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Reduction of Iron Oxide using Microwave Irradiation

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***Abstract*- The reduction of iron ore with carbon was investigated using a microwave heating techniques. The heat generated by the reaction mixtures while interacting with microwave, percentage, chemical composition and microstructure of iron produced were studied in detail. The experiments were carried out using 2.45 GHz microwave processor and reaction rates were analyzed at different power ratings: 540, 720 and 900 watts. The maximum reduction in weight obtained was 50.47% at 900 W power supply. The effect of varying the concentration of binders was also studied. Unlike conventional processing the microwave heating was found to be through the absorption of microwaves by reaction mixtures. The microwave processing showed considerable reduction in reaction time increases the yield and also reduces unwanted side reactions. However it showed uneven heating as the heating is due to the distribution of microwave absorbers present in the reaction mixture. Solid state reduction was observed at initial 10-40 minutes and molten iron produced above 40 minutes and the temperature attained was around 8000C. The microwave reduced mixture were characterized using SEM, EDS and XRD technique to find out the reduction of hematite. After the EDS analysis, it was found out that the iron content formed after the reduction process was 46.23 wt %.**

***Keywords*- conventional processing, EDS analysis, SEM, EDS and XRD technique etc.**

I. INTRODUCTION

Iron is believed to be the fourth most profuse element in the earth‘s crust up to 4.5% [1]. It is also the most useful in all the category of metals, comprising 95% of all the iron tonnage produced globally. Iron is generally produced from iron ores, mainly magnetite (Fe3O4), hematite (Fe2O3) and wustite (FeO) all these are reduced in the solid state in a array of reactors such as retorts, shaft furnaces, rotary kilns and fluidized beds to produce Direct Reduced Iron (DRI). The reducing agents used in this process are mainly reducing gases or coal based charcoal [2]. The content of Fe in the iron ore are limited to 25 – 70% Fe , or nearly 5 to 15 times the average iron content of the earth‘s crust. Recently, a growing interest in the field of microwave heating in minerals and metals treatment has emerge and a number of prospective applications of microwave processing have been investigated.

These included microwave assisted carbothermic reduction of metal oxides, microwave assisted ore grinding, microwave-assisted drying and an-hydration, microwave-assisted mineral leaching, smelting and roasting of supplied concentrate and many others microwave assisted process like pre-treatment of refractory gold concentrate, spent carbon regeneration and waste management [3]. Generally the common heavy metal oxides and carbon as charcoal (coke) give better response to microwave heating.So the microwave assisted carbothermic reduction of metal oxides is possible. Recently, the microwave heating becomes a novel method of supplying energy as compared heating with other conventional method. Microwave heating has been studied broadly to apply several chemical reductions and heating processes Microwave heating more advantagesious than conventional methods as follows.

1. Microwave rapidly heats the objects to high temperatures without any high speed blasting and a thermal drivingforce.
2. Microwave heats internally so it is a form of internal heating. A material can be heated to a high temperature uniformly in thefield.

So, the material can be uniformly-heated in a microscopical sense for better results [4]. Finally, the role of carbon was adequate to be the reducing agent, approximately half of the CO2 gas emissions can be reduced in comparison to the blast furnace, and pompously the microwave is excited from greenable electric power by hydro, solar and nuclear plants [5]. Microwave heating is more economical than cupola furnace process with respect to the production cost. A microwave can be operated in several of power supply and the irradiation time to get the optimal reduction process indicated by huge amount of Fe content.

Microwave is the coherent electromagnetic wave with a range of 0.3-300 GHz and it is absorbed through powder material with various types of losses such as electric polarization loss, eddy current loss and magnetic loss. The Microwaves with frequency 2.45 GHz, is mostly absorbed by the mixed powder of iron oxide with carbon which generates heat themselves to produce molten pig iron [6]. The interaction of material with microwaves can be classified as various types, depending on how the material is reacting [7].

1. Absorbers- Material which have strong affinity for microwaves.

2. Transmitters- Material which transmit all the incident microwave irradiations with minor heat losses.

3. Reflectors- It is the material which reflects all of the microwave radiations due to eddy current generation within the material.

4. Materials producing heat on interaction- The heating occurs due to one or combination of the following mechanisms [8]: rotation od dipole, resistive heating, dielectric heating and electromagnetic heating. And in an alternating field, the rotation of particles is 180° each time and the field got changes with time. Generally the frequency range of this field lies between 915 MHz to 2.45 GHz. Therefore the particle will re-align themselves 915×1010 time per second or 2.45 billion times - depending on the frequency available.

So it can be playing a cruicial role for creating the internal friction which, in turn, heats up the material. So the advantages of microwave heating over the conventional heating include quick and uniform heating, higher yielding and lower energy consumption. In these times with the areas of iron ore use increasing, it is very important to use raw materials that not only increase the productivity of plant but also produce a better quality iron. Blast furnace raw materials have changed in character greatly in the last 2 or 3decades.

Formerly, they used to be raw iron ores, raw lime stone and coke. There are now being increasingly being replaced by pre fluxed sinter and pellets and liquid and gaseous fuels. Few examples of revolutionary changes brought to the present day iron making industries by use of pellets are listed below:- • Essar steel has set up a palletizing plant in Vizag of 2.2 MT capacity which uses iron ore from Bailadila. By this the productivity of Essar steel, Surat, Hazir, has increased from 0.7 MT to1.1MT.

1. Trial at T T steel, Jamshedpur have shown that by using pellets a coke rate reduction of 20 Kg/tonne and increased in productivity by 0.7% has been achieved.
2. s a result of introduction of pellets in the Ijamudin blast furnace a coke rate decreases of 20-22 kg/tonne was expected.
3. Jindal has also set up a palletizing plant at Belgaon for pellets to be charged in COREX process.
4. 6.75 MT palletizing plant has been set up at Kudremukh, Karnataka which export pellets to various countries.

In our project we aim at studying the effect of size of iron ore pellet on its reduction kinetics and in the course of our experimentation we aim at arriving at particular pellet size that gives both increases in productivity and decrease coke rate.

5. History of Iron Production

Iron production is one of the most important discovery and it come to 2000 BC in ASIA. Iron became alternative instead of the bronze in the war arsenal The most crucial feature of the iron with mixture of carbon in comparison with bronze are harder, durable and it can be obtain sharper edge rather than previous metals. After some time, this invention became the basis of the civilization for other continents too. In the nature, it is not feasible to find pure iron itself, because it is highly reactive with oxygen. Generally, we can find out iron as iron oxides with other material like: sulphur, silicon, and phosphor, manganese etc. Iron oxide with other materials that exists in the nature, calls iron ore or iron stone, found in the form of magnetite, hematite, limonite or siderite, goethite. Crude iron ore need to be well pre-treated before fed into the DRI (Direct Reduced Iron) plant[9].

6. Direct Reduced Iron Production inIndia

Demand of sponge iron for the manufacture of different varieties of steel is increasing day by day and now days; new solid reluctant based sponge iron plans are being commissioned. A year-wise production of sponge iron for both India and the world is given in Table 1. According to recent analysis, it is observe that the beginning with a small production was 0.79 Mt in 1970; the production of world sponge iron went up to nearly 71 Mt in 2010, as shown in table-1., it is clear from the table that the world sponge iron production has been increased yearly.

At present scenario, India is the 5th largest producer (volume wise) of crude steel in the world and is expected to become the 2nd largest producer of crude steel in the world by 2015-16, and also India maintained its lead position as the world's largest producer of direct reduced iron (DRI) or sponge iron with around 22 Mt productions in 2009-10. In the year 2002 with the production capacity of 5.48 Mt, India became the largest producer of sponge iron in the world and still it has retained its 1st position in the world sponge iron production. In 2007-08 the contribution of coal based sponge iron units is around 14.14 Mt out of 20Mt and that of has based units are 5.84 Mt. This type of large difference is due to shortage of natural gases and huge availability of non-coking coal in India. This unparalleled growth of direct reduced iron (DRI) industries is driven by increasing demand of steel in India and as well as in the world. Approximately 16Mt per year of the aim of DRI capacity are currently under construction according to analysis by Midrex and other.

II. LITERATURE REVIEW

1. Iron Ore

Generally, availability of iron ore is in the form of rocks and minerals from which metallic iron can be extracted. The ores which are usually rich in iron oxides and they available in colours like dark grey, bright yellow, deep purple, to rusty red. Majorly rock types which are mined for the production of metallic iron are hematite, pisolitic goethite/limonite, which provide a high grade ore, and banded magnetite rich metasomatite; metasedimentary ironstone to a much lesser degree rocks which rich in siderite and rocks rich in chamosite which provide a low-grade ore. Ores containing high quantities of hematite or magnetite (> 60% iron) are known as natural ore or direct shipping ore and they can be fed directly into iron making blast furnaces for producing good quality Fe [11]. Basically the iron ore is the raw material for making the pig iron, which is one of the main raw materials to make steel. About 98% of the mined iron ore is used to making of steel. Definitely, it has been argued that iron ore is more integral to the global economy than other commodity except perhaps the oil.

2. Iron Minerals

Approximately 300 minerals contain some iron but only a few of them are considered to be important iron ore minerals they are frequently used for production of Fe [12]. The major iron ore minerals are tabulated below.

Table 1 Major iron bearing minerals and their therortical iron content

Mineral	Chemical formula	Theoretical iron content%
Hematite	Fe2O3	70
Magnetite	Fe3O4	72
Martite	α-Fe2O3	70
Geothite	FeO(OH)	63
Siderite	FeCO3	48
Chamosite	(Mg,Fe,Al)$_6$(Si,Al)4O14(OH)8	45
Pyrite	FeS2	47
Limonite	FeO(OH).n(H2O)	63
Lepidocrocite	γ-Fe2O3.H2O	60
Greenalite	Fe3Si2O5(OH)4	45
Ilmenite	FeTiO3	37

Hematite (Fe2O3) its name origin from the Greek word haimatite "bloodlike'" from allusion to dramatic red colour of the powder but it can be brown, reddish brown, balck or silver colour. Hematite is also known as "natural ore", name which indicates to very early years in field of mining, when some hematite ores contained up to 66% iron could be fed directly into iron-making blast furnaces [13]. Hematite deposits are mostly sedimentary in origin, such as the banded iron formations (BIFs).

These BIFs are consisted of alternate layers of chart (a finely grained re- crystallised quartz), hematite and magnetite. Their formation is not fully understood, though it is well known that they are formed by the chemical precipitation of iron from shallow seas about 1.8-2.6 billion years ago, during the Precambrian period [14]. On the Mohs scale, its hardness is given as 56. Hematite is brittle in nature, but it is harder than pure iron. It has the rhombododecahedral crystal structure and this shows an irregular or uneven fracture. At the lower temperatures hematite shows the antiferromagnetic nature. But at higher temperatures it shoes paramagnetism. There are few varieties of hematite viz. Hematite rose – its crystal is arranged in the shape of a rose flower. Kidney ore – it gives the appearance of kidney like masses. Tiger iron

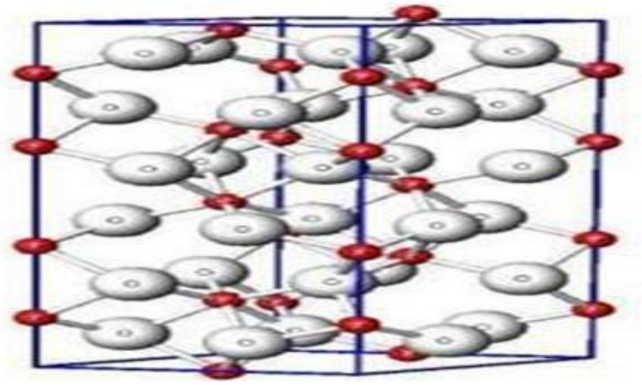

Fig. 2 Crystal structure of Hematite.

Magnetite (Fe3O4) is a naturally occurring metallic mineral which can be occasionally found in ample amount to be an ore of iron. It contains both iron oxide (FeO) and iron oxide (Fe2O3). High grade magnetite ore normally contains more than 60% iron with some impurities such as silica, alumina and phosphorus. Magnetite is beneficiated by crushing and then separating the magnetite from the gangue minerals with a magnet. According to the IUPAC nomenclature, it is named as iron (II, III) oxide. But, commonly we name this as ferrous ferric oxide. The iron ore, Magnetite got its name because it is magnet. Magnetite is black in colour. Its streak is also black and has a metallic to dull luster. On the Mohs scale its hardness is 5.5–6.5.Magnetite has octahedral crystal structure while rhombododecahedron types of structure is seen rarely. It shows an irregular, uneven fracture.

3.Martite (Fe2O3): It is a secondary hematite formed by chemical replacement of magnetite produced at in the depth and high pressure. The replacement proceeds from the outer edge towards the centre of the magnetite and grains generally along crystallographic planes[15].

4.Limonite [FeO(OH)]: Limonite is the names commonly given to hydrous iron oxides that mineralogicaly that have composition of different mixtures of minerals, like goethite or lepidocrocite. The chemical formula for goethite is HFeO2 and the same for lepidocrocite is FeO(OH). The mineral, Goethite contains 62.85% iron, 27.01% oxygen, and 10.14% water; has a specific gravity around 3.6 – 4.0, is commonly brown or yellow to nearly black in colour, and is compact to earthy and ocherous. In non-technical phrasing, the term ―limonite‖ is used to denote unidentified oxides with variable moisture content due to absorbed or capillary water action. Siderite (FeCO3) ore constitutes only a small proportion of the total world iron ore reserves. When pure, it contains 48.3% iron, but it is easily decomposed by heat to hematite with 70% iron. Chamosite [(Mg, Fe, Al)6(Si,Al)4O14(OH)8] ore occurs, together with limonite and siderite, in the relatively low concentration. This ore generally contains some sulphur, phosphorus and other minerals such as quartz and calcite[14].

Ilmenite have chemical composition of FeTiO3, corresponding to 36.80% iron, 31.57% titanium, and 31.63% oxygen. It is normally considered as an iron titanate. The ore, Ilmenite is often associated in small amounts with magnetite. Rather than as an iron ore , it is generally mined as a source of titanium. Well, iron may be recovered as a byproduct.

5.Requirements of Iron Ore

5.1 Physical Requirements

Strength and granulometry are the two important physical requirements in field of iron production. The iron ore should be hard and should have high strength. The tumbler strength of the ore should be minimum about 90%. Depending on the basis of reducibility, closely calibrated ore size is the range of 5 to 20 mm generallyused.

5.2 MetallurgicalRequirements

The important metallurgical requirement is that the ore should be thermally stable, reducible and have a low tendency for sticking and disintegration during the heating and reduction.

5.3 ChemicalRequirements

No other major chemical change takes place in direct reduction accepts removing the oxygen. There are the various gangue materials in sponge iron originating from iron ore, namely-silica, Alumina, sulphur and phosphorous contents adversely affect the economics in subsequent steel making operation. Therefore, the ore should be high in iron content and low in gangue.

5.4 Physical Specification

a. Size - 5-20mm
b. Oversize - < 5 %max
c. Undersize - < 5%max
d. Tumbler Index - +90
e. Silica - <3%
f. Contamination - <5%

6.Microwave and its Applications

Microwaves are the wave of electromagnetic energy and its range lies between radio wave and infrared. Wavelength (λ) of Microwaves has a range of 1m to 1mm with a frequency of about 300 MHz to 300 GH. Because of lower frequency band are congested and demand in research is continue increase that‘s why we choose microwave. The propagation of the microwave takes place in the space wave in view of the high gain and directivity in the form of a beam and is similar to that of light.The frequencies allocated for microwave heating are designated as ISM "industrial, Scientific and medical" [16]. ISM frequency bands are listed in Table 2.2. The most commonly used frequencies for heating purposes are 915 MHz and 2.45 GHz, which correspond to wave lengths of 32.78 and 12.24 cm. These frequencies were chosen by international agreement for minimizing the interference with communication systems. The longer wavelength of 915 MHz frequency is commonly used in industrial ovens where deeper penetration is required. The higher frequency 2.45 GHz is more practical for domestic ovens and is also used widely in industry as the wavelength of this frequency (12.2 cm) is more suitable for domestic ovens, where the cavity must be at least two wavelengths in two dimensions to work efficiently.

The word micro in microwave is not meant that a wavelength in the micrometer range; it actuallyindicates that the microwave is small as compared to the wave used in typical radio broadcasting technique, and they have shorter wavelengths. Boundaries between distant terahertz radiation, infrared radiation, microwaves, and ultrahigh frequency radio waves are quite arbitrary and are used variously between different fields of study and researches.The microwave also has a more technical and specific meaning in circuit and EM theory. Apparatus and techniques used can be described qualitatively as "microwave" when the frequencies used are high that wavelengths of signals are roughly the same as the dimensions of the equipment, thus the lumped element circuit theory is inaccurate.

Table 2. ISM Frequency Band

Designation	Band	Central frequency
UHF-Ultra high frequency	433.05-434.79MHz	433.92 MHz
UHF-Ultra high frequency	902-928 MHz	915MHz
UHF-Ultra high frequency	2400-2500MHz	2.450GHz
SHF-Super High Frequency	5725-5875MHz	5.8GHz
SHF-Super High Frequency	24-24.5GHz	24.125GHz
EHF-Extremely High Frequency	61-61.5GHz	61.25GHz
EHF-Extremely High Frequency	122-123GHz	122.2GHz
EHF-Extremely High Frequency	244-246GHz	245GHz

7.Microwave Sources

High-power microwave generation we used vacuum tubes. These devices are operated on basis of different principle, like low frequency vacuum tubes using the ballistic motion of electrons in a vacuum under the pressure of controlling magnetic or electric fields, and include the magnetron (used in microwave ovens), gyrotron, Traveling Wave Tube (TWT) and klystron. All these devices are work under the density modulated mode, instead of the current modulated mode. Low power

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microwave sources are used in solid state devices such as gunn diodes, field effect transistors, tunnel diodes, and IMPATT diodes [17]. Low power sources are available as embeddable modules, bench top instruments rack mount instruments, and in card level formats.

8.Effects on Human Health

Microwaves do not have sufficient energy for chemically changed substances through ionization hence it is an example of non-ionizing radiation [18]. The term "radiation" refers to energy radiating from a source not to a radioactivity. It is not seen finally that the microwaves have significant adverse biological effects at low level. The various studies suggested that long term exposure may have a carcinogenic effect [19].The risks related to very high intensity exposure which may cause heating and burns like a heat source and it is not a unique property of microwaves. Some other effects of microwave, directly seen are low blood pressure, slow pulse rate, chronic excitation of the sympathetic nervous system (stress syndrome) and high blood pressure. It is also often includes headache, dizziness, severe eye pain, high sleeplessness, irritability with anxiety, severe pain in stomach, high tension in nervous system, inability to concentrate, severe hair loss, an increased rate of appendicitis, cataracts, some reproductive problems, and may lead to different kinds of cancer. Chronic symptoms are succeeded by crises to the adrenal exhaustion, the ischemic heart disease such as blockage of coronary arteries and heart attack all these are the common effect which is found on human being.

III. EXPERIMENTAL DETAILS

1. Materials

For the reduction purpose, firstly a good quality iron ore powder is to be procured. Hematite (Fe_2O_3) powder contains high iron (Fe) content. So, a commercial grade hematite powder (Fe_2O_3) was used for the investigation of microwave reduction. The chemical composition of the iron powder used in the present study is given in table 3.1.

Table 3 Chemical composition of iron powder used in the present study

Element Present	Weight Percent
Fe2O3	98.5
Al2O3	0.21
SiO2	0.57
S	0.1
TiO2	0.12
P2O5	0.04

For reduction purpose a commercial grade activated charcoal powder was used. The typical phsico- chemical properties of the activated charcoal powder used in the present investigation is shown in table 3.

Table 3. Physico-chemical properties of the activated charcoal used in the present study

Element present	Weight percent
Fixed Carbon	77.70%
Ash	4.00%
pH	6.5
Fe	100 ppm
Ch	1000 ppm
Na	800 ppm
Acid Soluble	2.00%
Water Soluble	1.00%

ExperimentalSetup

A microwave oven (900W, 2450 MHz, LG, Model MC 8080 PRR) used for kitchen appliances was used for the studies. The photograph of the microwave oven modified as a reactor is shown in fig.3.

Fig 3.The photograph of the microwave reactor in the present investigation.

As shown, the microwave reactor is made of stainless with a rectangular cross section of 35 x 22 x 20 cm. The microwave source and controls are fixed at the right side of the reactor. The reactor has a provision to control the microwave power at three different levels. i.e. it is possible to set the microwave power at 400 watts, 750 watts and 900 watts respectively. The time of exposure of microwave can be adjusted from 0-99 minutes. The reaction crucible can be placed on a platform made to rotate during the process. The speed of rotation is fixed at 10 rpm. A provision is also given for the removal of exhaust gases. However there is no provision for temperature control. Thus an IR thermometer was used for measurement of temperatures. The details of the temperature measurements are explained in section 3.3.

2.Temperature Measurement andControl
The major challenge in microwave process is the measurement of temperature during microwave heating. Thermocouple wire cannot be used during operation as they interact with microwave and generate eddy current. Thus the temperature was measured by using Infrared thermometer at intermittent periods. The IR thermometer is capable of non – contact (Infrared) temperature measurement at the touch of a button. The built – in laser pointer increases target accuracy while the backlight LCD and handy push button combine for convenient, ergonomic operation. The Non – contact Infrared Thermometer can be used to measure the temperature of object's surface that is improper to be measured by traditional (contact) thermometer.

Fig.4 Infrared Thermometer used in the study

IV. RESULTS AND DISCUSSIONS

Interaction of Microwave with Reaction Mixtures and Heat Generation The reaction mixtures used in the present study consist of Fe2O3 and amorphous carbon (activated charcoal). Basically both are come into the category of subsector material i.e they absorb the microwave and generate heat energy. Fig. 4.1 shows the photograph of reaction mixtures generating high temperature while interacting with microwave in the present study.

Fig4.1 Photograph showing the temperature rise by the exposure of the reaction mixtures to microwave irradiation and flame generated by the microwave interaction.

Other researchers also carried out detailed investigations on microwave heating of ores and minerals [35]. The Liu Chunpeng et al. [36] had demonstrated that the temperature of hematite (Fe2O3) rises to 9800C by 4 miutes of microwave heating, while magnetite (Fe3O4) about 7800C in the same time According to previous researches information that the heating provided by the microwave can be stated as; Fe2O3 absorbs the Magnetic field of the microwave whereas the charcoal absorbs the electric (E) field. The heat generated by the loss of absorbed energy in the form of vibrations caused on the molecular level, by the dipole moment [37]. The microwave irradiation of the reaction mixtures generated a high temperature within a short period of time. However the major challenge in this process was measurement of temperature during microwave heating. Thermocouple wires cannot be used as they interact with microwave and generate eddy current. Thus the microwave was stopped at intermittent periods and the temperature was measured using an infrared thermometer. Fig. 4.2 shows the variation of temperature of the reaction mixture with time and its control during microwave reduction of iron ore.

V. CONCLUSION AND FUTURE SCOPE

Conclusion
1. Solid state reduction of hematite with activated carbon was successfully carried out using microwave technique.
2. The reaction mixture of iron oxide and activated charcoal showed good observation characteristics of microwave heating.
3. The microwave radiation had raised the temperature of reaction mixture to 7760C in 20 minutes of irradiation thus the heating rate is rapid in comparison to conventional heating.
4. 50 percent weight loss was observed when the reaction mixture exposed to microwave for about 55 minutes. The maximum recovery of iron ore was at 900 Watt and at 55 minutes. 720 W and 540 W showed lower wt.reduction.
5. With Increase in temperature for a pellet the percentage reduction increases with the increase in time.
6. Increasing the binder content leads to decrease in percent reduction of iron ore ball. Bentonite with 0.5 % concentration showed maximum reduction.
7. The EDX and XRD results conformed the formation of α-iron from the reaction mixture. EDX analysis showed that Fe content in the reduced sample was 46.23 % bymass.
8. XRD also showed presence of unreacted hematite and magnetite and phase in the reaction mixture. The crystallite size of Fe phase is 3.404 nm and lattice parameter, a, is 2.897A°
9. The SEM morphological analysis showed spherical and angular shaped particle distribution in the range of 0.6 μm to 7 μm. The average particle size was 3.44 μm. Porous structure was shown in SEMimages.

Future scope
1. Reduction of iron ore fines and coal fines.
2. This experiment can be carried out with different oxides like martite, geothite, pyrite etc. The

temperature measurement of reaction mixture can be carried out using thermal analyser. Separation techniques can be studied for density based separation.

3. Research and development will continue on direct iron and steelmaking concepts.

4.Direct reduction operations that utilize fine ores will increase due to the lower cost of the ore feed. Steelmakers without access to low-cost gas or ores will invest in DRI plants to control the cost of their iron units.

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