LOCATION AND EXTENT OF COHESIVE ZONE IN THE BLAST FURNACE AS RELATED TO THE DEGREE OF REDUCTION OF IRON ORE PELLETS/IRON ORE

THIS THESIS IS SUBMITTED IN THE PARTIAL FULLFILLMENT OF THE REQUIREMNT FOR THE DEGREE OF **BACHELOR OF TECHNOLOGY**

IN

METALLURGICAL AND MATERIALS ENGINEERING

BY

ABHIMANYU KADIYAN

(Roll No. 109MM0656)

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CERTIFICATE

This is to certify that the thesis entitled **"LOCATION AND EXTENT OF COHESIVE ZONE IN THE BLAST FURNACE AS RELATED TO THE DEGREE OF REDUCTION OF IRON ORE PELLETS/IRON ORE "** submitted by Abhimanyu Kadiyan (109MM0656) and Aneesh Singhal (109MM0644) in partial fulfilment 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 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.

Date: 8th May, 2013

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ABSTRACT

In addition to the chemistry, the degree of reduction of the iron bearing materials in the burden affects the characteristic temperature of the resulting primary slag in the cohesive zone of the Blast Furnace. Thus the location and extent of the cohesive zone which acts as a gas-distributor in the blast furnace are both influenced by the degree of reduction the iron bearing material undergo prior to their entry into the cohesive zone travelling through the granular zone. The present work is involved with sintering the iron-bearing materials, namely fluxed pellets and raw ore, to different degree of reduction and determination of the characteristic of these pre-reduced materials.

The softening temperature of the fluxed pellet increases with the increase of the degree of reduction up to 65% and then shows a reverse trend. In case of raw-ore the corresponding value is recorded to be 70%. Thus the cohesive zone supposed to be lowered with the increase in reduction of the fluxed-pellets, up to 65% reduction and similar trend is observed up to 70% for raw-ores. Due to certain experimental ambiguity the flow characteristic the flow-temperature, the softening-flow interval and thus the extent of the cohesive zone, as a function of the reduction of the iron bearing materials, is so difficult to be as curtained very accurately and only a likely trend has been reported.

Key words: Characteristic temperature, primary slag, location and extent of cohesive zone, degree of reduction, granular zone

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1. Introduction

The ironmaking blast furnace is a complex high temperature counter current reactor in which iron bearing materials (ore, sinter/pellet) and coke are alternately charged along with a suitable flux to create a layered burden in the furnace. The iron bearing material layers start softening and melting in the cohesive zone under the influence of the fluxing agents at the prevailing temperature which greatly reduces the layer permeability that regulates the flow of materials (gas/solid) in the furnace. It is the zone in the furnace bound by softening of the iron bearing materials at the top and melting and flowing of the same at the bottom.[14] A high softening temperature coupled with a relatively low flow temperature would form a narrow cohesive zone lower down the furnace.[15] This would decrease the distance travelled by the liquid in the furnace there by decreasing the Silicon pick- up.[16,17] On the other hand the final slag, that trickles down the Bosh region to the Hearth in the furnace, should be a short slag that starts flowing as soon as it softens. Thus fusion behavior is an important parameter to evaluate the effectiveness of the B.F. slag.

Fusion behaviour is described in terms of four characteristic temperatures [18]; IDT, the initial deformation temperature symbolising surface stickiness, important for movement of the material in the solid state; ST, symbolising plastic distortion, indicating start of plastic distortion; HT, the liquidus temperature, symbolising sluggish flow, playing a significant role in the aerodynamics of the furnace and heat and mass transfer; and FT, the flow temperature, symbolising liquid mobility.

 The slag formed in the cohesive zone is the primary slag formed with FeO as the primary fluxing constituent; the solidus temperature, fusion temperature, solidus-fusion interval being significantly affected by FeO [19]. This slag is completely different from the final slag where the fluxing is primarily caused due to the presence of basic constituents like CaO or MgO.

While it is not possible to obtain primary slag from the industrial blast furnace, it is always possible to prepare a synthetic slag in the laboratory resembling the primary slag and study its flow characteristics. We have kept this venture for future studies and the present study limits itself to the study of flow characteristics of the final slag as obtained from the industry. However, it must be noted that from the process point of view the final slag should be a 'Short Slag', a slag with a small difference between the ST and FT. Such a slag acquires liquid mobility and trickles down the furnace away from the site where it starts distorting plastically, as soon as possible. This action exposes fresh sites for further reaction and is supposedly responsible for enhanced slag-metal reaction rates, influencing the blast furnace operations and the quality of the metal.

Keeping the above in mind, in addition to employing two different experimental techniques for measurement of the flow characteristics of industrial blast furnace slags and comparing the liquidus temperatures so obtained the present work also involves itself with analysis of the data on the basis of the chemical composition of the slag.

2. Literature Review

Literature review reveal that formation of a narrow cohesive zone lower down the blast furnace improves the productivity and reduces the coke rate and that in majority of the cases, the iron bearing material in the blast furnace reaches the softening temperature (top end of cohesive zone) with a reduction degree higher than 50 percent[1]. The lowering and narrowing of the cohesive zone renders three distinct advantages:

1. It results in the extension of the granular zone enhancing the indirect reductions.
2. It results in a decrease in the distance travelled by the dripping metal thus reducing the silicon pick-up, improving the quality of hot metal.
3. It reduces the extent of the impermeable zone in the furnace that regulates the flow of gas/solid in the furnace.

While the final slag coming out of the blast furnace is a consequence of the primary slag in the cohesive zone travelling through the active coke zone and the bosh of the furnace being exposed to greater severities of prevailing conditions of temperature, pressure and slag-metal reaction potential ,the final slag does not reveal the characteristics of the primary slag. On the other hand it is impracticable to obtain samples of the primary slag from the cohesive zone for study of its characteristics. It can thus be appreciated that most of the work carried out concerning cohesive zone are model-based.

At this juncture it is only pertinent to discuss the most probable situation in the cohesive zone. The gangue and the flux in the blast furnace mainly consist of alumina, silica, lime and magnesia. At lower temperatures when these materials remain in the solid state the mutual interactions of these materials; especially the rate of lime dissolution, a process of solid state diffusion is very slow. On the other hand at high temperatures, alumina and silica will form alumino-Silicates which are more stable than either alumina or silica. Thus, it is most improbable that lime pick-up by the ore materials is significant before the reaction products are

liquid. The formation of the first slag, therefore, presumably occurs through fluxing of the alumino-silicates by unreduced FeO. The fluxed molten slag (liquidus temperature of alumino-silicates as evident from FeO-Al2O$_3$-SiO$_2$ system, above 40 percent FeO is not more than about 1200°C) flows over limestone lumps and coke and in the process picks up lime, forming the primary slag. The primary slag rapidly loses its ferrous oxide reacting with coke at the prevailing high temperature Fruehan et al. [1] on the other hand are of the opinion that depending on the phases present and their distribution, the first oxide melt forms at the interface of an iron oxide particle and other oxide. The first melt, thus formed, wets the ore particles as a result of the decreased interfacial energy [2], forms a barrier to the transport of reactants through it and slows down the reduction kinetics causing reduction retardation. The authors assume that the situation prevailing is that the liquid and solid oxides are confined within a solid, porous iron shell with a reduction degree of higher than 50 percent reduction. At the core of the shell the liquid slag covers the solid oxide particles, has a much reduced mechanical strength and the strength of the shell is determined by the porous iron outer shell. With the descent of the shell in the blast furnace, it encounters higher temperature, the liquid volume in the core increases, the shell gets carburized, its melting point decreases and finally the shell cracks resulting in the dripping of the liquid slag from the burden material. The crumbling of the carburized reduced iron shell under the action of temperature and pressure is known as softening. The softening coincides with the dripping of material from the burden component. During this process the reduction degree will tend to remain constant as a result of reduction retardation.

Various workers have varied opinion on the interdependence of softening temperature, the reduction degree and amount of FeO. Barnaba[3]opines that the softening temperature depends on the degree of reduction of the ore particle since the reduction degree determines the ability of the porous shell to retain the liquid slag in the core of the particle. Borider[4,5]and Ritz et al.[6], however, feel that the relationship between the softening temperature and the degree of

reduction of the iron ore particle is dictated by the reduction of the amount of FeO available for fluxing. Borider[4,5] even established through experiments that for acid pellets as well as pellets fluxed with olivine the metallic iron content had an effect on the softening and melting temperatures.

In the present work, therefore, an attempt is made to find out the relationship between softening, melting and flow temperatures of iron ore pellets/iron ore and their degree of reduction. It is obvious that such a study can throw light on the location and extent of the cohesive zone as a consequence of the degree of reduction of the concerned iron ore/pellet, since the cohesive zone is bound by the softening and flow temperatures at its top and bottom ends respectively.

3. Experimental Method

3.1. Sampling:

Coning and quartering sampling technique is used, which is the reduction in size of a granular or powdered sample, by forming a conical heap which is spread out into a circular, flat cake. The cake is divided radially into quarters and two opposite quarters are combined. The other two quarters are discarded. The process is repeated as many times as necessary to obtain the quantity desired for some final use (e.g. as the laboratory sample or as the test sample). If the process is performed only once, coning and quartering is no more efficient than taking alternate portions and discarding the others.
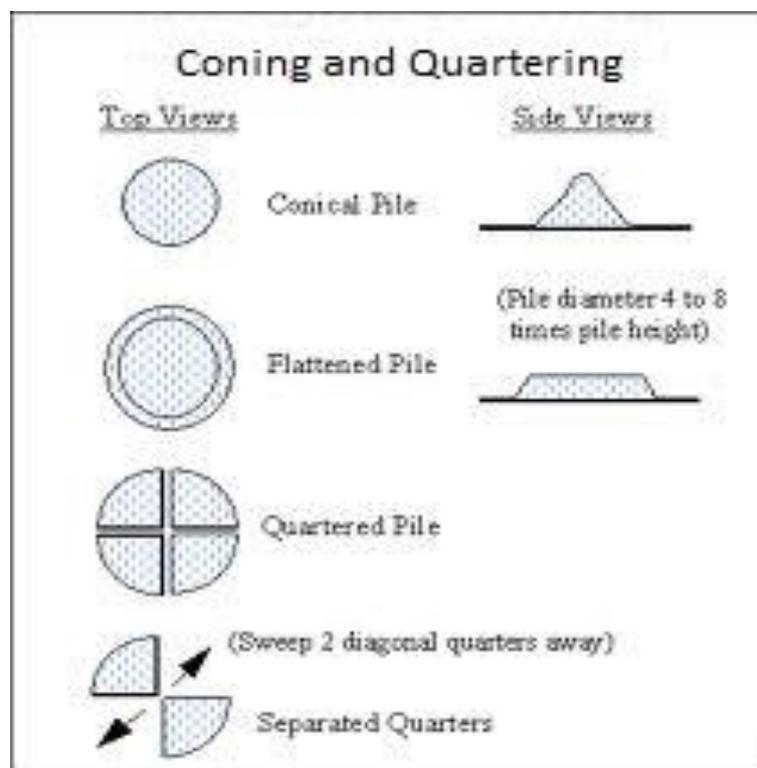

Figure 1. Coning and Quartering [13]

3.2. Testing:

High temperature microscope is used to determine flow characteristics of slag sample. It has got four characteristics temperatures to be studied:

- Initial deformation temperature (IDT)

- Softening temperature (ST)

- Hemispherical temperature (HT)

- Flow temperature (FT)

These temperatures have already been defined as per German Industrial Standards 51730.

The Heating Microscope method is adopted for recording the characteristic temperatures. A picture of the Leitz heating microscope is shown in Fig.2. Schematic diagram of the instrument is presented in Fig.4. The sample, in the form of a 3 mm cube, is heated in an 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. The four temperatures are reported as follows by following the German Industrial Standard 51730.
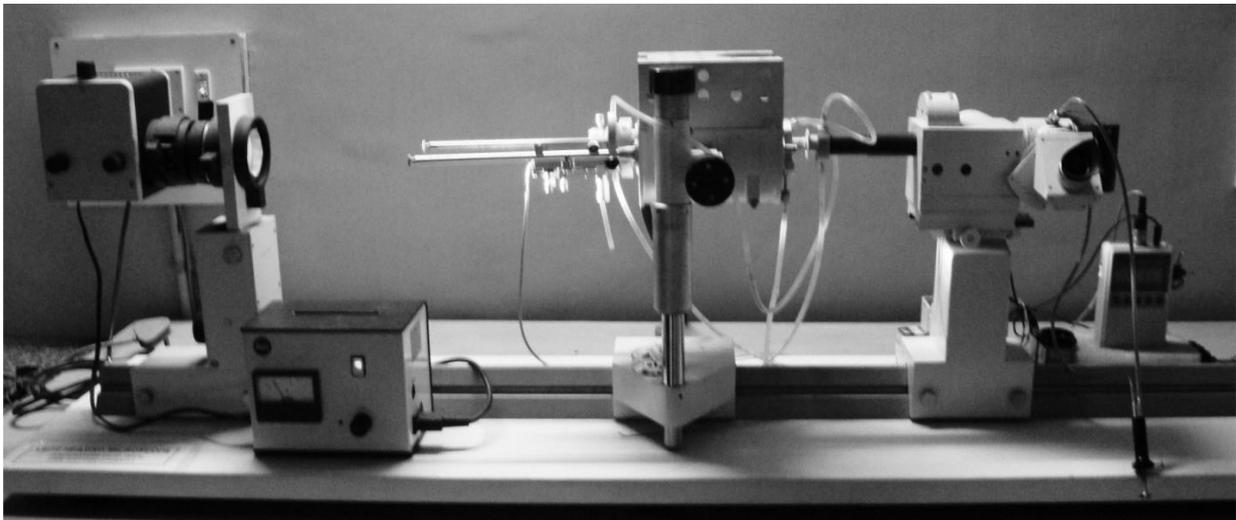

Figure 2. High Temperature Microscope

3.3 Preparation of iron ore pellets and reduction of pellets and iron ore to different reduction degrees

Pellets are prepared by using Iron ore, Lime stone, Olivine and Quartzite in different proportion with Bentonite as the binder. The analysis of the raw materials is given in Table-1.

Table 1: Analysis of raw materials for preparation of pellets.

Sl. No.	Item	LOI %	Fe_2O_3 %	Al_2O_3 %	SiO_2 %	CaO %	MgO %	TiO_2 %	Na_2O %	K_2O %
1	Iron Ore	06	86.5	3.75	2.5	-	-	0.5	-	-
2	Limestone	43	1.28	0.01	1.6	50.35	3.3	0.2	-	-
3	Olivine	03	7.7	1.61	37.39	0.4	49.5	0.22	-	-
4	Quartzite	01	-	-	98.5	-	-	-	-	-
5	Bentonite	18	13.36	15.1	48.85	-	-	0.2	03	0.03

Pellets of different compositions are made of diameter 10-20 mm by hand rolling. The pellets are designated as 1, 2, 3, 4 and 5. The chemical compositions of raw pellets are given in Table-2 on a loss free basis. These are subjected to drying in an oven at 110°C for 24 hours and then calcined at 950°C for 2 hours. The calcined pellets are subjected to reduction in a bed of coke-breeze contained in an alumina tube closed by porous alumina plugs at both ends with a supply

of air; the whole arrangement being heated in a tube furnace. The outgoing gases are analyzed to detect any escape of CO so that complete utilization of the gas for reduction is ensured.

Table 2: Chemical composition of the pellets (on loss free basis).

Sl. No.	Chemical Composition (wt. %)	Designated Pellets				
		1	2	3	4	5
1	Fe_2O_3	80.7	80.15	80.52	81.8	80.91
2	SiO_2	6.83	6.95	6.58	6.43	6.07
3	CaO	6.46	6.68	6.15	5.90	5.87
4	Al_2O_3	3.85	3.80	3.82	3.70	3.68
5	TiO_2	0.49	0.46	0.49	0.49	0.49
6	MgO	0.79	1.32	1.59	1.85	2.099

The reduced pellets are ground, the metal is separated using a magnet and the remaining slag is analyzed. The approximate slag analysis revealed the following:

CaO/SiO_2 (C/S) – 0.9

MgO content – 4, 6, 8, 10 and 12 wt. percent and

Al_2O_3 content – 20 wt. percent.

The degree of reduction for the pellets is calculated on a weight-loss basis. The degree of reduction of the pellets is provided in Table-3.

Table 3: Degree of reduction of the designated pellets.

Designated Pellets	Percentage Reduction
1	50
2	55
3	75
4	60
5	65

The designated pellets are then examined under the high temperature microscope for determining the characteristic temperatures.

In a similar way the iron ore samples (10-20mm size) of similar chemical composition as used for pellet making are subjected to reduction and the degree of reduction is calculated on the basis of weight-loss. These samples with different degrees of reduction are then examined to determine the characteristic temperatures. The percentage reduction of the designated iron ore samples are provided in Table-4.

Table 4: Degree of reduction of the designated iron ore samples.

Designated Iron ore	Percentage Reduction
1	65
2	70
3	75

3.4 Measurement of Characteristic Temperatures

The characteristic temperatures of the samples are measured using the Hot-stage microscope. The photograph of the instrument is provided in Fig. 1. A small cube (3 mm) of the sample material is prepared after adopting the method of coning and quartering for sampling. This small cube of the sample is gradually heated (2-3°C/min heating rate) and the profile of the sample is photographed. Fig. 3 gives the profile of the heated sample indicating various characteristics temperatures viz. softening temperature (ST); hemispherical temperature (HT) and flow temperature (FT).

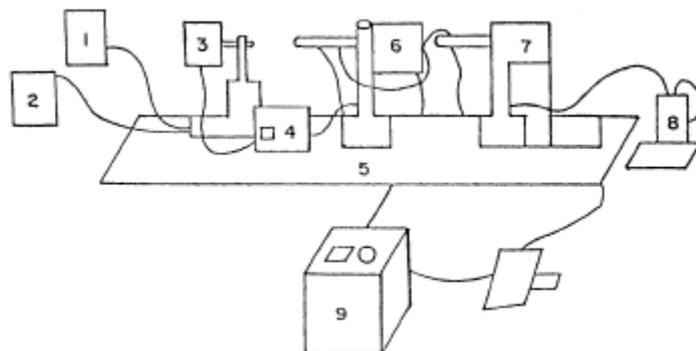Figure 3: Profile of the heated sample.

Figure 4. Line diagram of High Temperature 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 different steps for determining the flow characteristics are as follows.

- ✓ The sample, in the form of a 3 mm cube, is heated in an 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.

- ✓ The temperature, at which the sample just starts to deform a bit, is reported as IDT.

- ✓ 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 2:1, is reported as HT.

- ✓ The temperature, at which the height of the sample reduces to one-third of the height measured at HT, is reported as FT.

In present work, each slag has been tested twice (once each for each one of the alternate quarters).

4. Results and Discussion

The experimental findings are presented in Table-5 through Table-8 and illustrated in Fig. 5 through Fig. 8. The result can be analyzed as follows:

4.1 Iron Ore Pellets

4.1.1 Degree of Reduction

As evident from Table-5 and Table-6 and Fig. 5, the softening temperature of the pellets increases with degree of reduction till the reduction is 65 percent after which the ST decreases with the increase of the degree of reduction for the range of degree of reduction examined.

Table 5: Characteristic temperatures of pellets with metallic phase separated by magnetic separation.

Sample No.	MgO %	Degree of Reduction %	ST (°C)	HT (°C)	FT (°C)
1	4	50	1400	1515	1578
2	6	55	1423	1493	1562
3	8	75	1440	1490	1527
4	10	60	1455	1500	1535
5	12	65	1470	1475	1518

Table 6: Characteristic temperatures of pellets with metallic phase not separated.

Sample No.	MgO %	Degree of Reduction %	ST (°C)	HT (°C)	FT (°C)

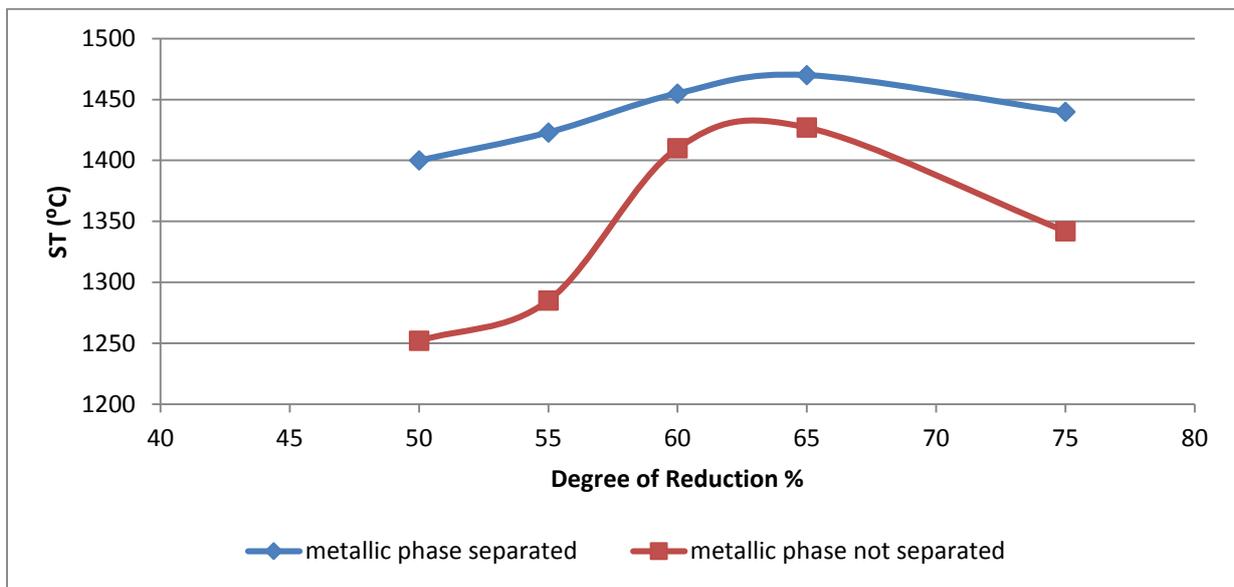|					
1	4	50	1252	1430	1567
2	6	55	1285	1425	1530
3	8	75	1342	1400	1500
4	10	60	1410	1438	1495
5	12	65	1427	1453	1486

Figure 5: Softening temperature vs. degree of reduction for pellets.

It is well understood that the softening temperatures, the degree of reduction and the amount of FeO in the burden material are interdependent. With the degree of reduction increasing, the proportion of FeO in the burden material gets lessened. According to literature the first oxide melt forms at the interface of an iron oxide particle and another oxide particle. Thus, as the amount of FeO is reduced as a consequence of an increased degree of reduction it is obvious that the formation of the first liquid phase is adversely affected, the possibility of formation of the interface between the iron oxide and another oxide particle being retarded. The first liquid phase formation is delayed resulting in the increase of the softening temperature. On the other hand the reduction of the iron ore particles renders a solid porous iron shell which envelops the

core containing the liquid slag that covers the remnant solid oxide particles. As the reduction degree increases the iron shell becomes more and more porous which enhances carburization of the shell, lowering its melting point in addition to reducing its strength owing to the increased porosity. As a result the shell crumbles at relatively less severe conditions of temperature and pressure. At this juncture the liquid slag starts dripping from the burden material. This phenomenon is known as softening of the burden material. Thus, the increased degree of reduction, after a certain degree of reduction, is responsible for assisting the process of softening there by bringing down the softening temperature.

To sum up, the mutually competing processes of lessening in the amount of FeO and the formation of the iron shell with increased porosity as a consequence of higher and higher degrees of reduction should explain the initial increase in the softening temperature with the degree of reduction and the subsequent decrease of the former with the increase of the later. The location of the cohesive zone in the blast furnace is thus related to the degree of reduction of the iron ore/pellet.

4.1.2 MgO Content

Data presented in Table-5 and Table-6 and Fig. 6 show the effect of MgO content of the pellets on the softening temperature. The data shows that irrespective of the degree of reduction an increase in the MgO content results in the increase of the softening temperature under the experimental conditions.
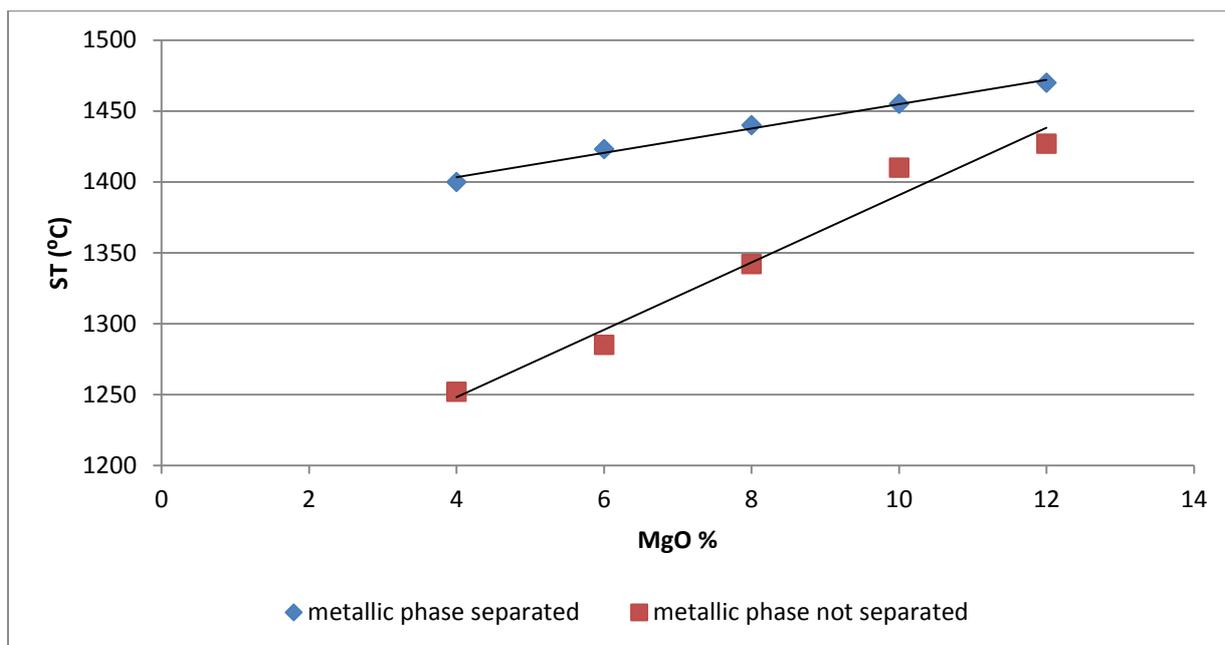
Figure 6: Softening temperature vs. MgO for pellets.

Literature seems to be divided on this aspect. While some agree with the findings some do not quite agree with the increased trend of ST with MgO content. Khakietal.[7] and Ritz et al.[6] establish experimentally that both the ST and the melting temperature (HT) increase with increase of basicity. Barnaba[3] reports that at basicities of 1.6 the softening and the melting temperature attain the respective maximum values. Ritz et al.[6] further reports that at lower reduction degrees (up to 50 percent) the ST and the HT depend on the expanded basicity (%CaO+%MgO)/%SiO$_2$). However, at higher reduction degrees when most of the FeO is removed, they report the HT of the acid pellets to be higher than that of the fluxed pellets, while the ST remains approximately the same. Bentel[8] is of the opinion that adding MgO in lieu of CaO helps forming high melting slag and this is beneficial for the process especially at high MgO/ SiO$_2$ ratios. They also note that at higher MgO/ SiO$_2$ ratios the FeO matrix melts earlier than the slag. The data obtained from a similar study by Jia-Shyan et al.[9], using high temperature microscopy, reveal that the softening temperature of the test sample increases with MgO content up to about 12 percent MgO. This finding coincides with the findings of the

present experiments. Shen et al. [10] conducted experiments to show that a slag containing high MgO cannot come to dripping easily. Therefore, high MgO is likely to increase the temperature range of the cohesive zone which intervenes with the permeability of the burden and thus is not favorable for the blast furnace process of iron making. However, the authors worked with MgO content of maximum 03 percent and suggested additional injection through the Tuyers to form a final slag with better fluidity and desulphurising abilities. Matsumura et al. [11] have experimental evidence to show that the melting point of CaO-FeO- SiO_2 slag increases with increase in the MgO content.

To sum up, notwithstanding the agreement/ disagreement of various workers with the findings of the present work concerning MgO content in the pellet, it is interesting to note that MgO content affects the characteristic temperatures of the slag in a complicated manner under different conditions and circumstances. Therefore, the study concerning MgO content should constitute an important component of future investigations.

4.1.3 Extent of the Cohesive Zone

The difference of ST and (Flow-Temperature) FT is a measure of the extent of the cohesive zone. While a small difference represents a narrow cohesive zone, a wide difference marks the presence of an extended cohesive zone which may cause a bad situation in the furnace by way of affecting the permeability of the bed being responsible for higher extents of pressure drop.
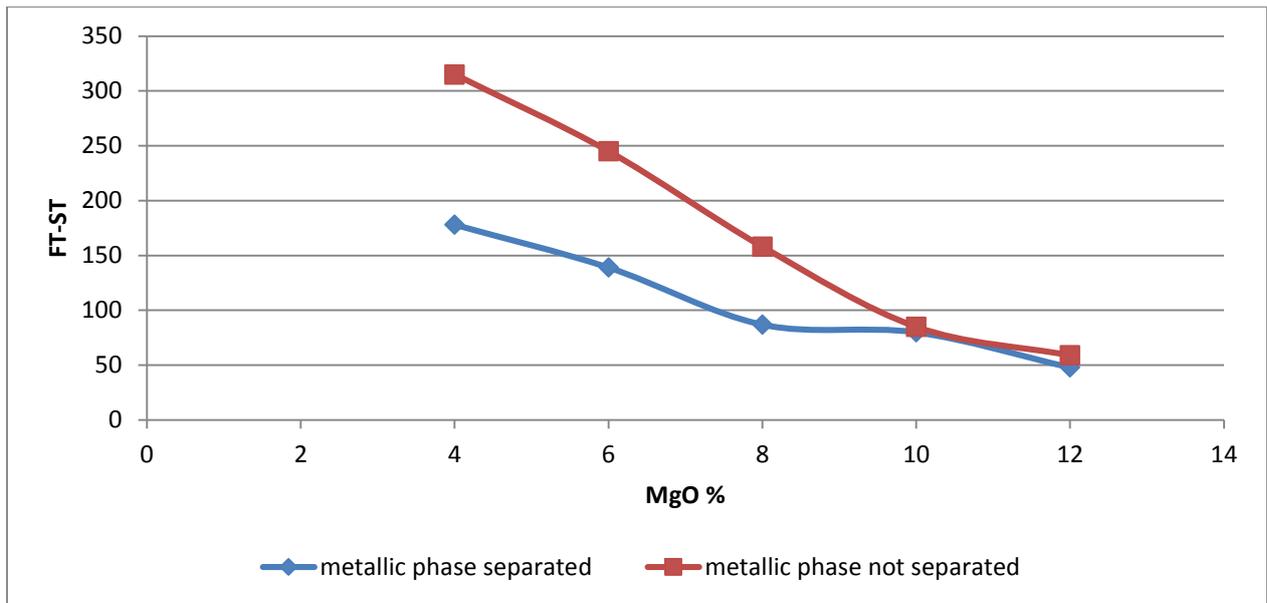
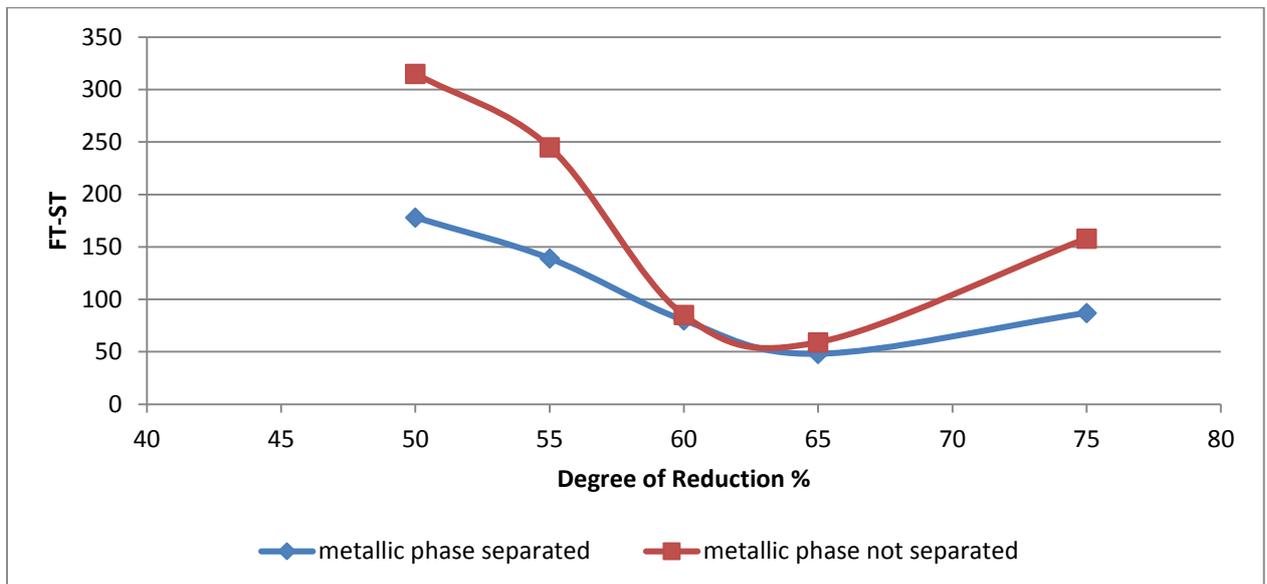Figure 7: FT-ST vs. MgO for pellets.

Figure 8: FT-ST vs. degree of reduction for pellets.

Fig.7 and Fig. 8 show that with the increase of both MgO content and degree of reduction the difference between the FT and ST decreases for pellets; though in the case of the degree of reduction the difference shows a little increase in FT-ST at greater than 65% reduction. This

indicates that with the increase of degree of reduction with the pellet charge, the cohesive zone narrows down till about 65 percent of reduction while a higher level of reduction reverses the situation. The decrease of the region of the pressure drop with MgO addition in the charge bed has been corroborated by Matsumura et al.[11]. They are of the opinion that the pressure drop is caused by the phenomenon of decreasing of the voids which is caused by melt formation. Therefore, they suggest, an increase in MgO content results in narrowing down the region of pressure drop by increasing the melting temperature of the burden material there by delaying the melting down. However, it may be noted that the FT measured in the present case is very high. This may be due to fact that the slag formation has only been initiated in the cohesive zone with the initiation of the softening and that most of the oxides have still not participated in the slag forming reactions and thus not stabilized in the slag-phase. Vacuum melting of the slag after subjecting the pellets to different degrees of reduction or conducting related measurements with synthetically prepared slag in the laboratory of appropriate chemical composition, may help the stabilization of the remnant oxide in the slag melt and provide a qualitative answer to the issue.

4.2 IRON ORE

By and large, the data for Iron ore for both the cases when the magnetic phase is separated and when it is not separated show that the analysis of the data made in the case of pellets almost hold good for the iron ore charge. There are two exceptions as can be noted from the data in Table-7 and Table-8.

1. The degree of reduction at which the softening temperature starts decreasing is 70 percent in the case of iron ore samples and not 65 percent as in the case of pellets. The reason for reversal of ST at a relatively high degree of reduction in the case of iron ore

may lie in the formation of Mullite ($3Al_2O_3.2SiO_2$) having a very high melting point (about 1850°C to 1900°C). The formation of Mullite is enhanced by the presence of TiO_2. At relatively high FeO content as a consequence of higher degrees of reduction of the Fe_2O_3 in the ore, FeO reacts with Mullite to form Fayalite ($FeO.SiO_2$) and Almandine (Al.Fe.Si oxide) having a comparatively lower melting point. Thus the resultant ST is decreased.

2. The respective temperature is so high in the case of iron ore that in some of the cases a measurement of the characteristic temperatures have not been possible.

Table 7: Characteristic temperatures of iron-ore with metallic phase separated by magnetic separation.

Sample No.	Degree of Reduction %	ST (°C)	HT (°C)	FT (°C)
1	65	1555	1582	-
2	70	1580	-	-

Table 8: Characteristic temperatures of iron-ore with metallic phase not separated.

Sample No.	Degree of Reduction %	ST (°C)	HT (°C)	FT (°C)
1	65	1530	1567	1584
2	70	1572	1585	-
3	75	1539	1563	1585

5. Conclusion

1. Degree of reduction of the iron bearing materials entering the cohesive zone affects both the location and the extent of the same in the blast furnace.

2. In the case of pellets a degree of reduction up to 65 percent may be beneficial while that in the case of iron ore is found to be 70 percent.

3. After the respective degree of reductions are reached a further enhancement of the degree of reduction may not be beneficial as it may raise the cohesive zone in the furnace causing an increase in the softening temperature, thus enhancing the formation of primary slag.

4. Under the ranges of MgO contents examined in the case of pellets, an increase in the MgO content seems to be beneficial by way of helping the formation of a narrow cohesive zone lower down the furnace.

Thus a pre-reduced pellet charge with appropriate MgO additions may help iron making in the blast furnace route influencing both the location and the extent of the cohesive zone in the furnace.

FUTURE SCOPE:

The work at this stage has certain limitations owing to the fact that the primary slag i.e. the cohesive zone lack stability. This concerns the continuous change in compositions as the slag descends from the cohesive zone to the bosh zone. In the process, the extent of FeO gets reduced and more and more CaO enters in the slag. Taking this fact into consideration we propose future investigation which are stated as below:

1. Composition of the pellets have to be changed over a wide range concerning MgO and

   CaO/SiO_2 ratio.

2. Degree of reduction has to be changed over a wide range

3. At every temperature of experimentation for change of degree of reduction sufficient time

   must be allowed to permit:

   a) Reduction of FeO.

   b) Entry of CaO into the slag, in order to attain stability.

4. The experiments may be conducted taking Iron Ore as the charge material for a

   comparison.

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