PREPARATION OF SYNTHETIC SLAG IN THE LAB RESEMBLING INDUSTRIAL BLAST FURNACE SLAG AND STUDY OF ITS FLOW CHARACTERISTICS

This thesis is submitted in the partial fulfillment of the requirement

for the degree of Bachelor of Technology in

Metallurgical and Materials Engineering

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

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By

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Under the guidance of

Prof. U. K. Mohanty

CERTIFICATE

This is to certify that the thesis entitled **"Preparation of synthetic slag in the lab resembling industrial blast furnace slag and study of its flow charecteristics"** submitted by **Hrudananda sahoo** (110MM0377) and **Jiten Kumar Behera** (110MM0349) in partial fulfillment of the requirements for the award of **BACHELOR OF TECHNOLOGY** Degree in **Metallurgical and Materials Engineering** at the **National Institute of Technology, Rourkela**

(Deemed University) is an original work carried out by them under my supervision and guidance.

The matter embodied in the thesis has not been submitted to any other University/ Institute for the

award of any degree or diploma.

Date: 7th May, 2014

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ABSTRACT

A study of the flow characteristics of blast furnace slag is important to record the softening and melting phenomena in blast furnace which greatly influence the extent and location of the cohesive zone having a direct say on the blast furnace operation, quality of hot metal and the coke consumption. In the present work, a noble technique (heating microscope) is adopted to determine the flow characteristics of synthetic slags with high alumina content resembling different indian blast furnace slags . It is also seen that the characteristics temperature are altered with the alteration of C/S ratio and also the MgO and TiO2 content of the blast furnace slag.

Keywords: Cohesive zone, Characteristic temperature, Quality of hot metal, C/S ratio.

CONTENT

INTRODUCTION

1) INTRODUCTION

The blast furnace can be considered to be both a chemical reactor as well as a counter-current heat exchanger in which iron bearing materials(lump iron ore or agglomerates like sinters ,pellets etc), coke (a reductant)to meet the thermal requirements and to support the burden during melting of the iron bearing burden along with coal(as an inject ant) and oxygen(primarily as air)are fed into the blast furnace . metals are generally extracted with ores which are always associated with some impurities, mainly oxides(usually alumina and silica) called gangue is removed by the addition of flux, the resultant liquid being insoluble in and lighter than the molten iron forms a separate layer. The gangue constituents being mainly acidic oxides, A basic flux is necessary to lower the melting points of the acid constituents added in the form of basic lime and magnesia. The solid burden descending from the top and the hot reducing gas flowing upwards interact in the following manner

a) heat exchange ,i.e. heat transfer from gas to solid

b) oxygen exchange , i.e. oxygen transfer from solid to gas.

Obviously this is affected by gas-pressure, composition and pressure of the burden which depends upon the rate and uniformity in gas flow. As the burden descends and its temperature rises on account of contact with the ascending hot gases, softening and melting of the iron bearing materials takes place in the so called cohesive zone (mushy zone).The cohesive zone is marked by the softening of iron bearing materials in the top till the bottom where melting and flowing of the iron bearing materials occur[**1**].This affects the bed permeability. The lumpy cokes stay solid and are responsible for maintaining the bed permeability even in front of the tuyeres but the softened or molten solids affect the bed permeability and restrict the flow of the ascending gases. Thus the ascending gases are able to flow through coke-grids only. When this happens pressure drops and influences the gas-solid-liquid interface greatly influencing the slag metal reaction rates and thus the blast furnace process of iron making.

From blast furnace aerodynamics point of view and uniform gas flow we require the cohesive zone to be narrow and lower down the furnace,which is aided by having a composition of high softening temperature and relatively low flow temperature[**2**]. This would decrease the distance travelled by the liquid in the blast furnace which would decrease the Silicon pick- up.**[3,4]** also the final slag formed in the bosh region should trickle down easily to the Hearth in the furnace.therefore it should be a short slag that will flow as soon as it softens. Thus fusion behaviour is very important parameter in evaluating the effectiveness of B.F. slag.

For describing the fusion behavior of slag four characteristic temperatures **[5]**;

a)IDT, the initial deformation temperature rheologically implying surface stickiness, important for movement of the material in the solid state.

b)ST, rheologically implying plastic distortion, indicating start of plastic deformation.

c)HT, the liquidus temperature, rheologically implying sluggish flow, playing a significant role in the aerodynamics of the furnace and heat and mass transfer.

d)FT, the flow temperature rheologically implying liquid mobility.

The slag that is formed in the cohesive zone is the primary slag .this type of slag has a low melting point because it contains large amount of FeO as the primary fluxing constituent. The fusion temperature, solidus temperature, solidus-fusion interval being greatly affected by FeO content **[6]**. This slag is completely different from the bosh slag as well as the final slag where the fluxing is primarily caused due to the presence of basic constituents like CaO or MgO.

As the primary slag is formed in the cohesive zone inside the blast furnace hence it is not feasible to get primary slag from the industrial blast furnace . But we can prepare a synthetic slag in the laboratory resembling the final slag and study its flow characteristics . However,it is desired that the final slag should be a ‘Short Slag’(a slag with a narrow difference between the ST and FT).for obtaining “short slag” it requires the slag to posses liquid mobility and should trickle down the furnace as soon as it is formed. This process will expose fresh sites for further reaction and is greatly responsible for intensifying slag-metal reaction rates, which will influence the blast furnace operations and finally affect the the hot metal quality.

Therefore, it was aimed at studying the flow characteristics of high alumina synthetic blast furnace slag with varying C/S, MgO and TiO2 so as to know what type of composition would lead to the formation of narrow cohesive zone lower down the furnace, which will affect the quality of hot metal in terms of Si content, permeability in the furnace for uniform gas flow, increased extent of indirect reduction.

2. Literature Survey

2.1. Blast Furnace Iron Making

Blast furnace iron making process is the most used process among all the iron making processes. This is because blast furnace iron making has a high production rate and a very high degree of heat utilization (85-90%). This is because the blast furnace is an extremely efficient counter current heat exchange apparatus. Modern high capacity furnaces are producing as much as 12000 tonnes of hot metal per day. **[7]** The sources of iron are its ores in which iron is contained mainly as its oxides such as hematite (Fe2O3) or magnetite (Fe3O4) and sometimes in small proportions as hydroxides and carbonates. Hematite constitutes the largest portion of all the ores used for blast furnace iron making.

When pure, hematite contains about 70% and magnetite about 72.4% of iron. But in actuality, the iron content of the ores ranges from 50-65% for rich ores and 30-50% for lean ores and the remainder is gangue which consists mostly of silica and alumina as well as minor amounts of moisture and chemically- combined water. The ores are normally charged as sinters or pellets.

Sintering and pelletizing are processes by which iron ore fines are agglomerated into larger pieces with or without incorporation of lime and magnesia as fluxes. The gangue materials are insoluble in liquid iron and possess very high melting points. However they fuse at lower temperatures in the
presence of fluxes and form a slag. Magnesia helps to lower the fusion temperature and increase the fluidity of the slag. Lime and magnesia are basic in nature and silica and alumina are acidic in nature and their ratio is known as basicity. **[7, 8]** The slag and the iron can only be separated completely from each other when they are in the liquid state which requires them to be heated to above their fusion temperatures. The heat is **usua**lly supplied
by burning of coke. The reduction of iron oxides also needs sufficiently high temperatures as well as adequate amounts of reducing agents (coke carbon). The iron also picks up 2-4.5% carbon from the coke which lowers its melting temperature from 1534oC by 200-300oC depending upon the carbon content. The coke contains ash which is mainly constituted of silica and alumina which require a further amount of lime and magnesia for fluxing. The ore, coke and flux contain compounds of Si, Mn, P, S and small amounts of other impurities like Pb,

Sn, Cu, Ni, Cr, Ti, alkali metals, etc., which get partly or wholly reduced and get taken up by iron. Manganese ores are usually deliberately added for manganese's beneficial effect on iron makingCoke is the only component of the charge materials which descends as a solid to the tuyere level. Apart from supplying the reducing agent and heat for processing the burden into finished products, the coke provides mechanical support to the burden especially where it is needed most, i.e. in the bosh region where the metal and slag are liquid. These liquids flow down to the hearth through the interstices of the coke particles (coke grid). The coke rate in the blast furnace per tonne of pig iron varies from 1000kg to as low as 45-500kg. Low coke rates are obtained with pre-fluxed sinters and pellets, high blast temperatures and uniform gas distribution. The hot blast of air entering the furnace through the tuyeres burns the coke carbon to CO2 immediately in front of them. The intense heat produced gives a flame temperature (tuyere gas temperature) of 1800-2000oC, depending upon the blast temperature. Since CO2 is unstable in the presence of carbon above 1000oC, CO is produced. The tuyere gas, therefore, consists only of CO and nitrogen, their contents being about 35% and 65% respectively when dry blast is used. The coke does not fall continuously but only periodically into the tuyere zone from above. The hot reducing gas rises through the active coke bed to the bosh, belly and the shaft and reduces the iron oxides.

Reactions in the upper zone

In this zone the burden is rapidly heated from the ambient temperature to about 800c within a distance of 4-6m from the stock level and the gas coming from the middle zone cools down from 900c to 100-200c as it leaves the furnace top.the main reactions that occur in this zone are narrated below:-

a)reduction of hematite and magnetite to its lower oxides(mainly wustite)partially or completely.

b)decomposition of carbonates of iron,manganese,magnesium and calcium-magnesium(dolomite) . since calcium carbonate decomposes at much higher temperatures,900-1100c depending upon its lump size.

c)carbon deposition remay occupyaction-

$$2CO = CO_2 + C + 41210cal$$

d)vapourization of hydrated water,i.e. water present chemically combined as water of crystallization or hydration.

e)vapourization of the chemically uncombined water entering the furnace with moist ore,coke,limestone or other charge material.

It generally takes about 6-8 hours for the burden to descend from the top to the level of tuyers which depends upon the production rate.

REACTIONS IN THE MIDDLE ZONE

This zone extends from the upper zone(4-6m below the stock level)downwards to 3-5m above the tuyere level .it is a moderate temperature zone where the temperature ranges between 800-1000c.the height of this zone is considerable and may occupy 50-60% of the shaft height(about 70% of the shaft volume) in modern furnaces working smoothly with properly sized charge materials and with uniform gas distribution.the charge passes through the zone in 2.5-3 hours.

From the kinetic point of view,this temperature range(800-1000c) is very suitable for indirect reduction of wustite,the phase left after reduction of hematite and magnetite in the upper furnace . within this zone there lies a chemically inactive zone where the gas composition does not change appreciably and the co and co2 contents approach equilibrium with wustite(nco,feo=25-30%).there also exists a chemically inactive zone where very little transfer of oxygen transfer of oxygen takes place.indirect reduction of all iron oxides takes place below 1000c and that of wustite at 800-1000c.

The importance of middle zone or indirect reduction zone in the blast furnace process can only be realized from an understanding of the role of direct/indirect reduction on the fuel economy. direct reduction is economical of coke but results in a thermal deficit. the blast works through an optimum balance between the thermal and reduction requirements. The larger the height of the 800-1000c temperature range , the longer the gas-solid contact time at these temperatures and the greater the degree of indirect reduction. similarly the higher the reducibility of the ore the more rapid the reduction. Another important reaction called as water-gas shift reaction occurs in this region:-

$$CO + H_2O = CO_2 + H_2$$

This reaction generates hydrogen which is a more active reductant than co.

REACTIONS IN THE LOWER ZONE

This zone extends from 3-5m above the tuyere level to the hearth bottom. The temperature of the lower zone is above 900 to 1000C . It takes about 0.7 to 3 hrs for the burden to decent from the belly to the tuyeres . The temperature of the molten materials reaches 1400-1450oC in this zone and the ascending gas cools down to a temperature of 800-1000oC. The combustion of coke in front of the tuyeres which results in a continuous creation of voids around the hearth periphery that is responsible for the flow of the charge materials downwards . This combustion results in an active area about a ring of 1-2m depth and is known as the raceway. Beyond the raceway, there is a closely-packed central column of coke known as dead man's zone. The coke column either remains suspended on the molten iron in the hearth or it reaches the hearth floor. Some iron particles and slag may remain entrapped within the interstices and voids of the coke pieces which needs to be drained out while tapping.

The gangue materials(mainly acidic oxides,such as silica,alumina etc) in combination with the flux added in the form of limestone or dolomite(basic oxides) starts to fuse or soften in the belly region where two distinct immiscible phases (partly carburized primary iron and FeO-SiO2-Al2O3-MnO primary slag containing some amount of CaO) begin to form at temperatures above 1200oC. Further down the furnace, these liquid phases are separated from each other, which infiltrate through the coke slits above the raceway and get collected in the hearth well from where they will be tapped periodically. Here the raceway ressembles a counter-current liquid-gas exchanger,because the tuyere gas rises through the voids or spaces present in the coke bed.the coke also provides the mechanical support to the burden.

The critical hearth temperature which is about 1500-1550oC is necessary for free running of the slag as well as to ensure some superheat in the hearth and make sure that both the iron and the slag are present in the liquid state under blast furnace operating conditions. The important physical and chemical reactions occurring in this zone are: -

1.Endothermic calcinations of limestone;

$$CaCO_3 = CaO + Coke$$

2. Endothermic direct reduction of FeO;

$FeO + C = Fe + CO$

3.Endothermic direct reduction of SiO_2;

$SiO_2 + 2C = Si + 2CO$

4. Endothermic direct reduction of MnO;

$MnO + C = Mn + CO$

5. Endothermic direct reduction of P_2O_5;

$P_2O_5 + 5C = 2P + 5CO$

6. Endothermic sulphur removal;

$FeS + CaO + C = CaS + Fe + CO$

7. Exothermic combustion of carbon;

$C + O_2 (air) = CO_2 + N_2$

8. Endothermic reduction of CO_2;

$$CO_2 + C = 2CO \ (>1000^oC)$$

9. Endothermic reduction of moisture in blast;

$$C + H_2O = CO + H_2 \ (>1000^oC)$$

BLAST FURNACE SLAG

Raw materials for a blast furnace consists of iron bearing materials,coke (a reductant and fuel supply). But these raw materials are not pure,they also include some impurities along with them,called as gangue.they are mainly oxides .coke ash which is the result of combustion of coke is rich in alumina and the iron ore lumps are mainly rich in silica as well as alumina.both the oxides alumina and silica are acidic oxides. The melting point point of these acidic oxides is very high(even greater than blast furnace operating temperatures).therefore they will not melt and their separation from the pure iron becomes difficult.hence to facilitate the removal of the gangue we add flux to lower their melting point.since the gangue is acidic the flux has to be basic. therefore we add limestone and dolomite as flux provider.in case if the gangue were basic or neutral then acidic flux(generally quartz)or neutral flux(calcium fluorite or fluorspar) has to be added.

A slag is a product of the chemical reaction between the gangue present in iron bearing raw materials(iron ore lumps or agglomerates like sinter and pellets) and coke ash with the fluxes added in the form of lime and magnesia. The slag formed has many important roles to play .it most have lower melting temperature than both the gangue and the flux,so that it will form immiscible liquids added to that the slag must posses lower density than the metal and it must be liquid at the operating temperatures to facilitate separation and removal.

Functions of slag:-

a)slag foremost duty is to react and retain the impurities by taking part in slag-metal reactions and controlling the metal quality,viz.,the Mn-,Si- and S- contents . apart from these four major non-ferrous oxides(lime,dolomite,silica and alumina) which forms about 95-96 % of the total,the slag also consists of many other minor constituents which depends upon the raw materials added and the quality of iron ore being smelted .they are mainly:-feo,tio2,mno,fes,cas,alkali oxides,etc.

b)the slag must control the metal temperature physically by forming a layer over the metal surface and chemically as the slag can enhance the reactions(either endothermic or exothermic or both) so that the temperature is under control.

c)it must have the ability to tolerate the fluctuations in the temperature and physical and chemical properties in the burden.

d)it must possess high sulphur retention potential to remove sulphur under the operating blast furnace conditions.

e)the slag volume should be kept as low as possible,because physically, the entrapment of hot metal in the slag is high and chemically,more the useless material we are handling more is the heat loss.

The volume of the slag can be controlled by controlling the amount of mgo and not cao(as it's content will depend upon the gangue content)

f)the slag must possess low viscosity as it will accelerate the slag-metal reactions but the refractories may get affected.a low viscosity slag is also desired for good flowability as it will be able to run out of the furnace freely and rapidly at the operating temperatures which will influence the fuel economy.

g)slag formation (fusion and melting) should be confined to a limited height of the shaft so that there is free movement of the stock and the furnace gases and the slag should be stable which in turns affect the productivity and the hot metal quality.

h)the slag should be fluid enough to provide good permeability in the zone of slag formation.

But if it is fluid enough then it immediately reaches the hearth and if viscous enough then the permeability gets affected . therefore the slag must possess optimum viscosity.

i)the melting point of the slag should be optimum. This is because in the hearth we need high temperature for certain reactions and if the melting point of slag is low, then the slag forms at the upper portion of the furnace and then falls to the hearth and due to low heat content takes the heat from the hearth and adversely affects the reactions and vice-versa happens when the slag is of high melting point.

Internal zones in a blast furnace

In an ideal blast furnace,it is assumed that the temperature and composition of the gas and the solid vary along the furnace height only and not along the horizontal,but in actual practice the temperature and composition vary along the horizontal direction as well.this is because the hot air blast introduced through the tuyeres results in localized combustion in that area . the resulting gases flow upwards and are not able to penetrate uniformly up to the furnace centre.another reason for this behaviour is that the solid burden charged from the top is heterogeneous , hence some non-uniformity in the size and density from the centre to the periphery cannot be avoided.these all phenomena leads to horizontal non-uniformity in the solids within the blast furnace at the belly and the bosh region,leading to the formation of different internal zones in the furnace.the different zones inside a blast furnace are:-

a)granular zone

b)cohesive zone

c)active coke zone

d)stagnant coke zone

e)tuyere zone

f)hearth zone

granular zone:it is present at the top of the furnace which contains the coke and the iron bearing materials charged, sometimes along with some quantities of limestone and other fluxes.no liquid formation is there , reaction(reduction) is there but in solid state and the iron bearing oxides charged gets reduced to wustite and metallic iron towards the lower end of this zone.

Cohesive zone:as the burden descends further, and its temperatures rises further on account of contact with hot ascending gases,softening and melting of the iron bearing solids takes place . it is marked by the onset of softening and extends till the onset of melting of the iron bearing materials.

Active coke zone:this is the region where coke actively takes part in the reactions involving direct reduction of iron oxide from trickling slag and its own interaction with carbon dioxide i.e., mainly bosh region.

Stagnant coke zone:since the hot air blast is unable to penetrate right upto the centre of the furnace.this results in the occurrence of a relatively inactive coke zone in the central region.here the coke is still solid and supports the overlying entire burden and through which the slag and metal trickles and the metal gets saturated with carbon.

Tuyere zone:it is the region where coke burns or carbon undergoes combustion by combining with the hot blast(oxygen) effectively to CO in the raceway of the tuyere zone.during combustion coke pieces swirl around before they get gasified, and then fresh pieces descend to take their place .

Hearth zone:it is the lowest region in the blast furnace where the hot metal and the slag clarify into two layers and are periodically tapped. It is here where they interact with eachother with respect to their chemistry to move, as far as possible,towards equilibrium.

Out of these six internal zones the most important is the cohesive (or mushy) zone. This is because once a muddy mass is formed,the permeability of the burden is hindered and the ascending gases flow through the coke slits. The cohesive zone acts as a gas distributer inside the furnace.the cohesive zone affects the smooth running of the blast furnace by influencing the gas flow both chemically(reduction) and physically(heat transfer).

We want a narrow cohesive zone, lower down the furnace i.e., the softening should be at high temperature and the gap between the softening and flowing should be low to get following advantages:

a)the granular zone is increased due to which maximum indirect reduction takes place . as these reactions are exothermic in nature , hence lesser amount of fuel is required.
b)the narrower the mushy zone, the lesser the permeability of the bed is affected.
c)if it is narrow then the liquids wil fall with a greater velocity and there will be more no of interfaces per unit time. Hence the productivity will increase.
d)it will result in better hot metal quality i.e., the corresponding hot metal will be low in silicon and phosphorus content.the low silicon content is required for further processing of hot metal to steel in steelmaking shop.

The course of slag formation in the blast furnace can be divided into three zones:-

(i) primary slag fusion zone
(ii) bosh slag fusion z
(iii) final slag or hearth slag

primary slag refers to the first liquid or first melt formed in the blast furnace.on reaching the cohesive zone iron has undergone a reduction of around

50% ,i.e., considerable amount of feo is present.the semi calcined flux enters the cohesive zone from the granular zone.as the cohesive zone is bound by the softening and melting of the iron bearing materials at its top and flowing at the bottom therefore the primary slag must form somewhere in the cohesive zone.

For cao to flux the oxides it has to diffuse into the oxides(by diffusion),but it cannot as it is a solid state diffusion process.moreover,at high temperatures alumina and silica undergoes sintering and form alumino-silicates which are much more stable than the individual oxides ,cannot be fluxed by cao.the only oxide which can flux them is feo and form the first liquid.hence the slag regime is feo-al2o3-sio2 . from the phase diagram of feo-al2o3-sio2 system we found that the liquidus temperature of the alumino-silicate slags containing above 40% feo is not more than 1200C.we have taken above 40% reduction of feo because iron ore on reaching the cohesive zone has undergone a reduction of 50% ,i.e., considerable amount of feo must be present.

As the %feo decreases , there is a rapid increase in the melting point to that of the feo free slags. From the ternary phase diagram of the primary slag ,the liquidus temperature rises progressively, especially towards the high alumina side.therefore the most favourable condition would be attained if the gangue content of an ore contains a silica/aluminaratio of 2.5-4.1 .

CHARACTERISTIC PROPERTIES OF THE BLAST FURNACE SLAG:

The hot metal quality in the blast furnace is directly influenced by the slag that is formed and by the mineralogical transformation undergone by the slag. The acidic components SiO_2 and Al_2O_3 (amphotoric) are responsible for increasing the slag viscosity whereas the basic components CaO and MgO decrease the viscosity. In determination of blast furnace productivity, the melting characteristics and fluidity of the slag play an influential role. The initial slag formed has a high FeO content and is low in CaO and MgO. However, as the slag descends, its composition varies significantly as it picks up CaO, MgO and coke ash. Thus the slag characteristics play a very important role in blast furnace operations. The most important characteristic properties of slag, which have attracted the attention of many metallurgists, are: Slag Viscosity, Slag Liquidus Temperature, and Sulphide Capacity of the Slag and Alkali Capacity of the Slag.

BASICITY OF SLAG

The oxides,e.g., sio_2, al_2o_3, which accept oxygen and form anion complexes in melts are called as acidic oxides. On the other hand the oxides which breakdown the anion complexes,e.g., cao,mgo,feo,mno, etc., by donating oxygen are called as basic oxides. the ratio of the sum of the concentrations of the basic oxides to that of the acidic oxides is called basicity of the slag.in ironmaking the ratio is usually depicted as cao/sio_2 or sometimes as $(cao+mgo)/(sio_2+al_2o_3)$.it should be maintained around 1.2 or 1.4 respectively for the smooth running of a blast furnace and the upper limit of the alumina content should be around 18% and lower limit should be around 12%. High viscosity of high alumina slags , hampers desulphurization . therefore , MgO which is added to decrease the viscosity , results in an increase in the volume of the slag which results in lower productivity of the furnace and increases the coke rate.

Slag Viscosity

Due to the presence of a range of shear stresses , the slag can be called as a Newtonian fluid.the viscosity of blast furnace slags is not governed due to the shear stresses present but due to the ionic and molecular structure .viscosity influences various important transport phenomena like heat transfer , mass transfer and various chemical reactions(slag-metal reactions).the slag should be free flowing at operating temperatures for ease of slag-metal separation.it should have less viscosity so that the permeability(uniform gas flow) is not affected which will affect the efficiency of the blast furnace.slag-metal reaction rates are accelerated when a slag is less viscous.tapping of slag becomes easy and fast if the slag is of low viscosity which directly affects the energy consumption and economy of the process. yet from the life of refractories point of view , the slag should posses high viscosity which may penetrate easily and decrease the life of refractories.also a less viscosity slag will reach the hearth immediately and take the available heat thereby reducing the temperature of the hearth essenstial for different reactions[**9**] .therefore, the slag should be of optimum viscosity, but generally we want the slag to be of low viscosity.

As the basicity increases , the viscosity decreases due to depolymerisation of silicate network. But after a certain level the viscosity will increase with increase in basicity due to increase in the chemical potential of certain solid phases[**9**].

Slag liquidus temperature

The german industrial standards 51730 defined liquidus temperature as the temperature at which the slag assumes a hemispherical shape on heating . a very small mass was noticed having a hemispherical form . it was shown by Osborn[**10**] and snow[**11**] that this temperature can also be defined as the temperature on cooling at which first liquid appears to form in the melt . according to ohno et al [**12**] all crystals vanish when the slag is heated to its liquidus temperature .by knowing the liquidus temperature we can know the shape , size and relative position of the cohesive zone inside a blast furnace . because the geometry and position of cohesive can influence the extent of indirect reduction , gas permeability, silicon pick-up by hot metal etc .

here we determine the liquidus temperature by leitz heating microscope from the characteristics temperatures defined by; the initial deformation temperature(idt), softening temperature(st), hemispherical temperature(ht) and flow temperature(ft).out of these four characteristics temperatures the hemispherical temperature(ht) , which is otherwise known as liquidus temperature and which implies sluggish flow .

Shortness of slag

The softening and melting phenomena of blast furnace slag is of vital importance in BF operation as it has a direct say over the productivity and efficiency of the process. The characteristic temperatures obtained from the flow characteristics of blast furnace slag includes; Initial Deformation Temperature (IDT), Softening Temperature (ST), Hemispherical Temperature (HT) and Flow Temperature (FT). The ST and FT rheologically signify the plastic distortion and liquid mobility of the blast furnace slag respectively. A slag is said to be short if the difference between softening and melting temperature is minimum . In a blast furnace operation, a short-slag is desirable as it trickles down the furnace as soon as it is formed thus exposing further reaction sites for better slag-metal reaction rates.it also affects the permeability in the so called cohesive zone and thus affects the uniformity gas flow. Shortness of the slag has a direct impact over the furnace productivity as it controls he slag-metal reaction rates.

2.4.2. Flow Characteristics of Blast Furnace Slag:

The flow characteristics of the blast furnace slag sample was determined with the help of a high temperature microscope. It is endowed with four characteristics temperatures required to be studied:

1.Initial deformation temperature (IDT)

2.Softening temperature (ST)

3.Hemispherical temperature (HT)

4.Flow temperature (FT)

The followings are defined as per German Industrial Standards 51730.

Initial Deformation Temperature (IDT):

Initial deformation temperature is the temperature at which the first rounding up of the edges of the cube-shaped sample specimen takes place. In fact this is the temperature at which the first sign where the change in shape appears.here the shape of corners change first because of stress concentration at the corners. Rheologically the initial deformation temperature symbolizes the surface stickiness of the slag.

Softening Temperature (ST):

Softening temperature is the temperature at which the outline of the shape of the sample starts changing and is defined as the temperature at which the sample will shrink by one division or the temperature at which the distortion of the sample will begin. Rheologically softening temperature symbolizes the onset of plastic distortion.

Hemispherical Temperature (HT):

Hemispherical temperature is the temperature at which the sample has fused down to a shape resembling a hemisphere and is measured by the temperature at which the height of the sample is equal to half the length of its base . This is defined as the melting point or the fusion point by the Germen Industrial Standards 51730 . Rheologically hemispherical temperature symbolizes the sluggish flow of the slag.

Flow Temperature (FT):

Flow tempera temperature is defined as the temperature at which the sample will liquify and is defined by the temperature at which the height of the sample will equal the one-third of the height that it had during HT (hemispherical temperature). Though some books report it as the temperature at which the height acquires one third of the initial height. but the former one is more accurate and is accepted universally. Rheologically the flow temperature symbolizes the liquid mobility or the flowability of the slag.

Effect of slag basicity on viscosity of the slag

y.s.lee et al[**13**] experimented and found that the viscous behavior of cao-sio2-al2o3-mgo slag under controlled conditions of c/s =1.15-1.6, by taking 10-13% al2o3, 5-10% mgo and 0-20% feo . it was concluded that due to polymerization of the silicate network there is increase in viscosity of the slag above c/s ratio of 1.3 and less than c/s ratio of 1.5. till the c/s ratio 1.3 the viscosity decreases due to an increase in the chemical potential of the dicalcium silicate which is a primary solid phase.the thermodynamic approach provided a good correlation between the viscosity and the slag components to find the the activity of primary solid components . therefore it was concluded that in highly basic slags the viscosity of slag can be approximated by the chemical potential of dicalcium silicate.

The study also shown that for low FeO content(<7.5%) the viscosity of slag showed minimum value with increasing MnO content , but if the FeO content was high(>7.5%) no particular influence was observed on the viscosity of slag. the study also confirmed that for a fixed CaO/SiO2 ratio the viscosity of blast furnace slag decreases with increasing FeO content. according to some tests done by

Japanese companies it was shown that by injecting flux into the blast furnace the slag basicity variation along with Si content in the hot metal can be minimized by less reduction of SiO2 to Si .

Y.S.Lee, J.R.Kim, S.H.Yi : 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.[20].

Their studies showed that the heat transfer, mass transfer, sio2 and feo reduction and gas permeability is controlled by the flow characteristics of the slag. This implied the effect of the viscous nature of the sag on the efficiency and productivity of the blast furnace. The viscous behavior of Cao-SiO2-Al2O3-MgOfeo slags was studied under the up to the Cao/SiO2 ratio 1.3, slag viscosity decreased with increase slag basicity. The FeO content in the slag if increases the viscosity of the slag decreases so when the Feo decreases from 10-15% so the viscosity of the slag at basicity of CaO/SiO2=1.5 increases from about 2.5 to 10 dpa.s with FeO content as mentioned above.

Amitabh Shankar et al[14] for the CaO-SiO2-MgO-Al2O3 and Cao-Sio2-MgO-Al2O3-TiO2 system by varying the C/S ratio between 0.72 and 1.23 in the temperature range of 1573-1873 k. alumina content (12-18%), manganese(2-8%) and titania(0-2%). They have shown that the viscosity increases with increase in basicity. It was also shown that the slope of the viscosity vs. temperature curve is steeper for low basicity slags. An increase in basicity decreases the slag viscosity, because silicate structure changes from network to discrete anionic groups containing simple chains or rings as basic oxides are increased.

J.-Y . JIA C.-G BAI, G.-B. QUI, D.-F. CHEN [15] based on the studies of the ternary slag system of CaO-SiO2-TiO2, it established a calculation model. Thus, they were able to outline a mass action concentration calculation model. Thus they were able to outline a mass action concentration calculation model based upon the exiting theories and documented data at different compositions.

With increase in TiO2 content the mass action concentration also increases.viscosity of slag decreases with increasing TiO2 contnt in the slag. With the rise in temperature viscosity decreases and running quality is improved.

Effect of MgO and Al2O3 content on the viscosity of the slag:

Seong-ho seok et al[16] the viscous behavior of CaO-SiO2-MgO-FeO and CaO-SiO2-FeO-Al2O3-MgO melts were studied which were saturated with di-calcim silicate with a MgO content of 8% under condition of high basicity and the temperature of around 1873k. the inferences drawn is that the viscosity of slag depends relatively on alumina content as the solid phase present is more in case of alteration in alumina content then MgO.

Y.S. Lee et al[**17**] studied the influence of MgO and Al2O3 alteration on the viscosity of BF slag. The viscous behavior of CaO-SiO2-Al2O3-mgo-feo slag was studied and the viscosity under conditions of C/S ratio 1.35-1.45, 10-18% alumina, 3.5 to 10% MgO and 5% FeO was measured, for a fixed MgO content and the basicity, the viscosity increases with increase in the alumina content. The slag also showed a minimum value of viscosity at around 7% of MgO at over the temperature of 1723K. the Mgo content did not significantly change the slag viscosity.

Yasuji Kawai [18] studied CaO-SiO2-Al203-MgO slags. When mgo was added Ca0-SiO2 slags, the viscosities decreased with increasing amount of MgO only up to about 20%, beyond which, however, it increased. The region of low viscosity was greater than that in CaO-SiO2-Al2O3 slag.

Noritaka saito et al[19] studied the effect of MgO on the viscosity of 40 CaO-40 SiO2-20 Al2O3-MgO slags. Through their studies they proposed that magenesia acts as a network modifier and also that the activation energy for flow also decreases with addition of MgO. Viscosity decreases with addition of MgO.

Masashi Nakamoto et al [20] to measure the viscous behavior of molten cao-sio2-mgo-al2o3 they used they used the rotating cylinder method and compared the result with its model that was created.their purpose of study was to obtain a specific compositin of slags that would melt at low temperatures (1673K) to improve the blast furnace operations. They hit upon a slag of the following composition (35%al203-43.1% cao- 7.5%mgo-14.4% sio2) which melts at below 1673k and has a viscocoty less than 0.6 pa.s.

Experimental work

Apparatus required

leitz heating microscope

These temperature is already defined by graham international standards 51730. For recording the characteristics temperature the heating microscope method is adopted. In fig.-4 leitz heating microscope is shown. Fig.-5 shows the schematic diagram of the instrument. The sample, in the form of a 3 mm cube, is heated in an electric furnace in the microscope assembly. A camera is used to photograph the shape change of the sample due to heating in the micro scope. A grid-division which is simultaneously photographed with the sample and the temperature to which the sample is being heated facilitate identification of the four characteristic temperatures. The four temperatures are reported as follows by following the German Industrial Standard 51730

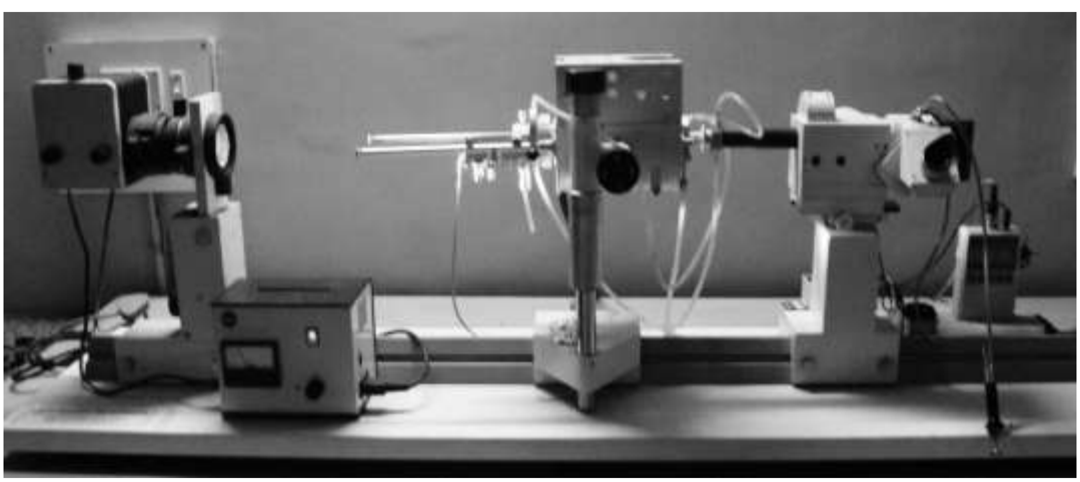


Figure 1

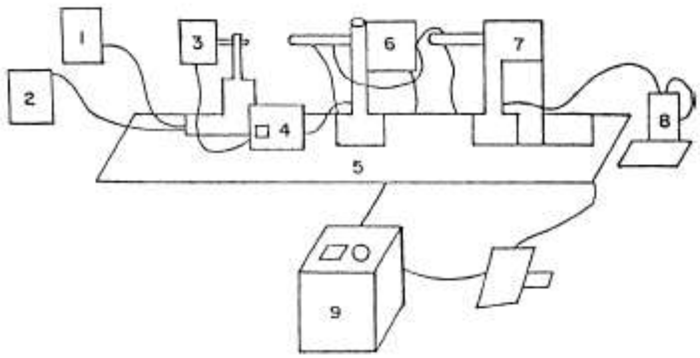


Figure 2

The components of leitz heating microscope are:

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

Planetary ball mill

These mills are also referred as centrifugal mills and are used to grinds samples into colloidal fineness by generating high grinding energy. Fig 7. 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 plate and then it is mounted in the machine. Once the vile are mounted and secured, the machine is functional. The bowls are independent of the rotable platform and the direction of rotation of the bowls is opposite to the direction of the rotable platform. The motion resembles the tea cup and saucer as seen in some of the amusement parks. Due to alternate subtraction and addition of the centrifugal forces, the grinding balls roll half way in the vile and then thrown across the vile and then impacting the opposite walls at a very high speed. 20g acceleration is reached due to the planetary action and the time of the grinding reduces about 2/3 times than a simple centrifugal mill.

Sintering furnace

The pellets obtained are sintered around 1680 degress Celsius in the sintering furnace to bind the slag materials by the process of diffusion. This temperature is selected as it is the tempreture that the sa;lg undergoes when comes out of the blast furnace, so firing is done at this temperature. Proper firing can be easily done when homogenous mixing is done. There are two crucibles holders where two platinum crucibles are palces each containing different slags. Thus at a time 2 slags can be prepared. After attaining the required temperature gradually, the slag is thenn quenched in water toi room temperature with the crucible in different containers.

Testing

For the determination of the flow characteristics of the slag sample high temperature microscope is used. There are four characteristics temperature for observaton.

1. Initial tempreture deformation (IDT).
2. Softening temperature (ST).
3. Hemispherical temperature (HT).
4. Flow temperature (FT)

Sample preparation

The oxides used for sample preparation were commercially oxides with 99% purity. The process involved heating the oxides first at about 150c in an oven to remove moisture content. After oven heating, the oxides were properly weighed in the weighing machine as per the required percentage composition and were mixed thoroughly in the planetary ball mill. The oxides mixed were in the powder form and they cannot be fired as such. So the powders were made as small pallets, thus making them compact and easier to charge. The pellets obtained were heated in the oven for some time to remove any moisture content in it. They were then introduced in the furnace at 1600c for duration of 30 minutes. The molten sample were quenched. The slag sample thus obtained was ground in the ball mill for duration of 30 minutes. The sample was then again introduced into the furnace at 1600c for a duration of 30 mins.it was subsequently water quenched and synthetic slag obtained was again ground.

Sampling

For sampling coning and quartering technique is used i.e. the reduction of size of the powdered or granular sample, by forming a conical heap which is spread out into a circular, flake cake. Then it is divided radially into quarters and two opposite quarter are combined. And the left quarters are discarded. This process is repeated until the desired quantity is obtained for final use. If the process is performed only once, coning and quartering is no more efficient than taking alternate portions and discarding the others

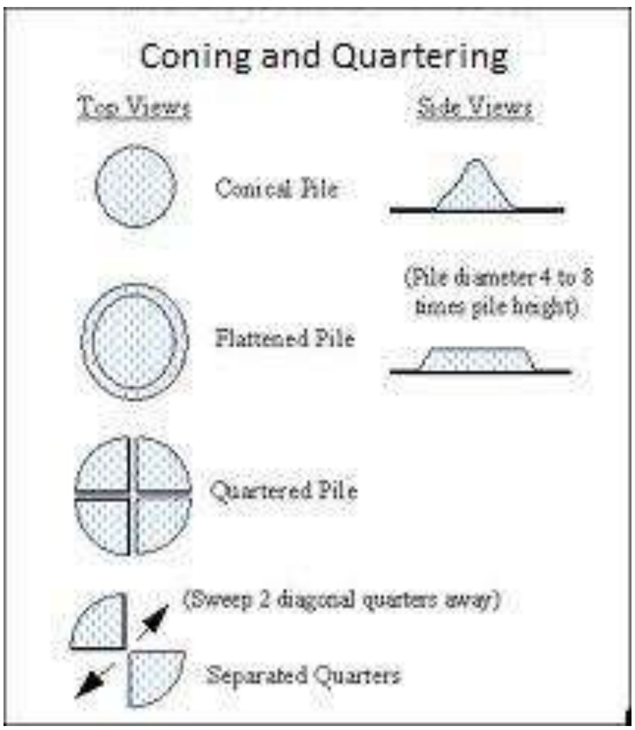


Figure 3

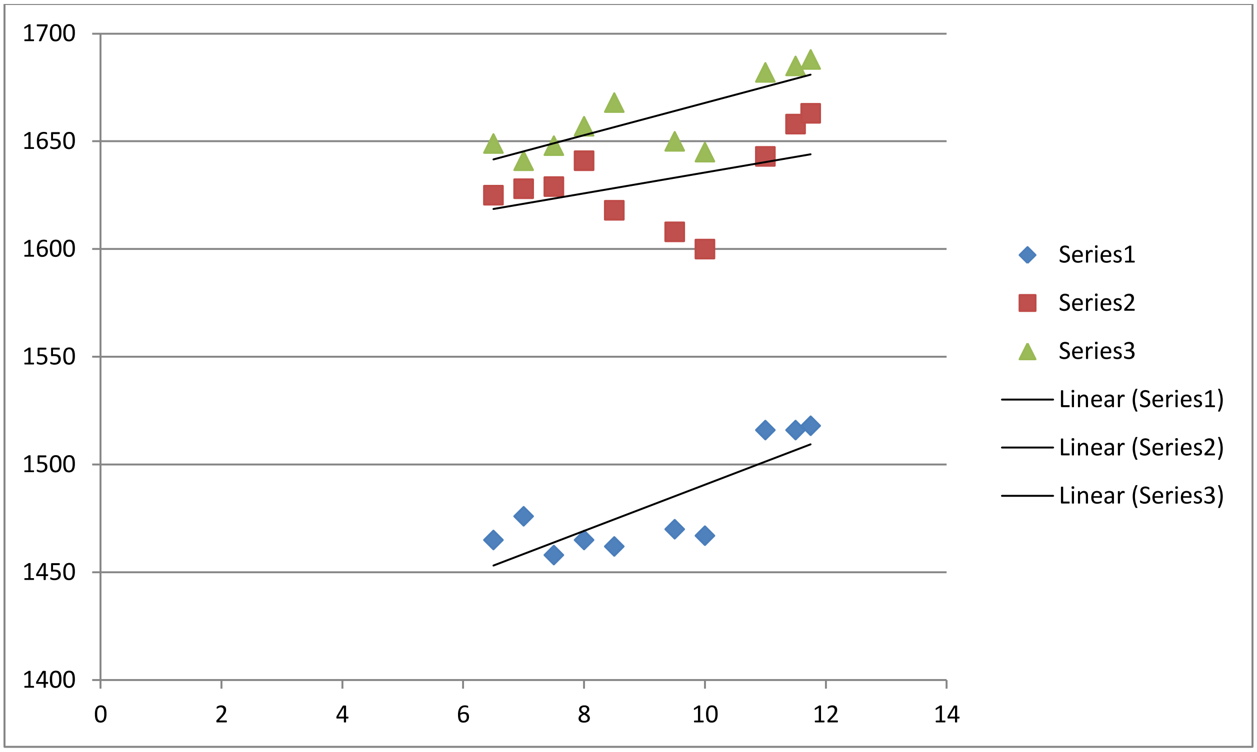
The different steps for determining the flow characteristics are as follows.

- The 3mm cube sample is heated in the electric furnace in the microscope assembly.
- The shape change of the sample as a result of heating is photographed by a camera.
- A grid-division which is simultaneously photographed with the sample and the temperature to which the sample is being heated facilitate identification of the four characteristic temperatures.
- IDT is reported as the temperature when the sample just start to deform.
- The temperature, at which the sample has already shrunk by exactly one division or at which the cube starts to swell, is reported as ST.
- The temperature at which the sample takes the shape of a hemisphere with its height to diameter ratio 1:2. Is reported as HT.
- FT is reported at the temperature at which the height of the sample reduses to one third the height measured at HT.

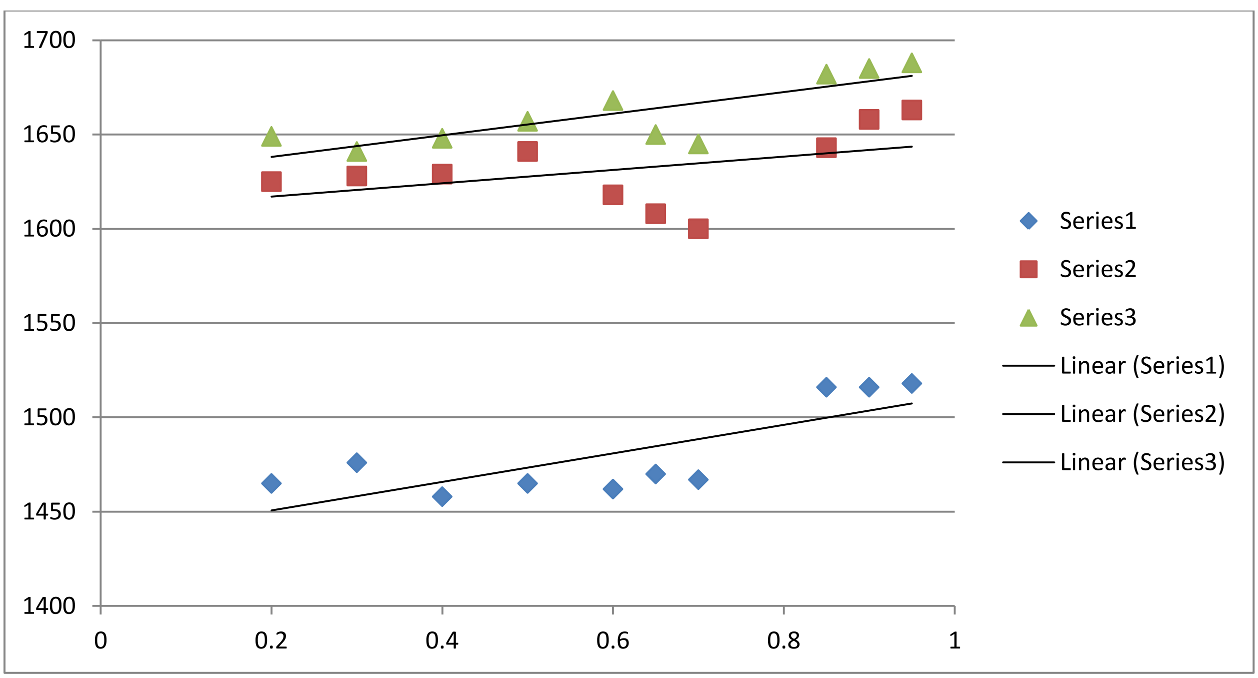
COMPOSITION OF SAMPLES PREPARED

slag	Al2O3	MnO	MgO	Na2O	K2O	Fe2O3	TiO2	CaO	SiO2	C/S
slag1	20	0.01	6.5	1.1	0.5	1	0.2	34.44	36.25	0.95
slag2	20	0.01	7	1.1	0.5	1	0.3	35.05	35.05	1.00
slag3	20	0.01	7.5	1.1	0.5	1	0.4	35.09	34.4	1.02
slag4	20	0.01	8	1.1	0.5	1	0.5	35.29	33.6	1.05
slag5	20	0.01	8.5	1.1	0.5	1	0.6	35.46	32.8	1.08
slag6	20	0.01	9.5	1.1	0.5	1	0.65	35.22	32.02	1.10
slag7	20	0.01	10	1.1	0.5	1	0.7	35.38	31.31	1.13
slag8	20	0.01	11	1.1	0.5	1	0.85	36.41	29.13	1.25
slag9	20	0.01	11.5	1.1	0.5	1	0.9	36.73	28.26	1.30
slag10	20	0.01	11.75	1.1	0.5	1	0.95	37.16	27.53	1.35

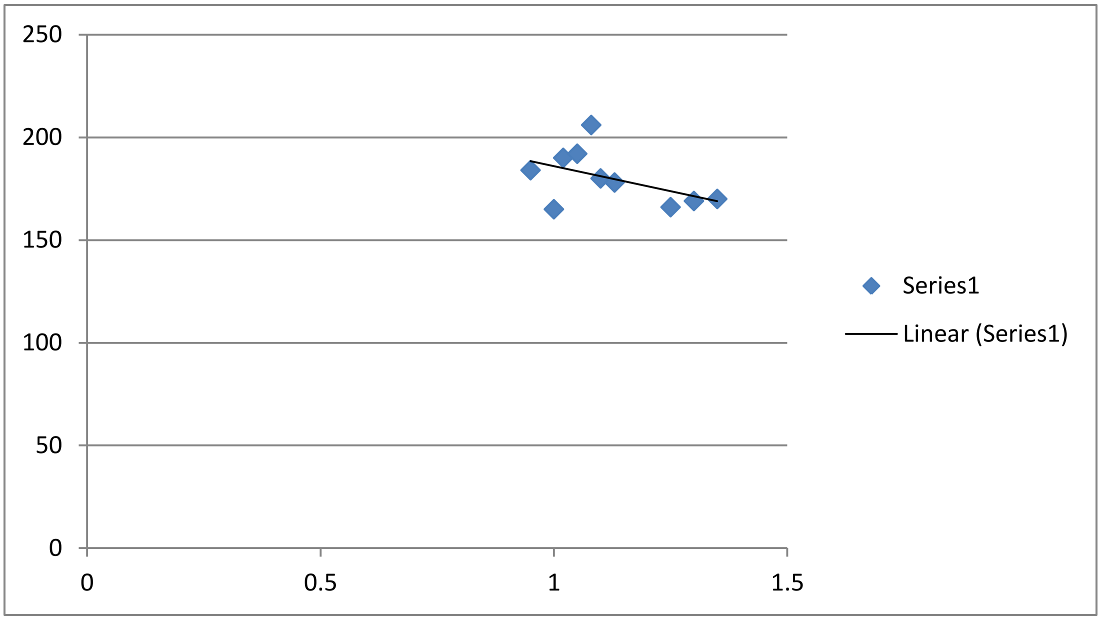
c/s	mgo	tio2	st	ht	ft	ft-st
0.95	6.5	0.2	1465	1625	1649	184
1	7	0.3	1476	1628	1641	165
1.02	7.5	0.4	1458	1629	1648	190
1.05	8	0.5	1465	1641	1657	192
1.08	8.5	0.6	1462	1618	1668	206
1.1	9.5	0.65	1470	1608	1650	180
1.13	10	0.7	1467	1600	1645	178
1.25	11	0.85	1516	1643	1682	166
1.3	11.5	0.9	1516	1658	1685	169
1.35	11.75	0.95	1518	1663	1688	170



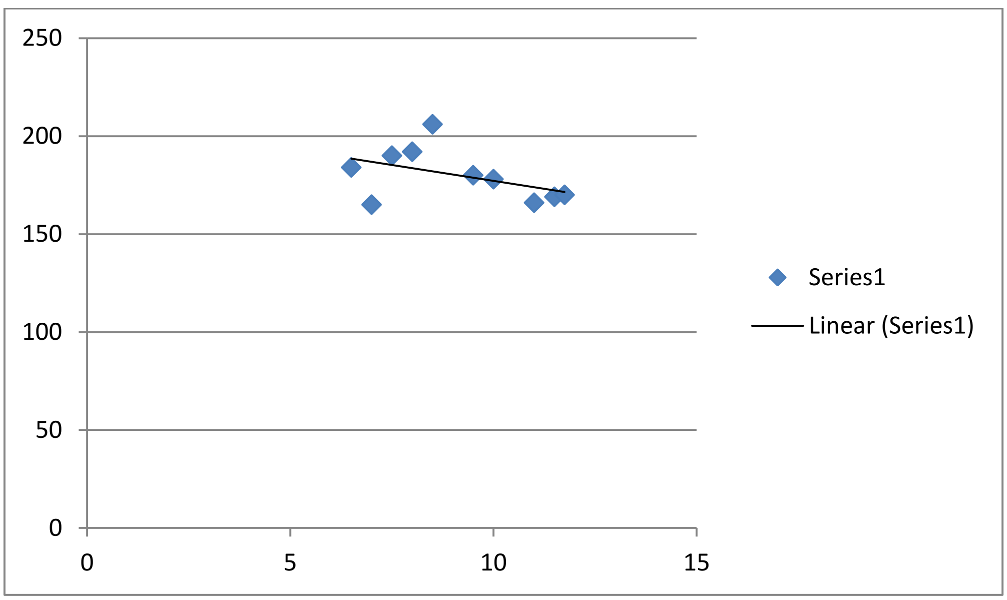
Effect of MgO on charecterictics temperature



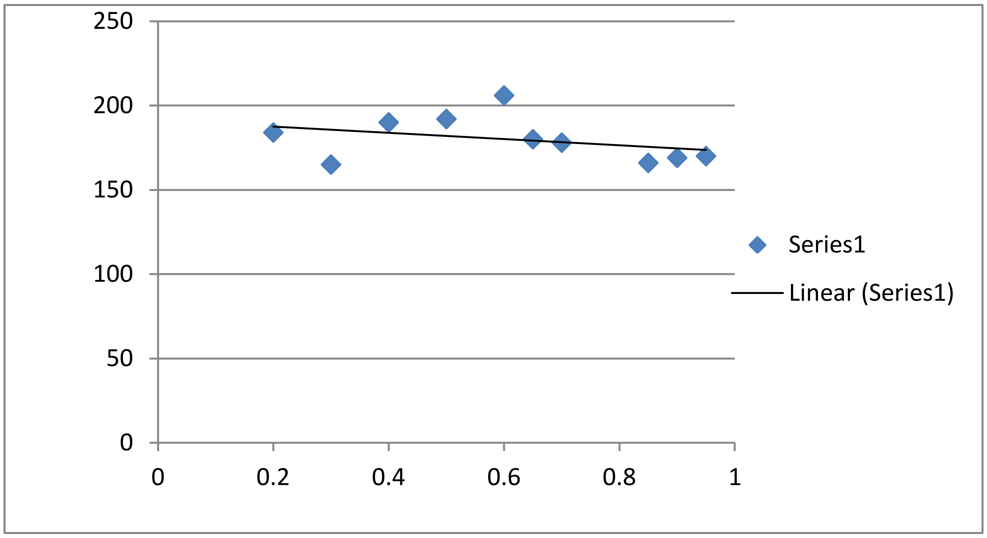
Effect of TiO2 on characteristics temperatures



Effect of C/S on FT-ST



Effect of mgo on FT-ST



Effect of TiO2 on FT-ST

RESULTS AND DISCUSSION

The flow characteristics of 10 blast furnace slag samples were measured using the high temperature microscope. The flow characteristics is determined by measuring the four characteristics temperatures ,viz. initial deformation temperature(IDT), softening temperature(ST) , hemispherical temperature(HT) and flow temperature(FT) .also the effect of c/s ratio, MgO content and TiO_2 on the flow characteristics was deduced.

Effect of C/S ratio on characteristics temperature

For all the lose ,the characteristics temperature tend to increase with the increase of the C/S ratio. The trend may be attributed to the presence of high Al_2O_3 as detailed below (alumina in all the cases are kept at 20 unit)

a. Alumina works as a network breaker when Al_2O_3/CaO ratio is greater than unity[21][22].in the present case Al_2O_3/ CaO ratio for all the slags investigated is below unity especially in increasing C/S ratio at the fixed alumina content, the Al_2O_3/CaO ratio further decreases. Therefore the total network forming ions are presented by (Al+Si), alumina contributing in the increase in the degree of polymerisation, thus hindering the free flowing nature of the slag. This explains increase in characteristic temperature with increase in c/s ratio.
b. Increase in C/S ratio means decrease in SiO_2 wt% resulting in increase in Al/Si ratio at fixed alumina content of 20wt%.this increase in Al/Si ratio is responsible for the increase in the viscosity of the slag[23][24].

Therefore the flow characteristics of the slg is adversely affected presented by general upward trend with increase in C/S ratio.

c. The flow characteristics due to the ppt. of a solid phase namely di-calcium silicate[25] (2CaO.SiO2) when C/S ratio is greater then 1.3.

This explains the increase of the characteristics temperature in general with the increase of the C/S ratio at the high content of alumina which amphoteric in nature.

Effect of MgO on characteristics temperature

In present case increased MgO results in an increase of the softening temperature of the slags. This is agreement with the findings of Dash et al[27].this trend is beneficial in the process of iron making.

Here the slags softens lower down the furnace within lesser possibilities of interfering with the permeability of the bed.

Effect of TiO2 on the characteristics temperature

TiO2 contents seems to increase the characteristic temperature at all levels. This may be due to the adverse effect on the depolymerisation of the slag.

Effect of C/S ratio, MgO & TiO2 contents in the difference between FT & ST

In all cases, the increase of the constituents (C/S ratio, MgO & TiO2) decreases the difference between the flow temperature and the softening temperature this difference is the measure of shortness of the slag. A short slag is defined as one which flows down soon after it softens without the requirement of higher heat input. This advantageous in iron making process through blast furnace process. this is because the flow of the slag. The route of the slag metal reaction heat and mass transfer reaction get auelanted

On the basis of the above it can be concluded that a careful selection of the composition of the blast furnace slag is essential for the smooth operation of the blast furnace. This is especially true in Indian contexts where the alumina content is high.

Summary of the results:

i) High ST means slag will soften lower down the furnace

Advantages

Less possibility to choke or interfere with the permeability of the bed. Thus heat and mass transfer accelerated.

ii) Significance of decreasing FT- ST

Short slag is obtained at higher values of three variables

Advantages

- Needs less thermal energy
- Trickles down as soon as softens
- More surfaces are exposed for accelerated reactions(slag metal property).

Conclusions:

i) The flow characteristics of the synthetic slag samples was measured using high temperature microscopy.

ii) It is seen that there in an increase in the characteristic temperature in general with the increase in C/S ratio at a high alumina content which is amphoteric in nature.

iii) Also, in the present case, increase in the MgO content led to increase in the softening temperature of the slag.

iv) It was also seen that the effect of TiO2 on the characteristic temperatures was an increasing one at all levels.

v) In all cases, the increase in the constituent elements decrease the difference between the flow, ie, it makes a short slag.

Future Scope:

In the present work, we were concerned with the flow characteristic of synthetic slag which resembles the final slag. This can lead to a new study where the effects of the constituents on the flow characteristic of the primary slag can be studied which directly influences the coke consumption, Si content, permeability etc.

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