

**AN ATTEMPT AT OPTIMIZING THE FLOW CHARACTERISTICS
OF BLAST FURNACE SLAG BY INVESTIGATION OF SYNTHETIC
SLAGS PREPARED IN THE LABORATORY USING PURE OXIDES**

A THESIS SUBMITTED IN PARTIAL FULFILLMENT

OF THE REQUIREMENT FOR THE DEGREE OF

**Bachelor of Technology
in
Metallurgical and Materials Engineering**

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2011



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CERTIFICATE

This is to certify that the thesis entitled, "**AN ATTEMPT AT OPTIMIZING THE FLOW CHARACTERISTICS OF BLAST FURNACE SLAG BY INVESTIGATION OF SYNTHETIC SLAGS PREPARED IN THE LABORATORY USING PURE OXIDES**" submitted by **Amlan Prateek Panda (107MM002), Sridhar Kumar Panigrahi (107MM028), Abhimanyu Pabba (107MM027), Kundan Raj (107MM033), Sidharth Sood (107MM014)** in partial fulfillments for the requirements for the award of **Bachelor of Technology Degree in Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ABSTRACT

In the cohesive zone the ore layer becomes compacted due to softening-melting and the gas passes predominantly through the sandwiched coke layer (coke slits). Minimizing the size and lowering the position of the cohesive zone will improve productivity and decrease coke rate. This work is designed at arriving at a slag composition through actual experimentations which will ensure the lowering of the cohesive zone of the blast furnace with simultaneous decrease in the difference between the softening temperature (ST) and flow temperature (FT) of the slag. Slag samples from SAIL, Rourkela were brought and their flow characteristics were measured under the heating microscope. With the same composition as obtained from the plant, a synthetic slag was prepared in the laboratory. It was melted to 1600 °C and then water quenched to form the glassy state. Flow characteristics for the same were also determined. In addition, all slag samples were sent to DISIR, Rajgangpur for slag analysis as well as to determine the mineralogical phases present in them. For this, they heated the slag samples to 1500 °C followed by slow cooling to convert them to crystalline form. Then, XRD analysis was done to determine the mineralogical phases. Finally, with relevant phase diagram study, we arrived at a composition which will ensure the lowering of cohesive zone. Moreover, flow characteristics data of synthetic slag was compared with that obtained from Blast Furnace slag.

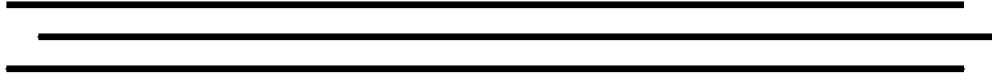
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CHAPTER 1



INTRODUCTION

1. INTRODUCTION

Slag is and has always been an integrated part of iron and steel making. It is so as 10% of total product in iron and steel making constitute of slag. In past, the quality of steel was decided by taking the slag quantity and quality in consideration. Now, the metallurgist and iron makers are looking forth to increase the quality and the economics of the steel.

Slag constitutes of the unwanted oxides, sulphides, nitrides, and phosphides. The slag composition must be controlled or else that may affect the quality of the products.

Slag formed must ensure

- That the impurities formed during the process must be removed
- It should enhance slag metal reaction
- To ensure less metal entrapment, better slag- metal separation should be maintained.

As the burden descends down the furnace, the quality of iron obtained is dependent on the formation of the slag and its transformations. Various c constituents affect the slag nature like silica and alumina increases the viscosity whereas it is reduced by calcium oxide. Melting characteristics greatly influence the productivity. Formerly slag of high iron content is produced but in the presence of CaO MgO and other compositions of flux changes the properties of slag.

Cohesive zone, shape and position influence the efficiency and productivity of the process. Cohesive zone is the region having low permeability and the gas flow takes place through the carbon slits. Phenomenon like softening and melting of oxides and metal iron phase take place simultaneously. For better slag-metal separation the slag formed should be of low fusion temperature. With change in the composition liquidus temperature also varies. Therefore a

detailed study of the liquidus temperature should be made for each slag since different compositions lead to different circumstances.

Many nomenclatures for the liquidus temperature have been reported. According to Osborn^[1] and Snow^[2], liquidus temperatures are the temperature when the first crystal forms as the cooling down the melt. Ohno^[3] stated as the temperature during heating, when last crystal disappears. As per the German Industrial Standards 51730, liquidus temperature as the hemispherical temperature; the temperature when the small quantity of the slag takes the shape of the hemisphere on heating. Heating microscope is used to measure this temperature.

The fusion behavior is characterized in four characteristic temperatures, (i) initial deformation temperature (IDT), (ii) the softening temperature (ST), (iii) the hemispherical temperature (HT) and (iv) the flow temperature (FT) describing the mobility of the liquid, which shows surface stickiness, plastic distortion, slow flow and liquid mobility respectively. Fusion behavior of the slag defines the smelting and refining process. For the movement of slag-state materials, the IDT indicating the surface thickness of the material is important. Similarly the start of plastic distortion is indicated by ST and HT represents the fusion or the liquid temperature of the material has a important role in the mass and heat transfer. Cohesive zone in the blast furnace would be lower if it has a high softening and fusion temperature of the iron bearing materials. Due to this the distance travelled by the molten metal in the droplet form is decreased which in turn reduces the silicon pickup.

The characteristic behavior of slag and the operating conditions is determined by the composition and various constituents present in the slag. Hence an attempt has been made in

lowering of the cohesive zone which in other words mean decreasing the difference between the HT and FT of the slag.

1.1 BLAST FURNACE

The blast furnace is a large steel stack, lined with refractory brick, where the charge consisting of coke, iron ore and limestone are charged from the top and preheated air is blown from the bottom. The blast furnace is utilized to physically convert and chemically reduce the iron oxide into liquid iron known as “hot metal”. The time taken by raw materials to move to the bottom of the furnace where they take 6 to 8 hours to finally give slag and liquid iron as the final product. The liquid products are drained at regular intervals from the furnace. After going through numerous chemical reactions the hot air reaches the top in about 6 to 8 seconds. Baring the short stops for maintenance once started the blast furnace will continuously ranging from four to ten years.

1.2 BLAST FURNACE PROCESS

Iron oxide comes to the blast furnace plant as raw ore, pellets and/or sinter. After removing the raw ore from the earth’s crust, it is sized into pieces which range from 0.5 to 1.6 inches. The ore can be either Magnetite (Fe_3O_4) or Hematite (Fe_2O_3), where Fe ranges between 50% to 70%. In order to increase the iron content, purification processes are undertaken for low iron containing ores. Low iron content ore are used to produce pellets. Gangue is removed by crushing the ore in powdered form and then purifying them. Small, marble-sized pellets containing 60 to 70% iron are produced by rolling the remaining iron rich powder into balls and firing them in a furnace. Sinter produced contains small amount of iron. Sintering is a process of agglomeration of fine particles of raw materials by placing the mixed raw materials on the sintering strands and

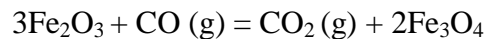
igniting them by gas fire and the fines get fused by the heat to form large size blocks varying from 0.6 to 2.0 inches. The iron ore, pellet and sinter form the molten metal and the rest, basically the impurities join the slag.

Coke is produced from the coal. This is done by crushing and grinding of the coal and then charging it into the furnace. Cooking of the coal takes place in the furnace and the volatile matters are removed, and the product obtained is called coke. Screening of the cooled coke is done ranging from 1 to 4 inches. The coke constitutes of 90 to 93 % carbon, ash and sulfur and the strength of the coke is more than that of coal.

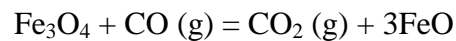
Lastly the limestone constitutes the raw material for the iron making. Explosives are used to break the limestone from the earth. Then limestone are crushed and screened to a size ranging from 0.5 to 1.5 inches, which becomes the flux for the blast furnace.

The reduction of iron ore, pellet and sinter takes place in the sinter by a series of the chemical reactions. The reactions are as follows:

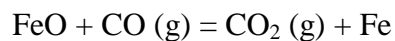
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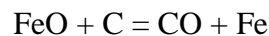
- Starts at 1100° F



- Starts at 1300° F

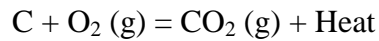


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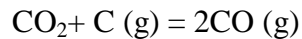


The above reactions are simultaneously followed by the softening and melting of iron which finally trickles down as the liquid iron through the layers of coke present at the bottom of the blast furnace.

The coke moves to the bottom, to the level where the preheated air enters the furnace. By this hot blast the coke is ignited and it immediately reacts to produce heat, as shown from the reaction below:



As the reaction is taking place in excess of carbon at a high temperature, CO_2 is reduced to CO by the reaction given below:



The product obtained from the above reaction; carbon monoxide is used to reduce the iron ore.

The limestone remains a solid as it moves down the blast furnace where the first reaction takes place, as shown:



It is an endothermic reaction, its starts at about 1600°F. The CaO formed removes sulfur from the iron. The sulfur is removed by the undersigned reaction:



The CaS joins the slag. The slag comprises of Alumina (Al_2O_3), Silica (SiO_2), Magnesia (MgO) or Calcia (CaO) that entered the furnace during charging. Then the slag which is in the liquid state trickles down the furnace across the coke layer and floats over the molten iron.

Hot dirty gases are another by-product of the iron making. These gases leave the furnace from the top and are then taken for gas cleaning. Gas cleaning equipments remove dust particles .the heat left in the gases have considerable amount of energy, so they are used as fuel in the hot blast stoves and are used for preheating the air entering the furnace also known as hot blast. The unburnt gases are sent to the boiler to produce steam which rotates a turbo blower which in-turn generates cold blast that flows to the stoves.

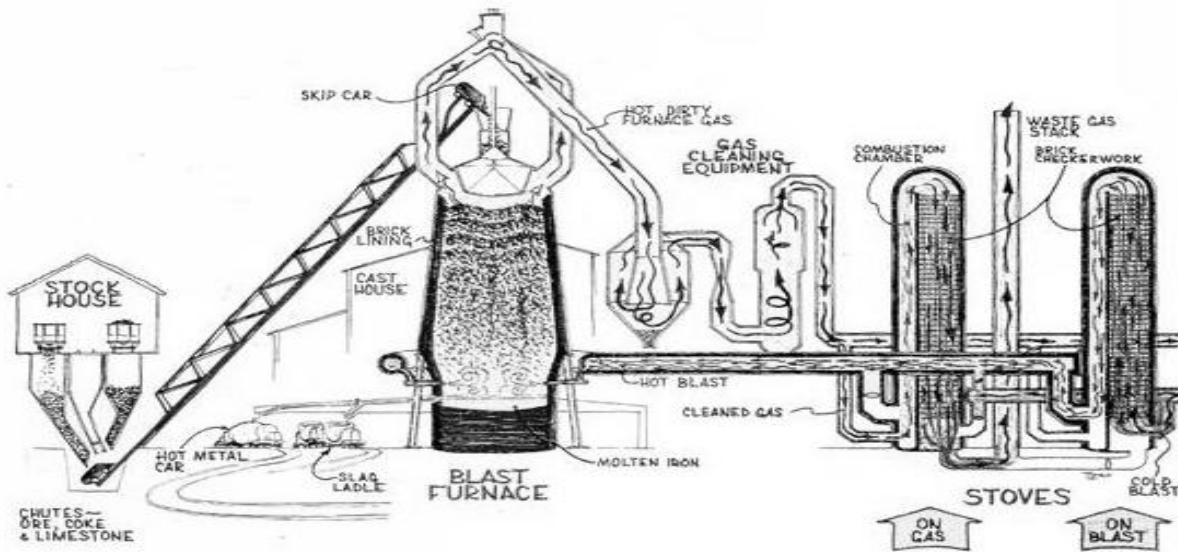


Fig. 1-A schematic diagram showing Blast Furnace Process

To summarize; the blast furnace is a counter-current process where solid moves down and gases moves up. General hot metal chemistry follows: ^[5]

Iron (Fe)	= 93.50-95.00%
Sulfur (S)	= 0.025-0.050%
Silicon (Si)	= 0.30-0.90%
Phosphorous (P)	= 0.03-0.09%

Manganese (Mn)	= 0.55-0.75%
Titanium (Ti)	= 0.02-0.06%
Carbon (C)	= 4.10-4.4%

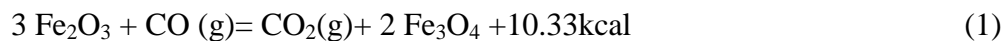
1.3 BLAST FURNACE REACTIONS

In the iron blast furnace mainly reduction and smelting is done. The charge material primarily comprises of iron ore, flux, and coke. By blowing preheated air into the coke bed from the bottom of the furnace, the reducing gas carbon monoxide and the heat for the smelting of the ore are produced. The slag and metal are collected as two separate layers at the bottom of the blast furnace. Specific thermal requirements must be satisfied to allow the endothermic reactions to take place and to maintain the products to their final temperatures. The blast furnace is divided into three zones, to study of the physical and chemical reactions taking place in them. The zones are as shown below:

- The upper or pre-heating zone.
- The middle or indirect reduction or isothermal zone.
- The lower or direct reduction zone.

1.3.1 REACTIONS IN THE UPPER ZONE

The reduction reactions of iron oxides are of main concern in the blast furnace. The product layer formation is well known during the iron ore reduction. More the driving force of reduction, greater is the chemical reaction rate and product layer formation is more pronounced.





The iron oxides undergoing purification reactions also start to soften and finally melt to trickle in form of liquid iron to the bottom through the coke. The reaction below states the carbon deposition by carbon monoxide.



The deposition takes place in temperature range of 440-600° C, the iron and its oxides present catalyses the reaction. It is also possible for carbon monoxide to reduce steam in the upper zone of the furnace



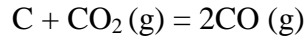
At a low temperature of about 400°C, carbonates other than calcium decompose.

1.3.2 REACTIONS IN THE MIDDLE ZONE

This is the medium temperature zone, with temperatures between 800 to 1000°C. Reduction of wustite is (equation 3) take place in this zone. The ratio of Ca/CO₂ is 2.3 which exhibit equilibrium with Fe-FeO (equation 3). The indirect reduction will be more if the height of this zone is large since there is longer gas/solid contact time. As is the case with reducibility of the ore.

1.3.3 REACTIONS IN THE LOWER ZONE

The temperature of this zone is 900-1000° C. Different chemical and physical processes takes place in this zone. Unreduced iron oxide moves into this zone as calcium ferrites or fayalite, in the primary slag. The direct reduction of iron oxide takes place above 1000 ° C according to:



The Si and Ti reduction require high temperatures, while the oxides of Mg, Ca and Al are highly stable such that they are reduced to a negligible amount. Reduction of Mn from its monoxide is difficult and takes place at very high temperature. Cr and V behaves in similar manner as Mn.



Phosphorus is present as tri- or tetra- phosphate of Calcium.



1.4 DIFFENT ZONES OF BLAST FURNACE

In the late seventies the data collected from the dissection of numerous quenched blast furnaces in Japan lead to establish significance of melting and softening features of the blast furnace operation. By providing detailed account of physical and chemical phenomena taking place inside the furnace helped in revolutionizing the understanding of the blast furnace process. It was found that internal state of furnace comprises of five distinct but interrelated zones. The zones are given below:

- Granular/isothermal zone
- Cohesive zone,
- Active coke zone,
- Stagnant coke zone and
- Hearth.

The most important achievement of the above studies was finding the existence of cohesive zone between the temperature range (1000 to 1500°C).

The blast furnace productivity, hot metal quality, fuel consumption, stability and lining life depends on the cohesive zone shape and location. The pattern of cohesive zone was found out to be a function of iron bearing material properties. Thus the softening and melting properties of the iron bearing materials were considered to be important.

1.4.1 COHESIVE ZONE

The region of softening and melting of the charge materials takes place is stated as the cohesive zone. Cohesive zone is the region where maximum solid volume disappears driving the solid flow. The performance and efficiency of the blast furnace is decided by the two factors which are position and thickness of the cohesive zone. The thickness and position of cohesive zone affects the following things:

- Gas permeability
- Extent of indirect reduction
- Si content of the pig iron

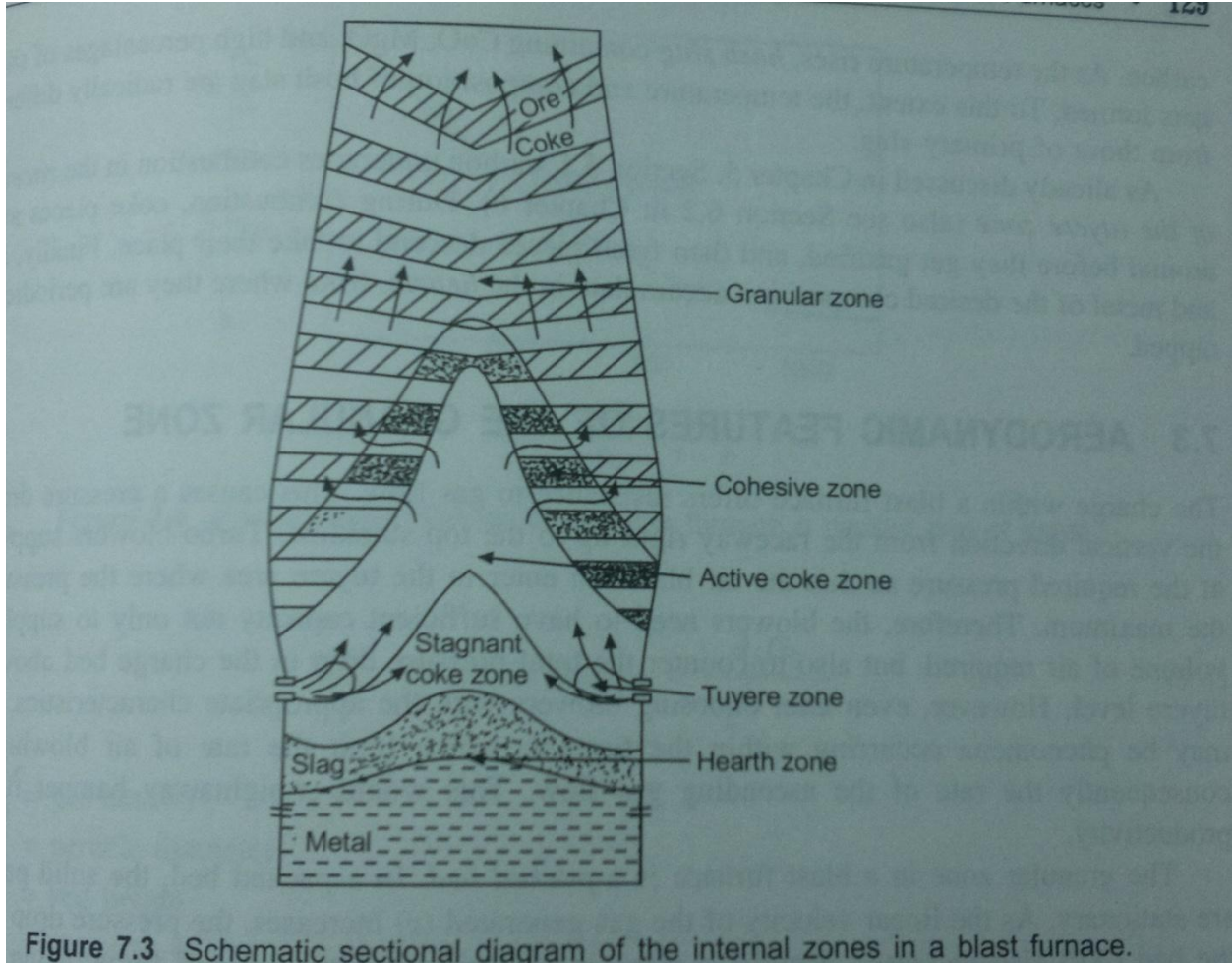


Fig. 2-Cohesive Zone

1.4.1.1 GAS PERMEABILITY

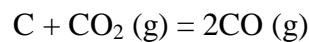
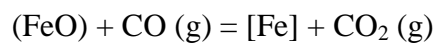
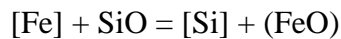
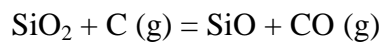
Cohesive zone thickness is directly related to gas permeability. Cohesive zone constitute of alternative impermeable, semi fluid layers resisting the flow of ascending gases. More and more gases pass through if the thickness of cohesive zone is less. The wind volume is related to the viscosity, length of the coke slit and the band volume of the melt.

1.4.1.2 EXTENT OF INDIRECT REDUCTION

Extent of indirect reduction depends on the place of the cohesive zone. Granular zone increases if the zone is location deeper into the furnace; hence the descending materials stay longer in the granular zone. Thus rising carbon monoxide is better utilized to reduce the wustite indirectly. And by lowering of the cohesive zone, the coke consumption for 1 tonne of pig iron gets highly reduced.

1.4.1.3 Si CONTENT OF PIG IRON

Si gets into the metal when SiO_2 reduction takes place. SiO_2 reduces to SiO and finally to SiO . Silicon Oxide is reduces in bosh or dripping zone. Hence by lowering the location of cohesive zone in blast furnace, the Si content in the pig iron is reduced as Silicon Oxide has got less chance to reduce.

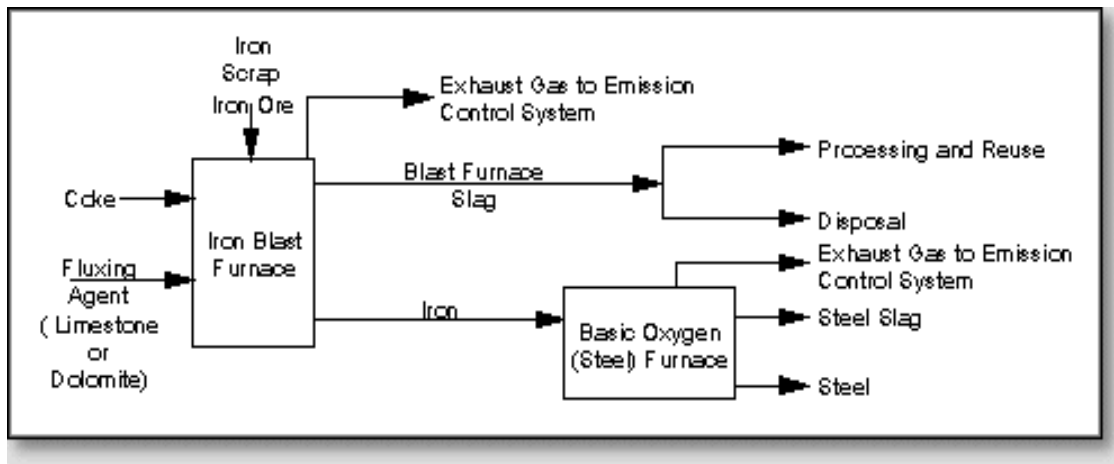


1.5 BLAST FURNACE SLAG

1.5.1 ORIGIN

In the production of iron, iron ore, fluxes (limestone and/ or dolomite) and scrap are dumped into the furnace accompanied with coke for fuel. CO, reduces the iron ore to produce molten iron, is generated by the combustion of coke. This molten iron is casted into iron and used for steel production.

Blast furnace slag is a nonmetallic co product produced in the process. Primary constituents of blast furnace slag are silicates, aluminosilicates, and calcium-alumina silicates. The molten slag, which absorbs sulphur from the charge material, constituting of 20 percent by mass of iron produced. **Fig. 3** shows a schematic of blast furnace operation which represents the blast furnace feed stocks and the production of blast furnace co products (iron and slag).^[8]



**Fig. 3-Schematic of blast furnace operation
and blast furnace slag production**

1.5.2 BLAST FURNACE SLAG STRUCTURE

The liquid slag consists of oxides of silicon and other elements which are homogenous in nature and having electrical properties and consisting of simple and/or complex ions. Crystal structure analysis of solid silica depicts that the silicon ion is located at the centre of a tetrahedron which is bounded by 4 oxygen atoms, one at each corner. The network is continuous and in three dimensions where each oxygen atom is bonded to two silicon atoms. Since these tetrahedra can share only corners so that when each corner oxygen atom is collective, the product formed will have a general formula of SiO_2 . Si atom has 4 valences.

Symmetry is obtained in the structure of tetrahedral arrangement of the silicon and oxygen atoms. In the molten state the distorted structure is observed but all most all the corners are shared. These are obvious from **Fig. 4**.

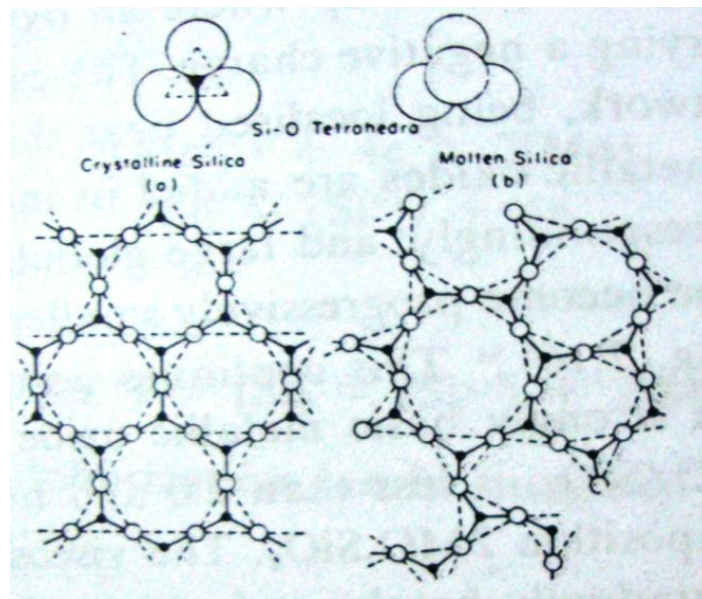


Fig. 4- Diagram of the silicate tetrahedron, molten and crystalline silica. Oxygen is represented by white and silicon atoms by black.

The (SiO_4) is having oxygen at the four corners and silicon at the centre. The attraction between silicon and oxygen is the driving force for the breakdown of silicon-oxygen network, which depends on their valancies and ionic radii. ^[9]

1.5.3 SLAG COMPOSITION

20 % of the total mass in iron production is formed into slag. Different slags are a result of different cooling methods applied. Basicity ratio in a range of 1.25 to 1.55 is a result of calcium and magnesium content. The slag chemistry of overall components reported as equivalent to Calcium oxide plus magnesium oxide divided by silica, is given below:

- CaO - 38%
- SiO_2 - 36%
- MgO - 10%

Minor elements are as follows:

- Iron Oxide (FeO & Fe_2O_3) - <1%
- Sulfur(S) - 1%
- Alumina (Al_2O_3) - 6-12%.

Table-I shows the chemical composition of blast furnace slag.

Table-I: General composition of blast furnace slag^[10]

Constituents	Percentage (%)							
	1949 ^a		1957 ^a		1968 ^a		1985 ^a	
	Mean	Range	Mean	Range	Mean	Range	Mean	Range
Calcium Oxide (CaO)	41	34-48	41	31-47	39	32-44	39	34-43
Silicon Dioxide (SiO ₂)	36	31-45	36	31-44	36	32-40	36	27-38
Aluminum Oxide (Al ₂ O ₃)	13	10-17	13	8-18	12	8-20	10	7-12
Magnesium Oxide (MgO)	7	1-15	7	2-16	11	2-19	12	7-15
Iron (FeO or Fe ₂ O ₃)	0.5	0.1-1.0	0.5	0.2-0.9	0.4	0.2-0.9	0.5	0.2-1.6
Manganese Oxide (MnO)	0.8	0.1-1.4	0.8	0.2-2.3	0.5	0.2-2.0	0.44	0.15- 0.76
Sulfur (S)	1.5	0.9-2.3	1.6	0.7-2.3	1.4	0.6-2.3	1.4	1.0-1.9
<p>a. Data source is the National Slag Association data: 1949 (22 sources); 1957 (29 sources); 1968 (30 sources) and 1985 (18 sources).</p>								

1.5.4 SLAG VISCOSITY

Blast furnace slag should be essentially homogenous liquid at the working temperatures in for being most effective and practicable. It should also have enough fluidity so that it can run out of the hearth unreservedly without any loss of manufacture time.

The quality of hot metal is greatly influenced by the development of slag and the mineralogical conversion that the slag undergoes during the descend of the burden inside the furnace. Viscosity of the slag is increased by the presence of components like silica and alumina whereas calcium oxide reduces it. The cohesive zone is determined by the melting zone therefore the melting and flow characteristics of slag play a very important role in the blast furnace productivity. Silica and alumina of ash, generated from the combustion of coke is assimilated as the slag trickles down the blast furnace. This process of trickling down depends on the viscosity of slag. This in turn is governed by the temperature and composition of melt.

As the flow pattern of molten slag has a major influence on gas permeability, heat transfer and the reduction of SiO_2 and FeO . Thus viscous behavior of slag plays an important role in furnace operation. Since iron ores reduction and also slag formation takes place in blast furnace, basicity and FeO percentage in the slag changes frequently from the initial sintered ore composition. They exhibit high CaO/SiO_2 (C/S) ratios of =1.4–1.6 and 5–20% FeO , which is comparable with C/S-1.2 and $\text{FeO} \leq 0.20$ mass% in tapped blast furnace slag. The main causes of slag formation problem are the relatively high C/S ratio of the slag in the bosh regions.

1.5.5 FLOW CHARACTERISTICS OF SLAG

The high temperature microscope is used for determining the flow characteristics of slag. It has got four characteristics temperatures:

- Initial deformation temperature (IDT)
- Softening temperature (ST)
- Hemispherical temperature (HT)
- Flow temperature (FT)

The followings are defined as per 51730, German Industrial Standards.

1.5.5.1 INITIAL DEFORMATION TEMPERATURE (IDT)

IDT is the temperature where primary rounding of the cube sample edges takes place. Here first indication of shape change appears. Basically IDT shows the surface stickiness of the slag sample.

1.5.5.2 SOFTENING TEMPERATURE (ST)

ST tells us about the onset of plastic deformation. It is the temperature where the specimen shrinks by one division. The outline of the sample changes during ST.

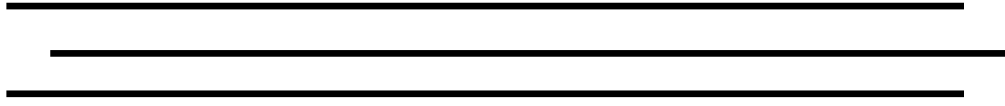
1.5.5.3 HEMISPHERICAL TEMPERATURE (HT)

When the specimen has melted down to hemispherical shape then the temperature responsible for the above change is referred as HT. Here half of the base length equals height of the specimen. Basically HT shows the sluggish flow of slag.

1.5.5.4 FLOW TEMPERATURE (FT)

Here the specimen liquidates and the height equals to 1/3 of the initial height. FT refers to the mobility of slag.

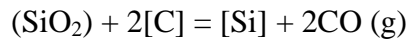
CHAPTER 2



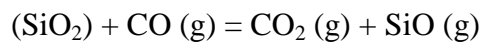
LITERATURE STUDY

1. B.Ozturk and R.J.Fruehan: “Kinetics of the Reaction of SiO (g) with carbon saturated Iron”: Metall.Trans.B. Vol.16B, 1985, p.121

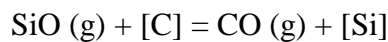
- This paper basically deals with the importance of Silicon transfer in the Iron blast furnace. The main reaction which takes place in blast furnace for the Silicon transfer is given below :-



- SiO reaction with carbon is the key factor in silicon transfer in Iron blast furnace. SiO is generated in the tuyere zone from the reaction of coke with its ash. When carbon saturated iron droplets pass through the furnace, it reacts with the SiO (g) to put Si into the metal.
- At the slag-gas interface CO reduced the SiO₂ to SiO (g)



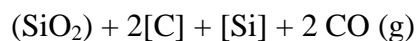
At metal-gas interface SiO reacts with dissolved carbon



CO₂ oxidizes carbon to form CO



So, the overall reaction taking place is summarized to,



- Rate controlling mechanism : the rate of reaction of SiO with C can be controlled by
 - Gas phase mass transfer of SiO (g) to the metal surface.

- Liquid phase diffusion of the reactant (carbon) to the surface or product (Silicon) into the metal.
- Chemical kinetics on the metal surface.
- Gas phase mass transfer of the product CO away from the surface.

2. B.Ozturk and R.J.Fruehan: “The Reaction of SiO (g) with liquid slags”: Metall.Trans.B. Vol.17B, 1986, p.397.

- This paper discusses the transfer of Silicon in the blast furnace. Earlier investigation on the rate of the transfer of Silicon and carbon saturated iron. Results based on the rates obtained from slag-metal reaction show that the rate of silicon transfer is too slow to obtain the actual silicon contents in the blast furnace. But now, alternative mechanism to the slag-metal reaction SiO gas is used.
- SiO generated travels in the blast furnace reacts with carbon saturated iron droplets and as well as with slag droplets.
- The kinetics of the reaction between SiO (g) and carbon dissolved in iron is controlled by gas phase mass transfer.
- Rate of transfer if silicon increases with decreasing diffusion distance.

3. Y.S.Lee, J.R.Kim, S.H.Yi and D.J.Min: “Viscous behavior of CaO-SiO₂-Al₂O₃-MgO-FeO slag”. Proceedings of VIII international conference on molten slag, fluxes and salts, The South African Institute of Mining and Metallurgy, 2004, p.225.

- Viscous behavior of blast furnace slag plays as important role that affects efficiency as the flow nature of molten slags have a major influence on heat transfer, gas permeability, SiO₂ and FeO reduction. The viscosity of CaO-SiO₂-Al₂O₃-MgO-FeO

slags were measured under $\text{CaO/SiO}_2 = 1.15-1.6$, 10-13 % Al_2O_3 , 5-2. % MgO , 5-20% FeO .

- Slag viscosity decreased with increasing slag basicity upto 1.3.
- Repolymerization of silicate network, where silicate structure changes from 30 to discrete anionic groups containing simple chains resulting in the decrease in basicity.
- Viscosity of slag at basicity of $\text{CaO/SiO}_2 = 1.5$ increases from about 2.5 to 10 dPa.s as FeO content decreases from 10 to 15 percent.

4. Paulo Nogueira, Richard Fruehen: “Blast Furnace Burden Softening and Melting Phenomena”- Part I Pellet Bulk Interaction Observation, Metallurgical and Materials Transactions B. Vol 35B, 2004, pp.829.

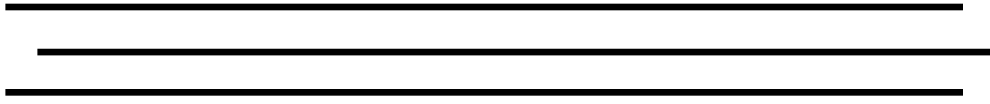
- An experimental technique was developed in which the deformation of pellets under load and temperature was studied while simultaneously X-ray pictures of the phenomena. When coupled with an accurate control of the reduction degree, this technique allowed the study of the phenomenon involved in the softening and melting of different fluxed pellets.
- There are indications that the initial deformation can be related to the reduction degree alone, with small interference from the amount and nature of the fluxing components. This suggests that the deformation of the pellet is the result of the softening of the metallic shell only. However, both dripping and meltdown were affected by the reduction degree and the chemical composition, indicating the competition between the effect of the metallic iron content and of the fluxing components.

- The softening and meltdown temperature from the displacement vs temperature curves were sufficiently close for both pellets reduced to 80 %. However, the X-ray pictures indicate that their behavior is different.

**5. P.F.Noguira and R.J Fruehen: “Blast Furnace Softening and Melting Phenomenon”
– Melting Onset in Acid and Basic Pellet:, IUSS-AIME Iron making Conference,
2002, pp.585.**

- In this experiment with only acid pellets, the samples showed that the metallic phase had coalesced, and a slag layer surrounded the exterior of the metallic phase. In addition, a remaining oxide core was observed. None of the experiments indicated the exudation of liquid from the basic pellets.
- The analysis of the samples of the mixed burden experiment showed an interaction between the acid and basic pellets along their interphase. The depth of interaction ranged from .5 to 1.3mm increasing with temperature and reduction degree.
- From the results of the single burden experiments with acid pellets as well as the considerations of the viscosity of these slags, it appears that the liquid slag from the acid pellet exuded and interacted with the periphery of the basic pellet.

CHAPTER 3



EXPERIMENTAL DETAILS

3. EXPERIMENTAL DETAILS

3.1 EXPERIMENTAL PROCEDURE

Firstly, blast furnace slag samples were collected from Rourkela Steel Plant. Slag analysis and charge history were also collected. **Table III** depicts the compositional **Table IV** depicts the charge history. One slag specimen was collected from every Blast Furnace (4 in total). Big chunks were broken to small pieces. The above process was done for all the samples. Planetary ball mill was used to ground the hammered samples. The mill was operated at 300 rpm for a total of I lakh rotation. Five zirconia balls were used in each vile. Four quarters were divided from a cone. The samples prepared were heated in heating microscope and subsequently the flow characteristics, depicted in **Table V**.

Table III: Chemical Composition of Slag as obtained from SAIL, Rourkela

Blast Furnace	Cast No.	Chemical Composition of Slag as obtained from SAIL, Rourkela.				
		SiO ₂ (%)	Al ₂ O ₃ (%)	CaO (%)	MgO (%)	Minor constituents(FeO,MnO,Na ₂ O,TiO ₂)
I	21306	33.5	20.8	31.9	10.4	3.4
II	18582	33.7	20.6	31.9	10.5	3.3
III	7046	33.9	20.6	31.6	10.6	3.3
IV	11833	33.8	20.6	31.8	10.5	3.3

Blast Furnace	Cast No.	Amount of coke charged (Kg)	Amount of sinter charged (Kg)	Amount of ore charged (Kg)	Amount of flux charged (Kg)	Hot blast temp. (° C)	Hot metal temp. (° C)	Blast volume (Nm ³ /hr)	Blast pressure (Kg/cm ²)
I	21306	808	1730	728	5	980	1428	81000	1.09
II	18582	723	1845	688	6	964	1430	90000	1.14
III	7046	780	1953	767	0	985	1422	88000	1.11
IV	11833	1201	2873	1154	5	985	1436	126000	1.49

Table IV: Details of the charge generating the slag as obtained from SAIL, Rourkela

Table V: Flow Characteristics of Blast Furnace slag samples as measured in the laboratory using the Hot-Stage microscope

Blast Furnace	Cast No.	Quarter No.	IDT (° C)	ST (° C)	HT (° C)	FT (° C)	Difference between HT and FT
I	21306	1	818	1212	1331	1372	41
		2	817	1217	1331	1373	42
		3	813	1217	1333	1367	34
II	18582	1	822	1198	1324	1367	43
		2	819	1193	1322	1408	86
		3	821	1200	1319	1393	74
III	7046	1	813	1218	1328	1398	70
		2	813	1215	1324	1383	59
		3	820	1216	1324	1398	74
IV	11833	1	814	1230	1326	1395	69
		2	822	1219	1323	1392	69
		3	820	1219	1323	1392	69

Secondly, hot air oven was used to heat pure oxides to remove the moistures. Mixture of oxides weighing 10 grams was made. In given proportion:

CaO - 34.5%

SiO₂ - 0.5%

Al₂O₃ - 21.5%

Fe₂O₃ - 33%

MgO - 10.5%

Similarly two more sets were prepared and the three sets altogether were mixed in the machine with agate as the medium. The above stated procedure was repeated nine more times, in order to make a mixture of oxides of combined weight of 300 grams. The mixture was heated to 1600°C and subsequently quenched to prepare synthetic slag. A photograph of the slag made is shown in **Fig. 5**. Again planetary ball mill was used to ground synthetic slag. Heating microscope was used to measure the flow characteristics of the synthetic slag. **Table VI** shows the composition of the synthetic slag.

Table VI: composition of synthetic slag

CaO	SiO ₂	Al ₂ O ₃	MgO	BASICITY
36.67	33.33	20	10	1.10021
35	35	20	10	1
33.16	36.842	20	10	0.90006
34.5	33	21.5	10	1.045455



Fig. 5-Synthetic slag prepared in the laboratory

Thirdly, all the samples sample was sent to DISIR, Rajgangpur for slag analysis where the composition of slag was also determined. **Table VII** depicts the compositional of the sample slags. Samples were hearted to 1500°C followed by slow cooling. XRD technique was used to determine the mineralogical phases present in the slag.

Table VII: Chemical Composition of Slag as obtained from DISIR, Rajgangpur

Blast Furnace	Cast No.	Chemical Composition of Slag as obtained from DISIR, Rajgangpur					
		SiO ₂ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	CaO (%)	MgO (%)	TiO ₂ (%)
I	21306	39.48	-	20.89	34.39	5.23	-
II	18582	34.97	0.73	19.31	34.39	9.8	0.6
III	7046	33.9	0.17	25.31	33.01	7.53	-
IV	11833	26.99	0.27	30.98	33.94	7.85	-

Finally, melting temperature and the related phase diagrams were found out. an attempt was made to vary the composition of slag in order to reduce the difference between HT and FT.

3.2 EXPERIMENTAL APPARATUS

3.2.1 HEATING MICROSCOPE

The temperature characteristics can be recorded by heating microscope. Leitz heating microscope picture is shown in **Fig. 6**. plan diagram of the apparatus is represented in **Fig. 7**. A thorough explanation of the instrument and its operating principles are presented elsewhere ^[33]. The sample was formed in cubes fo 3 mm, and heated in an electric furnace incorporated in the heating microscope.

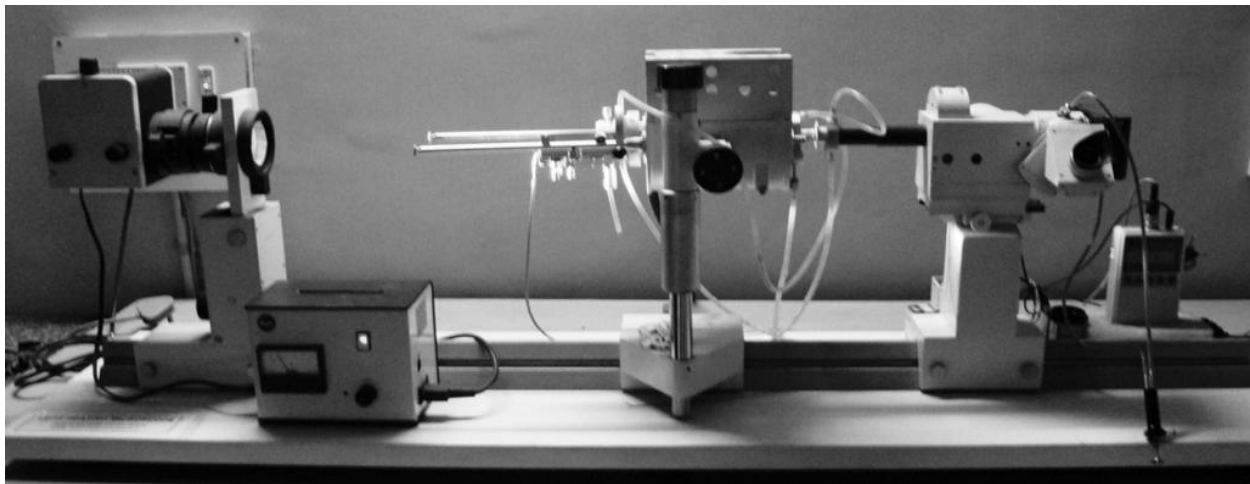


Fig. Symbolic view of Leitz heating microscope

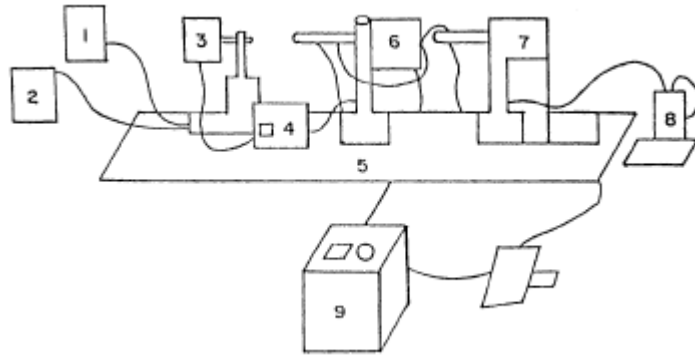


Fig. 7-A plan diagram of the heating microscope

1. Cooling water tank,
2. Cooling water recirculating tank,
3. Light source,
4. Regulating transformer for light source,
5. Optical bench,
6. High temperature electrical furnace with specimen carriage,
7. Observation and photo microscope,
8. Digital thermometer,
9. Regulating transformer for high temperature electrical furnace

The maximum temperature of upto $10,000^{\circ}\text{C}$ is reached by a rate of rise in temperature maintained at 100°C , after which it is kept at 40 to 60°C per minute. If the rate of heating is much higher than the mentioned rate, then this decreases the action of charge thus increases the characteristics temperature, especially HT & FT. Two samples are collected per slag for study. In case the difference between two is less than 50°C , then the mean of two readings is shown, but if it exceeds 50°C then experiments are carried out with fresh samples. ^[4]

3.2.2 PLANETARY BALL MILL

These mills are also referred as centrifugal mills and are used to grind samples into colloidal fineness by generating high grinding energy.

Fig.8. represents a four stationed planetary mill presented by Gilson Company. The samples are placed in one of the vile and numerous balls are added as shown. The vile is covered by the cover plate and then it is mounted in the machine. Once the viles are mounted and secured, the machine is functional.

The bowls are independent of the rotatable platform and the direction of rotation of the bowls is opposite to the direction of the rotatable platform. The motion resembles the teacup and saucer as seen in some of the amusement parks.

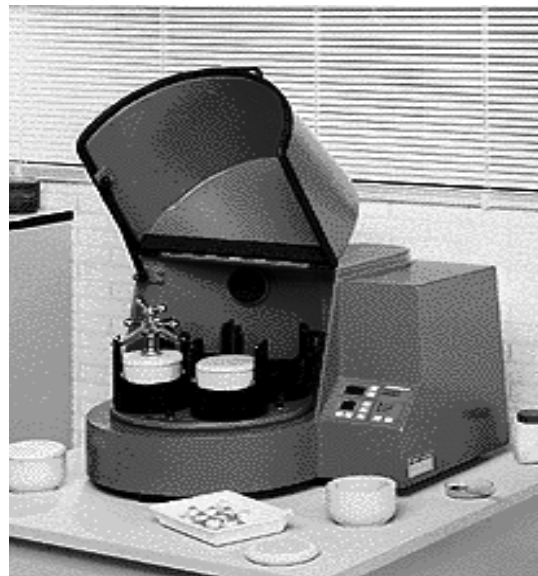


Fig. 8- A four station planetary mill

Due to alternate addition and subtraction of the centrifugal forces, the grinding balls rolls halfway in the vile and then thrown across the viles and then impacting the opposite walls at very

high speeds as shown in **Fig.9**. 20g acceleration is reached due to the planetary action and the time for the grinding reduces about 2/3 times than a simple centrifugal mill.

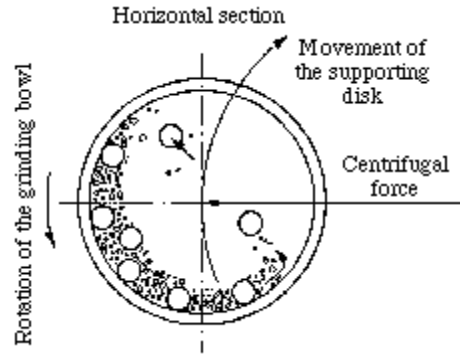



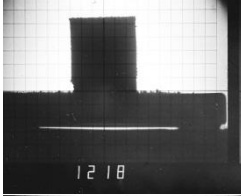
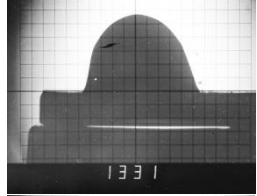
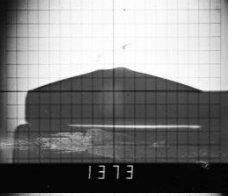
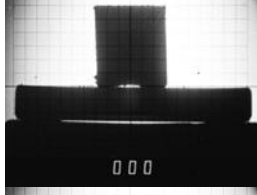




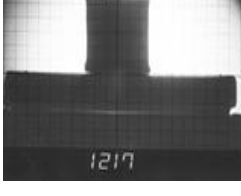


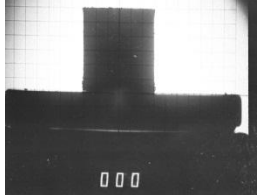
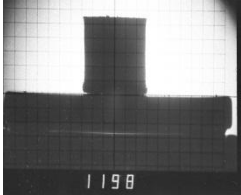
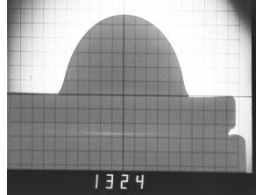
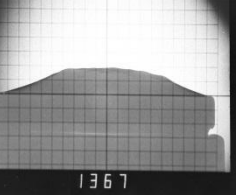



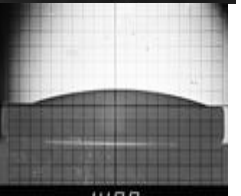
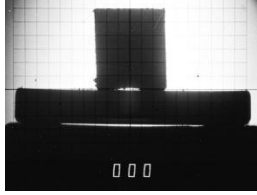



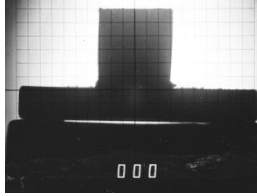

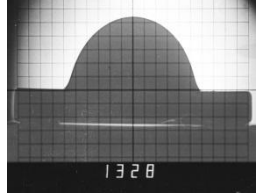
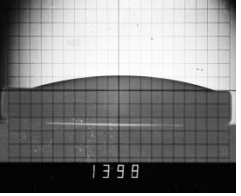
Fig. 9-Mechanism inside the planetary ball mill

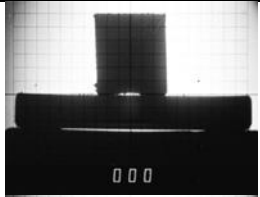
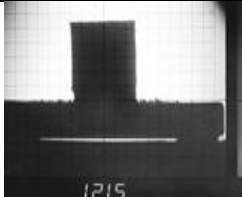
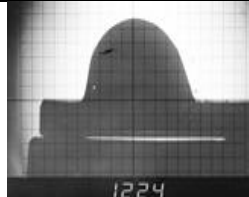

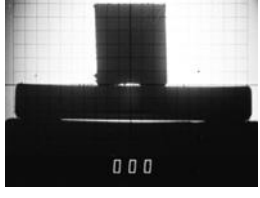

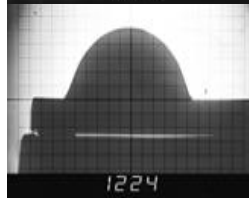

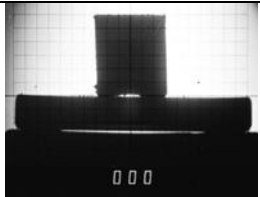
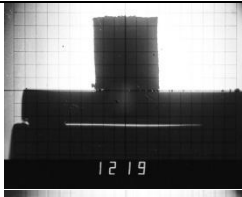
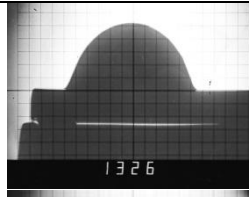
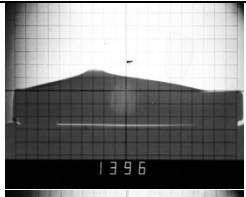





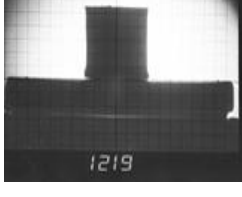


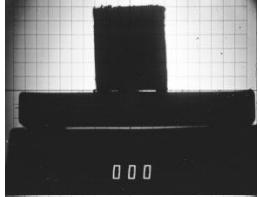
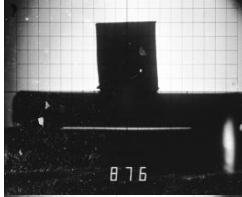
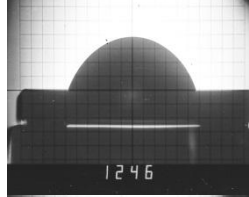

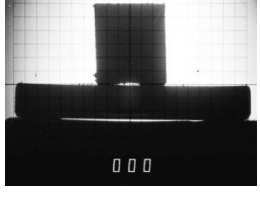


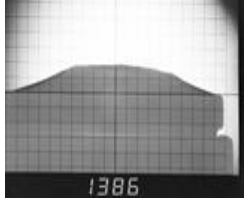
Grinding media utilized sintered corundum, tempered chrome steel, tungsten carbide, zirconium oxide, stainless, and polyamide plastic. Type of material being ground relates the type of vial and balls to be used. As with any grinding method, contamination of the sample with the grinding media is a problem. ^[34]

3.3 EXPERIMENTAL RESULTS AND DISCUSSION

3.3.1 FLOW CHARACTERISTICS MEASUREMENT

Heating microscope was used to determine the flow characteristics of all slag samples. The primary goal was to find out melting temperature (HT) and flow temperature (FT). Here we utilized the flow characteristics data of blast furnace slag, to make comparisons with the synthetic slag prepared by us. Flow temperatures and melting temperature were also compared with variations in the slag compositions. Lastly, attempts were made using relevant phase diagram to obtain a slag with less difference between HT and FT. The photographs of slag samples examined are shown below.

Sample		Original shape of the sample	ST (°C)	HT (°C)	FT(°C)
Blast Furnace I	1				
	2				
	3				
Blast Furnace II	1				
	2				
	3				
Blast Furnace III	1				

	2				
	3				
Blast Furnace IV	1				
	2				
	3				
Synthetic slag	1				
					

3.3.2 RESULT

Four typical slag samples obtained one each from the 4 numbers of Blast furnaces in SAIL Rourkela. The chemical composition of these slags are presented in Table no. III. While the major constitution CaO, MgO, SiO₂, Al₂O₃ together accounted for 96-97%, the rest accounts for the minor constituents such as FeO, MnO, and TiO₂ etc.

Four numbers of synthetic slags are prepared in the laboratory using pure oxides. The chemical compositions of such slags are presented in Table -VI. The major constituents (CaO, MgO, SiO₂, and Al₂O₃) together amounted for 100%.

The chemical composition as reported in Table-III and Table -VI are obtained by actual chemical analysis as carried out in DISIR, Rajgangpur.

The chemical composition of the synthetic slags are decided approximating that of the slags obtained from the industry, concerning the major oxides. The MgO content in all of these cases is kept at 10% since it is reported that high MgO content is not beneficial from the viscosity point of view of the Blast Furnace slag ^[22].

Table-VII and Table-VIII present the fusion behavior of these two sets of slags separately. The fusion behavior data, mainly the HT and the FT have been presented in figure no. 10 through figure no. 13, for the industrial slags as affected by Al₂O₃/CaO ratio, CaO/SiO₂ ratio, CaO% and MgO% respectively in that order. Figure no.14 through Figure no.16, present the fusion behavior variations (HT and FT) of the synthetic slags as a function of Al₂O₃/CaO ratio, CaO/SiO₂ ratio and CaO% respectively. It must be noticed that MgO in all these cases does not vary and is kept at 10%.

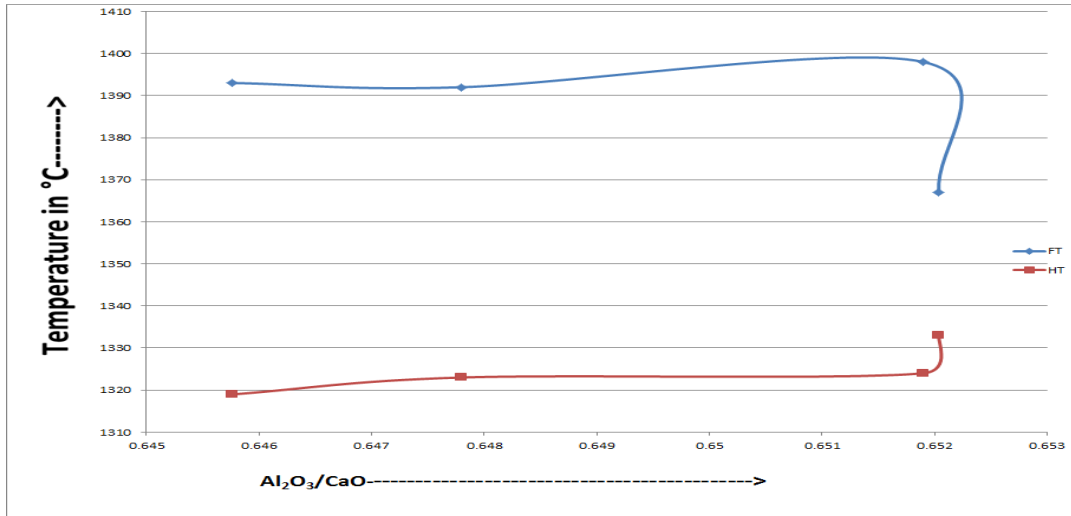


Fig 10:-Variation of HT and FT with Al₂O₃/CaO ratio for the SAIL, ROURKELA slags

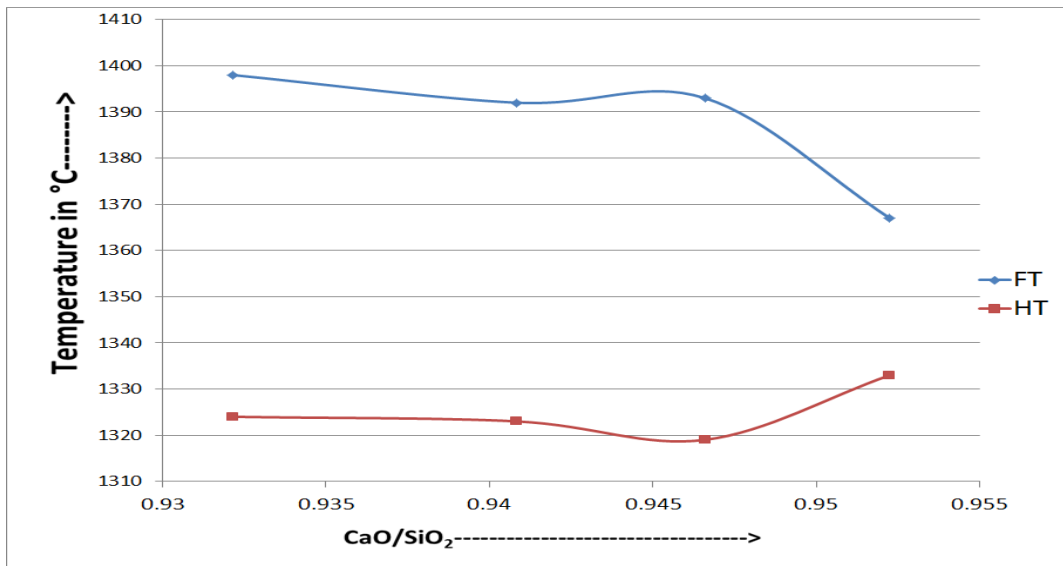


Fig 11:-Variation of HT and FT with CaO/SiO₂ ratio for the SAIL, ROURKELA slags

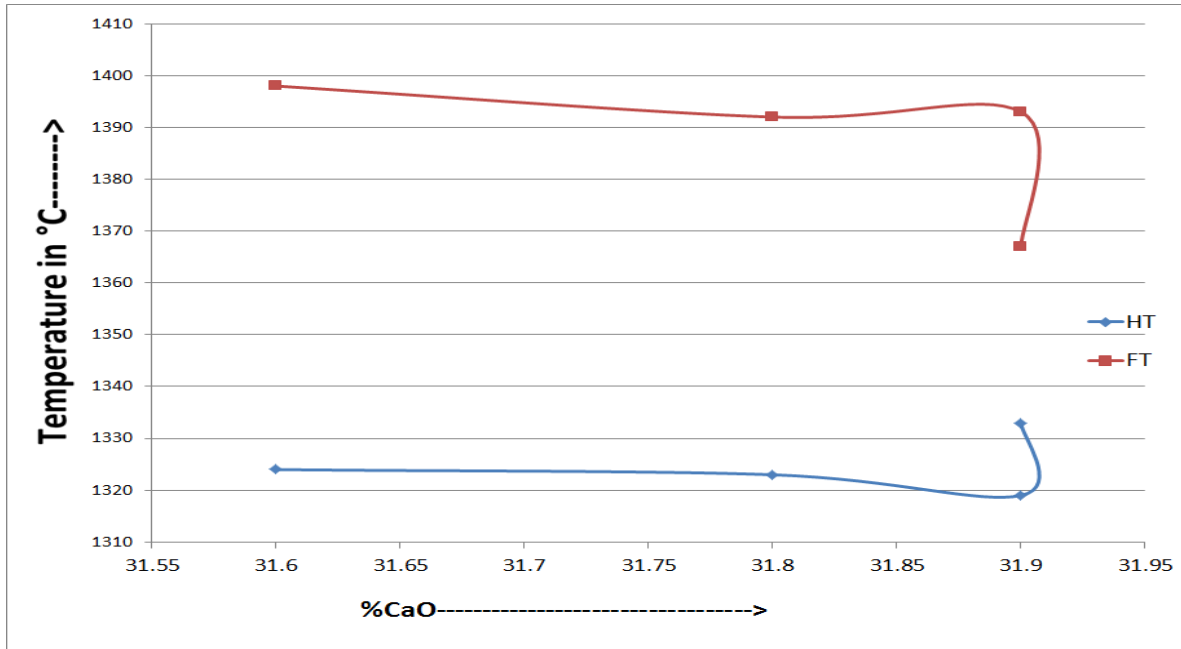


Fig 12:-Variation of HT and FT with %CaO for the SAIL, ROURKELA slags

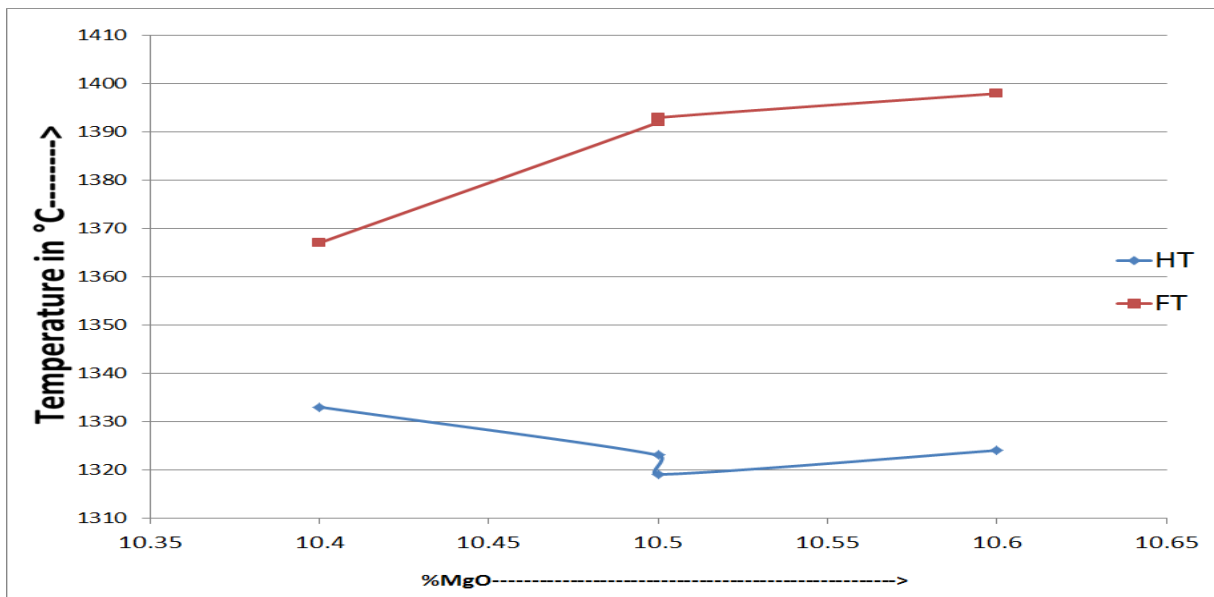


Fig 13:-Variation of HT and FT with %MgO for the SAIL, ROURKELA slags

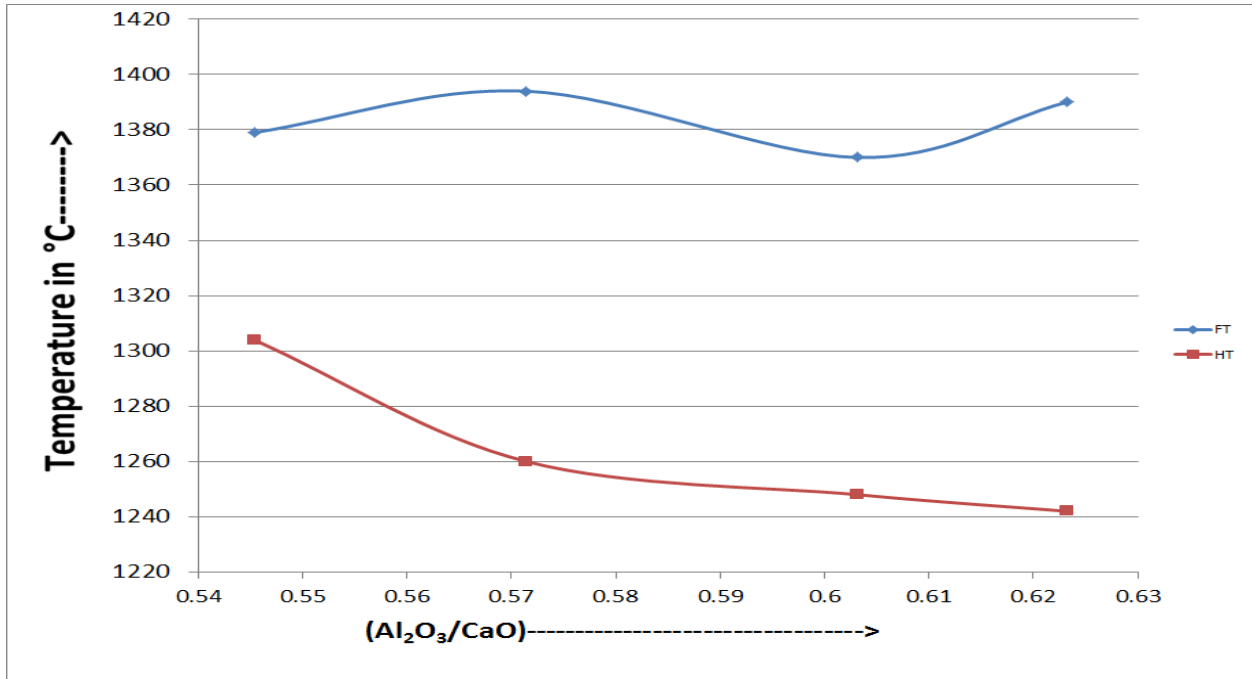


Fig 14:-Variation of HT and FT with Al₂O₃/CaO ratio for the synthetic slags

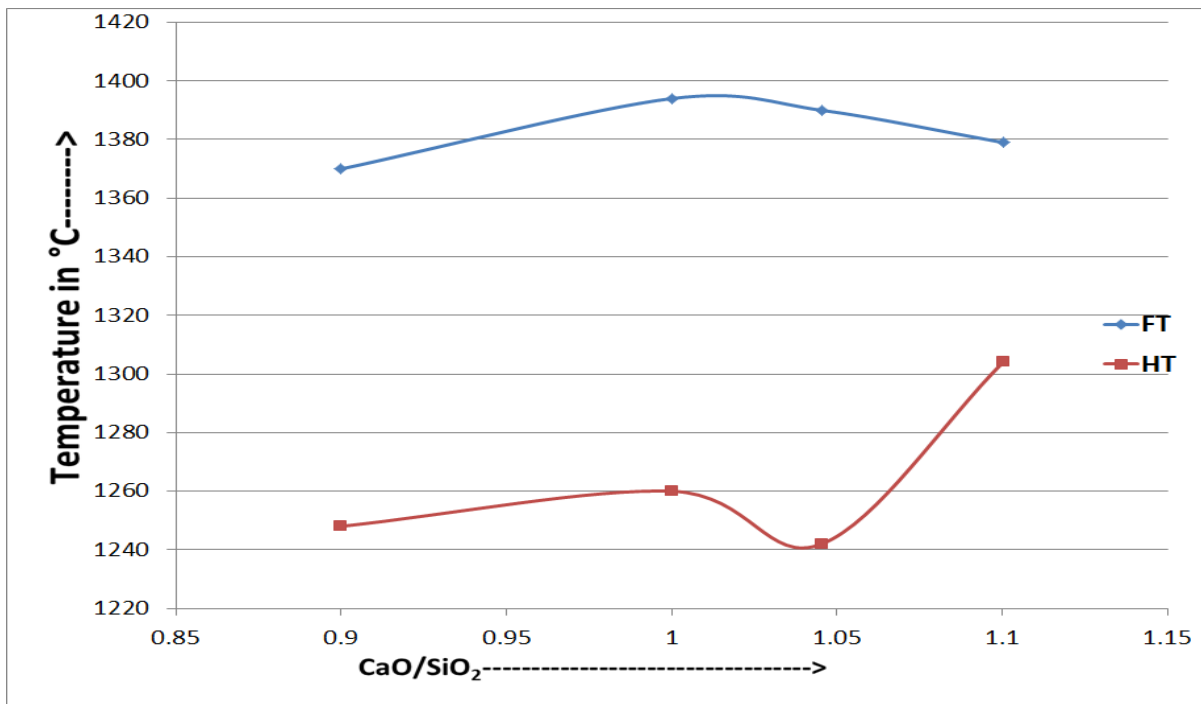


Fig 15:-Variation of HT and FT with CaO/SiO₂ ratio for the synthetic slags

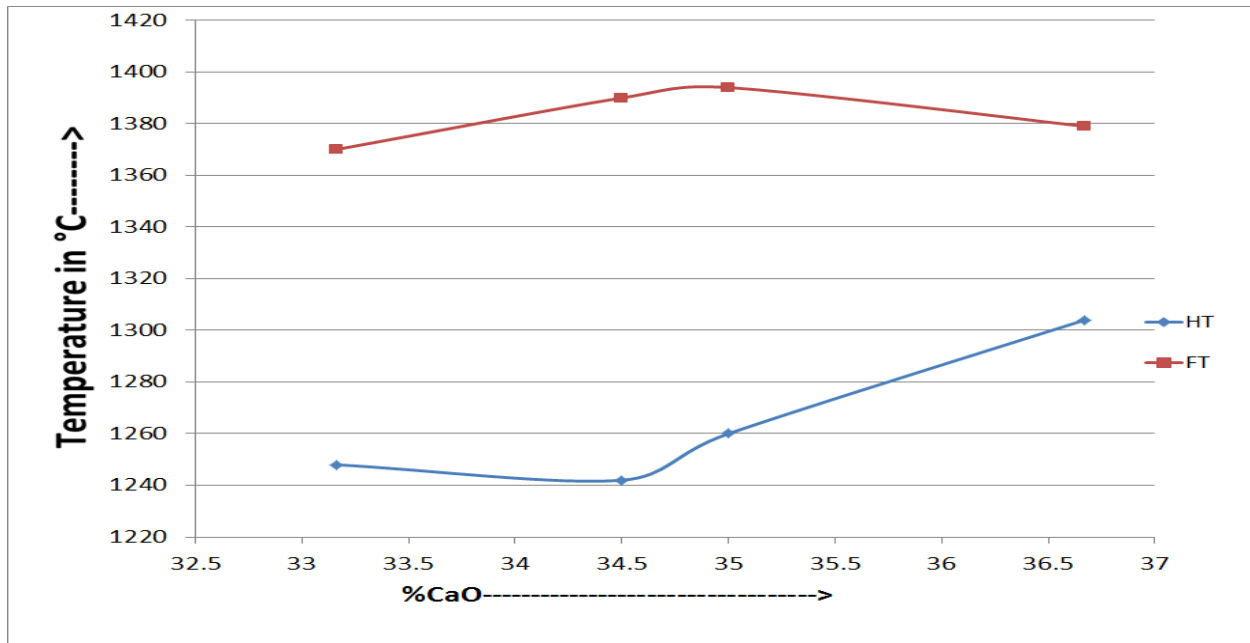


Fig 16:-Variation of HT and FT with %CaO for the synthetic slags

3.3.3 DISCUSSION

(a) Al₂O₃/CaO Ratio

The Binary Al₂O₃-CaO diagram reveals that CaO addition brings down the melting temperature of Al₂O₃ considerably. However, the B.F. slag, in addition to having Al₂O₃ and CaO as major components and also containing other oxides like MgO, SiO₂, etc. Hence it is important to examine how these oxides combined influence the flow characteristics of these slags at various Al₂O₃/CaO ratios and hence influence the characteristics of resultant slags.

For the synthetic slags the highest HT (1340°C) is obtained at Al₂O₃/CaO ratio of 0.545. This is the lowest ratio for the group of slags investigated. At this ratio the FT assumes the second lowest value and the difference between the FT and the HT is the minimum (75°C). It is also recorded that at this ratio of Al₂O₃/CaO the ST is maximum (867°C).

From the B.F. operators point of view this slags which $\text{Al}_2\text{O}_3/\text{CaO}$ ratio of 0.545 serves to be the best slag. This has the highest ST and HT, meaning that the cohesive zone in the B.F. bound by the softening-melting zone forms down the furnace. This would increase the extent of the granular zone with the possibility of concurrent increase in the extent of indirect reduction in the furnace resulting in energy saving.

The minimum difference of FT and HT and the relatively low FT value would indicate a narrow cohesive zone. This would ensure a smooth operation of B.F. with a minimum inference on permeability of the charge.

A high softening (ST) and a high fusion (HT) temperature of the iron-bearing material would lower the cohesive zone in the blast furnace thereby decreasing the distance travelled by the molten metal droplets in the blast furnace thereby decreasing the silicon pick-up^[23]. This is found to enhance the quality of hot metal considerably. Further, the low difference between FT and HT will result in an enhanced mobility of the slag and ensure better slag-metal separation. The relative low FT means a considerably low viscosity at the relatively low temperature. In operation, the low slag viscosity is indicative of the ease with which slag would be tapped from the furnace and therefore relates to the energy requirements and profitability of the furnace^[24].

On the other hand, for the industrial slags the second highest ST(1217 C), the highest HT(1333C) the lowest FT(1367 C) and the minimum difference between the FT and the HT(34 C) are obtained for the slag with $\text{Al}_2\text{O}_3/\text{CaO}$ of .652

From all counts, as discussed above, this industrial slag sums to be more advantageous than the synthetic slag notwithstanding the fact that this industrial slag has a relatively high $\text{Al}_2\text{O}_3/\text{CaO}$

ratio and that in particular the $\text{Al}_2\text{O}_3/\text{CaO}$ ratio is much higher compared to the concerned synthetic slag.

It is further noticed from the investigation of Nityananda and Fine^[25] that in the presence of 5% TiO_2 the effect of $\text{Al}_2\text{O}_3/\text{CaO}$ ratio is erratic. In this connection it must be borne in mind that the industrial slags considered contain 3-4% of minor constituents while the synthetic slags have none.

These facts ,must be kept in mind while deciding the constituents of the slags for a better result from the energy saving, smooth operation and hot metal quality

(b) CaO/SiO_2 :-

CaO/SiO_2 ratio can be considered to be the basicity of the slag. Results show that the synthetic slag with 1.1 CaO/SiO_2 ratio shows the best result from all the three counts as discussed above. However, in the case of industrial slag the test results are envisaged at a CaO/SiO_2 ratio of .95.

In practice the BF operates with the basicity of the slag around 1. This aspect needs to be further investigated keeping in mind that the industrial slags also contain minor constituents which have not been considered in case of the synthetic slags prepared in the laboratory.

(c) MgO content:-

So far as MgO content is beneficial the best result are obtained at 10% MgO for the synthetic slag which is not a variable in this case. Similarly in the case of the industrial slags the best result is obtained at the lowest MgO content(10.4%) of the slag.

Mohanty *et al*^[26] examined the effect of MgO at high $\text{Al}_2\text{O}_3/\text{CaO}$ ratios (2.5). They found that MgO content in the range of 4-8 % decrease the HT whereas the trend was reversed beyond 8.0

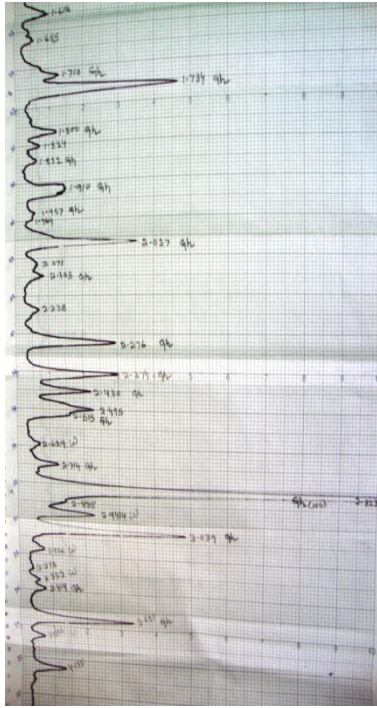
wt% MgO. Similar trends were observed in the case of BF slags within the range of MgO content from 0-16% as reported by Athappan ^[22] and 0-30% MgO as reported by Singh *et al* ^[27]. Also it was reported that beyond 6.0 wt% MgO additions the FT-HT value goes on increasing. Considering all these, the MgO content was kept at 10% for the synthetic slags.

3.3.4 XRD AND MICROSCOPIC ANALYSIS

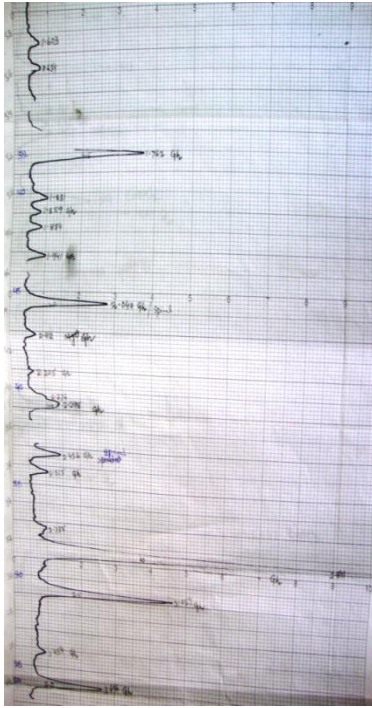
To know the chemical characteristics of the minor phases present in the slag, XRD analysis was carried on. This was done by first heating it to 1500 °C for one hour and then cooling it slowly. This was done to convert the slag on crystalline form, then the XRD analysis was done and the result is shown in **Table VIII** and the graphs are presented in **Fig. 17**.

Table VIII: Microscopic and XRD results of slag specimen as obtained from DISIR, Rajgangpur

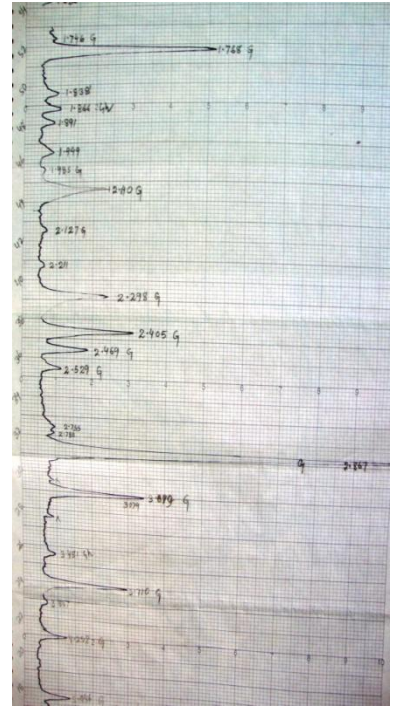
Specimen	Traces obtained from XRD results	Microscopic result		
		Crystalline (%)	Glass (%)	Opaque (%)
BF I	Gehlenite, Anorthite, Spinel	93.0	6.9	0.1(Trace)
BF II	Monticellite, Gehlenite, Anorthite, Spinel	93.4	6.5	0.1(Trace)
BF III	Monticellite, Gehlenite, Anorthite, Spinel	92.0	7.9	0.1(Trace)
BF IV	Gehlenite, Anorthite, Spinel	91.4	8.5	0.1(Trace)
Synthetic slag	Gehlenite, Anorthite, Spinel	91.6	8.3	0.1(Trace)



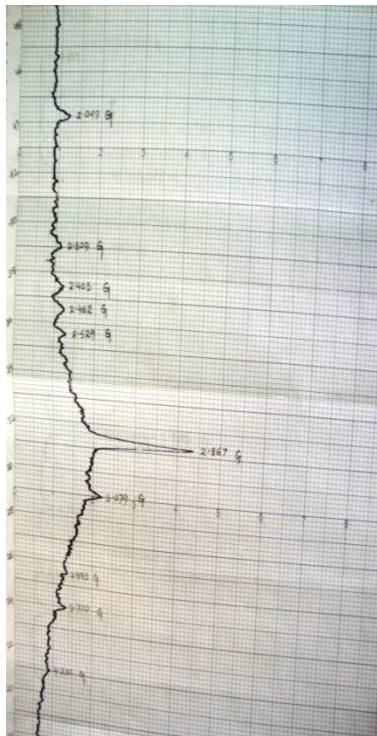
(a)



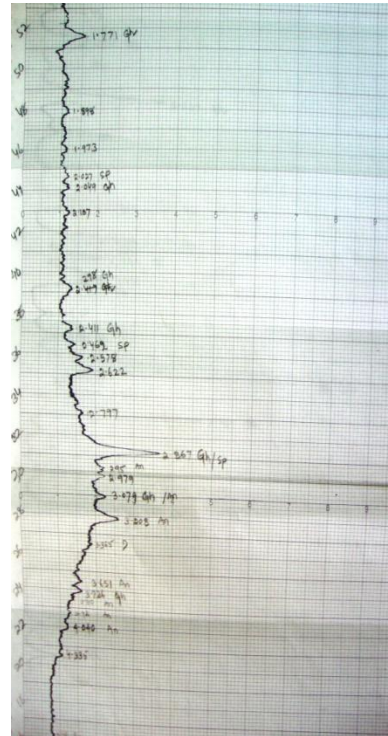
(b)



(c)



(d)



(e)

Fig. 17- XRD plots representing traces of mineralogical phases in case of (a) BF I, (b) BF II, (c), BF III, (d) BF IV, and (e) Synthetic Slag. In every case, Gehlenite was establish to be the main phase. However, some traces of Anorthite and Spinel were also present.

XRD analysis was done to deduce the major traces present in the slag sample. The traces found may not be the actual cause for the variation of the result but may be the cause for the variation of the melting point. Gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$) was found to be the main phase in all slag specimens. This crystalline phase was confirmed in the SEM micrographs as depicted in **Fig. 18**. As the cause of the lowering of the difference between the HT and FT cannot not be known until and unless the the major composition responsible for this is not found, so further investigation have to be done before reaching to a conclusion.

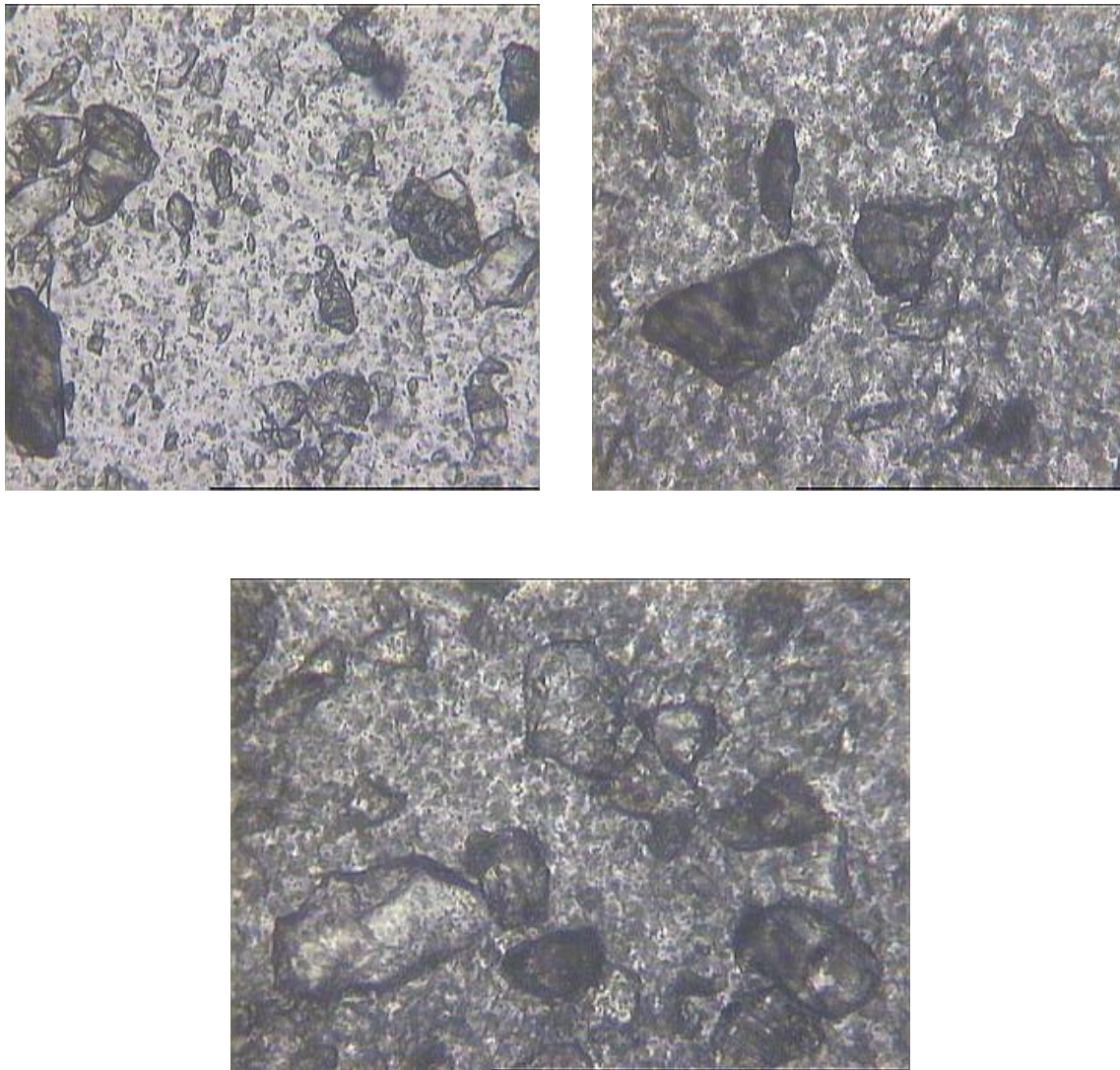


Fig. 18-SEM micrographs having magnification 100X, depicting that the crystalline phase is Gehlenite

3.3.5 PHASE DIAGRAM STUDY

Major trace elements found after the XRD analysis were Gehlenite, Anorthite and Spinel. As we were not able to get the phase diagram for Gehlenite-Anorthite system so we used Anorthite-Spinel system, shown in the **Fig. 19**.

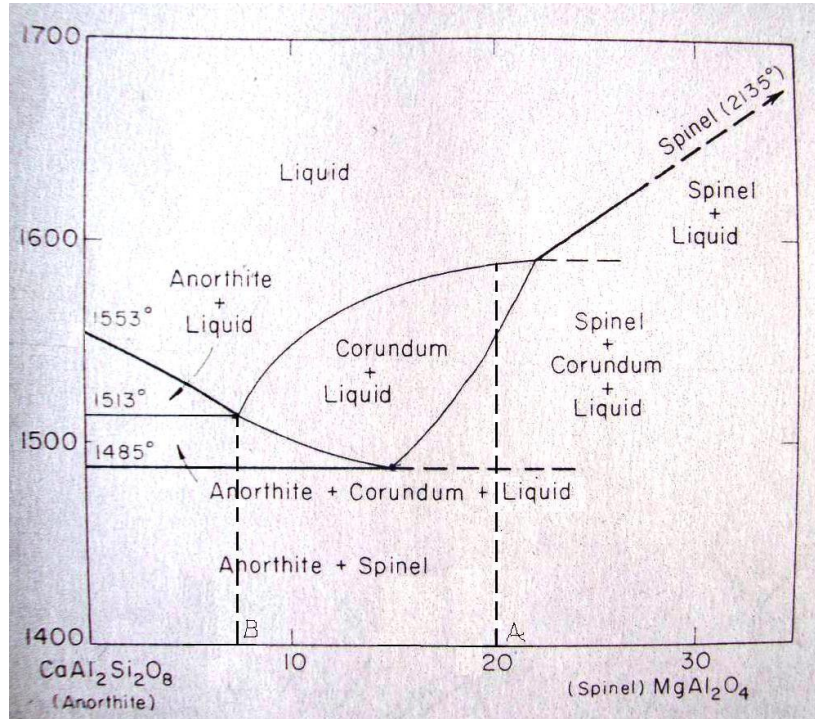
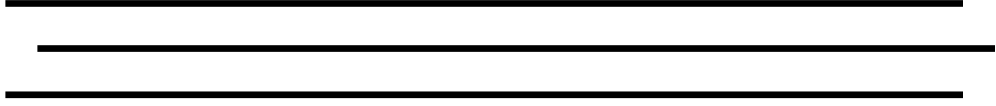


Fig. 19-System $\text{CaAl}_2\text{Si}_2\text{O}_8\text{-MgAl}_2\text{O}_4$. Heavy lines specify binary relationships

As shown in the phase diagram, it is confirmed that the melting point of Anorthite is 1553°C while that of Spinel is 2135°C . Now we assume a point in the graph with 80% Anorthite and 20% Spinel. It was found out that the melting starts at 1485°C but the flow temperature is around 1580°C . As our aim is to reduce the difference between FT and HT, we move on to the other side of the graph with 8% Spinel and 92% Anorthite, where the melting starts at 1573°C and flow temperature is reduced to 1573°C . Therefore, altering the composition around 8% Spinel, we can

get the desired result, i.e., by altering the amount of MgO and Al₂O₃ such that the composition of Spinel remains around 8%. The cohesive zone lowers down when the melting temperature increases and flowing temperature decreases, thus leading to low Si pickup in the hot metal.

CHAPTER 4



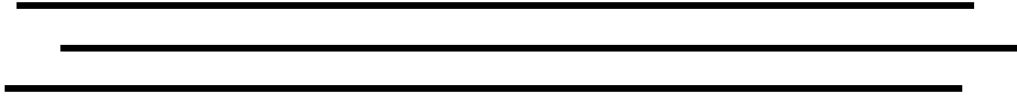
CONCLUSION

4. CONCLUSION

With our result obtained from our work, we deduce that with an appropriate selection of raw materials, desired slag can be obtained which leads to lower coke consumption and gives a greater control on metal composition by control of slag-metal reaction. The flow characteristics obtained after the experiment and concluded that addition of MgO content around 9.80 % is beneficial for a thin cohesive zone down the blast furnace. The result of the XRD analysis showed the presence of some major and minor oxides. The data obtained was used to draw related phase diagram and the phase diagram gave the idea of the slag composition which could be used to in lowering of cohesive zone.

The future prospect of the project is to use the data provided and to alter the composition in the slag in order to lower the cohesive zone. This can be done by preparing synthetic slag of different composition and of different weight percentage and then studying their flow characteristics. The characteristics obtained should be compared with the slag from different steel plants of India. From the data obtained a related phase diagram should be deduced and then it should be used in lowering the difference between HT and FT and thus finding out the characteristics for a narrow cohesive zone. On the basis of final slag obtained, burden distribution will be formulated. Finally, we can arrive to a specific slag composition which will have some industrial significance.

CHAPTER 5



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5. REFERENCES

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