi-burnt coal, discharged from kiln is cooled in water-cooled cylindrical rotary cooler to a temperature of 100–200 ^{0}C. The discharge from cooler consisting of sponge iron, char other contaminations are passed on through magnetic separators so that sponge iron can be separated from other impurities. Later the sponge iron is screened into two size fractions i.e. –3 mm & +3 mm. +3 mm fraction directly goes for usage, -3 mm fraction can be either used directly where ever it is possible or is to be briquetted by using molasses and hydrated lime as binders.

$$C + O_2 = CO_2$$

$$CO_2 + O_2 = 2CO$$

$$Fe_2O_3 + CO = Fe_3O_4 + CO_2$$

$$Fe_3O_4 + CO = FeO + CO_2$$

$$FeO + CO = Fe + CO_2$$

Raw Material Requirements

As in case of any process, here also quality of raw materials plays a vital role in obtaining the rated capacity and product quality. The iron ore shall have resistance for physical and thermal degrading and good reducibility and the coal should be of reactive nature along with the characteristics of high fusion temperature and less amount of ash.

Chemical and Physical Composition of Iron Ore

Chemical Composition	%
Total iron	65 - 67 %
$SiO_2 + Al_2 O_3$	2 - 3 %
CaO + MgO	0.5 - 1 %
Sulphur	0.02 % max.

Physical Composition	
Size	5 - 20 mm
Shatter index	+ 95 %
Tumbler index	+ 88 %
Abrasion index	+ 5 % max
Reducibility index	+ 94 %
Thermal degradation index	5%

Typical Chemical Analysis of Coal

Constituent	**%**
Moisture	8 % max
Total iron	27 - 30 %
Ash	23 -25 %
Fixed carbon	+ 40 %

Size	0 - 20 %
Ash softening point	1250 deg cent
Calorific value	5200 k . cal mole
Reactivity	2.2 CO / gm . deg

Typical Physical Analysis of Coal

CaO	40 - 45 %
L.O.I	40 - 45 %
Insoluble’s	5 - 7 %
Size	0.5 - 4 mm

Typical Analysis of Sponge Iron Produced from Coal Based Process

Total iron	91 - 93 %
Metallic iron	80 - 84 %
Metallisation	90 - 92 %
Gangue	3 - 4 %
Carbon	0.25 %
Sulphur	0.02 - 0.03 %
Phosphorous	0.035 - 0.05 %

1.2.2 Gas-Based Process:

This process utilizes natural gas as the reducing agent. Natural gas is reformed to enrich with H_2 and CO mixture and this enriched and reformed gas mixture is preheated in gas-based process. A vertical retort is used for the reduction of iron ore as against a rotary kiln in coal based sponge iron process. When the gases are travelling upwards the charge moves downward by gravity. Gas-based sponge iron is not subjected for magnetic separation, as no contamination with non–magnetic is possible either it can be cooled indirectly or briquetted in hot condition to get hot briquetted iron (HBI)

The basic reactions are as follows:

$$Fe_2O_3 + 3H_2 = 2\,Fe + 3\,H_2O$$

$$Fe_2O_3 + 3CO = 2\,Fe + 3\,CO_2$$

Here Are Only Two Well Established Processes

1. **MIDREX**
2. **HYL**

In gas-based process more preferable feed is pellets but lump ore is also used for with proper size distribution to have required permeability.

Physical and Chemical Composition

Chemical Property	**%**
Total iron	65 - 67 %
$SiO_2 + Al_2O_3$	2 - 3 %
CaO + MgO	1 - 2 %
Sulphur	0.025 %
Phosphorous	0.045 %

Chemical Analysis of Iron Ore Pellets

Total iron	64 - 65 %
$SiO_2 + Al_2O_3$	4 - 6 %
CaO + MgO	2 - 3 %
Sulphur	0.028 %
Phosphorous	0.045 %
Size	12 - 16 mm
Avg. Cold cr. Strength	200
Bulk density	2.2 t / m3

Typical Chemical Analysis of the Natural Gas

Constituent	% After reformation
Nitrogen	CO + H_2 - 90 %and above 0.3 -1.0%
Carbon Dioxide	0.1 - 5 %
Methane	85 – 93 %
Ethane	3 – 8 %
Propane	1 – 4 %

Typical Analysis of Sponge Iron produced from Gaseous Process

Total iron	92 %
Metallic iron	81 - 85 %
Metallization	85 - 93 %
Gangue	6 - 8 %
Carbon	1.2 - 2.5 %
Sulphur	0.003 % max
Phosphorous	0.06 % max

1.3 DIRECT REDUCED IRON (DRI) [5]

Sponge iron is the metallic product formed by the reduction (removal of oxygen) of iron ore at temperature below the fusion point of iron while it is still in solid state. It is also called direct reduced iron (DRI). Direct-reduced iron (DRI) is produced from direct reduction of iron ore (in the form of lumps, pellets or fines) by a reducing gas produced from natural gas or coal. The reducing gas is a mixture of majority of hydrogen (H_2) and carbon monoxide (CO). This process of directly reducing the iron ore in solid form by reducing gases is called direct reduction.

A broadly used iron source is also a product known as direct reduced iron ("DRI") which is produced by the solid state reduction of iron ore to highly metalized iron without the formation of liquid iron.

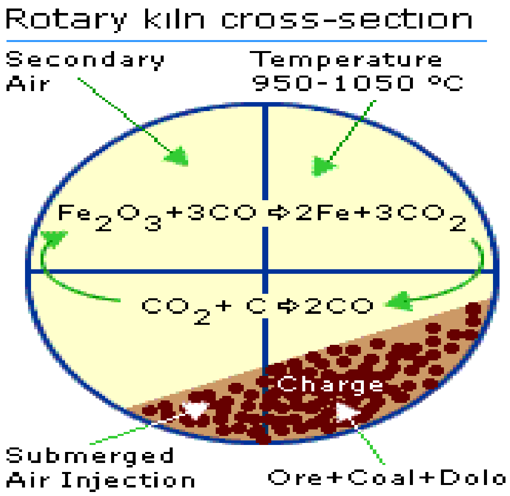

Figure.1.2: DRI rotary kiln cross –section

Sponge iron is the product created when iron ore is reduced to metallic iron, in the presence of coal, at temperatures below the melting point of iron. The external shape of the ore is retained with 30% reduction in weight due to oxide reduction resulting in change in true density from 4.4 gm/cc to 7.8 gm/cc in this product. **[6]**

1.3.1 Type of DRI Processes:

Table 1.3: Type of DRI process

Reducent used	**Reactor design**	**Process**	**Charge type**
Reducing gas produce from natural gas or Neptha	Static bed	HYL	sized lump/pellets
	Continuous retort	MIDREX	do
		PUROFER	do
		ARMCO	
	Fluidised bed	Hi Iron	sized ore
		Esso Fior	briquettes
		Nu Nron	
		HIB	
Solid and liquid or gaseous	Rotary kiln	SN/RN	sized lumps or pellets

1.4 SPONGE IRON

Hematite and magnetite are found on in earth crust as oxide form. Containing more than 27% oxygen. Oxygen particle occupy space in solid ore body in compound form in nature. In solid state reduction oxygen is removed from ore body making number of holes or cavities in the solid. During direct reduction, oxygen is removed from iron ore in solid state. This procedure results in a spongy structure of the product makes "sponge iron", with a high porosity. The manufacturing of sponge iron is highly sensitive to raw material characteristics. Depending on the raw material and the reduction process applied, apparent product density is approx. 2 g/cm³ associated with a very high specific surface area. The latter is typically around 1 m²/g. [8]

Figure 1.3: Sponge iron

Sponge iron is the product created by heating an iron ore at a temperature high enough to burn off its oxygen and carbon content but below iron's melting point.

1.4.1 Concept of Sponge Iron: [7]

The appearance of iron ore is completely changed when it gets converted to sponge iron of its composition. The hematite iron ore is most suitable ore whose chemical formula is Fe_2O_3 is most suitable raw material for DRI. Composition of hematite iron ore as follows

Fe, O_2, SiO_2, Ai_2O_3, CaO, MgO, Pb, Cu, Zn, V, S, P, LOI

The oxygen concentration in hematite iron ore is as follows:

$$Fe_2O_3 \rightarrow 2Fe + 3O_2$$

When hematite iron ore of 65% Fe oxygen combined is calculated as follows

111.70 part Fe combined with → 48 part O_2
So 1 part of Fe combined → 48 / 111.70
65 part Fe combined → 48 ×65 / 111.70 =27.93 part O_2

During reduction reaction only O_2 and LOI are removed and all other element remains intact. After reduction reaction the hematite ore is called DRI /sponge iron and the weight of the product become lighter due to removal of O_2 and LOI. The chemical composition of 65% Fe hematite iron ore is supposed to be as follows:

Fe = 65%
O_2 = 27.93%
SiO_2+Al_2O_3 = 4.52%
CaO+ MgO etc = 1.0%
LOI =1.55%

Total 100.00

After 100% reduction of oxygen, the sponge iron contains:

Fe = 65.00 %
O_2 = → 27.93% Goes out gas
SiO_2 = 4.52%
LOI = → 1.55% Goes out as gas
CaO MgO etc = 1.00%

Total = 70.52% + 29.48% =100%

So like scrap the Fe iron content in sponge goes high depending on Fe % in ore.

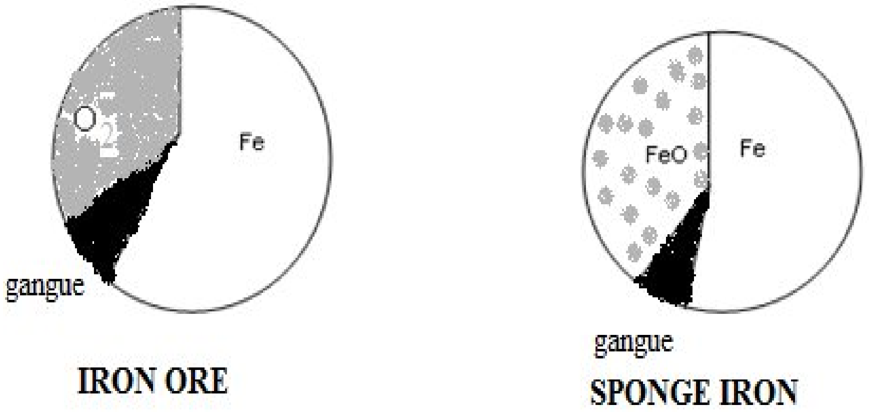

Figure.1.4: Reduction and Unreduction part

1.4.2 Kinetics of Iron Ore Reduction:

It is reduction reaction that iron oxide over come during transformation of phases under pressure and temperature in the kiln at reducing atmosphere. Rate at which reduction is complete, determines the rate of reduction in the kiln. The reaction of metal oxide by carbon start at a temperature at which the line for free energy of CO formation resume. This indicates that at 1000 ^{0}C carbon can reduce all common metal oxide except Mn, Si, Ti, Mg and Ca. In the reactor large number of iron ore granules (used in lumpy form) is exposed to reductions. The large gaps between the granules are known as macropores and smaller gaps with in groups are known as micropores. The reduction gas flow between the macropores and micropores of lumps of ores and hence boundary layer is formed around the individual lump So the gaseous reaction continues as follows:

Table 1.4: Kinetics of Iron Ore Reduction

Equation of Reaction	**Heat of reaction Cal/mole 1000 ^{0}C**	**Heat of reaction Cal/mole 1400 ^{0}C**
1.$C + O = CO_2$	-94320	-94512
2. $C + CO_2 = CO$	+40780	+39750
3.$C + 1/2O_2 = CO$	-26770	-27380
4.$C + H_2O = CO + H_2$	+32440	+32360
5.$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2$	-11250	-11630

6.$Fe_3O_4+CO=3FeO+CO_2$	+3750	+4270
7.$Fe2O_3+3CO=2Fe+3CO_2$	-3583	-316
8.$FeO+C=Fe+CO$	+56050	+35520
9.$3Fe_2O_3+H2=2Fe_3O_4+H_2O$	-2910	-4240
10.$Fe_3O_4+H_2=3FeO+H_2O$	+12090	11660
11. $FeO+H_2=Fe+H_2O$	+3590	+3160

1.4.3 The Reduction Mechanism for Porous Iron Ores:

1. Diffusion of the hydrogen and CO gas across the boundary, macropores and Microspores in the ores.
2. Phase boundary reaction.
3. Diffusion of water vapour through the micro pores and macropores
4. Diffusion of water vapour across the boundary layer migration of Fe++ and 2e to the iron nucleolus.

The diffusion of iron and electron the fallowing pathway:

$$4Fe_2O_3 + Fe + 2e^- = 3Fe_3O_4$$

$$Fe_3O_4 + Fe + 2e^- = FeO$$

According to above mechanism O_2 is removed from the iron oxide at the iron wustite interface only. CO and Hydrogen diffuse inwards through the iron layers and the product gas CO_2 and water vapors diffuse outwards. The flow rate of gases through the bed of solid particles must be large enough so that a stagnant layer of gas cannot build up around each solid particle. For continuous effect of reaction, the reduction must get into the interface and solid may attain desired reduction.

The coal gasification process in air to CO_2 entirely depends on rate of removal of carbon or coal reactivity which mainly depends on pore structure of coal, particle size carbon concentration of active carbon, coal rank, ash of coal and other condition like charging.

1.4.4 Thermodynamics of Carbon Oxygen Equilibrium System (Fe- O-C System):

Carbon reacts with O_2 in the following reaction:

$$\left.\begin{array}{lr} 2C + O_2 = 2CO & (1) \\ C + O_2 = CO_2 & (2) \\ 2CO + O_2 = 2CO_2 & (3) \\ C + CO_2 = 2CO & (4) \end{array}\right\} \text{Boundouard's reaction}$$

The burning solid coal with molecular oxygen or oxygen from air produces CO which is utilized in the reduction of iron ore as well as provides heat to the kiln. In addition to it hydrogen is also used as reductant in the process.

The reductant CO reacts with oxide iron ore in the following manner:

$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2$ H=12636 Cal (Exothermic)

$Fe_3O_4 + CO = 3FeO + CO_2$ H=-8664 Cal (Endothermic)

$FeO + C = Fe + CO$ H= -4136 Cal (Endothermic)

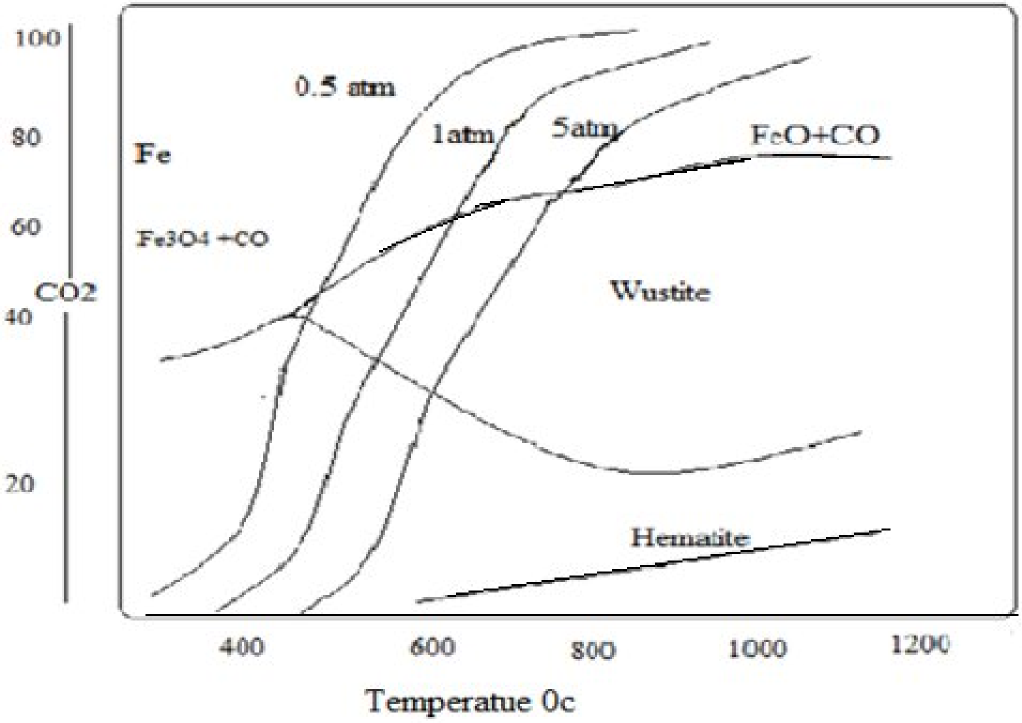

Figure 1.5: Fe-O-C System

It's found that at temperature of 700^0 C when gas composition is 20 % CO and 80% CO_2 the reduction of hematite to magnetite takes place. At this gas composition magnetite remains in stable phase. Similarly at 1000^0C if gas composition is 50% CO and 50% CO_2 then the wustite is stable phase. For reduction of FeO (Wustite) at 1000^0C and above the gas composition is expected to be 75% CO and 25 % CO_2. Here iron oxide is reduced if CO is more and will get oxidized if CO_2 is more. If however Nitrogen is present the ratio of CO/CO_2 will remain the same and only the % CO and CO_2 will change.

1.5 DRI IN ROTARY KILN PROCESS (SL/RN PROCESS) [8]

1.5.1 Process out Line:

The process builts up on solid state reduction of iron ore by using sub-bituminous coal as a reductant under controlled atmosphere at desired temperature and pressure. The rotary kiln of more than 80m length and more than 4m dia, is designed in such a manner that solid feed stock move under kiln bed in touch with gaseous atmosphere throughout the length of the kiln.

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Figure 1.6: Rotary kiln

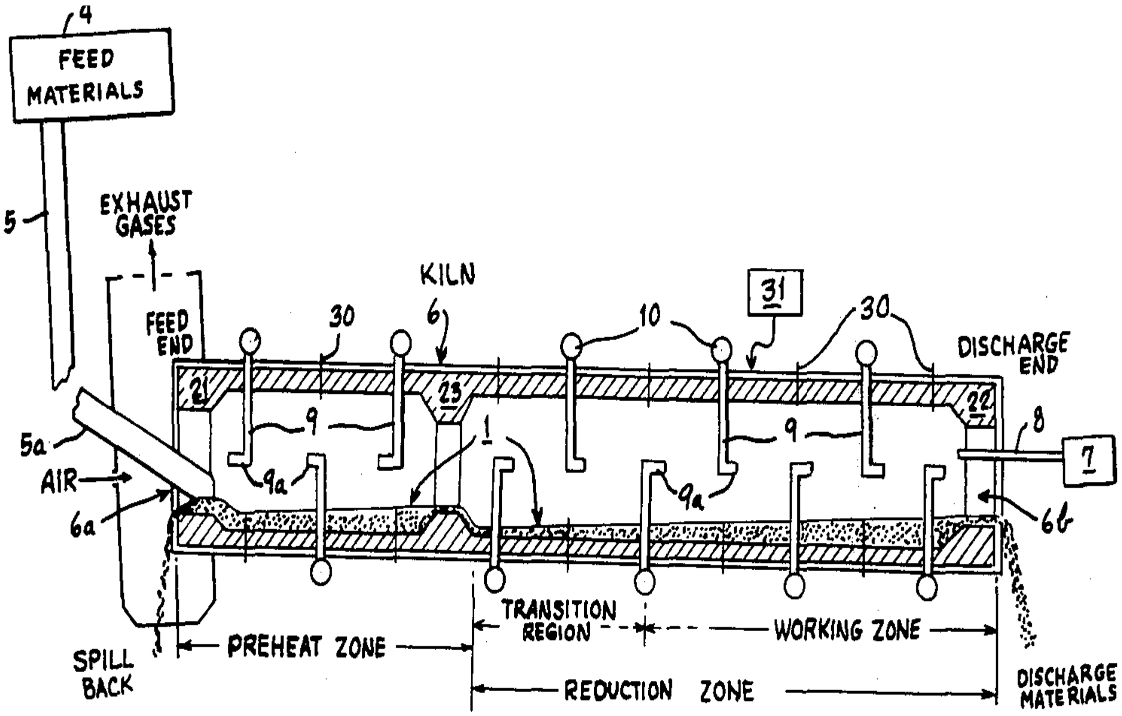


Figure.1.7: Direct reduction in a rotary kiln

1.5.2 Principle of Reaction in Rotary Kiln:

In a rotary kiln carbon is used as reducing agent and reaction takes place mostly through CO intermediate. The reduction reaction of solid iron ore in rotary kiln is heterogeneous in nature. The iron oxide in ore usually takes place in step which may be generalized as follows:

$$m\ Fe_XO_Y + CO(g) = n\ FeOz(S) + CO(g)$$

Where m,n,x,z are integer 0,1,2,3,4,----- depending on specific iron oxide or metallic iron.

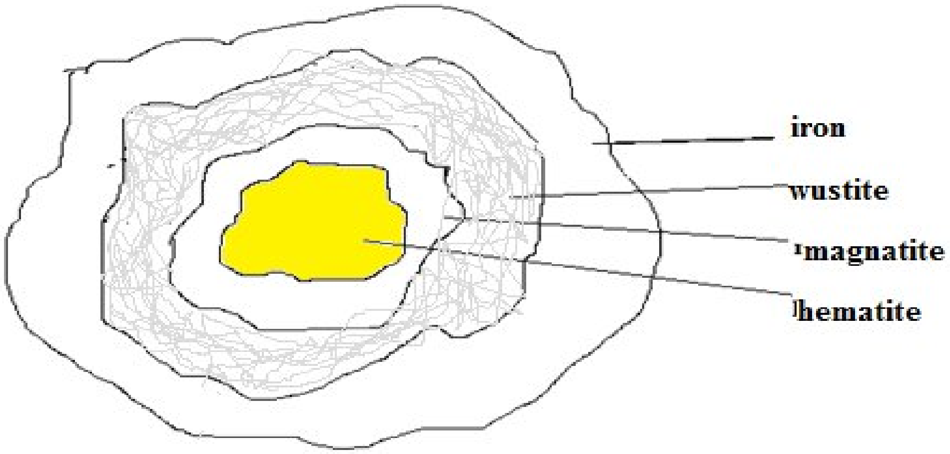


Figure.1.9: Reduction section of iron ore

The whole mechanism or any one of the step can be a rate controlling step are as follows:

1. Mass transport between bulk gas flow and the solid specimen surface i.e. boundary diffusions.
2. Mass transfer between sample surface and reaction interface.
    a. Solid diffusion through a lower oxide.
    b. Solid diffusion through a dense metallic iron phase.
3. Interparticle diffusion of gas species.
4. Nucleation and growth of iron phase
5. Heat transfer to reaction interphase.

In rotary kiln where solid and gas phase are separated, the atmosphere is almost stagnant. For reducible reaction the reducent must get in touch with the interface otherwise reaction cannot be completed. Some of rate determining factor in iron ore reduction are associated with the nature of the reaction are associated with nature of reaction system and contact between the reducing phase while others are associated with nature of ore. The latter determine the ease with oxygen can be removed from the oxide in ore by the reducing gases.

1.5.3 Reducibility Factor of Sponge Iron:

This property of the iron ore depends on following factors:-

1. **Nature of Iron Oxide**

    The major deposition of iron oxide in nature is mainly in the form of hematitend magnetite. The oxygen enrichment in hematite iron ore is more than the magnetite,

.hematite iron ore is the saturation point of oxide ore which is more stable in nature. The purest hematite iron ore may contain maximum 1% of magnetic or magnetic in nature.

2. Ore Size

The deposition of iron ore in nature is usually in hard and massive form. But when the ore is considered for DRI process its size and shape are very much important for the of reduction reactions.

3. Porosity

The porosity of ore is one of the important factor which affects the reducibility. It is observed that the reducibility of soft hematite iron ore is greater than that of hard hematite and magnetite ore. The reducibility of natural ore show that for 90% reduction, reciprocal of time varied linearly with percentage porosity.

4. Nature And Composition of Gangue

It observed that natural ore contains gangue oxide of Si,Al,Cr,Ti etc.in different from. In some of the ores wustite is present as complex compound such as $2FeOSiO_2$, $FeOAl_2O_3$, $FeOCr_2O_3$ where wustite exist in a state of low activity. These oxides tend to the decrease the reducibility of iron ore in kiln in reducing atmosphere.

5.Swelling

Some ores show abnormal increase in volume at temperature btween 900 to 1000 ^{0}C which is refered as swelling of ore . The cause of swelling has not been understood fully. Various investigatiors have in opinion that it is associated with mechanism of nucleations and groth having of wustite.

6.Linear Gas Velocity

It is an established fact that increase in linear velocity of reducing gas increase the rate of reduction. It remains constant after a certain critical veloity of gas reductent gas.the figure plotted with hyrogen gas as reducent shows the efect of increasing linear velocity of the reducing gas on reducent ore.

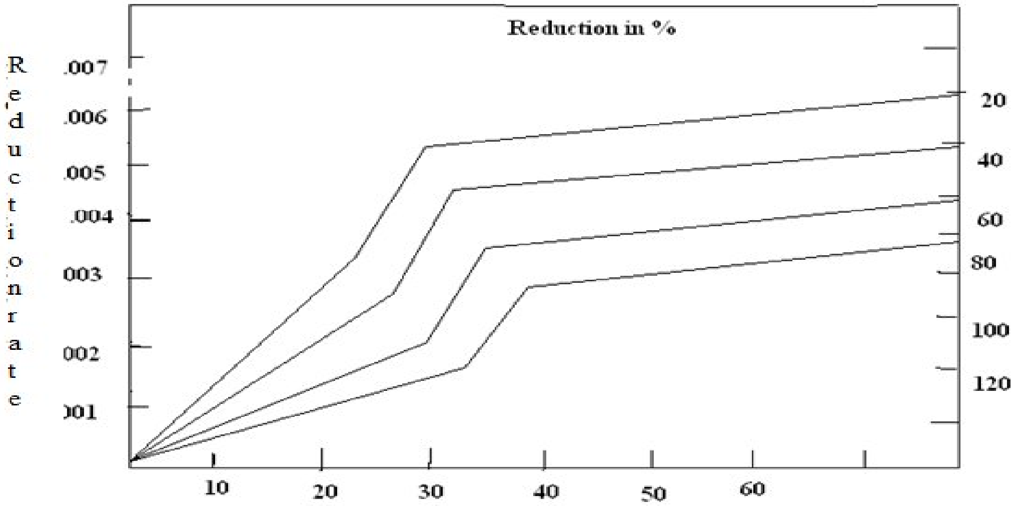


Figure 1.10.: Linear gas velocity of hydrogen

6. Temperature

The rate of reduction of iron oxide increases with increase in temperature of iron oxide.

In solid gas reduction it is advantageous to use a temperature above 1000 ^{0}C because boundary reaction is maximum for producing CO with CO_2 and C combination. It is observed that if iron ore is reduced to metallic iron at low temperature, this form a layer of dense metallic iron on the ore body which affects reduction at higher temperature region.

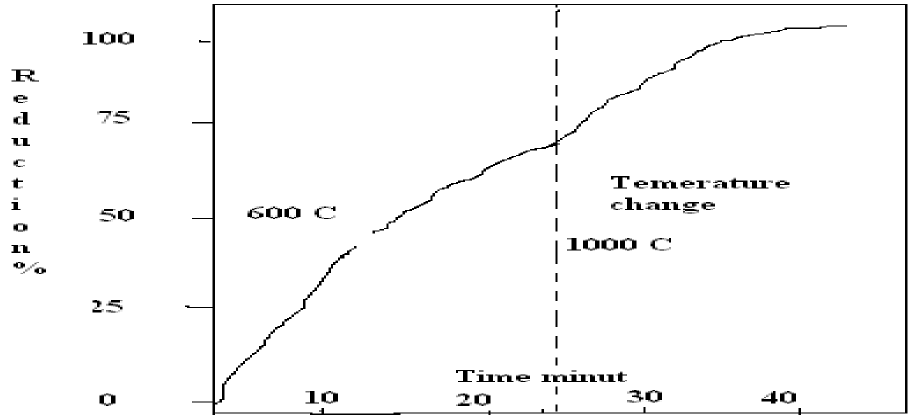


Figure1.11 Reduction time of iron ore

8. Gas Compositions

The coal after gasification becomes a composition of CO_2, H_2, some water vapours, CO_2 etc. It is observed that by increasing the partial pressure of reducing gases the rate of reduction can be increased. It has been proved that hydrogen is better reducing gas than CO but at higher degree of reduction CO reduce iron oxide at faster rate

9. Pressure

The increase in pleasure is expected to increase the rate of reduction as increase the partial pressure of the reducing gas.

Grade of sponge iron

1) **A Grade Sponge Iron**

Sponge iron having total Fe more than 88% and metallization get up to 98% then sponge iron is called A grade sponge iron. .

2) B Grade Sponge Iron

Sponge iron having total Fe in between 80% to 87% and metallization get up less than 90% then sponge iron is called B grade sponge iron.

3) C Grade Sponge Iron

Sponge iron having total Fe is less than 80% and metallization less than 84% then sponge iron is called C grade sponge iron.

1.6 PROPERTIES OF SPONGE IRON [12]

1.6.1 Physical Properties:

There are several parameters to be monitored for improving the quality of sponge iron for steel making operation, these are listed below:-

(a) **Size** - A very fine sized material (1 mm to 2 mm) would be quickly oxidized during falling to the slag or may be lost in fume extraction system. Extremely large size (exceeding 30 mm) poses problem during continuous feeding. the size fraction less than 2 mm needs to be limited for continuous feeding and hence the size plays an important role.

(b) **Density** - Sponge iron after falling should have the ability to penetrate into the slag layer and reside at the slag/metal interface for effective heat transfer and chemical reaction. Sponge iron with lower density tend to float on the slag while, high density material readily penetrates into the metal. Hence, it is desirable to have the density of sponge iron in the range 4 - 6 gm/cc.

(c) **Unit Weight** – The transition time of the sponge iron pellets through the slag is dependent on the momentum. If the pellet stays in the slag layer for too long a time, the phenomenon of slag boiling occurs. Slag fluidity is highly important. However, a heavier sponge iron pellet does not require close control in slag fluidity.

(d) **Crushing Strength** - Sponge iron should possess good crushing strength to prevent generation of large amounts of fines.

(e) **Weather Resistance** - Sponge iron is prone to oxidation and heat builds up in contact with atmosphere. The storage of sponge iron for long periods of time affects its metallization, partially due to surface re-oxidation caused by the porous structure of sponge iron pellets or lumps.

(f) **Carbon Contents** - During continuous feeding, an active carbon oxygen boil is necessary to shield the arcs. It has been observed that to achieve the aforesaid, sponge iron should possess a minimum of 0.60% carbon.

(g) **Metallization** - High metallization helps in lower power consumption but severely reduces the bath activity and results in flat bath conditions. For low metallization levels, increased carburization is required to compensate for the extra oxygen in sponge iron.

1.6.2 Chemical Properties of Sponge Iron:

1. The sponge iron retains the size as that of iron ore and contain no oxygen or little amount of oxygen as FeO.
2. It is attracted by magnet and looks black with metallic lusture.
4. It picks up little carbon during process.
5. Highly reactivity with moisture.

- Magnetite-Wustite $\underline{Fe_3O_4}+\underline{H_2}\leftrightarrow\underline{3FeO}+\underline{H_2O}$
- Iron-Wustite $\underline{FeO}+\underline{H_2}\leftrightarrow\underline{Fe}+\underline{H_2O}$

Effect of Moisture on Sponge Iron Material [13]

Main reason of high sensitivity of DRI towards rapid reoxidation is because of its high porosity leading high surface to volume ratio .The chemical reaction involved in oxidation of DRI with air moisture leads to heat formation. The presence of hydrogen gas (if due to moisture) compounds the problem.

Factor promoting the rate of oxidation

The rate of oxidation depends on:-

(1) Concentration of chemical ingredients
(2) Surface area available for reaction
(3) Temperature at reaction site .

1.7 PROBLEMS OCCURING DURING DRI PRODUCTION BY KILN [14]

1.7.1 .Accretion Formation:

Accretion or ring formation in coal base DRI rotary kiln is the catastrophic accumulation of sintered particles of solid bed which form rings at places along the length of the kilns. It is a regular phenomenon in the DRI rotary kiln. This narrows down the opening of the kiln and gives hindrance to material flow. The system needs shutdown for cleaning. The accretion in frequent intervals affects the economy of the process where campaign life is reduced. Productivity is decreased, kiln lining are damaged and cost of production goes high. So it is necessary to minimize the causes which attributes the formation of semi-molten masses in the rotary kiln during process and causes accretions.

Reason for Accretions:

The rotary kiln of DRI process rotates at certain RPM in clockwise along with the feed stock like iron ore and coal at in particular bed height. The reduction is well within this limit. The accretion in the kiln is built-up when solid particles diffuse with semi-molten compounds formed due to either chemical reaction or rise of temperature beyond the limit or both during operation of DRI kiln. The accretion formed due to high temperature shows distinct indication on the body surface itself. The coal has different ash fusion temperature of initial deformation basing on its compositions and deposits. It is always advisable to operate the kiln at 150^0C less temperature than the ash fusion temperature so that accretions can be avoided. But ash when moves in kiln does not move in the purest form and due to contamination of sponge fines, Dolomite fines etc., the ash also sometimes fuses in lower temperature than the real fusion temperature of ash.

1.7.2 Hot Spot in Kiln Cell:

The DRI rotary kiln is operated at temperature 1050 to 1100 ^{0}C continuously under reducing atmosphere. The kiln cell is made to protect heat loss. Such lining for insulation is usually made with high aluminous refractory material place in a compact form or as a single piece so that system can run for longer period without break. This lining also gives protection to the metal clad out of direct heat and maintains longer life of the kiln. The refractory lining in the kiln some time detaches or collapses in the form of patch and hot spot on kiln cell is marked. This hot spot is not fully stopped by patch of insulating material rather it may cause further problem and weaken the kiln cell which may lead to cracking or swelling of the cell.

1.7.3 Generation of Fines:

The iron ore is composed of iron oxide, silica and alumina as major constituents. These elements are usually in oxide form for which the ore is non-conductive and heat transmission from grain to grain usually takes time.

When solid expands due to rise of temperature, the solid iron ore in the reactor owing to much higher temperature also expands. Such expansion is so sudden that the heat does not get time to be distributed equally throughout ore body. The thermal shocks develop number of cracks on ore body. This generates -3 mm fines in side reactor which may be cause for accretion.

1.8 ADVANTAGES OF USING SPONGE IRON IN STEEL MAKING [15]

Use of sponge iron:

- Increases productivity through shorter tap-to-tap time and refining time.
- Simultaneous melting and refining with continuous charging.
- Faster metallurgical reactions and Improved as well as more stable power consumption
- Less electrode consumption due to stable power.
- More precise of steel compositions and quality advantages.
- High degree of metallization (up to 92%) and consistent chemical composition.
- Deep drawing steel grades can be produced in electric arc furnace (EAF).
- Flat products and alloy steels of international standards can be made in electric arc furnace (EAF).
- Reduction in number of off-grade heats.

Recently much interest has been shown internationally in the use of in electric arc furnaces made of sponge iron in the form of reduced pellets V. I. Trakhimovich, et al. [16], used pellets with a diameter of up to 10 mm. The pellets are continuously fed into the furnace daring melting. Such a method, though it has a number of advantages, requires re-equipment of the furnace. In operating furnaces, sponge iron can be remelted by charging it in the place of scrap. To avoid welding and excessive oxidation, size and bulk density of sponge iron must be higher than those of pellets. The use of sponge iron as the charge in place of scrap must, first of all, affect the parameters of the melting period because of the difference in the chemical composition and physical properties. Melting of sponge iron was started in a manner similar to scrap melting. It must be noted that sponge iron shows little tendency to get welded during heating and melting.

With regard to the contemporary problem of raising the quality of metal, E. F. Mazurov, S. M. Gnuchev,[17] have given them a review article on the use of sponge iron as a charge material the main reason for producing and using sponge iron. An increase in the silica content of the sponge iron leads to the formation of a large amount of acidic slag during the melting period, which destroys the lining of the furnace. The large amount of slag caused by the high content of silica in the sponge iron was the cause of the low output of molten steel due to repeated removal of the slag which carried beads of metal suspended in it during the process of intense boiling thus, experience indicates the expediency of using sponge iron as a charge material for producing a number of steels in electric-arc furnaces. The necessary conditions for such production are a large content of total iron and a high degree of its reduction, a low percent of silica, and granules not less than 40-45 mm in size. The great merit of sponge iron when produced by the correct technology is the constancy of its chemical composition and the cleanness with respect to admixtures of nonferrous metals.

Pargeter et al, [18] has mentioned the process of manufacturing sponge iron with low sulphur content by depositing a charge made up of superposed layers of finely divided material on a moving hearth, at least one of the layers being substantially made up of iron oxides and at least another of the layers being made up of a mix of a solid reducing agent containing carbon and a desulphurizing agent, after heating the said charge, it induces at least partial gasification of the said solid reducing agent containing carbon in gaseous compounds of carbon and sulphur. It induces the reduction of the said iron oxides by means of at least one part of carbon monoxide (CO) contained in the said gaseous compounds of carbon. In that one fixes by means of the said desulphurising agent at least one part of the sulphur of the said gaseous compounds of sulphur, the reduced oxides are seprated from the layer containing residues of the said solid reducing agent containing carbon and of the said desulphurising agent.

It is well known that the gasification of the solid reducing agent containing carbon requires heating to a high temperature in the order of at least 900° c. The group of Duong, H. V. and Johnston, R. F., [19] has carried out the heating in such a way that the gaseous carbon compounds contain essentially carbon monoxide (CO) which is a very reducing gas. The heating system necessary for the gasification is advantageously assumed by burners preferably installed in the vault of the furnace containing the moving floor which produce CO_2 necessary to start boudoirs reaction. This group has give a process for manufacturing sponge iron with low sulphur and high carbon content by exposing hot sponge iron to a gas containing gaseous hydrocarbon, the thermal cracking of the said gaseous hydrocarbon in contact with the said hot sponge iron was observed.

The production of sponge iron by the reduction of iron oxide at a low temperature is engaging the attention of many experiments. Iron and steel metallurgists want sponge iron for subsequent conversion into foundry iron or steel, and copper metallurgists want it for precipitating copper from solutions resulting after leaching operations. Both are interested in the metallic iron content of the sponge iron, although the copper metallurgists have been satisfied with the “copper-precipitating value”-that is, the amount of copper that is precipitated by a unit quantity of the sponge iron. Clyde E. Williams and Arvid E. Anderson [20]. Are using the Methods for determining the metallic iron in ferrum reducturn (a pharmaceutical and chemical reagent obtained by the reduction of pure iron oxide with hydrogen) have been used for years and should apply equally well for sponge iron:

John D.H., Matthew S.P., And Hayes P.C [21], have examined wustite single crystals reduction in CO/CO_2 and H_2/H_2O gas mixture and have shown that in all cases a dense iron layer is formed initially on the oxide surface and that the porous growth of iron which is obtained under certain experimental condition occurs as a result of the breakdown of this initial dense iron layer. Possible mechanism of the iron layer breakdown are examined and compared with the experimental observations. A qualitative model of the breakdown process involving the nucleation of gas bubbles and the expansion of this bubble and the expansion of this bubble in the iron layer is presented.

D. K. Biswas (NML), S. R. Asthana (NCL), V. G. Rau (IIT KGP) [22], estimated the scope of energy savings in some areas, in and around the rotary kiln, as this is the main energy consuming part of a noncoking coal based sponge iron plant. The maximum temperature of the smaller kiln is 1100°C where as that of larger sized kiln is about 1170°C. This is due to the fact that in the larger diameter kiln, the preheating time increases significantly as the ratio of surface area of charge bed across which heat transfer takes place and the volume charge decreases. Increasing heat transfer using higher temperature for sponge iron making may lead to inefficient operation and operational problems such as localized heating and liquid phase formation

Mirko Komatina, Heinrich W. Gudenau [23], described possible chemical reactions and their thermodynamic analysis during direct reduction. The sticking mechanism during direct reduction in the fluidized bed was analysed, and the reasons for the sticking appearance are explained. The investigations could be performed in fluidized bed reactor by using Coal an inert material. Separately, the influence of volatile content in the coal on the reduction process and sticking appearance is analysed.

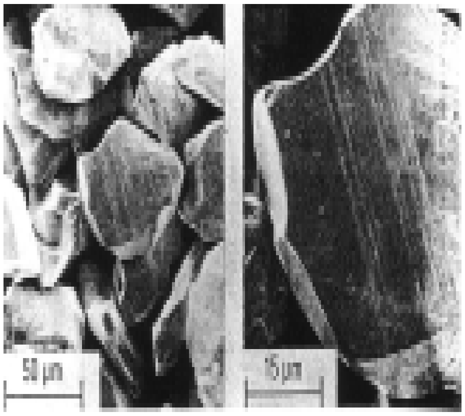


Figure.2.1: Hematite iron ore

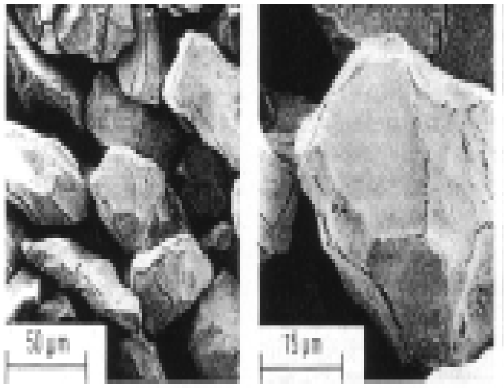


Figure.2.2: Hematite iron ore reduced to magnetite

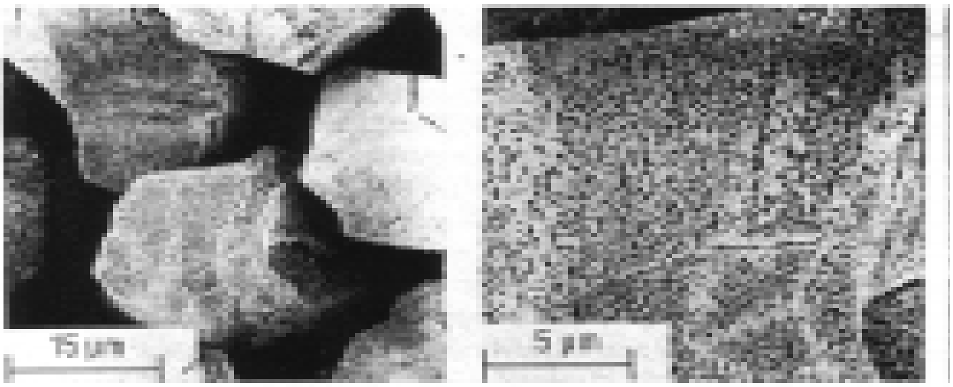


Figure.2.3: Hematite iron ore reduced to wustite

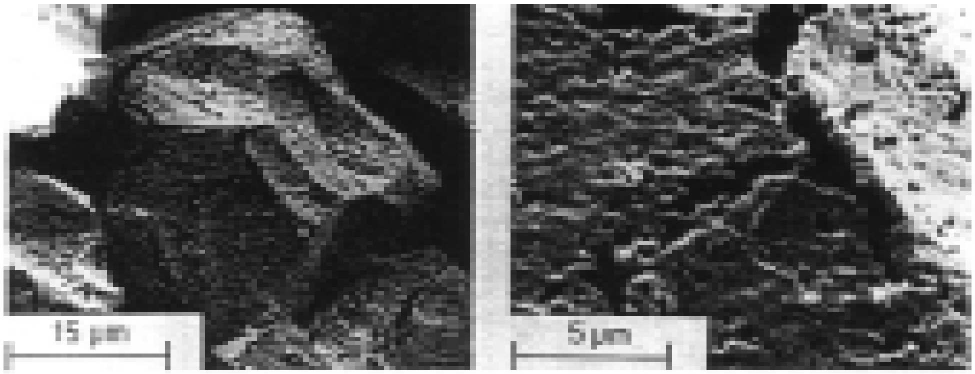


Figure.2.4: Hematite iron ore reduced to porous iron

Sticking could be caused by many reasons such as: low melting eutectic mixture iron grains soften and stick together, a little gangue in the iron ore and under special conditions fibrous precipitates grow, two fibrous iron become hooked to each other and finally crystallize, etc.

S.K. Dutta, A.B. Lele and N.K. Pancholi [24], Studied direct reduced iron melting induction furnace. Unlike scrap and even pig iron, DRI is characterized by high porosity, low thermal and electrical conductivities which, in turn, poses problems in its melting. Attempts were made to study melting of DRI in a laboratory size induction furnace using molten steel bath as hot heel. The induction stirring accelerates the transfer of heat and promotes the melting of DRI. The effect of partial replacement of scrap by DRI on various melting parameters has been studied. Also kinetic studies were made to evaluate net melting rate. It was concluded that higher proportion of DRI, as a replacement to scrap, contributes to improve mechanical properties with no segregation of carbon content and the decrease in sulphur and tramp elements in the product that improves steel quality.

Shalini Sharma, T., Saxena, V. K., and L.N. Upadhyaya,. [25] proposed that irrespective of charging mode, DRI is always charged after initial formation of molten pool by melting of steel scrap. Melting of DRI in EAF / IF is greatly influenced by factors like carbon content and degree of metallization of DRI.4 Carbon content of DRI reacts with unreduced iron oxide content of DRI giving CO evolution from liquid bath i.e. carbon boil takes place, which results into subsequent removal of hydrogen and nitrogen gases, ultimately producing clean steel. Carbon boil occurs at slag metal interface by the reaction

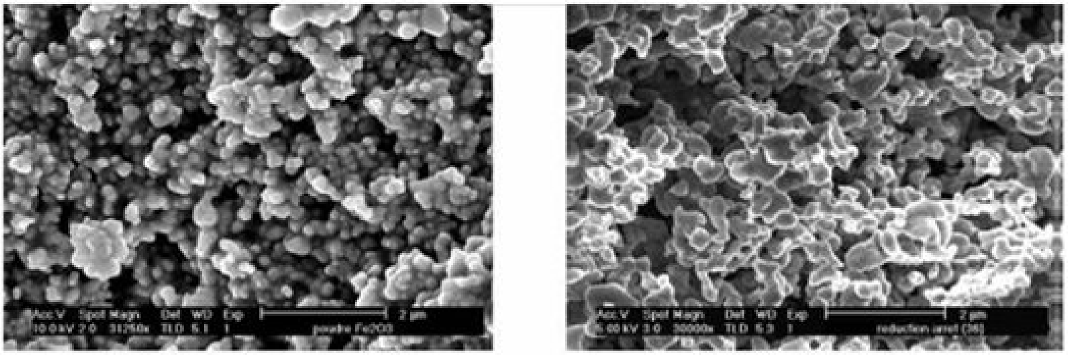
$$[FeO]+ [C] = [Fe] + [CO] \quad (1)$$

For this very reason, steelmakers always prefer higher carbon content in DRI. The amount of carbon required (C, in Kg) to reduce the FeO content of the DRI is

$$C = 1.67 \, [100 - \%M-\{(\%Sl/100) \times \%Fe\}] \quad (2)$$

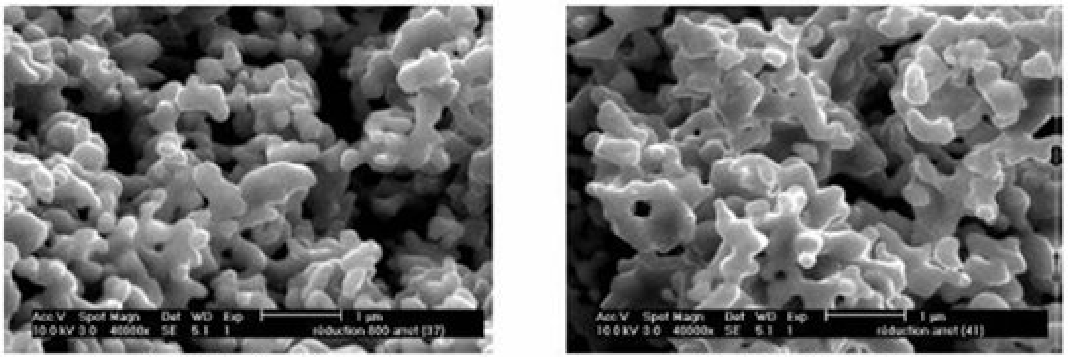
Where, M is degree of metallization, and slag Fe is amount of iron in the slag.

F. Kongoli and R.G. Reddy, [26] worked on DRI (Direct Reduced Iron) using hydrogen as the reducing gas instead of carbon monoxide. In this context, the reduction of pure hematite by hydrogen was studied at the laboratory scale, varying the experimental conditions and observing the rate and the course of the reaction. All the reduction experiments were performed in a thermobalance and supplementary characterization methods were used like scanning and transmission electron microscopy.The influence of rising temperature in the range 550-900°C is to accelerate the reaction; no slowing down was observed, contrary to some literature conclusions. A series of experiments consisted in interrupting the runs before complete conversion, thus enabling the characterization of partially reduced samples. Interpretation confirms the occurrence of three successive and rather separate reduction steps, through magnetite and wustite to iron,



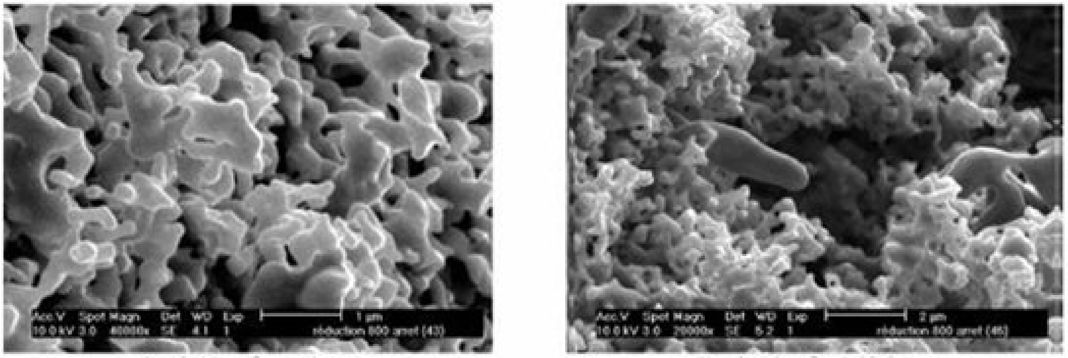
Initial hematite powder P1

Red 36, after 152 s



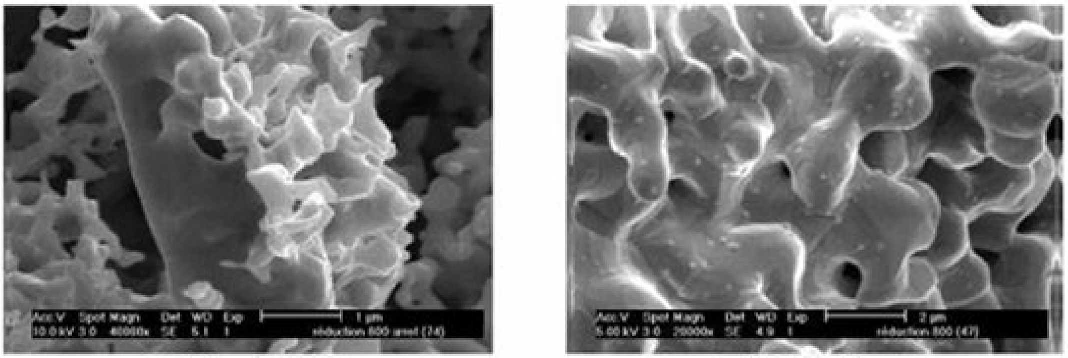
Red 37, after 189 s

Red 41, after 260 s



Red 43, after 427 s

Red 46, after 686 s



Red 74, after 935 s

Red75, final iron

Figure 2.5 . SEM phographs for different reduction times

M. Kumar S. Jena ’S. K. Patel [27], studied on chemical and physical properties, and the reduction behavior (in coal) of hematite iron ores procured from 10 different mines of orissa to provide information for the iron and steel industries (sponge iron plants in particular). The majority of the iron ores were found to have high iron and low alumina and silica contents. All these iron ores were free from the deleterious elements (S, P, As, Pb & alkalies etc.). The results indicated lower values of shatter and abrasion indices, and higher values of tumbler index in all the iron ore lumps except serazuddin (previous) and khanda bandha omc ltd. For all the fired iron ore pellets, the degree of reduction in coal was more intense in the first 30 min, after which it became small. Slow heating led to higher degree of reduction in fired pellets than rapid heating. All the iron ores exhibited more than a 90% reduction in their fired pellets in 2-h time interval at a temperature of 900°c.

M. Kumar and S. K. Patel [29], also studied reduction kinetics (with *F* grade coal) in fired pellets of hematite iron ores, procured from four different mines of Orissa, were carried out in the temperature range of 850–1000◦C to provide information for the Indian sponge iron plants. The rate of reduction in all the fired iron ore pellets increased markedly with a rise of temperature up to 950◦C, and thereafter it decreased at 1000◦C. The results showed a pronounced effect of temperature on the extent of reduction in all the studied hematite iron ore pellets. The reduction rate was enhanced substantially by an increase in temperature up to 950◦C and then decreased at 1000◦C. At all studied temperatures, the degree of reduction increased with heating time, the rate being faster in the first 30 minutes because of greater influence of volatile matter.

2.1 AIMS OF WORK

During the course of this present study to meet the following objective:

- Characterization of the chemical and physical properties of selected iron ore.
- Characterization of the properties of selected noncoking coals.
- Study of the effect of time on the degree of reduction of iron ore.
- Study of the effect of temperature on the degree of reduction of iron ore.
- Study of the effect of coal type on the reduction characteristics of iron ores.

To achieve better quality of sponge iron.

Chapter3 EXPERIMENTAL PROCEDURE

3.1 EVALUATION OF RAW MATERIALS:

3.1.1 Selections of Materials:

For packed bed reduction studies, iron ore sample was collected from Zenith iron ore mine of Orissa and non coking coals were collected from Lingaraj Mine, Ananta Mine, and Jagannath Mine in Orissa.

3.1.2 Determination of Chemical Composition and Loss on Ignition of Iron Ore:

The chemical composition of the iron ore was determined by wet chemical technique. The loss on ignition values of the iron ore is determined by heating 1gm powder of air dried samples at a temperature of 900 ^{0}C for 1hr, followed by air cooling. Loss in weight was taken as the % loss in ignition.

3.2 PROXIMATE ANALYSIS OF NON-COKING COAL:

Analysis for moisture, volatile matter, ash and fixed carbon contents were carried out on sample ground to pass through 5-25 (mm) mesh test sieve as follows.

3.2.1 Moisture Determination:

1 gm. of air dried sample was placed in an air oven maintained at a temperature of 110^{0}C and kept there for 1 hour. The loss in weight, expressed as the percentage of initial weight of coal/coal char, gives the percentage of moisture content in the sample.

3.2.2 Volatile matter determination:

1 gm. of air dried sample was taken in a volatile matter crucible (made of silica) covered with a lid**.** The crucible was introduced in the furnace maintained at a temperature of 950^{0}C and kept at this temperature for 7 minutes. The crucible was then taken out and loss in weight of sample was determined. The % loss in weight minus % of moisture content in the sample gives the value of percentage volatile matter in the sample on air dried basis.

3.2.3 Ash determination:

1 gm. of air dried sample was taken in a Silica disc and placed in the furnace maintained at a temperature of 775 ^{0}C, and kept there till complete burning. The weight of ash obtained expressed as the % of initial weight of the sample gives % of content in the sample on air dried basis.

3.2.4 Fixed carbon determination:

It was simply calculated as follows: % Fixed Carbon (FC) = 100 - % (moisture + volatile matter + ash).

3.3 EVALUATION OF PHYSICAL PROPERTIES OF IRON ORE:

3.3.1 Determination of Cold strength:

Assessments of cold strength of the selected iron ore was carried out by determining their tumbler, abrasion and shatter indices.

(i) Tumbler and Abrasion Indices:

The tumbler tests for lump ore sinter and pellets were carried out for the determination of resistance to degradation or breakage by impact and abrasion. 15 kg of oven dried lump iron ore Size of sample 6-15 mm was placed in circular drum of 100 cm inside diameter and 50 cm inside length and the door of the drum was closed tightly. The drum was rotated at 25 rpm for a total 200 revolutions. All the materials were removed gently from the drum by slowly opening the door and sieved on a 6.3 mm sieves. The weight of the fraction retained on and passing through 500μm were taken. The tumbler index (T) and Abrasion index (A) values were calculated by using the following formula.

Formula used

$$\text{Tumbler Index \%} = \frac{\text{Wt of +6 mm seizer}}{\text{Total wt of material}} \times 100$$

$$\text{Abreaction Index \%} = \frac{\text{Wt of -6 mm seizer}}{\text{Total wt of material}} \times 100$$

(ii) Shatter index:

A 10 kg dried iron ore sample of size 5 to 20 mm was dropped three times from a height of 2m. The material was then screened and shatter index was expressed as the percentage of – 0.5 mm fraction produced.

Formula used

$$\text{Shatter Test} = \frac{\text{Wt of -5 mm fraction seizer}}{\text{Total wt of material}} \times 100$$

3.4 PROCESS OUT LINE FOR MAKING SPONGE IRON: (500 ton per day capacity of kiln)

Known quantity of iron ore, dolomite and feed coal are mixed with one another and the material was feed from inlet side in rotary kiln by conveyer belt. All these materials pass through the different temperature zone of kiln. To maintain the zone temperature of kiln coal was pumped through pipe with high pressure from outlet side of kiln.

The process build up on solid state reduction of iron ore by using sub-bituminous coal as a reductant under controlled atmosphere at desired temperature and pressure. The rotary kiln of 84 m length and diameter of kiln 4.8 m was designed in such a manner that solid feed stock move under kiln bed in touch with gaseous atmosphere throughout the length of the kiln where reduction reaction continues to get desired metallisation at the time when it reaches discharge end. Hot reduced materials have to be cooled to avoid reoxidation. For it hot material was pressed in cooler of 50 m long and 3.8 m diameter with positive pressure and brought to desired temperature earlier in rotary action. [9]. At last it passes through magnetic separator for separation of sponge iron and charcoal then separated sponge iron was store in search bin. Figure 3.1 describe the diagram of the entire process.

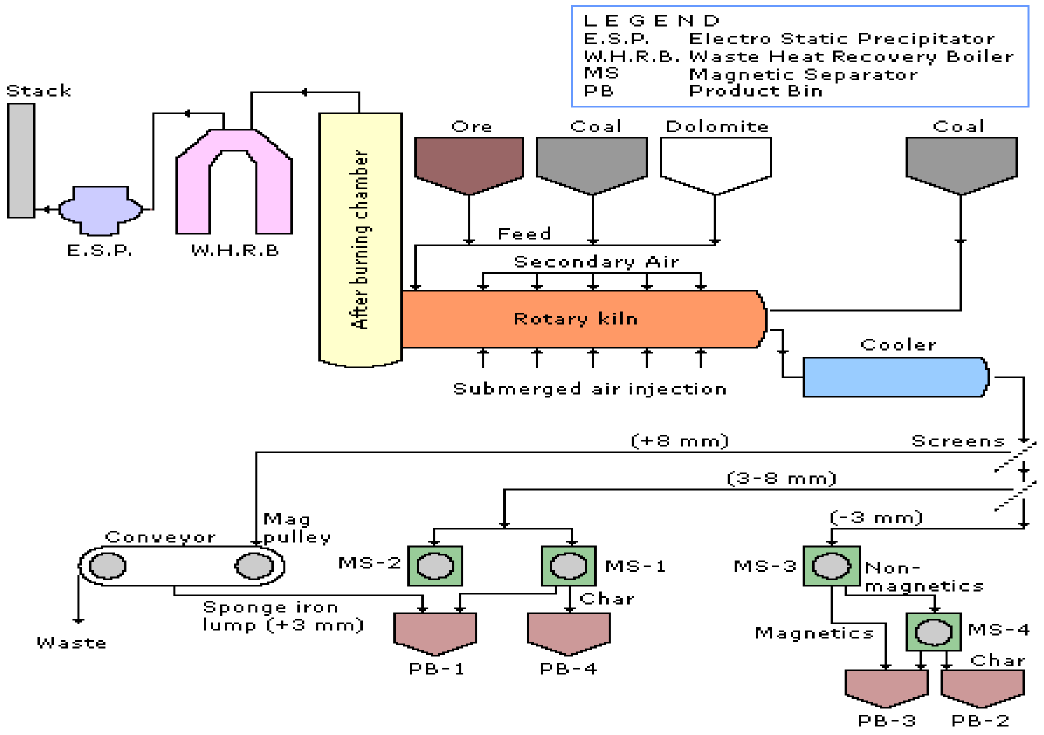


Figure.3.1: Manufacturing process of sponge iron

3.5 PROCEDURE FOR REDUCTION:

Before reduction, the iron ore were dried for 1 hr at 110 ^{0}C.The iron ore surrounded by coal were reduced in a metallic container (size 5 cm diameter,10 cm length) kept inside a muffle furnace until temperatures 850 ^{0}C, 900 ^{0}C, 950 ^{0}C, and 1000 ^{0}C. Out four containers three were taken out of the furnace in 15 minutes interval, after the furnace reached the predetermined reduction temperature and the last one was taken out at 30 minute interval. The containers were allowed to cool to room temperature in air. The initial and final weights of pellets were noted, from which weight loss (equal to oxygen removed) was calculated. The degree (%) of reduction was calculated using the given formula:

$$\% \text{ of reduction} = \frac{\text{weight loss of the iron ore x 100}}{\text{Total oxygen present in iron ore}}$$

3.6 CHARACTERIZATION TECHNIQUES:

3.6.1 X-ray diffraction analysis:

X-ray diffraction analysis (XRD) is a non-destructive, very versatile technique to determine the crystalline phases and their volume fractions. The sample is irradiated with monochromatic X-rays and the reflected radiation is recorded by the counters. In these techniques various forms of the samples could be used and very less amount is required for the phase determination. The X-ray diffraction pattern were recorded using Rigaku model Geiger diffractogram with CuK_{α} radiation (λ = 1.5418 Å) obtained from the copper target using as in built Ni filter. The 2θ values for XRD patterns were generally taken in the range of 5° to 100 ° for most of the samples at a scan rate of 5 ° per min. The inter planar spacing (d) values of samples were calculated using Bragg's law.

$$\boxed{\mathbf{2d\sin\theta = n\,\lambda}}$$

Where λ is the wavelength of the incident X-ray, d is the inter planar distance and θ the diffraction angle. The XRD patterns were identified using Powder Diffraction files (PDF). The geometric representation of Bragg's law is given in the Figure 3.2.

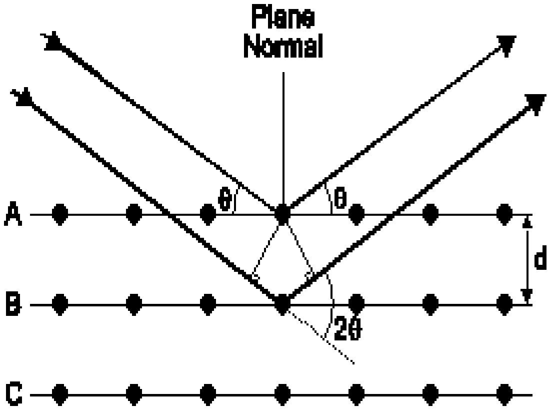


Figure 3.2: Geometric representation of X-ray diffraction

3.6.2 Scanning Electron Microscope:

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons on a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.

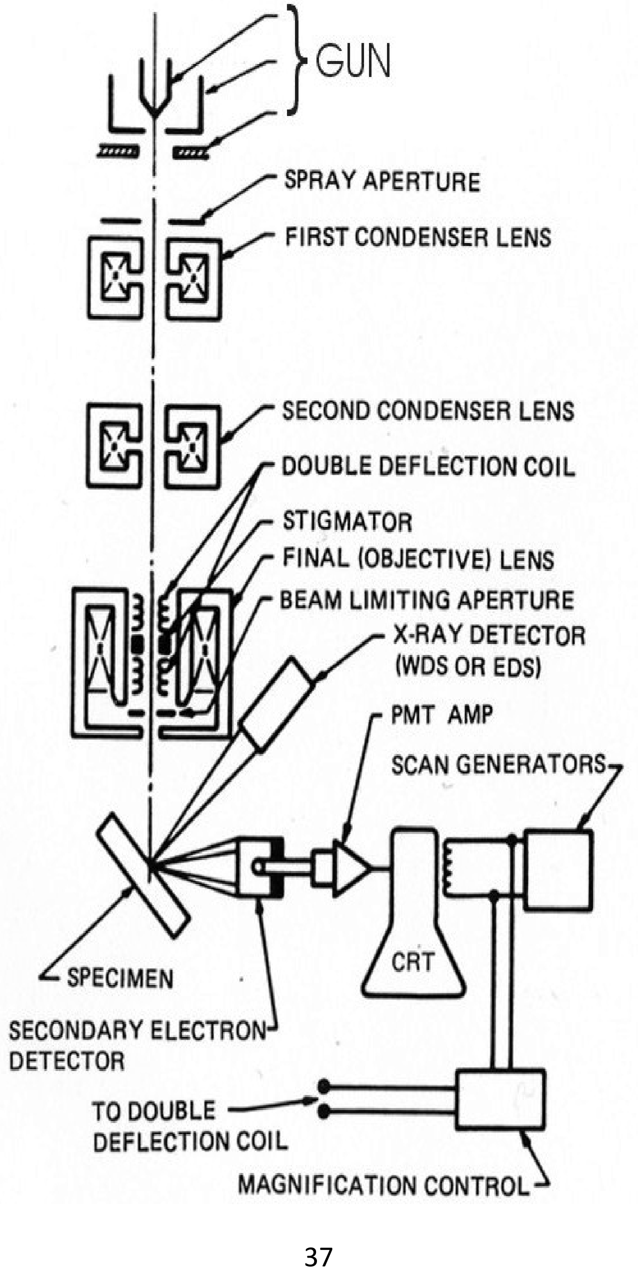


Fig 3.3: Scanning Electron Microscope

The types of signals made by an SEM can include secondary electrons, back scattered electrons, characteristic X-rays and light (cathodoluminescence). These signals come from the beam of electrons striking the surface of the specimen and interacting with the sample at or near its surface. In its primary detection mode, secondary electron imaging, the SEM can produce very high-resolution images of a sample surface, revealing details about 1 to 5 nm in size. Due to the way these images are created, SEM micrographs have a very large depth of focus yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. This great depth of field and the wide range of magnifications (commonly from about 25 times to 250,000 times) are available in the most common imaging mode for specimens in the SEM.

The operation of a SEM is schematically depicted in Figure 3.3. A beam of electrons is generated by an electron gun located at the top of the beam column. This beam is accelerated to the anode, condensed with a condenser lens, and focused to a very fine spot on the sample by the objective lens.

The scan coils, by varying the voltage produced by the scan generator, create a magnetic field, which deflects the beam back and forth in a controlled pattern. The varying voltage is also applied to the coils around the neck of the cathode-ray tube (CRT), which produces a pattern of light deflected back and forth on the CRT. In this way, the pattern of deflection of the electron beam is the same as the pattern of deflection of the spot of light on the CRT. SEM images can be generated by any signal produced by the interaction of a finely focused primary beam of electrons as it is scanned over the sample surface.

3.7 CALCULATION FORMULA USED:

Fe(T)= Total Iron in sponge iron

Fe(M)= Metallic Iron

Mtz= Metallization of sponge iron,

FeO= Wustite

V.M= Volatile material

F.C= Fixed Carbon

LOI= Loss of ignition

Fe (t) = Total iron in iron ore

1. Mtz% $= \frac{Fe(M)}{Fe(T)} \times 100$
2. Gangue $= 100 - Fe(T) + O_2$
   or Gangue $= 100 - Fe(M) + FeO$
3. F C =100 - Ash+ V.M
4. V.M+F.C =100%
5. Fe(m) $= \frac{Mtz \times Fe(t)}{100}$
6. FeO $= Fe(T) - Fe(M)$

Chapter 4 RESULT AND DISCUSSION

4.1 CHARACTERISTICS OF IRON ORE

The chemical compositions and loss on ignition (LOI) values of the hematite iron ore is given in table 4.1:

Table 4.1 Chemical analysis of iron ore

Fe	Al_2O_3	SiO_2	S	P	LOI
64.51	2.34	1.55	.006	.03	1.69
65.4	2.12	1.26	.009	.028	1.53
64.34	2.10	1.19	.015	.021	1.47

This indicates that the gangue materials in this iron ore are mainly alumina and silica. Screen analysis of all categories of iron ore (feed 1, feed 2 and feed 3) is given in table 4.2, table 4.3 and table 4.4 and their analysis is shown in figure 4.1, 4.2 and 4.3 respectively.

4.2 SCREEN ANALYSIS TEST

Table 4.2 Screen Analysis Test for feed 1

Feed iron ore		Feed coal		Coarse coal		Medium coal		Coal fine	
Size	%	Size	%	Size	%	Size	%	Size	%
+18	NIL	+25	0.51	+25	NIL	+15	1.13	+6	1.26
+15	8.89	+20	30.49	+15	88.80	+10	40.10	+3	46.01
+12	29.18	+15	16.52	+10	9.23	+6	32.05	+2	15.15
+10	18.04	+10	27.77	+6	1.18	+3	17.89	+1	15.13
+6	29.84	+5	32.49	-6	0.79	-3	8.83	-1	NIL
+5	7.56	-5	2.22	NIL	NIL	NIL	NIL	NIL	NIL
-5	6.49	SHALE	7.58	SHALE	4.43	SHALE	5.88	NIL	NIL

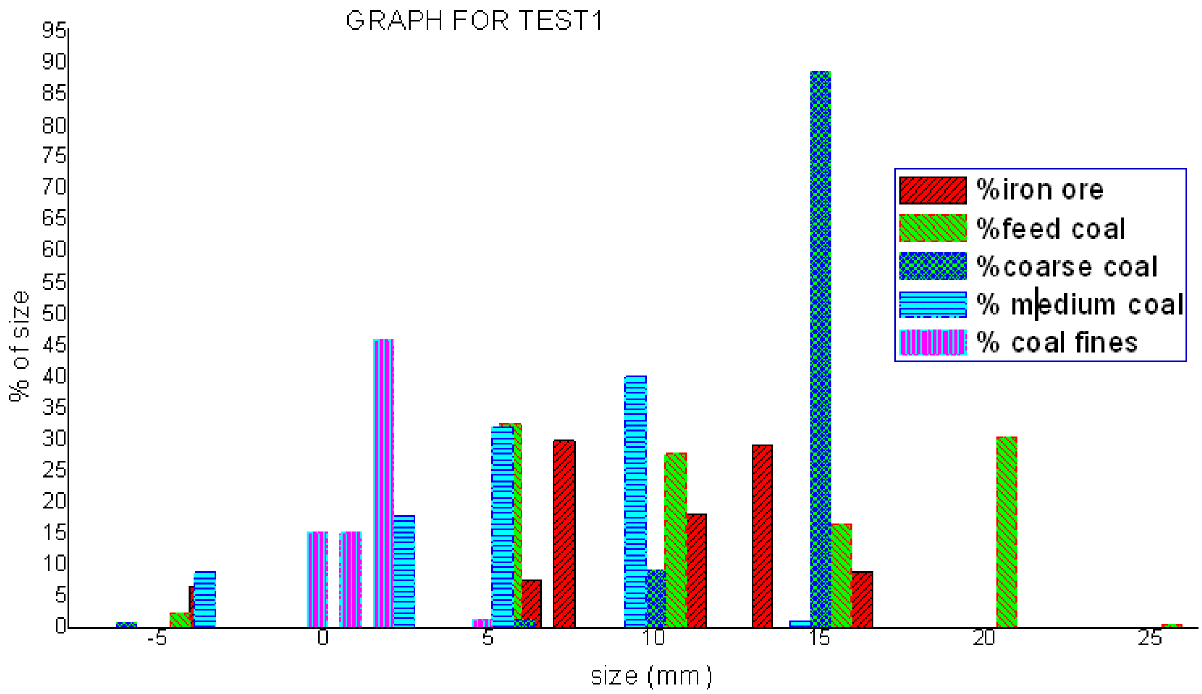


Figure 4.1 screen analysis for feed 1

From the data screen analysis of feed1 shows that the size of majority of iron ore lies in between 5 to 15 mm and coal size is also compatible to it which provides better reduction. But the size of coarse coal needs to be increased to produce better compatibility.

Table 4.3 Screen Analysis Test for feed 2

Feed iron ore		Feed coal		Coarse coal		Medium coal		Coal fine	
Size	%	Size	%	Size	%	Size	%	Size	%
+18	0.89	+25	1.16	+25	0.64	+15	1.84	+6	6.48
+15	14.50	+20	29.14	+15	31.22	+10	56.76	+3	47.93
+12	29.26	+15	17.02	+10	6.21	+6	34.71	+2	11.92
+10	16.41	+10	25.73	+6	0.42	+3	5.06	+1	13.73
+6	27.10	+5	23.31	-6	1.51	-3	1.63	-1	19.94
+5	6.36	-5	4.20	NIL	NIL	NIL	NIL	NIL	NIL
-5	5.48	SHALE	8.32	SHALE	8.1	SHALE	7.25	NIL	NIL

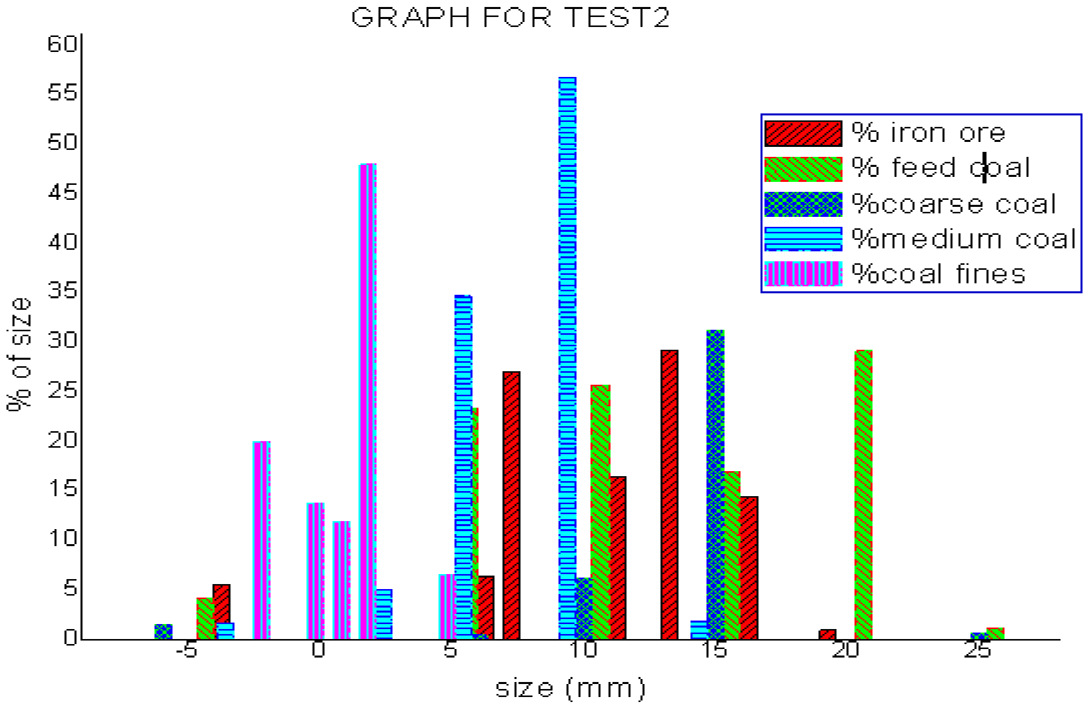


Figure 4.2 screen analysis for feed 2

From the data screen analysis of feed2 its observe that size of iron ore is compatible and lies within the required range for better reduction. But the percentage size of medium coal should be reduced as in this case it comes out to be higher and is not good for reduction. By increasing the fixed carbon percentage in feed coal we can achieve better reduction of iron ore.

Table.4.4 Screen Analysis Test for feed 3

Feed iron ore		Feed coal		Coarse coal		Medium coal		Coal fine	
Size	%	Size	%	Size	%	Size	%	Size	%
+18	0.36	+25	0.19	+25	1.68	+15	33.9	+6	4.60
+15	15.97	+20	30.37	+15	91.83	+10	61.3	+3	54.24
+12	25.84	+15	15.86	+10	5.29	+6	30.51	+2	9.20
+10	17.40	+10	25.73	+6	0.24	+3	3.67	+1	9.69
+6	25.03	+5	23.79	-6	0.96	-3	1.13	-1	22.27
+5	6.56	-5	4.06	NIL	NIL	NIL	NIL	NIL	NIL
-5	5.84	SHALE	8.83	SHALE	7.83	SHALE	7.14	NIL	NIL

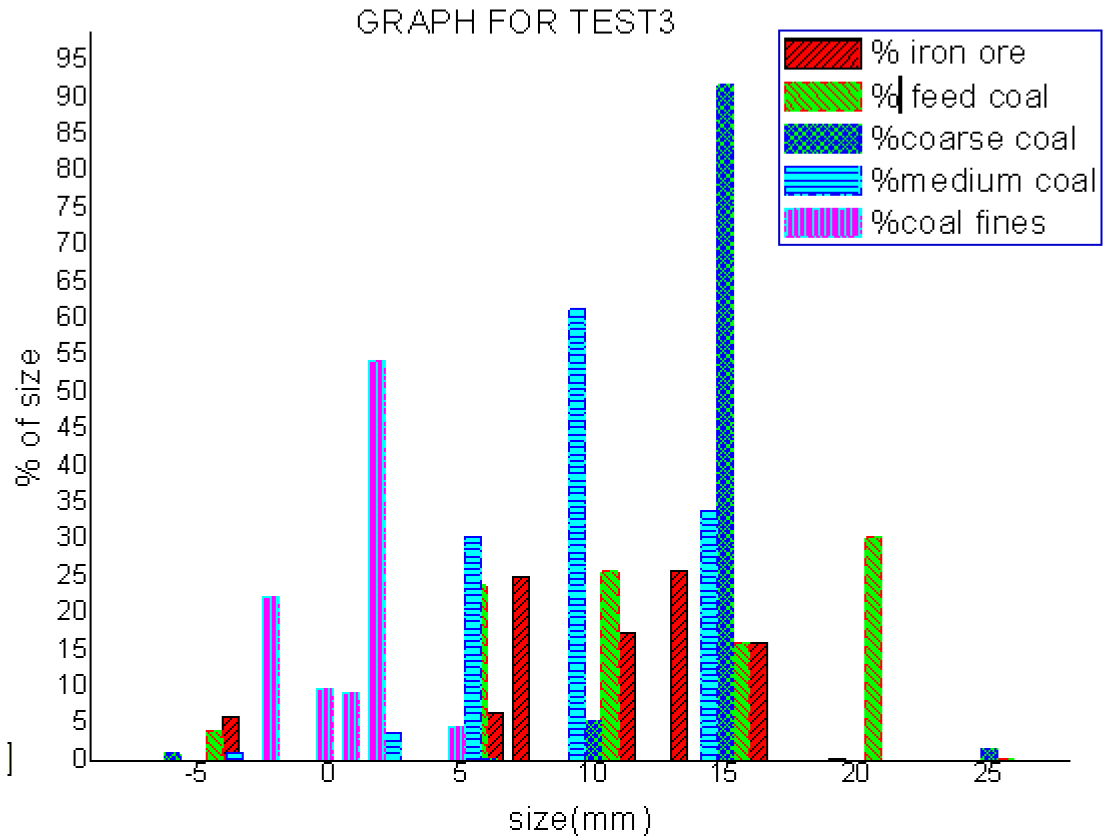


Figure 4.3 screen analysis for feed 3

From the screen analysis of feed3 its observe that due to high variation in size between medium and coarse coal, there is bad effect on the reduction of iron ore and it may result in poor grade quality. The less % of fixed carbon (FC) and volatile matter (VM) in coal can also be a reason for bad quality of sponge iron.

Size fraction of raw material is most important parameter for better production of sponge iron as mentioned above it was observed that if iron ore size lies in between 10-15 mm then it will provide better reduction and prevents sticking problem inside the kiln commonly known as ring formation problem.10-15 mm sized material is capable to absorb temperature and pressure shocks in ore body without much fluctuation of temperature and generation of fines. [29].

By adding high grade coal with ore, a good solution is formed which avoids sticking during reduction. The volatilities in the coal play an important role during initial steps of iron ore reduction, providing enough energy for complete reduction of hematite to magnetite and later for reduction of magnetite to wustite. The reduction of wustite to iron occurs by gases as product of gasification and production will increase.

Data for strength properties (tumbler, abrasion, shatter indices) of this iron ore have been presented in table 4.5. Tumbler, abrasion and shatter indices are the most popular properties to assess the resistance of iron ore towards degradation. As shown in table the iron ore offers higher resistance to abrasion and tumbling, because of their hard fine grained structure. For poor crushing strength it may be due to weak bonding between ore particles.

Table 4.5 Physical Testing of Iron Ore

Name of test	Feed 1 (%)	Feed 2 (%)	Feed 3 (%)
Tumbler Index	84	86.4	94.32
Abreaction Index	4.8	4.54	5.57
Shatter Test	85.87	89.93	92.66

4.3 CHARACTERISTICS OF NON COKING COALS

The non coking coal samples used in the present investigation have been characterized in terms of their proximate analysis of coal. The respective data have been shown in table 4.6, 4.7 and 4.8. The results in table clearly indicate that all the studies coals having no caking index. This is due to the high inertinite contents (fusinite, semifusinite etc.) in these coal samples. The proximate analysis results as shown in table 4.6, 4.7 and 4.8 reveals the fixed carbon contents in these coals are in the range of 27 – 40 % and ash contents are in the range of 30 – 45%. It apparent that all types of coals have high ash fusion temperature which is in good agreement with the data for sponge iron making. Higher ash fusion temperatures are expected to be due to the presence of Al_2O_3, SiO_2, TiO_2, and K_2O.

Table 4.6 Approximate Coal test for feed 1

TYPE OF COAL	%MOISTURE	%ASH	%V.M	%F.C	Caking index
FEED COAL	5.48	43.74	26.79	29.47	NIL
COARSE COAL	5.39	39.17	26.32	34.51	NIL
MEDIUM COAL	5.79	40.37	28.44	31.19	NIL
COAL FINE	6.43	38.51	28.41	32.78	NIL
CHAR COAL	NIL	19.27	2.91	18.54	NIL

Table.4.7 Approximate Coal test for feed 2

TYPE OF COAL	%MOISTURE	%ASH	%V.M	%F.C	Caking index
FEED COAL	5.75	46.04	25.28	28.68	NIL
COARSE COAL	5.82	41.21	27.25	31`.54	NIL
MEDIUM COAL	5.90	42.29	27.21	30.50	NIL
COAL FINE	6.35	45.09	25.09	29.86	NIL
CHAR COAL	NIL	80.18	2.19	17.65	NIL

Table 4.8 Approximate Coal test for feed 3

TYPE OF COAL	%MOISTURE	%ASH	%V.M	%F.C	Caking index
FEED COAL	5.39	47.96	24.73	27.31	NIL
COARSE COAL	5.54	44.07	26.51	29.42	NIL
MEDIUM COAL	5.84	44.13	26.51	29.25	NIL
COAL FINE	6.19	45.74	25.54	28.72	NIL
CHAR COAL	NIL	79.79	2.42	17.79	NIL

Dolomite is used in reduction of iron ore it helps in slag formation and thus reducing the sulphur content in iron. Dolomite is mainly used as a desulphurising agent to prevent the pickup of sulphur by the sponge iron from the sulphur released by the burning of coal inside the furnace. The chemical analysis of dolomite is given table 4.9.

Table 4.9 Chemical Analysis of Dolomite

Constituents	Feed 1 (%)	Feed 2 (%)	Feed 3 (%)
LOI	44.12	43.50	44.12
CaO	27.78	28.25	27.78
MgO	20.16	19.40	20.18
SiO_2	4.32	4.22	3.92

4.4 EFFECT OF REDUCTION TIME ON DEGREE OF REDUCTION OF IRON ORE:

In the above part of this chapter a detailed analysis of raw material have been presented. These raw materials have been used see the feasibility of reducing iron ore to sponge iron. For our easy analysis the data presented in sieve analysis as feed 1, feed 2 and feed 3 are the base constituents used in the reduction experiments. In total 3 sets of experiment (feed 1, 2 and 3) ware conducted. In order to have an average value each set of experiment were further sub divided into nine segments. The average of each has been considered in the presentation of the data which are given in the next section. For this study 19 ton iron ores, 10.5 ton feed coal and half ton of dolomite mixture was blended and fed inside the kiln and 5 ton coarse coal, 4.5 ton medium coal and 1.5 ton fine coal mixture was fed from outlet side of the kiln. Effect of percentage weight reduction of iron ore is presented graphically in figure 4.4.

Illustrate the effect of time for different reduction temperature 900, 950, 1000 and 1050 ^{0} C for hematite iron ore reduced by three types of different non coking coals under non-isothermal condition. It can be observed from this that the reduction time has an approximately identical effect on the reduction behavior of all the studied iron ore-coal combination. With increase in reduction time, the degree of reduction increased at every temperature under consideration the rate of reduction in general was observed to be high up to about 70 – 80 % reduction and then after that it decreases formation of solid reductant at the point of contact on the surface of pellet which produces CO/CO_2 gas. The CO_2 produced combines with solid carbon and gets converted into CO gas. The CO gas diffuses into the pellet and takes part in the reduction. The higher reduction rate in initial conditions may be attributed to the combined effect of less resistance offered to the flow of reducing gas into the iron ore and a significance contribution of volatile matter released initially, as suggested by Bodsworth et al. [29]. The released volatile matters of coal get almost reformed completely into H_2, H_2O and CO during the initial stage of reduction. It might be expected that the pressure of H_2 and CO as reducing gas gives boost in the reduction rate. As the reduction progresses with time, the thickness of the product iron layer increases and offers greater resistance to the diffusion of carbon and reducing gas onto the surface of unreduced iron oxide. This is the reason for lower rate of reduction in the later stages at all the temperatures.

Table 4.10 Effect of time and temperature on degree of reduction percentage

Time in (min)		**Reduction**, %		
	900°**C**	**950 °C**	**1000 °C**	**1050 °C**
15	35.82	45.69	49.21	59.20
30	57.19	63.67	68.10	84.18
45	65.14	72.33	74.98	84.18
60	67.61	72.33	81.51	86.21
75	72.64	82.70	83.98	86.86

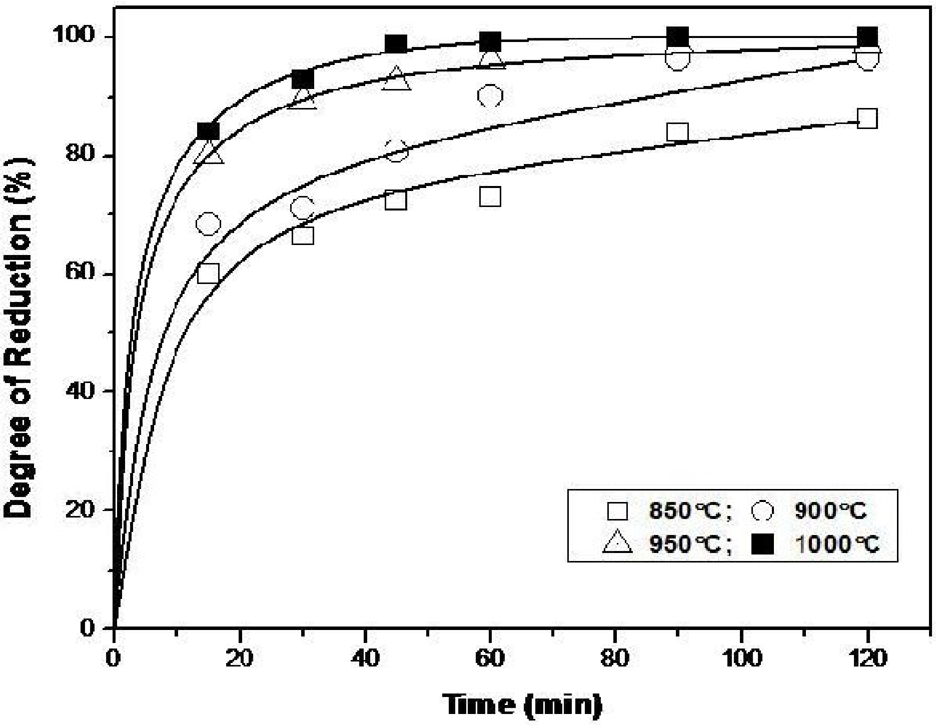


Figure 4.4 Effect of time and temperature on degree of reduction

$$\% \text{ of reduction} = \frac{\text{weight loss of the iron ore x 100}}{\text{Total oxygen present in ore ore}}$$

The analysis of the data indicate that the reduction is faster up to about 70-80 % and then slows down at the latter stages. This is undoubtedly due to formation a dense metallic iron layer over unreduced iron oxide as the described earlier on the other hand, reduction (%) was enhanced with the increase in temperature. Reduction at 900 and 950 ^{0}C was not complete within the studied time period (75 minutes) and stopped at about 73% reduction at 900 ^{0}C and 83% reduction at 950 ^{0}C, where reduction stopped at about 84% at 1000^{0}C and about 87% at 1050 ^{0}C. This may be due to the catalytic effect of newly formed metallic iron layer on the reduction rate of the iron ore. The greater influence of temperature was observed up to 60-70 minutes time period. It is believed that as the temperature increases, the initially formed iron layer grow through further reduction leading to higher degree of reduction at higher temperatures.

4.5 EFFECT OF COAL TYPE ON THE DEGREE OF REDUCTION OF IRON ORE In order to study the effect of coal type on the degree of reduction of hematite iron ore, the degree of reduction of hematite iron ore in the three different coals at different times are presented graphically at four different temperatures (850, 900, 950 and 1000^0C)**.** The proximate analysis and reactivity data of three type's coals are already given in table 4.5, 4.6, 4.7.

Table 4.11 Comparison of Degree of Reduction (%)

Temperature ^{0}C	Time (minute)	Reduction % Bigger size (18mm)	Reduction % Smaller size (5 mm)
850	30	13.93	58.32
	45	19.30	70.79
	60	20.0	68.51
	90	21.50	76.73
900	30	28.44	68.26
	45	35.83	75.38
	60	36.10	87.63
	90	39.04	90.20
950	30	36.91	87.14
	45	37.50	89.66
	60	49.33	90.82
	90	5070	93.17
1000	30	53.23	86.35
	45	71.10	94.20
	60	71.11	100.0
	90	79.80	100.0

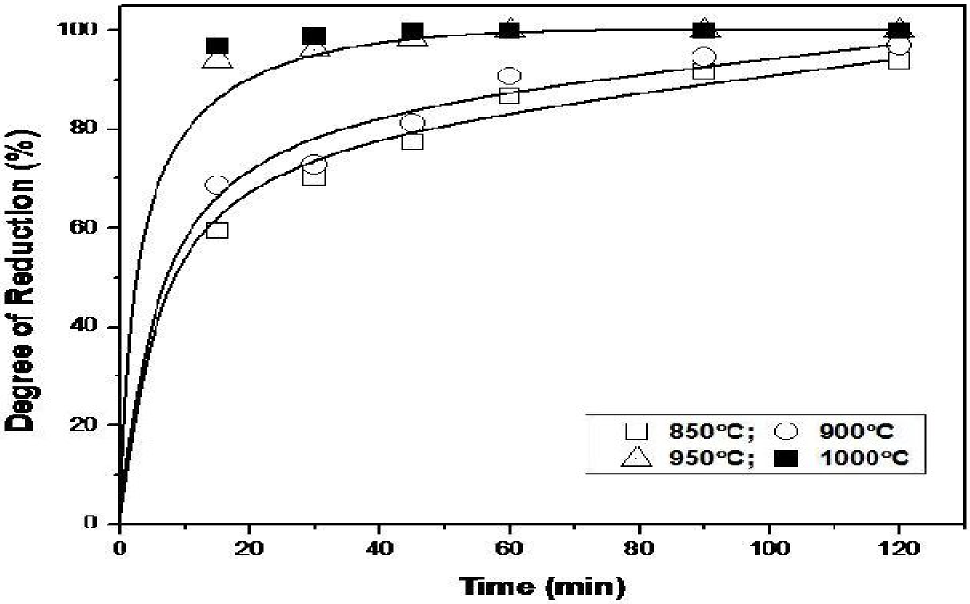


Figure 4.5 Effect of coal on the degree of reduction with time smaller size (5 mm) iron ore

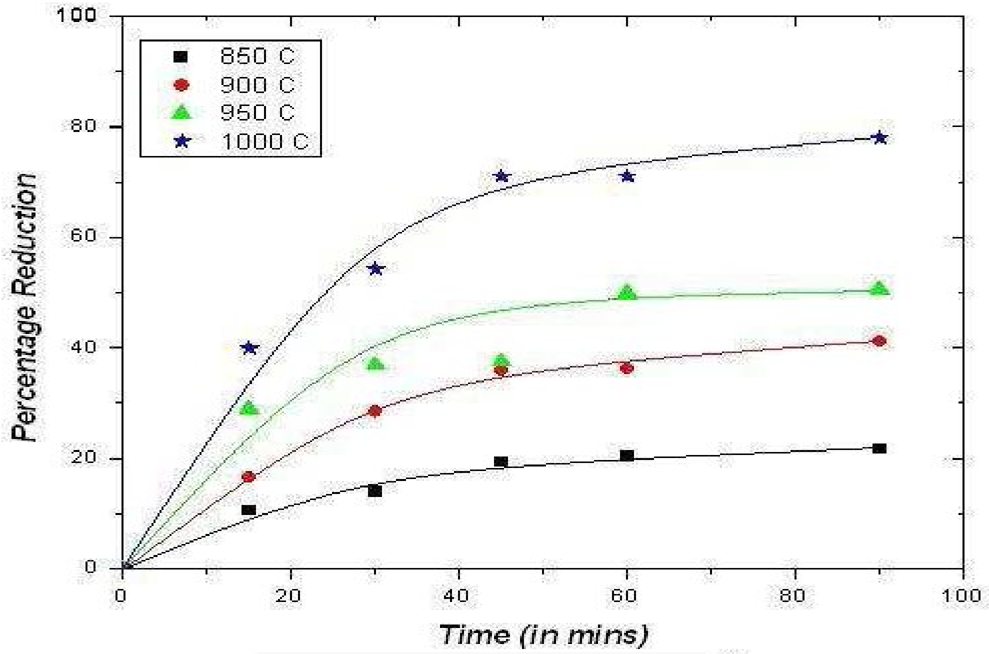


Figure 4.6 Effect of coal on the degree of reduction with time bigger size (18mm) iron ore

It is clear from the graph that, the reduction behavior of iron ore is nearly identical in these three non coking coals at the four different temperatures. This may be due to generation of sufficient amount of reducing gas (CO) in case of high FC and V.M. value of coals. Hence, the effect of reactivity of coal appears to be not pronounced. But however, the effect of reactivity may appear if, the time interval of reduction at a particular temperature is reduced.

Percentage of reduction of iron ore increased with increase in temperature and time as the increase in temperature and time leads to an increase in the rate and quantity of diffusion.

4.6 COMPARISON OF PERCENTAGE REDUCTION OF HIGH ASH COAL WITH LOW ASH COAL SAMPLE

To study the effect of quality of non-coking coal samples from were it was taken in as received from (with 42% ash) and another (with 46% ash). The reduction tests were carried out with these two coals at 1050°C. The results obtained have been tabulated. Reactivity of low ash coal having 42% ash is higher than another (high ash) coal. Also the volatile matter (VM) content of low ash coal is higher (26%) than another (high ash coal) 21%. Volatile matter also plays an important role in reduction of iron oxide. This may be the reason for higher reduction rate of low ash content of coal.

Table 4.12 Comparison of percentage reduction of high ash coal sample with low ash coal sample

Time, min %	Reduction at 1050 °C with high ash sample, %	Reduction at 1050^0Cwith low ash sample%
15	59.20	54.12
30	74.22	78.68
45	83.18	86.90
60	86.21	89.76
75	86.86	90.85

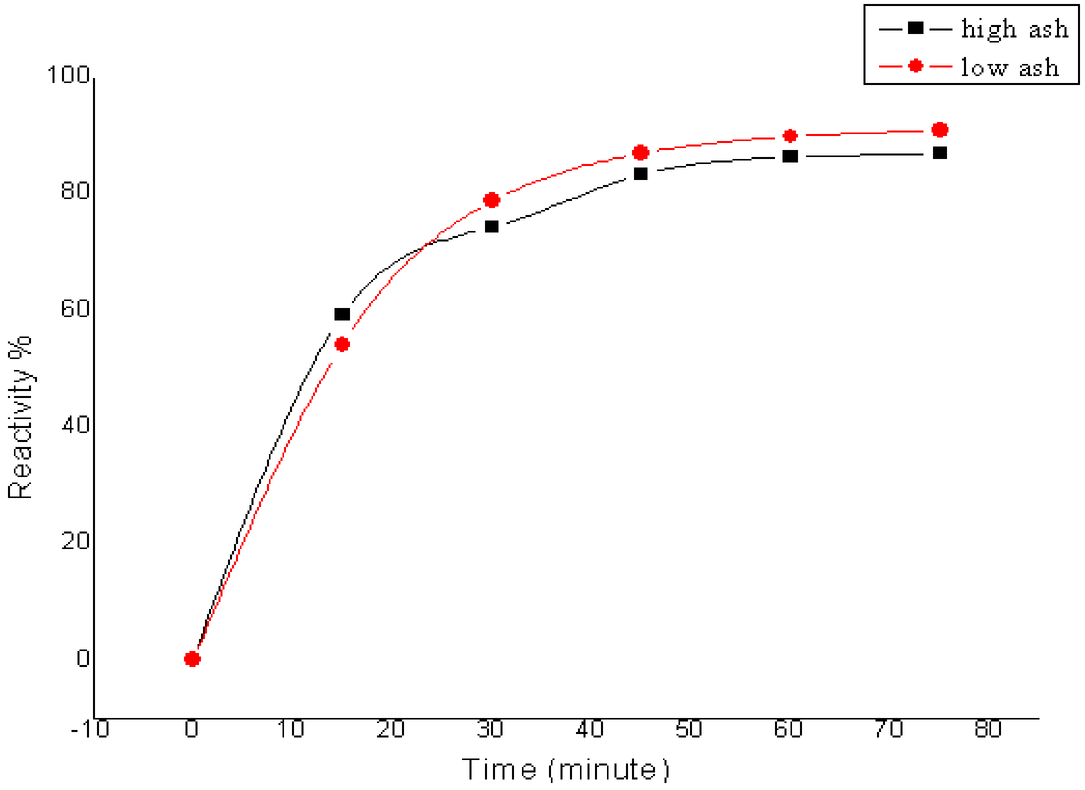


Figure 4.7 Reactivity between high and low ash coal

4.7 COMPRISION OF REDUCTION BEHAVIOR SAMAL SIZE Vs LUMP OF IRON ORE

This study provides more reliable information on the reduction behavior of lumps and small size for a better application in the actual industrial reactors. In this study, reduction behavior of dried iron ore lumps have been compared with those of hard iron ore reduced under identical non isothermal condition. The results (Degree of Reduction) obtained have been presented. It is evident from this that the iron ore lumps have lower degree of reduction than the corresponding small size iron ore. This clearly indicates that the dried iron ore lumps have much lower porosity values than those of corresponding small size iron ore. The appreciably lower porosity in iron ore lumps appears to be most likely the reason for its lower reducibility. As outlined in the literature [27], hematite pellets tend to be more disordered and hence more reactive wustite, which enhance the rate of wustite reduction**.** This may be another reason for relatively higher reducibility of small size, as observed in the present investigation.

4.8 Chemical Analysis Test

The sponge iron produced was subjected to magnetic separator which eliminates the non magnetic elements. In order to grade the sponge iron different test as described in experimental section were done. The result are summarized in table 4.13 and presented in figure 4.8.

Table 4.13 Sponge Iron Test prepared from Feed 1

TIME(Hrs)	% NON MAGNETIC	% Fe(M)	%Fe(T)	%METALLIZATION (Mtz)	%+5 mm. Mag	GRADE
1	42.31	84.00	92.53	90.70	26.51	A
2	41.95	85.80	93.16	92.10	24.78	A
3	40.72	84.20	92.57	90.96	26.29	A
4	41.34	85.00	92.73	91.64	27.18	A
5	40.81	84.50	92.64	91.21	28.49	A
6	40.31	86.50	93.31	92.70	30.46	A
7	40.32	88.60	93.77	94.49	30.58	A
8	39.87	84.00	93.83	94.85	30.70	A

% OF SULPHUR =0.031, % OF CARBON =0.05.

(% Wt= %Weight)

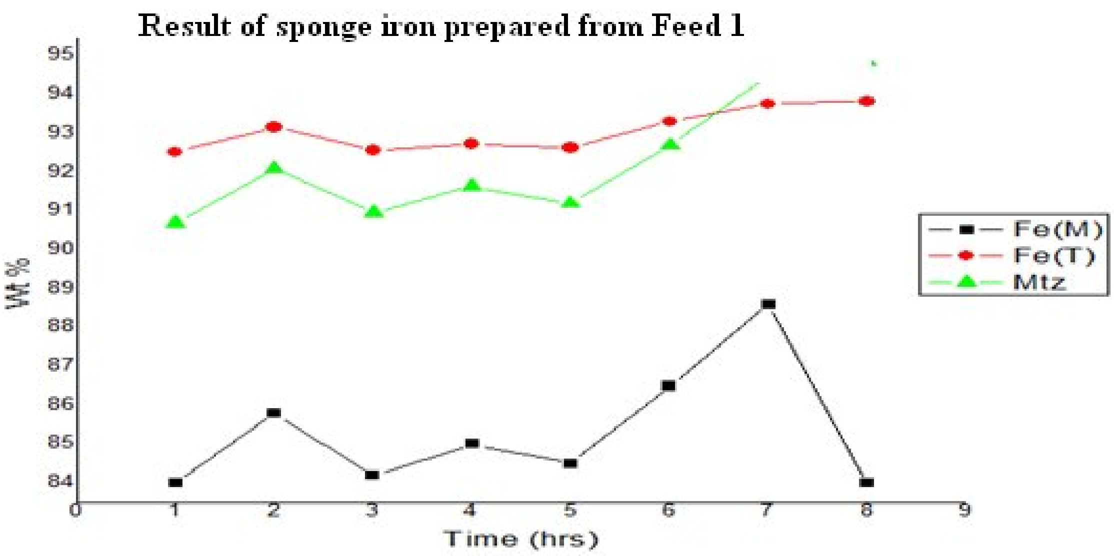


Figure 4.8 chemical test results from Feed 1

From this figure it was observed that if Fe (M) increases then metallization also increases because FeO content is getting decreased which leads to subsequent fall in oxygen content providing A grade sponge iron

Table 4.14 Sponge Iron Test prepared from Feed 2

TIME(Hrs)	% NON MAGNETIC	% Fe(M)	%Fe(T)	%METALLIZATION (Mtz)	%+5 mm. Mag	GRADE
1	39.71	82.00	91.88	89.25	30.29	A
2	39.25	81.00	91.56	88.47	30.45	B
3	41.98	83.00	92.20	90.02	30.13	A
4	41.76	82.00	91.88	89.25	30.07	A
5	40.45	84.50	92.64	91.21	28.62	A
6	40.17	83.50	92.37	90.40	28.36	A
7	40.38	85.00	92.73	91.64	29.41	A
8	40.53	81.50	91.50	88.86	29.28	B

% OF SULPHUR =0.027, % OF CARBON =0.08

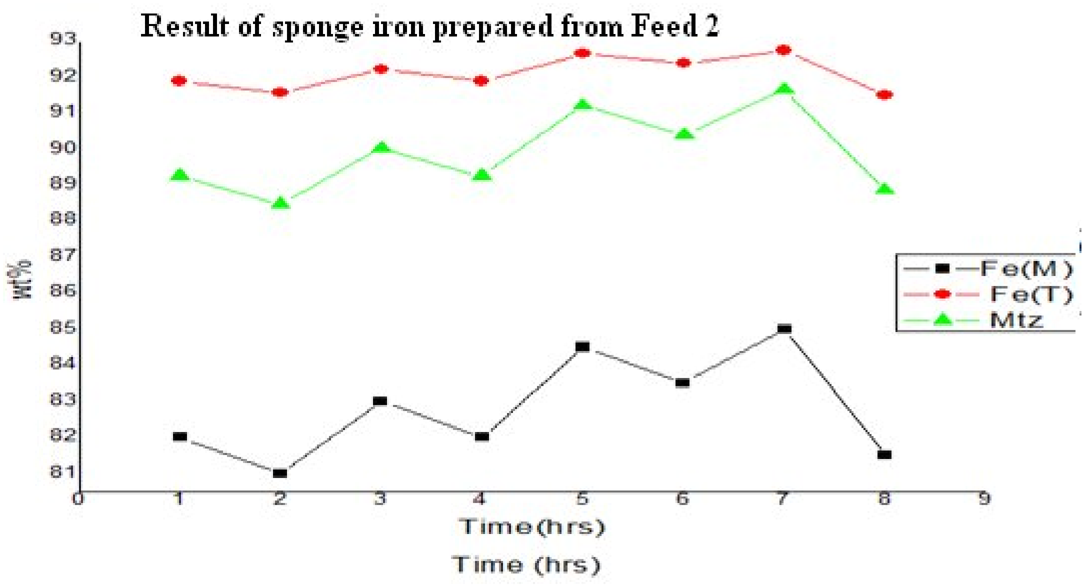


Figure 4.9 chemical test results from Feed 2

From the figure it is observed that Fe (M) decreases as FeO content increases. Hence the increase in oxygen content inside sponge iron leads to the production of B grade sponge iron. Fe (T) also shows inverse effect on metallization. In graph Fe (T) does not show a constant value because coal consumption are varying with time.

Table 4.15 Sponge Iron Test prepared from Feed 3

TIME(HRS)	% NON MAGNETIC	% Fe(M)	%Fe(T)	%METALLIZATION (Mtz)	%+5 mm. Mag	GRADE
1	40.57	78.50	90.09	87.15	32.46	B
2	38.73	80.5O	91.22	88.25	24.46	B
3	40.73	82.50	92.10	89.75	24.97	A
4	39.42	82.70	92.14	89.58	32.84	A
5	39.38	82.50	92.4O	88.58	31.78	B
6	39.93	80.76	91.36	88.33	3780	B
7	36.78	81.50	91.36	88.86	37.77	B
8	38.27	82.00	91.88	89.25	25.80	A

% OF SULPHUR =0.025, % OF CARBON =0.06

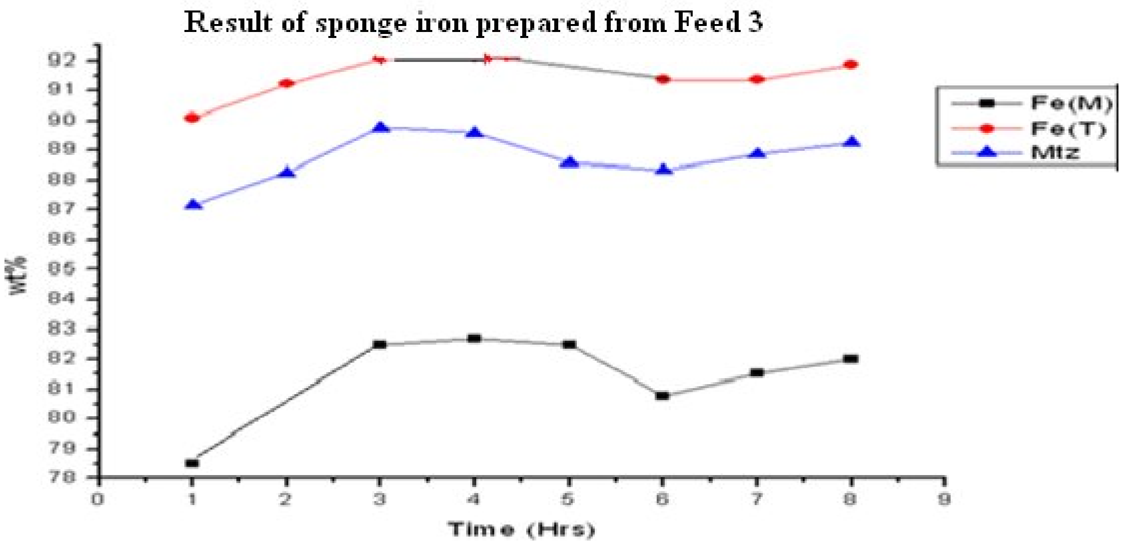


Figure 4.10 chemical test results from Feed 3

It is very clear from the above graph that FeO content increases and metallization decreases. Further if Fe (M) increases then Fe (T) also increases. But the decreasing values of Fe (T) effects grade of sponge iron.

4.9 XRD Analysis of Sponge Iron

Figure 4.11 produced the x ray diffraction of sponge iron in figure 4.11 is A grade sponge iron produced from feed 1 and figure 4.12 is B grade sponge iron produced from feed 2

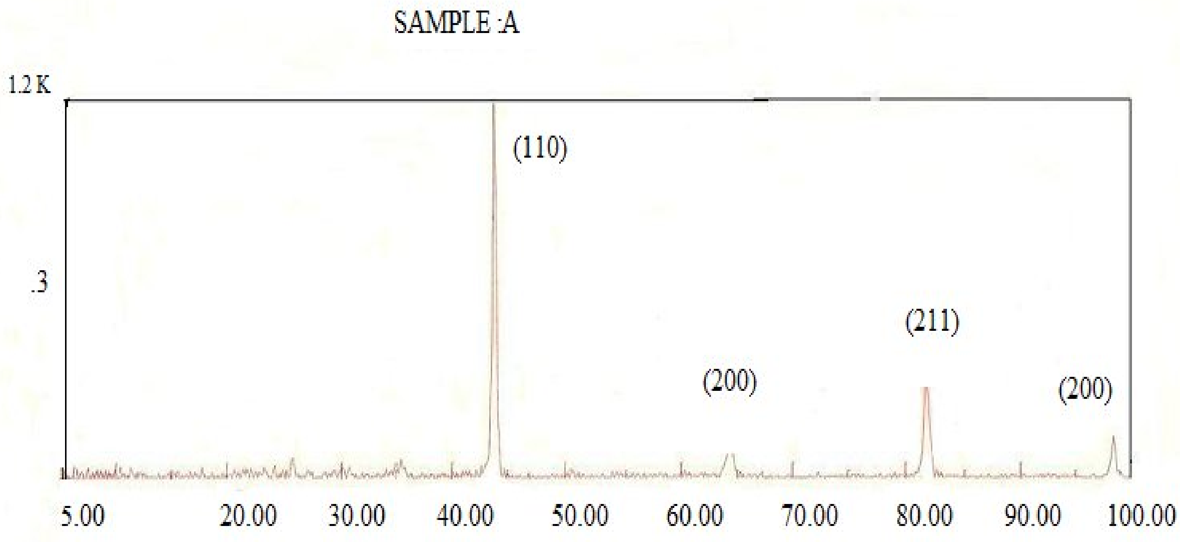


Figure 4.11: XRD pattern for A grade sponge iron.

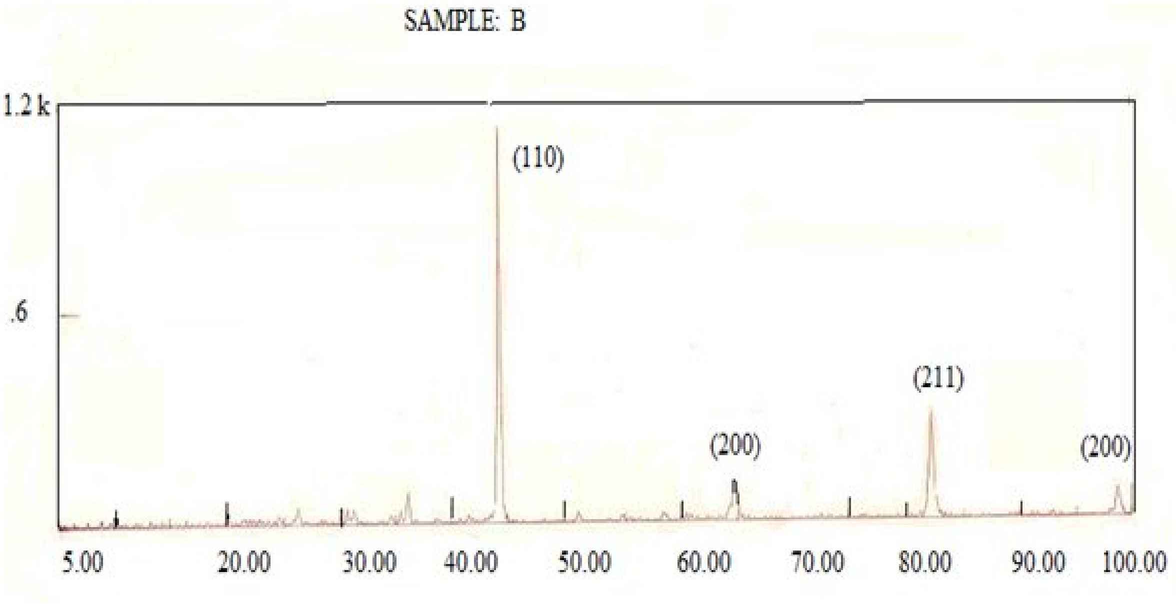


Figure 4.12: XRD pattern for B grade sponge iron

The XRD graphical data where compared with JCPDF data file no 60696. It was observed that highest intensity of peaks is of pure iron phase and their planes are indicated above their respective peaks in the graph as shown in figure. Some others peaks are found having lower intensity and are not clearly visible. So we can't compare them with any data file because these peaks are mostly due to impurities or mixture of phases.

Figure presented X ray diffraction of B grade sponge iron of feed 2 only some variations in intensity have been observed in A grade and B grade samples. Pure iron phase have been detected in both of them.

4.10 Microscopic Examination of Sponge Iron

The produced sponge iron of both grade (A & B) are analysed by SEM. Three distinct phases were identified in sponge iron by SEM which appeared as grey, white and mottled white black areas. Figure 4.13 presents the micrograph of A grade.

Examination by scanning electron microscopy showed that the grey phase was of quartz, the white phase hematite, and the mottled areas intergrowths were of hematite and magnetite. Samples of the iron ores, partially and fully-reduced both by hydrogen and carbon monoxide are shown in micrographs 4.13(a, b) respectively.

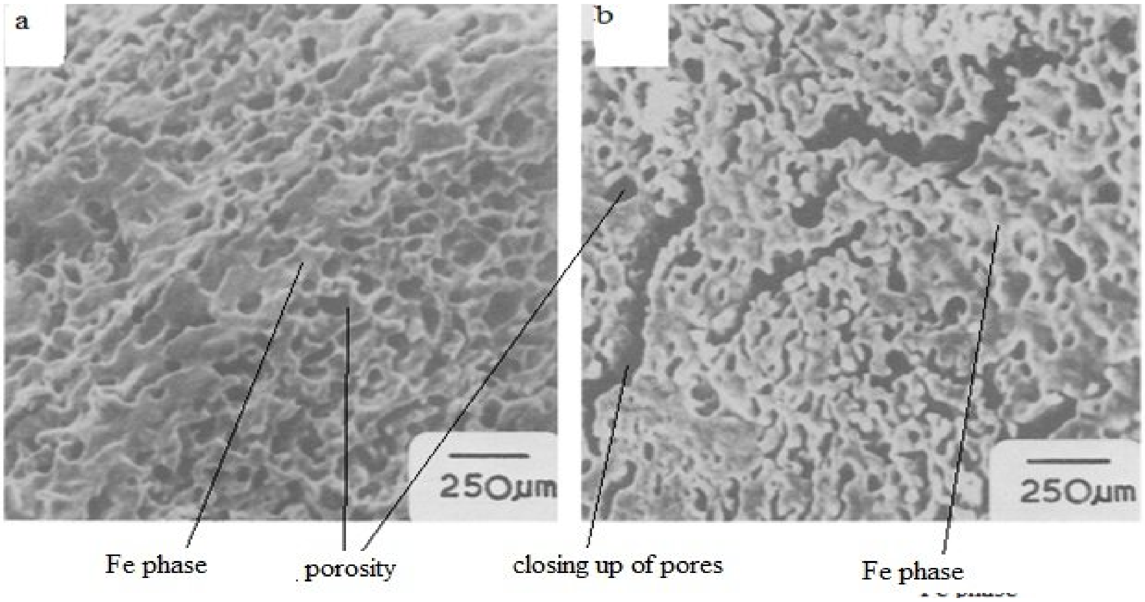


Figure 4.13 Micrograph for A grade sponge iron (a, b).

The most significant feature observed is the slight sintering and closing-up of pores that occur when the sponge iron is fully reduced as the figure 4.13 (a & b). The morphologies of the products obtained by hydrogen reduction were similar to those obtained in carbon monoxide although the products from hydrogen reduction were more sintered in nature at all temperatures than those obtained in carbon monoxide. Both lower-grade ores gave spongy (although very impure) iron products with many similar features.

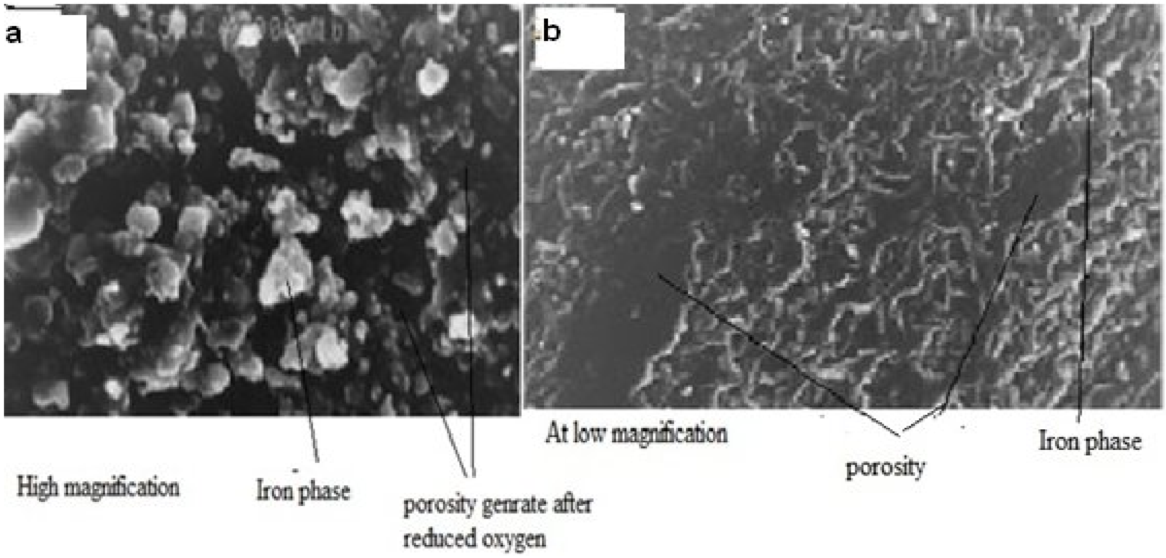


Figure 4.14: Micrograph for A grade sponge iron (a, b).

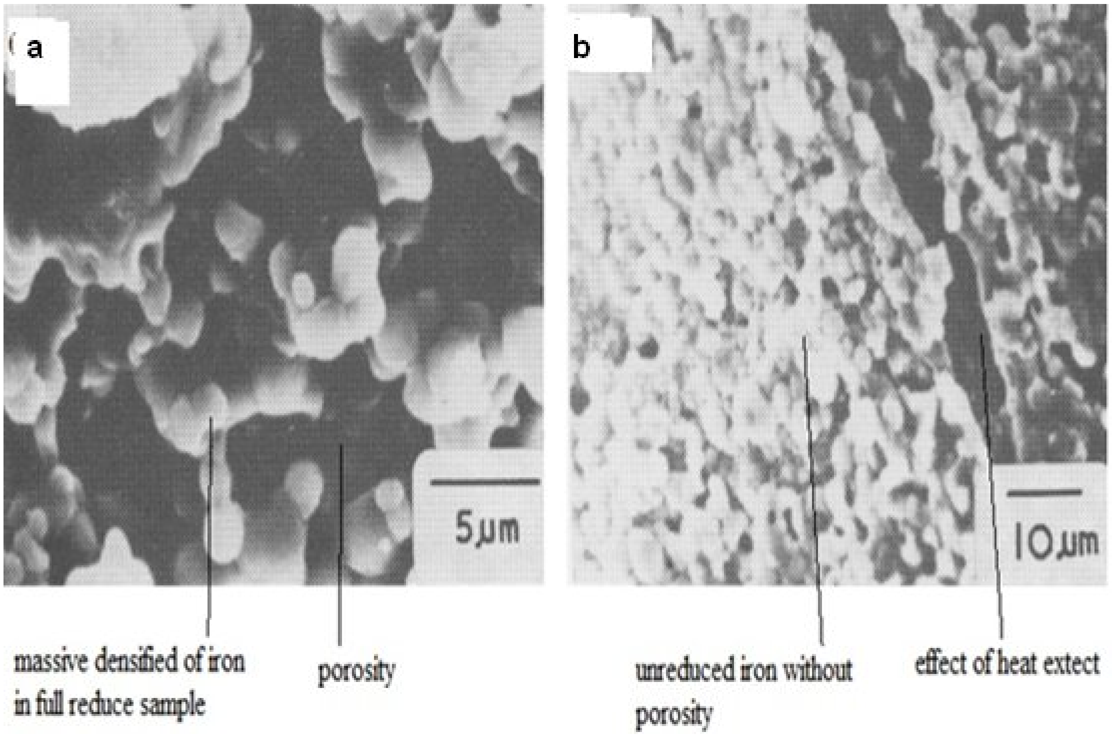


Figure 4.15: Micrograph for B grade sponge iron (a,b).

In figure micrograph 4.15 (Figure a & b) shows B grade sponge iron fig (a) shows that fully-reduced samples were found to contain massively densified iron inside the structure and even more significantly sintering together of the individual particles. At 1100 ^{O}C sintering of the product causes diffusion to become the rate-controlling step. According to 4.15 (Figure b) unreduced ore shows minority of porosity and increase in the oxygen content inside that is responsible for reducing the porosity. In micrograph Fig 4.15 (a & b.) the lighter phase is now metallic iron which, at higher magnifications, can be seen as individual grains.

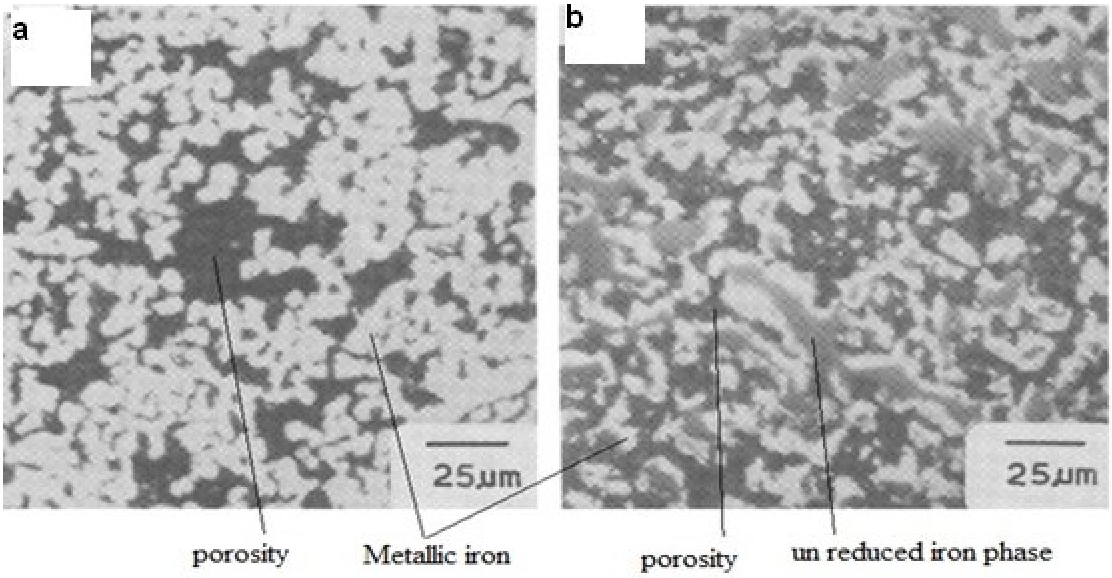


Figure4.16: Micrograph for B grade sponge iron (a, b).

The topochemical nature of the reduction process was clearly seen in all partially-reduced samples. Fig.4.16 (b) shows iron rims encapsulate the remaining unreduced oxide (in this case wustite) .Sintering is not so extensive during reduction with carbon monoxide because of carbon deposition.

Microscopy and image (quantitative) analysis could be very useful for detecting the change size of iron ore during reduction process. The change of size could be due to transformation lattice structure during reduction and due to sticking phenomena as well as fully reduced iron ore having sufficient number of porosity which is obtained by removing oxygen as shown in above micrographs. If porosity is high according to micrograph, then oxygen reduction is better and quality of sponge iron gets improved.

The solid oxide iron moves under gravity in rotary kiln where time is permitted for completion of reaction based on size of iron oxide. Finer particles reduce rapidly due to more exposure of surface area but takes longer retention time in the kiln. When finer particles are generated then accereation problem occurs. By physical testing of material, we can determine the possibility of generation of fines inside kiln. Depending on this, the operating and temperature is lowered and fusion point of coal is increased for better production.

These semi-reduced and reduced finer particles occupies bottom of the kiln due to heaviness and ash of the coal covers the top of such materials with formation of dense layers, this will form low melting point compound which will cause for accereation. So it has been observed that ash content in coal should be less whereas fixed carbon content and volatile component in coal must be high which helps in the formation of reducing gas.

SHAIL quantity in coal should be less in coal for better production of sponge iron. The quality of sponge iron is primarily ascertained by the percent of Metallization (removal of oxygen), which is the ratio of Metallic Iron to the total iron present in the product.

The Indian non-coking coals are unsuitable for many applications but appear ideally suited for treatment in rotary kiln sponge iron making processes and this trend is likely to continue for a few decades to come.

Chapter 5 CONCLUSION AND FUTURE SCOPE

The above results indicate that the iron ore gets reduced and easily. The rapid reduction of this ore can be explained on the basis of its purity and the presence of iron in hematite ore. The results showed a pronounced effect of temperature on the extent of reduction in all the studied hematite iron ore. The reduction rate was enhanced substantially by an increase in temperature up to 950 ^{0}C and then decreased at 1000 ^{0}C.At all studied temperatures, the degree of reduction increased with heating time, the rate being faster in the first 30 minutes because of greater influence of volatile matter. The majority of hematite iron ore reduced at 900 and 950 ^{0}C, contained a substantial number of broad cracks, most probably because of build-up of high gas pressure at the iron-wustite interface. The presence of 64 -66% Fe (T) (total iron) in ore is most suitable ore for sponge iron making. Produced Sponge iron has very low levels of contaminant such as, sulphur and phosphorus so that it is most suitable for steel making. The reduction rate increases with increase in heating time, the reduction could be achieved by gases generated in gasification process as well as influence of volatile content in coal.

Enhancement carbon is a bigger problem in sponge iron. There is no way to improve carbon in sponge iron. This area is attracting many works toward it. In non magnetic, charcoal goes to outlet from kiln having more than 20% FC (fixed carbon) waste which is not used as reduction of ore.

The various slag-forming constituents (silica, alumina, lime and magnesium oxide) are so closely associated with the iron-bearing constituents that separation is impossible to achieve by simple physical means. Furthermore, the high phosphorus content (about 2.5%) would probably give rise to problems in steel production.

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